

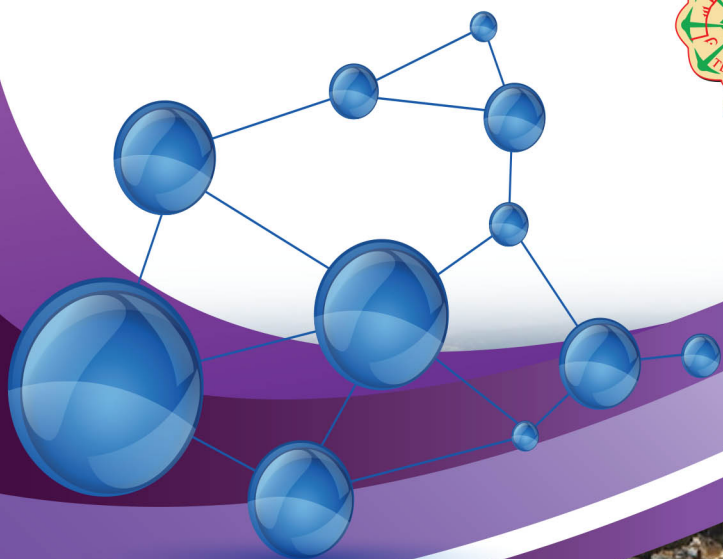
General Directorate for Scientific Research and Technological Development

University Aboubekr Belkaid - Tlemcen

Faculty of Sciences

Department of Chemistry

Laboratory of Catalysis and Synthesis in Organic Chemistry



2nd International Symposium on Catalysis and Specialty Chemicals ISCSC-2018

Book of Abstracts

October 01-03, 2018

Tlemcen - ALGERIA

Chair's welcome

On behalf of the Organizing Committee, we take great pleasure in welcoming you to the 2nd International Symposium on Catalysis and Specialty Chemicals ISCSC-2018, Tlemcen, Algeria, October 1-3, 2018. The symposium is organized by the Laboratory of Catalysis and Synthesis in Organic Chemistry (LCSCO), and hosted by the Palais de la culture of Tlemcen city. It takes place under the auspices of the University of Tlemcen and the General Directorate for Scientific Research and Technological Development (DGRSDT).

The first ISCSC event took place in 2012, and was a real success thanks to participants, speakers and organizers. This new edition aspires to renew this opportunity for learning and exchange of our young researchers with leading figures in the fields of catalysis and organic chemistry.

The 2nd edition of ISCSC follows the path of the previous one by including topics such as Catalysis for a cleaner and sustainable future, catalytic processes for energy, catalysis for water treatment, biomass and agroresources for chemistry, design and characterization of innovative catalytic materials, nanomaterials in catalysis, products design, green solvents for chemical synthesis, green routes for chemical synthesis, organic synthesis, heterocycles and fine chemicals and pharmaceuticals.

Our technical program is rich and varied with 5 plenary conferences, 10 keynote speeches and around 200 communications split between 4 oral and 2 poster sessions. The ISCSC aims to bring academics, scientists and industrial partners together to discuss the recent developments in the areas of catalysis and specialty chemicals.

Our special thanks go to our guest speakers for coming from the USA, Germany, France, Portugal, Italy, Spain and our neighbors from Tunisia and Egypt to share this enjoyable event with us. We also wish to thank all the authors for choosing this venue to present their research work, and all other participants who are taking part in this scientific event. We would like to thank all the members of the International Scientific Committee, Local Organizing Committee and the students involved for their great efforts in contributing to the success of the conference.

Finally, we would also like to thank Palais de la culture of Tlemcen for hosting the symposium and acknowledge the sponsors financial support, especially the University of Tlemcen, DGRSDT, PAU, SAIDAL, SONELGAZ, SINAL, EVLAB, PROCHIMA-SIGMA, MANSOURAH and ICB.

We hope you will enjoy the content, renew old friendships, make new friends, get new ideas, take some time to visit the artistic heritage and wonderful landscape of Tlemcen and above all, have a good time. With best wishes,

Noureddine CHOUKCHOU-BRAHAM

LECTURES

NANOTECHNOLOGY ENABLES HOT GOLD NANORODS TO KILL CANCER CELLS AND TO STOP ALIVE CELLS FROM MIGRATING AND KILLING MORE SICK PEOPLE I

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TRANSITION METALS : POWERFUL TOOLS FOR THE CONSTRUCTION AND FUNCTIONALIZATION OF HETEROCYCLES II

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THE ROLE OF SPECTROSCOPY IN UNDERSTANDING HETEROGENEOUS CATALYSTS III

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CYCLOADDITION AND CONJUGATE ADDITION REACTIONS ON CHROMONES LEADING TO OTHER OXYGEN AND NITROGEN HETEROCYCLIC COMPOUNDS IV

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CATALYTIC DOMINO REACTIONS FOR THE ECOLOGICALLY BENIGN SYNTHESIS OF NATURAL PRODUCTS AND MATERIALS V

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RECENT ADVANCES IN THE CHEMISTRY AND SYNTHETIC USES OF LITHIATED SMALL RING HETEROCYCLES VI

Saverio FLORIO

Consorzio CINMPIS, University of Bari "Aldo Moro", I-70125 Bari, Italy

ALKANE PROTOLYTIC CRACKING BY ZEOLITES: IN SEARCH OF THE REACTANT STATE VII

Arnaud TRAVERT

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A JOURNEY IN CYCLOPEPTOIDS' CHEMISTRY VIII

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SYNTHESIS AND DESIGN OF FEW LAYER GRAPHENE-BASED COMPOSITES FOR CATALYSIS AND ENERGY RELATED APPLICATIONS IX

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N-HETEROCYCLIC CARBENES (NHCs) COMPLEXES OF Zn(II) AND GROUP 13 METALS: USE IN POLYMERIZATION CATALYSIS FOR BIOMATERIALS PRODUCTION AND FOR CO₂ FUNCTIONALIZATION	X
Samuel DAGORNE <i>Institut de Chimie de Strasbourg – CNRS-University of Strasbourg, Strasbourg, France</i>	
SYNTHESIS OF FIVE-MEMBERED AROMATIC HETEROCYCLES APPLICATION TO THE PREPARATION OF POTENTIAL ACTIVE COMPOUNDS AND NEW SCAFFOLDS	XI
Gilbert KIRSCH <i>UMR CNRS 7053 L2CM Université de Lorraine 1, boulevard Arago 57070 METZ</i>	
SUPPORTED GOLD NANOPARTICLES. EFFICIENT HETEROGENEOUS CATALYSTS FOR OXIDATION REACTIONS AND C-C COUPLINGS IN LIQUID PHASE	XII
Redouane BACHIR <i>Laboratory of Catalysis and Synthesis in Organic Chemistry, Université de Tlemcen Algeria</i>	
RECENT PROGRESS IN THE SYNTHESIS OF AZAPHILONES AND ANALOGUES	XIII
Xavier FRANCK <i>Normandie University, CNRS UMR 6014 COBRA, 76000 Rouen, France</i>	
PHOTOCATALYTIC PATHWAY TOWARD DEGRADATION OF VARIOUS PHARMACEUTICAL MOLECULES: KINETICS, MECHANISMS AND TOXICITY ASSESSMENT	XIV
Mohamed KSIBI <i>Laboratory of Environmental Engineering and Eco-Technology, National School of Engineers (ENIS), University of Sfax, Route de Soukra Km 3,5, P.O. Box 1173, 3038 Sfax, Tunisia</i>	

TOPIC I: CATALYSIS

ORAL COMMUNICATIONS

O-1	X-RAY CRYSTAL OF AURATE SALT COMPLEX OF DI-PROTONATED TETRA (2-PYRIDYL) PYRAZINE (H₂ TPPZ)	3
Mohsen M. MOSTAFA		
O-2	ACTIVITY OF MONO AND BIMETALLIC GOLD CATALYSTS ON ADIPIC ACID PRODUCTION	4
Nawal AMEUR, Redouane BACHIR, Sumeya BEDRANE, Abderrahim CHOUKCHOU-BRAHAM		
O-3	NOVEL CLEAN ROUTE OF ADIPIC ACID SYNTHESIS THROUGH CYCLOHEXANONE OXIDATION OVER METAL SUBSTITUTED POLYOXOMETALATES H_{3-2x}Sn_xPMo₁₂O₄₀ (x =0.25-1.5)	5

Abdenour BOUMECHHOUR, Siham BENADJI, Tassadit MAZARI, Sihem MOUANI ,
Leïla DERMECHE , Lynda MOUHEB , Chérifa RABIA

- O-4 EFFECT OF REDUCING GAS ON THE HYDROGEN PRODUCTION BY THERMO-
OXIDATION OF WATER OVER 1%Rh/Ce_{0.6}Zr_{0.4}O₂** 6
Bachira SOUKHAL-BENAZIZA, Aïcha BENAMAR, Farida SADI
- O-5 Ni AND/OR Co MOLYBDATE MATERIALS PREPARED BY MODIFIED CO-
PRECIPITATION METHOD: STRUCTURAL, TEXTURAL APPLICATION IN
ETHANE OXIDATIVE DEHYDROGENATION TO ETHYLENE** 7
Hamza BOUKHLOUF, Akila BARAMA, Rafik BENRABAA, Samira AMOKRANE,
Axel LOFBERG, Jesus GUERRERO CABALLERO, Elisabeth BORDES-RICHARD
- O-6 SYNTHESIS OF A SERIES OF NEW PLATINUM ORGANOMETALLIC
COMPLEXES DERIVED FROM BIDENTATE SCHIFF BASE LIGANDS AND
THEIR CATALYTIC BEHAVIOUR TOWARDS THE HYDROSILYLATION OF
ALKENE** 8
Mohammed Belhadj LACHACHI, T. BENABDALLAH, M. HADJ YUCEF and J.
LYANM
- O-7 PHOTOCATALYTIC DEGRADATION OF METHYLENE BLUE THROUGH Au-
Ag/TiO₂ AND Au-Ag/TiO₂/GRAPHENE** 9
Hicham ATOUT, Abdellah BOUGUETTOUCHA, Derradji CHEBLI, Miguel MONGE,
José M López-de-Luzuriaga
- O-8 REMOVAL OF POLLUTANTS FROM WASTEWATER USING SILVER DOPED
MESOPOROUS TiO₂** 10
Hanane CHAKER, Leïla CHÉRIF-AOUALI, Sophie FOURMENTIN
- O-9 PHOTOCATALYSIS WITH Fe/Ti-PILLARED CLAYS FOR THE OXIDATION OF
ALKYLAROMATICS** 11
Houria REZALA, Jose Luis VALVERDE, Amaya ROMERO, Alessandra MOLINARI,
Andrea MALDOTTI
- O-10 SYNTHESIS, CHARACTERIZATION AND OPTIMIZATION OF
HETEROGENEOUS CATALYTIC CYCLOHEXENE OXIDATION VIA THE LATIN
SQUARE DESIGN (TAGUACHI L₉) METHODOLOGY** 12
Mostapha ZERROUKI, Souheyla BOUDJEMA, Abderrahim CHOUKCHOU-
BRAHAM, Ilhem REKKAB, Sanaa ELKORSO

POSTER COMMUNICATIONS

- P-01 IN-SITU HYDROGASIFICATION/REGENERATION OF NiAl-HYDROTALCITE
DERIVED CATALYST IN THE REACTION OF CO₂ REFORMING OF METHANE:
A VERSATILE APPROACH TO CATALYST RECYCLING** 14
Z. ABDELSADEK, M. SEHAILIA, , F.TOUHARA, N. AIDER, B. DJEBARI, D.
HALLICHE, V.M. GONZALEZ-DELACRUZ, J.P. HOLGADO, K. BACHARI, A.
CABALLERO, O. CHERIFI
- P-02 GREEN SYNTHESIS OF Cu-Ni ALLOY NANOPARTICLES USING ROSMARINUS
OFFICINALIS PLANT EXTRACT: APPLICATION TO ELECTROCATALYTIC
ACTIVITY** 15
Dyhia AIT ABDESSELAM, Razika AITOUT, Laid MAKHLOUFI

P-03	EFFECT OF ALKALINE TREATMENT OF A COMMERCIAL ZSM-5 ZEOLITE ON H₂ PRODUCTION FROM CH₄ DECOMPOSITION OVER Ni SUPPORTED HZSM-5 CATALYSTS	16
	Lilia ALALGA, Aicha BENAMAR,	
P-04	DEACTIVATION BY SINTERING OF COBALT CATALYST IN THE FISCHER-TROPSCH REACTION	17
	Dounia ALIHELLAL, Lemnouer CHIBANE	
P-05	HYDROGEN PRODUCTION FROM DRY METHANE REFORMING OVER Ru SUPPORTED CATALYSTS	18
	Djaouida ALLAM , Simona BENNICI and Smain HOCINE	
P-06	Cu-Zn BASED CATALYSTS, PREPARATION, CHARACTERIZATION AND ACTIVITY IN THE CATALYTIC HYDROGENATION OF CARBON DIOXIDE	19
	Djaouida ALLAM , Mohamed Ameziane BOURKACHE, Simona BENNICI and Smain HOCINE	
P-07	CATALYTIC DEHYDROGENATION OF ETHANOL TO ACETALDEHYDE AND ETHYL ACETATE	20
	Samira AMOKRANE, Hamza BOUKHLOUF , Ghazi OTMANINE	
P-08	PHOTOCATALYTIC DEGRADATION OF ORGANIC POLLUTANT USING HYDROTALCITE-LIKE COMPOUNDS	21
	Farid AOUDJIT, Ouiza CHERIFI, Djamilla HALLICHE	
P-09	PREPARATION OF GOLD NANOPARTICLES SUPPORTED ON NICKEL-CONTAINING HYDROTALCITE CATALYSTS AND THEIR ACTIVITY IN CO OXIDATION REACTION	22
	Loubna ARAB, Mokhtar BOUTAHALA, Véronique PITCHON	
P-10	HYDROGEN PRODUCTION FROM ETHANOL STREAM REFORMING OVER Ni-BASED CATALYSTS	23
	Koubra ARIBI, Madani GHELAMALLAH, Vicente CORTES CORBERAN	
P-11	DEEP OXIDATION OF TOLUENE OVER Pd AND/ OR Au SUPPORTED ON MESOPOROUS Co₃O₄	24
	Hajer AZZI, Leila Chérif-Aouali, Amine Zioueche.	
P-12	NANOCAST ORDERED MESOPOROUS CERIA AS SUPPORT FOR HIGHLY ACTIVE SILVER CATALYSTS FOR PROPYLENE TOTAL OXIDATION	25
	Zohra BAILICHE, Sihem BENAÏSSA, Leila CHERIF-AOUALI, Stéphane SIFFERT, Abdelkader BENGEDDACH	
P-13	SYNTHESIS, CHARACTERIZATIONS OF THE LAMELLATE DOUBLE HYDROXIDES AND THEIR APPLICATION CORROSION INHIBITOR	26
	Naima BAKHTAOUI, Omar BENALI, Chewki SELLES	
P-14	SYNTHESIS AND CHARACTERISATION OF SUPPORTED GOLD CATALYSTS FOR CATALYTIC APPLICATION	27
	Meriem BANOU, Charaf BOUNEKTA, Meriem CHENOUF	

P-15	ADVANCED METHANOL TRANSFORMATION INTO FORMALDEHYDE SENSIBILIZED BY RHODAMINE-B WITH THE PRESENCE OF CU AND ZN PHOTOCATALYSTS	28
	Wissame CHETTAH, Siham BARAMA, Anne DAVIDSON, Mohamed-Salah MEDJREM .	
P-16	SYNTHESIS AND CHARACTERIZATION OF IRON OXIDE NANOPARTICLES FOR HETEROGENEOUS PHOTOCATALYSIS APPLICATION	29
	S. BELAIDI, L. LAMERA, K. CHORFI, L. MAMMERI, W. REMACHE, T. SEHILI	
P17	REMOVAL OF REACTIVE BLACK 5 FROM AQUEOUS SOLUTIONS BY NANO-TiO₂ IMMOBILIZED ON ACTIVATED CARBON	30
	Hanane BELAYACHI, Sarra BOURAHLA, Fadéla NEMCHI, Mostefa BELHAKEM	
P-18	BIOINSPIRED SYNTHESIS OF SILVER NANOPARTICLES	31
	Mouhammed BELDJILALI, Smain BOUSALEM	
P-19	SYNTHESIS OF ZnO-BENTONITE AS PHOTOCATALYSIS TO DEGRADE CATIONIC YELLOW DYE	32
	Nassima BELGAID , Mohamed Redha MENAN I, Kamel Eddine BOUHIDEL	
P-20	EFFECT OF FLUORINATED MEDIUM ON THE SYNTHESIS OF AIPO-34	33
	Salima BELLATRECHE, Mohammed Abdelkrim HASNAOUI, Abdelkader BENGEDDACH	
P-21	CO₂/CH₄ HYDRATES FORMATION KINETICS INFLUENCED BY Al₂O₃ AND SiO₂ NANOPARTICLES	34
	Said SAMER, Mohamed BELLOUM, Mohamed KHODJA, Jean-Michel HERRI	
P-22	PHOTOCATALYTIC ACTIVITIES FOR HYDROGEN EVOLUTION OF NEW LAYERED COMPOUND Cu_{0.5}Nb₃O₈	35
	N. Belmokhtar, R. Brahimi , R. Nedjar and M. Trari	
P-23	SYNTHESIS AND CHARACTERIZATION OF THE [Zr] ZSM-5 COMPOSITE FROM Ludox SILICA AND USE AS A CATALYST FOR OXIDATION OF 2-MERCAPTOBENZOTIAZOLE	36
	Imen BENCHIKH , Khadra MOKADEM , Fatiha DJAFRI, Attalah DAHBI, Affaf TABTI , Ayada DJAFRI	
P-24	THE EFFICIENT REMOVAL OF XYLENOL ORANGE DYE IN AN AQUEOUS SOLUTION BY MAGADIITE	37
	Soumia BENKHATOU, Amel DJELAD, Mohamed BOUCHEKARA, Abdelkader BENGUEDDACH, Mohamed SASSI	
P-25	H₄SiMo₁₂O₄₀ AS A CATALYST FOR THE SYNTHESIS OF 4-HYDROXY-4-METHYL COUMARIN	38
	Leila BENNINI , Tassadit MAZARI, Malika MAKHLOUFI	
P-26	FENTON AND FENTON-LIKE PROCESSES FOR INDUSTRIAL EFFLUENTS TREATMENT- A REVIEW	39
	Khedidja BENOUIS, Rokia BOURI	
P-27	GOLD/CHITOSAN CATALYZED THREE MULTICOMPONENT COUPLING OF ALKYNE, CH₂CL₂ AND AMINES	40

	Meriem BENSAAAD, Amina BERRICHI, Redouane BACHIR, Sumeya BEDRANE, Nouredine CHOUKCHOU-BRAHAM	
P-28	BOROSILICATE ZSM-5 ZEOLITE SYNTHESIS IN THE PRESENCE OF N,N-DIMETHYLANILINE AND ITS PERFORMANCE IN CO₂ ADSORPTION	41
	Boumediéne BENSAFI, Nadjat CHOUAT, Rajaa ELAHMAR, Fatiha DJAFRI	
P-29	SYNTHESIS OF TITANIUM-BASED HETEROGENEOUS CATALYSTS FOR PHOTOCATALYSIS APPLICATION	42
	Zineb BENSMAINE, Sanaa EL-KORSO, Abderrahim CHOUKCHOU-BRAHAM, Ilhem REKKAB, Souheyla BOUDJEMA	
P-30	VALORIZATION OF THE BIOMASS-BASED PLATFORM MOLECULE ON CLAY	43
	Ghezlane BERRAHOU, Redouane BACHIR, Nihel DIB	
P-31	EFFECT OF SBA-15/SH MOLAR RATIO ON Pb(II) and Cd(II) IONS ADSORPTION FROM AQUEOUS SOLUTION	44
	Zayneb BOUCHERIF, Rokia BOURI, Leïla CHERIF-AOUALI, Renaud DENOYEL	
P-32	PRELIMINARY INVESTIGATION OF M-DOPED PILARED CLAY FOR VOCs TOTAL OXIDATION	45
	Khadidja BOUDADI, Abderrahim BELLIFA, Madani GHELAMALLH, Amine ASSENINE, Céline FONTAINE.	
P-33	PHOTOCATALYTIC ELIMINATION OF MALACHITE GREEN BY MATERIALS BASED ON TRANSITION METALS	46
	Hasna BOUDGHENE STAMBOULI, Fouad GUENFOUD, Malika MOKHTARI	
P-34	HYDROGEN GENERATION OVER COBALT CONTAINING MICROPOROUS ALUMINOPHOSPHATES PHOTO-CATALYST	47
	A. BOUCHAMA, A. BOUDJEMAA, H HENTIT, J.C JUMAS, K. BACHARI, M.S OUALI	
P-35	ACIDO BASIC CATALYTIC HYDROLYSIS OF N,N-DIMETHYL-4-((4-NITROPHENYLIMINO) METHYL)ANILINE IN THE PRESENCE OF CYCLODEXTRIN : EXPERIMENTAL AND THEORETICAL STUDIES	48
	Hadjer BOUGHERARA, Sara SOLTANI ,Mekki KADRI,Charif BOUMAZA	
P-36	PHOTODEGRADATION OF PHENOL USING mont-La (6%)-Cu_{0.6}Cd_{0.4}S CATALYST UNDER NUV-VISIBLE IRRADIATION	49
	Horiya BOUKHATEM, Lila DJOUADI, Hussein KHALAF, Ruffino Manuel NAVARRO, Moisés CANLE LOPEZ.	
P-37	ACETYLENE HYDROGENATION OVER Ni-Cu BIMETALLIC CATALYSTS: CHARACTERISATION AND REACTIVITY	50
	Mouhssin BOULBAZINE, Abdel-Ghani BOUDJAHM	
P-38	SYNTHESIS OF FUNCTIONALIZED CARBON BASED MATERIALS FOR HMF PRODUCTION WITHOUT SOLVENT	51
	Charf Eddine BOUNOUKTA , Meriem BANOU, Meriem CHENOUF , Fatima AMMARI	
P-39	EFFECT OF EXCHANGEABLE CATION IN MONTMORILLONITE ON THE MULTICOMPONENT REACTION OF BIGINELLI FOR THE SYNTHESIS OF DIHYDROPYRIMIDONES	52
	Farida BOUREMMAD, Shalima SHAWUTI, Mehmet Ali GULGUN	

P-40	MESOPOROUS AI-MCM-41 MOLECULAR SIEVE OR TiCl₄ AS CATALYST FOR THE ALLYLATION OF AROMATIC ALDEHYDES WITH ALLYLTRIMETHYLSILANE	53
	Lamia BRAHMI, Tewfik ALI-DAHMANE, Rachida HAMACHA, Salih HACINI	
P-41	VISIBLE-LIGHT PHOTOCATALYTIC PERFORMANCES OF TiO₂ NANOBELTS DECORATED WITH IRON OXIDE NANOCRYSTALS	54
	Samiha CHAGUETMI, Nadjah SOBTI, Philippe DECORSE, Sophie NOWAK, Fayna MAMMERI, Slimane ACHOUR, Souad AMMAR	
P-42	GOLD CATALYSTS AU/C IN BASE-FREE AEROBIC OXIDATION OF GLUCOSE TO GLUCONIC ACID	55
	Meriem CHENOUF, Charf eddine BOUNOUKTA, Meriem BANOU , Fatima AMMARI	
P-43	NICKEL CATALYSTS FOR HYDRODEOXYGENATION OF FURFURAL-ACETONE CONDENSATION ADDUCTS TO BIO LIQUID ALKANES	56
	Neyla CHERIF BENMOUSSA, Chaima TABET ZATLA, Asma ZITOUNI, Wahiba BENDEDDOUCHE, Redouane BACHIR, Sumeya BEDRANE	
P-44	EFFECT OF THE METAL LOADING ON THE CATALYTIC OXIDATION OF CYCLOHEXENE OVER RUTHENIUM SUPPORTED ON TITANIUM-PILLARED CLAY	57
	Ahmed DALI, Farah GUITOUNI, Ilhem REKKAB-HAMMOUMRAOUI, Abderrahim CHOUKCHOU-BRAHAM	
P-45	COORDINATION OF MODELING AND EXPERIMENTAL RESEARCH OF NANOMATERIAL CATALYSTS FOR THE ADSORPTION AND OXIDATION REACTIONS OF ORGANIC MOLECULES	58
	Atallah DEHBI, Meryem Imene AMRANE , Nadia TOUMI, Abdelkader BENGUEDDACH	
P-46	ANDERSON-TYPE POLYOXOMETALLATES AS CATALYSTS IN ADIPIC ACID SYNTHESIS	59
	Leila DERMECHE, Mouheb LYNDA, Tassadit MAZARI, Siham. BENADJI and Chérifa RABIA	
P-47	EFFECT OF POSITION OF NICKEL ON THE Ni/Mg/Al HYDROTALCITE STRUCTURE FOR THE DRY REFORMING OF METHANE	60
	B. Djebbari, F.Touahra, N. Aider, Z. Abdelsadek, F. Bali, J.P. Holgado, A. Caballero, D. Halliche , K.Bachari.	
P-48	REMOVAL OF 4- (2-PYRIDYLAZO) -RESORCINOL FROM AQUEOUS SOLUTIONS BY AN ADSORPTION TECHNIQUE USING AN ALGERIAN KAOLIN	61
	Toufek METIDJI, Hacene BENDJEFFAL, Abdelkrim DJEBLI, Hadjer MAMINE, Nacer REBBANI, Yacine BOUHEDJA	
P-49	BIOCONVERSION OF LIGNOCELLULOSIC BIOMASS FOR THE PRODUCTION OF BIOETHANOL FROM AGRICULTURAL WASTE	62
	Sohaib DJEDDOU, Khalida BOUTEMAK, Amel HADJ-ZIANE, M.Ayoub FKAIr, Benamar CHEKNANE	
P-50	SYNTHESIS OF METAL-CONTAINING MCM-41 AND ITS APPLICATION FOR CATALYTIC OXIDATION OF PRIMARY ALCOHOL WITH H₂O₂	63
	Houda DOUBA and Ourida BEKHTI-MOHAMMEDI	

P-51	DEVELOPMENTS IN HETEROGENEOUS CATALYSIS FOR THE SUSTAINABLE PRODUCTION OF BIODIESEL	64
	Radjaa EL AHMAR, Zahira Bouyakoub, Pr.Soufi KACIMI	
P-52	SYNTHESIS, CHARACTERIZATION OF CeO₂/SBA-15 MESOPOROUS MATERIAL AND ITS PHOTOCATALYTIC PERFORMANCE IN THE 2,2'-BIPYRIDINE DEGRADATION UNDER VISIBLE-LIGHT IRRADIATION	65
	Abdelkader ELAZIOUTI, Nadjia LAOUEDJ, Naceur BENHADRIA and Bouhadjar BOUKOUSSA	
P-53	HETEROGENEOUS FENTON CATALYSTS UNDER VISIBLE IRRADIATION	66
	Nassira FERROUDJ, Sébastien ABRAMSON, Anne DAVIDSON, Delphine TALBOT, Aude MICHEL	
P-54	EFFICIENT CATALYTIC DEGRADATION OF 4 NITROPHENOL USING SiO₂@Cu CORE@SHELL MATERIALS	67
	Imane GHIAT, Amel BOUDJEMAA, Adel SAADI	
P-55	EFFECT OF THE ELECTROLYTE CONCENTRATION FOR CO₂ PHOTO-ELECTROCHEMICAL REDUCTION BY CuO PHOTO CATALYST: A CYCLIC VOLTAMMETRY STUDY	68
	Fethi GHRIBI, Adlane TAHAR, Amel BOUDJEMAA, Nabila CHREFI-OURADI	
P-56	ULTRASOUND-ASSISTED BIOSORPTION OF BASIC BLUE 9 FROM AQUEOUS MEDIA	69
	El-Khamssa GUECHI, Lamis BEGGAS, Soulef BENABDESSELAM, Oualid HAMD AOUI	
P-57	QUANTITATIVE STUDY OF HYDROPHOBICITY OF MATERIALS IN CATALYSIS	70
	Mouna Wissem GUELLIL, Hafsa RABAH, Abderrahim CHOUKCHOU-BRAHAM, Sanâa EL-KORSO, Souheyla BOUDJEAMA	
P-58	SYNTHESIS OF CYCLIC ENAMINES CATALYZED BY POROUS MATERIALS	71
	Yasmine Rahma HACHEMI, Nabila BOUCHIBA, Abdelkader BENGUEDDACH, Mohammed HAMADOUCHE	
P-59	SYNTHESIS, CHARACTERIZATION AND REACTIVITY OF MESOPOROUS MATERIALS FE-TUD-1	72
	Saliha HADDOUM, Ioana FECHETE, Bertrand DONNIO, Francois GARIN, Doina LUTIC, Chems Eddine CHITOUR	
P-60	GREEN OXIDATION OF CYCLOHEXENE WITH HYDROGEN PEROXIDE OVER GOLD SUPPORTED ON CLAY	73
	Abdelkader HAKKOUM, Radouane BACHIR, Ritha BENABADJI-SOULIMANE, Sumeya BEDRANE, Aderrahim CHOUKCHOU-BRAHAM	
P-61	REMOVAL OF TEXTILE DYES BY ADSORPTION ON ACTIVATED CARBON AND ADVANCED OXIDATION PROCESSES	74
	Sabrina HALLADJA, Naima BENCHIKHA	
P-62	COBALT ALUMINATE NANOPARTICLES SUPPORTED ON MIL-101 STRUCTURE: CATALYTIC PERFORMANCE INVESTIGATION	75
	Mohamed KARMAOUI, Lemia AMAROUCHE and Mohamed HENNOUS	

P-63	HETEROGENEOUS PHOTODEGRADATION OF METHYL ORANGE BY PHOTO-FENTON USING A NATURAL CLAY POWDER	76
	Badis KHENNAOUI, Moulay Abderrahmane MALOUKI, Moisés CANLE LOPEZ, Insaf BATAICHE, Zakaria REDOUANE SALAH	
P-64	ELIMINATION OF HEAVY METALS FROM AQUEOUS SOLUTION USING NATURAL SORBENT (CHITIN AND CHITOSAN)	77
	Imane LANSARI, Khadidja TIZAOUI, Belkacem BENGUELLA	
P-65	DEGRADATION OF AZO DYE IN AQUEOUS SOLUTION BY SEVERAL ADVANCED OXIDATION PROCESSES	78
	Nour El Houda LARBI, Djellali Redha MEROUANI, Abelkader IDDOU ;Amine KHELIFA	
P-66	PHENOL CATALYTIC OXIDATION BY NEW $\text{Bi}_{1.4950}\text{Sb}_{1.415}\text{Co}_{0.44}\text{Fe}_{0.52}\text{O}_7$ PYROCHLORE COMPOUND	79
	Fadia MERABET ; Mayouf SELLAMI ; Kheira ZOUAOUI ; Cherifa MEKKI-DOUADJI, Naceur BENHADRIA	
P-67	HYDROGEN STORAGE OF POROUS SILICON	80
	Saloua MERAZGA, Nour Eddine GABOUZE	
P-68	EFFECT OF DEPOSIT POTENTIAL OF CUXOY NANOCOMPOSITE ON PHOTOCATALYTIC DEGRADATION OF PHENOL	81
	Z. MEZINE, A. KADRI, L. HAMADOU, A. CHAOUCHI, N. BENBRAHIM	
P-69	LA-PROMOTED TUNGSTATED ZICRONIA CATALYST FOR N-BUTANE ISOMERIZATION	82
	Amina MEZOUAGH, Souhila BENDENIA, hanaa DIB, Chahra BENDENIA, Mhamed DJENNAD, Zahira MOHAMED SEGHIR*	
P-70	HE NANOSTRUCTURED $\gamma\text{-MnO}_2$ FILM ELECTROCHEMICALLY PREPARED FOR USED AS NEW PHOTOCATALYST FOR THE DEGRADATION OF AN ORGANIC POLLUTANT	83
	Fatsah MOULAI, Ouarda FELLAHI, Toufik HADJERSI	
P-71	PHOTOCATALYTIC DEGRADATION OF DYES BY MESOPOROUS MgO UNDER SOLAR LIGHT	84
	Souad NACHET, Karima BENDAHOUE, Fatiha HAMIDI, Leila CHERIF - AOUALI	
P-72	KINETIC STUDY OF THE SPIRAMYCIN REMOVAL BY SUPPORTED TiO_2/ACTIVATED CARBON	85
	Amel OUNNAR, Radia DJOUDER, Fayrouz KAIDI, Lidia FAVIER, Abdelkrim BOUZAZA and Fatiha BENTAHAR	
P-73	DEGRADATION OF CONGO-RED BY TI PILLARED CLAYS	86
	Hafsa RABAH, Khadidja KHALDI, Abderrahim CHOUKCHOU-BRAHAM, Djahida LERARI-ZINAI and Khaldoun BACHARI	
P-74	CATALYTIC OXIDATION OF AROMATICS COMPOUNDS OVER CU-CONTAINING LAYERED DOUDLE HYDROXIDES	87
	Ghania RADJI, Amina K Aidari, Abdellah BAHMANI, Nourredine BETTAHAR	

P-75	FFECT OF CALCINATION ON COORDINATION AND REACTIVITY OF MOLYBDENUM SUPPORTED BY SULFATED ZIRCONIA Sahar RAISSI	88
P-76	AN EFFICIENT MICROFLUIDIC REACTOR NANO-ENABLED FOR WATER PURIFICATION FROM VOLATILE ORGANIC COMPOUNDS (VOCs) Imadeddine AZZOUZ, Mohammed Reda RAMDANI, Yamin LEPRINCE, Amel BOUDJEMAA, Djahida LERARI, Khaldoun BACHARI, Tarik BOUROUINA	89
P-77	SYNTHESIS OF HIGHLY ORDERED NONMATERIAL FROM ALGERIAN DIATOMITE MCM-41 AND SBA-15 CATALYSTS FOR ESTERIFICATION OF LAURIC ACID Fatma SAHEL, Rachida HAMACHA, Abdelkader BENGUEDDACH	90
P-78	THE USE OF ADVANCED OXIDATION PROCESSES FOR THE DEGRADATION OF DYES: CASE OF METHYL VIOLET 2B Meryem SAIDI, Meriem TEBSI, Amina BENNOMARA, Malika MOKHTARI, Leila BOUKLI-HACEN	91
P-79	CaTiO₃ -TiO₂ NANOCOMPOSITE PRODUCED BY CLASSICAL AND MICROWAVE HYDROTHERMAL METHOD Nadjah SOBTI, Samiha CHAGUETMI, Slimane ACHOUR	92
P-80	IN THE SELECTIVE OXIDATION OF ORGANIC MOLECULES – SOLVENT INFLUENCE Ritha SOULIMANE, Souhila KHERBOUCHE, Abdelkader HAKKOUM, Redouane BACHIR	93
P-81	A DFT STUDY OF THE PROTOLYTIC CATALYTIC CRACKING REACTION OF N-PENTANE ON ALUMINOSILICATE ZEOLITE Youcef YOUSFI, Wafaa BENCHOUK, Sidi Mohammed MEKELLECHE	94
P-82	IMPROVEMENT CATALYTIC PROPERTIES OF BaCuO₂ DELAFOSSITEIN THE GAS PHASE HYDROGENATION OF BENZALDEHYDE. SYNERGY WITH PARTIAL SUBSTITUTION OF Cu by Ni,Co AND Fe H. Zazoua, F. Touahra, R. Chebout, D. Lerari, K. Bachari, A.Saadi	95
P-83	TEXTILE DYES REMOVAL FROM AQUEOUS SOLUTION USING KAOLIN-BASED ZEOLITE A Nor-el-houda FARDJAOUI, Ibtissem SLATNI, Soumia ZEN, Fatima Zohra EL BERRICHI	96
P-84	KINETIC STUDY OF MOLYBDENUM BASED KEGGIN TYPE CATALYST IN THE CYCLOHEXANONE OXIDATION Lynda Mouheb, Leila Dermeche, Riad Guerroudj, Tassadit Mazari, S. Benadji, Nadine Essayem and Cherifa Rabia	97
P-85	VALORIZATION OF MOLECULAR SIEVE'S END-OF-LIFETIME IN THE REMOVAL OF PHENAZIN DYE IN THE SIMULATED WASTE WATER Nadjia Laouedj, Abdelkader ELAZIOUTI	98

TOPIC II : SPECIALTY CHEMICALS

ORAL COMMUNICATIONS

- O-1 ENANTIOSELECTIVE ORGANOCATALYTIC CONJUGATE ADDITION OF NUCLEOPHILIC IMINES TO α,β -UNSATURATED ALDEHYDES** 101
Houcine CHOUBANE, Alberto F. GARRIDO-CASTRO, Cuauhtemoc ALVARADO, Ana MARTÍN-SOMER, Andrea GUERRERO-CORELLA, Mortada DAAOU, Sergio DÍAZ-TENDERO, M. Carmen MAESTRO, Alberto FRAILE and José ALEMÁN
- O-2 A NEW STRATEGY FOR THE SYNTHESIS OF NOVEL CHROMENO[2,3-*d*]PYRIMIDINE DERIVATIVES** 102
Fatima BELHADJ, Zahira KIBOU, Mohammed BENABDELLAH, Nouraddine CHOUKCHOU-BRAHAM and Didier VILLEMIN
- O-3 PHENYLBORONIC ACID CATALYZED FACILE ONE-POT SYNTHESIS OF 1-AMIDOALKYL-2-NAPHTHOL** 103
Khawla BOUDEBBOUS, Zineddine ZEBBICHE, Abdelmadjid DEBACHE
- O-4 LIPASE-CATALYSED DIRECT C-P BOND FORMATION IN ONE POT SYNTHESIS VIA KABACHNIK-FIELDS REACTION** 104
Samia Guezane-Lakoud, Martial Toffano, Louisa Aribi-Zouioueche
- O-5 SYNTHESIS OF 1,10-DICHLOROBENZO[C]CINNOLINE, AND ITS N-OXIDE WITH SOME STRAINED MOLECULES RELATED TO BIPHENYLENE** 105
Ali LARKEM, Hamama LARKEM.
- O-6 VISIBLE LIGHT-MEDIATED SYNTHESIS OF ORGANOPHOSPHORUS COMPOUNDS: SYNTHETIC AND MECHANISTIC INVESTIGATIONS** 106
Sami LAKHDAR
- O-7 ADVANCED SYNTHESIS OF PHARMACEUTICALS CATENA VIA MACROCYCLIC CYCLONES** 107
Hamama LARKEM, Ali LARKEM
- O-8 *CANDIDA ANTARCTICA* LIPASE-B CATALYZED ALKALINE-HYDROLYSIS OF A SET OF ACETATES IN NON AQUEOUS MEDIA** 108
Mounia MERABET-KHELASSI, Louisa ARIBI-ZOUIOUECHE, Olivier Riant
- O-9 A MILD AND EFFICIENT CATALYST USED FOR THE SYNTHESIS OF 5-ARYLIDINE BARBITURIC ACID DERIVATIVES** 109
Imene SEHOUT, Abdelmadjid DEBACHE
- O-10 CATECHOLASE ACTIVITY OF IN SITU TRANSITION METALS COMPLEXES OF A THIOSEMICARBAZIDE DERIVATIVE LIGAND** 110
Oussama Kheireddine NEHAR, Samira LOUHIBI

POSTER COMMUNICATIONS

- P-01 FORCED DEGRADATION STUDY OF AN ANTIHISTAMINE SYRUP BY HPLC** 112
N. MAKAOUI, S. BOUBEKEUR, M. ALAIMIA, S. KHELIFAOUI, H. IMOUDACHE, H. BENCHAAABANE
- P-02 REMOVAL OF PHENOL FROM AQUEAS SOLUTION WIYH ADSORPTION MUDDY SEDIMENTS: EQUILIBRIUM, KINETIC AND THERMODYNAMIC STUDY** 113
Djelloul ADDAD, Amel DRIF, Fatima Zohra MEBDOUA, Belkhadem MOKHTARI
- P-03 A NEW 1,2,3-TRIAZOLE-BASED LIGAND CONATNING AN ARYLSULFONAMIDE : SYNTHESIS, CRYSTAL STRUCTURE AND COMPUTATIONAL STUDIES** 114
Yassine AIMENE, Achour SERIDI, Eric BENOIST
- P-04 ORTHO-LITHIUM/MAGNESIUM CARBOXYLATE-DRIVEN AROMATIC NUCLEOPHILIC SUBSTITUTION REACTIONS ON UNPROTECTED NAPHTHOIC ACIDS** 115
Regadia AISSAOUI, Aicha DERDOUR, Anne-Sophie CASTANET, and Jacques MORTIER
- P-05 ACETALIZATION OF BENZALDYHEDA AND ACETONE CATALYZED BY ACID ACTIVATED CLAY** 116
Kouider ALALI, Foued LEBSIR , Sondes AMRI, Ali RAHMOUNI, Ezzeddine SRASRA , Néji BESBES
- P-06 PHOSPHOTUNGSTIC ACID TYPE DAWSON AS GREEN CATALYST FOR THE SYNTHESIS OF NEW CYCLIC IMIDES** 117
Sabrina ALIOUA, Chafika BOUGHELOUM
- P-07 SYNTHESIS OF SOME GLYCOSTEROIDAL BOLAPHILES** 118
Fahima ALI-RACHEDI
- P-08 DEPROTO-METALLATION OF *N*-ARYLATED PYRROLES AND INDOLES USING A MIXED LITHIUM–ZINC BASE** 119
Yacine Ameur Messaoud, Ghenia Bentabed-Ababsa, Aicha Derdour, Florence Mongin
- P-09 SYNTHESIS OF SULFONYL ANALOGUES OF FOTEMUSTINE** 120
Zineb AOUF, Sarah BOUGHABA, Monique MATHÉ-ALLAINMAT, Jacques LEBRETON, Nour Eddine AOUF
- P-10 COMPOSITION AND SYNERGISTIC EFFECT OF ESSENTIAL OILS OF *THYMUS FONTANESII* AND *SYZIGIUM AROMATICUM* AGAINST *CANDIDA ALBICANS*** 121
Imane AZZOUNI, Maghnia BOUSSAID, Félix TOMI, Chahrazed BEKHECHI
- P-11 SYNTHESIS OF NOVEL TETRAHYDROBENZO[*b*][1,8]NAPHTHYRIDINES DERIVATIVES** 122
Ikram BABA AHMED, Zahira KIBOU, Nouredine CHOUKCHOU-BRAHAM
- P-12 SYNTHESIS AND ELECTROCHEMICAL PROPERTIES OF 2,5-BIS(2-PYRIDYL)-1,3,4-THIADIAZOLE AND ITS NICKEL COMPLEX** 123

Yamina BABA HAMED ,Amel FELLAH , Yahia HAREK

- P-13 SYNTHESIS AND CHARACTERIZATION OF NEW POLY (4', 4 METHYLENEDIANILINE) NANOCOMPOSITE USING MAGHNITE-Co AS CLAY CATALYST** 124
S.BAHOUSI , A.BELMOKHTAR, A. BELLIFA , AND A.BENYOUCEF
- P-14 A GREEN METHOD FOR THE SYNTHESIS OF BODIPY FROM ARYLDIPYRROMÉTHANE** 125
Nawal BAYOU-KHIER, Mohamed AMARI, Mokhtar FODILI
- P-15 SYNTHETIC STUDIES AND ANTIBACTERIAL ACTIVITIES OF CADIOLIDES ANALOGUES** 126
Sarrah BEKRI, Yazid DATOUSSAID, Stéphane LELEU, Xavier FRANCK, Nouredine CHOUKCHOU-BRAHAM.
- P-16 SYNTHESIS OF THE 1,2,4-TRIAZOLES. BIOLOGIC ACTIVITY** 127
Leila BELHABRI, Adel Ali OTHMAN , Fouzia BENALI
- P17 CHEMICALLY & ENZYMATICALLY SYNTHESIS OF SOME CARBOXYLIC AMIDES** 128
Nour el Houda BENAMARA , Amna ZAÏDI , Mounia MERABET-KHELASSI , Louisa ARIBI- ZOUÏOUECHE, Olivier RIANT
- P-18 ANTI-INFLAMMATORY DRUG ACTIVITY OF THE SEEDS EXTRACTS OF ALGERIAN PRICKLY PEAR** 129
Farah Kenza BENATTIA, Zoheir ARRAR, Youssef KHABBAL
- P-19 DEVELOPMENT OF STUDY ON THE EFFECT OF INHIBITION CORROSION IN A CORROSIVE ENVIRONMENT BY A NEUTRAL POLYMER** 130
Abbes BENCHADLI, Tarik ATTAR, Amel BENKHALED, and Esma CHOUKCHOU-BRAHAM
- P-20 ELECTROCHEMICAL CATHALYSIS OF INHIBITING EFFECT OF NATURAL PROTECT ON HARD GROWTH WATER** 131
Yasmina BENDAOU-BOULAHILIB, Falestine SOUIAD, Samah BOUDIBA, Aissa CHIBANI.
- P-21 CHARACTERIZATION AND TREATMENT OF USED LUBRICATING OILS** 132
Abdelmadjid Mahfoudh BENDJERAD, Naoual CHEIKH, Houcine BENMAHDI, Belkacem DRAOUI
- P-22 MICROWAVE-ASSISTED SYNTHESIS OF N-BENZYLAMINES CATALYZED BY COPPER** 133
Mohamed El Hadi BENHALOUCHE, Abdellah MILOUDI
- P-23 SYNTHESIS OF A NEW FAMILY OF GLYCOLIGANDS BY A SIMPLE AND EFFICIENT REACTION** 134
Chahrazed BENHAOUA, Mustapha RAHMOUNI
- P-24 ELIMINATION OF CHROMIUM (VI) WITH NEW CATALYSTS MATERIALS: POLY(N-VINYLEPYRROLIDONE-SODIUM BENTONITE** 135

Anissa GADIRI, Amel BENKHALED, Djawhar HEDDI and Esma CHOUKCHOU-BRAHAM

- P-25 ZIRCONIUM COMPLEX OF THE ANTIBIOTIC SULFANILAMIDE, THE X-RAY SINGLE CRYSTAL STRUCTURE OF [Zr (C₁₂H₁₇N₄O₄S₂)]₂. NO₃** 136
Sabrina. BENMEBAREK, Hasna. BOUCHAREB
- P-26 AZA-DIELS-ALDER REACTIONS OF CYCLOPENTADIENONES** 137
Amel BENMEDDAH, Bachir MOSTEFA-KARA, Didier VILLEMIN, Nathalie BAR, Jean Francois LOHIER, Remy LEGAY
- P-27 AN EFFICIENT SYNTHESIS OF QUINOLINES AND NAPHTHYRIDINES USING HETEROPOLYACIDE CATALYSTS** 138
Yamina BENTARZI , Bellara NEDJAR-KOLLI
- P-28 SYNTHESIS OF NOVEL CHIRAL INTERMEDIATES THROUGH REGIOSELECTIVE REDUCTIVE CLEAVAGE OF BIS-BENZYLIDENE ACETAL: A NEW ACCESS TO CARBASUGARS** 139
Samiya BENYAMINA, Anne WADOUACHI, W, BOUZZOUI, J.KAJIMA MULENGI
- P-29 ISOXAZOLIDINE'S SYNTHESIS BY INTRAMOLECULAR REACTION** 140
Ahmed Yacine BENZAIM, Ismahène BAZINE , Zinelabbidine CHERAEIT, Abbès BOUKHARI
- P-30 POMEGRANATE (*PUNICA GRANATUM* L.) SEED OIL: COMPOSITION AND PHYSICOCHEMICAL PROPERTIES** 141
Salima BENZERDJEB, Bachir MOSTEFA-KARA, Farid CHEMAT ,Didier VILLEMIN
- P-31 INFLUENCE OF LIPIDS IN FORMULATION OF NANOPARTICLES BY HOT MELTED SONICATION TECHNIQUE** 142
Sarah BOUAMEUR, Wahiba CHAIBI, Fatma Zohra GHANASSI
- P-32 INDIUM-MEDIATED ALLYLATION OF CARBONYL COMPOUNDS WITH FUNCTIONALIZED ALLYLIC HALIDES AND DEHYDRATIVE CYCLISATION TO TETRAHYDROPYRANS DERIVATIVES** 143
Linda BOUARATA, Awatef TABBI, Dahmane TEBBANI
- P-33 SYNTHESIS OF 1, 3, 4- OXADIAZOLE DERIVATIVE AND STUDY OF ITS SURFACTANT PROPERTIES** 144
Mansouria BOUCHAKOUR , Hocine CHOUBANE, Mortada DAAOU
- P-34 SELECTIVE SYNTHESIS AND POLYMORPHISM OF A NEW FERROCENYL ALLYLPHOSPHONIUM SALT** 145
Rafika BOUCHENE, Sofiane BOUACIDA, Jean-CLAUD DARAN· Eric MANOURY,
- P-35 H₆P₂W₁₈O₆₂.14H₂O CATALYZED SYNTHESIS, SPECTRAL CHARACTERIZATION OF α-AMINOPHOSPHONATES CONTAINING AMINOTHIAZOLE MOIETY** 146
Sara BOUGHABA, Zineb AOUF, Nour-Eddine AOUF
- P-36 MICROWAVE-ASSISTED ONE-POT SYNTHESIS OF NEW SULFONAMIDES CONTAINING BENZOTHAZOLE MOIETY CATALYZED BY HETEROPOLYACIDS** 147
Chafika BOUGHELOUM, Abdelrani MESSALHI

P-37	SYNTHESIS AND EVALUATION OF THE ANTIOXIDANT ACTIVITY OF TRIPODAL STRUCTURES BASED ON PYRAZOLE	148
	Nadia BOUHASSANE, Nouria MERAD.	
P-38	ORGANOCATALYTIC 1, 4-CONJUGATE ADDITION OF N-SUBSTITUTED BARBITURIC ACID ON α,β-UNSATURATED KETONES: SYNTHESIS, NMR AND SINGLE-CRYSTAL X-RAY DIFFRACTION STUDIES OF NOVEL 2,4-DIMETHYL-7,11-DIARYL-2,4-DIAZASPIRO[5.5]UNDECANE-1,3,5,9-TETRAONE DYADS	149
	Leïla BOUKENNA, Oualid TALHI, Ridha HASSAINE, Yahia RACHEDI, Nadia TAIBI, Khaldoun BACHARI and Artur M.S.SILVA.	
P-39	SYNTHESIS AND REACTIVITY OF FUNCTIONALIZED 5-ARYLIDENE-2-THIOXOTHIAZOLIDIN-4-ONE	150
	Chaima BOUREGHDA, Rima LAROUM, Abdelmadjid DEBACHE	
P-40	EFFECT OF 5 METHYL 1H, BENZOTRIAZOL ON THE CORROSION INHIBITION OF API N 80 STEEL IN ALBIAN WATER	151
	Fatiha CHELGHAM, Nouredine BOUZID, Mokhtar SAIDI, Souheyla BOUDJEMA, Messaouda DAKMOUCHE, Adel TAABOUCHE and Noura MEDDOURA	
P-41	QUANTITATIVE CONFORMATIONAL STABILITY HOST-GUEST COMPLEX OF METHIONINE WITH B-CYCLODEXTRIN: A THEORETICAL INVESTIGATION	152
	Mouna CHERIET, Abdelkrim GUENDOUI, Leila NOUAR	
P-42	SYNTHESIS OF A PHOTOCROSSLIKABLE MACROMONOMER PROMOTED BY A GREEN CATALYSIS	153
	Zakaria CHERIFI, Aniss ZAOUI, Rachid MEGHABAR	
P-43	SYNTHESIS OF NEW TRIAZINE DERIVATIVES BY CYCLIZATION OF 2-PYRIDYL HYDRAZONES	154
	Zahia Cheurfa, Mohamed Khodja	
P-44	A CASCADE SYNTHESIS, BIOLOGICAL EVALUATION AND MOLECULAR MODELING OF SOME NOVEL TACRINE-PYRANOPYRAZOLE DERIVATIVES AS CHOLINESTERASE INHIBITORS	155
	Chamseddine DERABLI, Imen BOUALIA, Ahmed B. ABDELWAHAB, Raouf BOULCINA, Chawki BENSOUICI, Gilbert KIRSCH and Abdelmadjid DEBACHE	
P-45	A GREEN SYNTHESIS AND ANIONIC POLYMERIZATION OF METHACRYLIC MONOMERS N-ALKYL METHACRYLAMIDE CATALYZED BY AN ECOLOGIC AND FRIENDLY CATALYST MAGNITE H⁺	156
	Samira DERKAOUI, Mohammed BELBACHIR	
P-46	IODINE-PROMOTED SYNTHESIS OF AROYLBISINDOLIZINES FROM N-PHENACYLPYRIDINIUM SALTS AND ACTIVATED ALKINES	157
	Andreea-Veronica DEDIU, Bianca FURDUI, Rodica Mihaela DINICĂ	
P-47	SYNTHESIS AND STUDY OF FIELD HETEROCYCLIC DERIVATIVES CONTAINING NITROGEN ATOM	158
	Madiha DJAHIECHE, Abbas BOUKHARI, Smail KHELILI, Cianluigi BROGGINI	
P-48	ACTIVATED CARBON FROM JUJUBE STONES FOR THE REMOVAL OF RHODAMINE-B FROM AQUEOUS SOLUTION	159

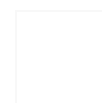
	Radia FENNOUH, Oumessaad BENTURKI	
P-49	SYNTHESIS, CHARACTERIZATION, BIOLOGICAL ACTIVITY AND DOCKING STUDY OF NOVEL SCHIFF BASE	160
	Mariem GHAMRI ; Dalal HARKATI	
P-50	ELECTIVE SYNTHESIS OXAZABOROLIDINE-BORANE REDUCTION OF INDANE-1,3-DIONE	161
	Hind GUEMMOUR , Djaffar KHEFFACHE, Ahmed. BENABOURA	
P-51	A NOVEL AND GREEN PROCEDURE FOR THE SYNTHESIS OF SULFAHYDANTOINS UNDER ULTRASOUND IRRADIATION	162
	Meriem GUERFI, Khawlla BECHLEM, Malika BERREDJEM	
P-52	CHEMICAL SYNTHESIS AND STUDY OF H-BONDING INTERACTIONS IN FUNCTIONALIZED O/P-XYLYL IMIDAZOLIUM DICATIONIC IMIDAZOLIUM IONIC LIQUIDS	163
	Boumediene HADDAD, Mokhtar DRAI , Mostefa BOUMEDIENE ,Didier VILLEMIN , Annalisa PAOLONE Johannes KIEFER , Mustapha RAHMOUNI	
P-53	NOVEL MULTI-COMPONENT SYNTHESIS OF AMRINONE ANALOGUES	164
	Wassila HADJ SLIMANE, Zahira KIBOU, Nouredine CHOUKCHOU-BRAHAM	
P-54	REACTION OPTIMIZATION OF THIOALKYLATION AND THIOARYLATION BY CLASSICAL WAY AND MICROWAVE	165
	Adnane HALIMA-SALEM, Abdellah MILOUDI	
P-55	SIMULATION STUDY ON H₂S REMOVAL FROM ALGERIAN NATURAL GAS ON HIGH DENSITY POLYETHYLENE MEMBRANE	166
	Abdel Illah HAMMOU and M'hamed. DJENNAD	
P-56	SYNTHESIS, CHARACTERIZATION, CONFORMATIONAL AND COMPLEXATION STUDIES OF DI-SUBSTITUTED CALIX[4]ARENE DERIVATIVES BEARING N-ACYLHYDRAZONE UNITS	167
	I. HAMZI, T.M. BARHOUMI-SLIMI, R. ABIDI	
P-57	SOLVENT-FREE SYNTHESIS OF BIOLOGICALLY ACTIVE AMINOPHOSPHONATES UNDER MICROWAVE IRRADIATION	168
	Abdelkader HELLAL, Salah CHAFAA, Lasnoui TOUAFRI	
P-58	VALORIZATION OF AN AGRO-FOOD WASTE FROM N'GAOUS ORANGE JUICE FACTORY IN WATER DEPOLLUTION	169
	Maya KEBAILI, Souad DJELLALI, Manelle RADJAI, Hakim LOUNICI	
P-59	SYNTHESIS, PHARMACEUTICAL FORMULATION and EVALUATION OF ANTI-ADDICTION OF NEW ANALOGUES OF BACLOFENE® AS ANTI-CRAVING AGENTS	170
	Assia KENICHE, Mohammed EL AMINE SI SAID, Joseph KAJIMA MULENGI , Ibtissem LOUAR	
P-60	SYNTHESIS AND BIOLOGICAL EVALUATION OF MONO AND DISUBSTITUTED KETOAMIDES	171
	Riad Mustapha KERBADOU, Assya TALEB, Salih HACINI, Hadjira HABIB ZAHMANI.	

P-61	ONE-POT SYNTHESIS OF IMIDAZO[1,5-a]IMIDAZOLE DERIVATIVES	172
	Nassima KESRAOUI, Yahia RACHEDI	
P-62	SYNTHESIS, CHARACTERIZATION AND ANTIBACTERIAL ACTIVITY STUDIES OF POLY(ESTER-AMIDE)	173
	Yasmina KHANE, Lahcen BELARBI, Fatma DAR KEBIRA, Benali MOUFFOK.	
P-63	PHASE EQUILIBRIUM PROPERTIES OF BINARY SYSTEMS CONTAINING 1-BUTYL-3-METHYLIMIDAZOLIUM THIOCYANATE [BMIM][SCN], WATER, 1-PROPANOL, OR 1-BUTANOL) AT SEVERAL TEMPERATURES	174
	Asma KHELASSI-SEFAOUI, Fabrice MUTELET , Ilham MOKBEL , Jacques JOSE , Latifa NEGADI	
P-64	TOTAL SYNTHÈSE OF MACROCYCLIC MARINE ALKALOIDS, HALICLAMINE A	175
	Ibrahim KHETTAR, Ayyoub SELKA , Ghaniya FAROUANI , Chewki ZIANI-CHERIF	
P-65	SYNTHESIS AND PHARMACOMODULATION ALDIMINES: ALDIMINES 2-METHYL IMIDAZO [1,2-a] PYRIDINE-3-CARBOXALDEHYDE	176
	Abderrahmane KRIM, Saïd GHEZALI	
P-66	1,3,4-OXADIAZOLE, 1,2,4-TRIAZOLE and 1,3,4-THIADIAZOLE SYNTHESIZED FROM 2-FLUOROBENZOIC ACID: PHYSICO-CHEMICAL CHARACTERIZATION AND PRELIMINARY EVALUATION OF ANTIBACTERIAL PROPERTIES	177
	Fatima Zahra LAHMAR , Adel ALI-OTHMANE	
P-67	SHORT AND EFFICIENT SYNTHESIS OF PEPTIDOMIMETICS WITH SATURATED HETEROCYCLICS	178
	Imane LAZOUNI, Wassila DRICI, Joëlle PERARD	
P-68	SYNTHÈSE DES TETRAHYDROQUINOLEINES 2- SUBSTITUEES <i>via</i> L'-AMINONITRILE	179
	Fadila LOUAFI, Saurabh SHAHANE, Jean-Pierre HURVOIS	
P-69	DETERMINATION OF THE MICROBIAL ACTIVITY OF A GLYCOSIDE ANALOGUE DERIVATIVE OF A CARBOXYLIC ACID (LACTIC ACID)	180
	Fatima Imane MAHI, Adel ALI OTHMANE	
P-70	EFFECT OF PYRIDO [2,3-D] PYRIMIDINES ON T CELL PROLIFERATION	181
	Amel MEDJDOUB , Fatima BELHADJ, Amel SAIDI MERZOUK, Nouredine CHOUKCHOU BRAHAM, Hafida MERZOUK	
P-71	SYNTHESIS OF NEW SUBSTITUTED BIS-2-PYRIDONES DERIVATIVES FROM ENAMINONITRILES UNDER FREE-MILD CONDITIONS	182
	Nawel MEHIAOUI, Zahira KIBOU, Nouredine CHOUKCHOU-BRAHAM	
P-72	KEY FACTOR AFFECTING THE BASICITY OF MESOPOROUS SILICAS MCM-41: EFFECT OF SURFACTANT EXTRACTION TIME AND SI/AL RATIO	183
	Amel MEKKI, Bouhadjar BOUKOUSSA, Aboubakr HAKIKI, Zahira KIBOU, Nouredine CHOUKCHOU-BRAHAM, Abdelkader BENGUEDDACH, Rachida HAMACHA .	

P-73	EMULSION IONIC LIQUID MEMBRANE FOR RECOVERY PROCESS OF LEAD. COMPARATIVE STUDY OF EXPERIMENTAL AND RESPONSE SURFACE DESIGN	184
	Meryem MESLI, Nassereddine BELKHOUCHE ,Zineb SEFROU	
P-74	SYNTHESIS OF ARYLIDENES PYRROLIDI-3-YL)-1,2,4-TRIAZOL-4-AMINE AS POSSIBLE CDK'S KINASES INHIBITORS	185
	N. METAHRI ; A.SAAD EL HACHEMI AMAR ; A.SAFER	
P-75	SYNTHETIC OF NEW POLYMERS FROM POLYSTYRENE MERRIFIELD VIA COOPER IODIDE CATALYZED CLICK CHEMISTRY AND APPLICATION IN TREATMENT OF WASTEWATER : EXTRACTION OF NITRATE AND METALS	186
	Abid OUERGHUI, Mokthar DARDOURI , Hichem ELAMARI and Christian GIRARD	
P-76	ANTI TUMOR MOLECULES: SELECTIVE DESIGN FOR SYNTHESIS OF NEW HETEROCYCLIC COMPOUNDS CALLED FROG AND FOX CATALYZED BY MAGHNITE (ALGERIAN MMT)	187
	Abdelkader RAHMOUNI , Mohammed BELBACHIR	
P-77	SYNTHESIS OF NEW PYRAZOLONE AND FUSED PYRAZOLONE HETEROCYCLIC DERIVATIVES	188
	Lydia SAIDI, Yamina BENTARZI, Ricardo F.MENDES, Filipe A. ALMEIDA PAZ, Bellara NEDJAR-KOLLI & Artur M. S. SILVA	
P-78	CYCLIZATION REACTION OF THIOSEMICARBAZONE FOR FUNCTIONALYZED 4-THIAZOLIDINONES. EVALUATION OF ANTIOXYDANT ACTIVITY	189
	Lydia SALHI, Samia ACHOUCHE-BOUZROURA, Bellara NEDJAR-KOLLI, Sophie POULAIN-MARTINI, Elisabet DUNACH	
P-79	SYNTHESIS, CHARACTERIZATION AND ANTIOXIDANT PROPERTIES OF NEW 2-AMINOPYRIDINE DERIVATIVES	190
	Fadhila SALHI, Naoual CHEIKH,Didier VILLEMIN, Nathalie BAR, Siham BENANANE, Nouredine CHOUKCHOU-BRAHAM and Bachir MOSTEFA-KARA	
P-80	SEM AND FTIR STUDY OF ADSORPTION CHARACTERISTICS USING XANTHATE (PIPX) SYTHESISED COLLECTORS ON GALENA	191
	Zohir NEDJAR, Asma NOUIOUA , Lazhar SALMI, Djamel BARKAT	
P-81	ENCAPSULATION OF THE ASCORBIQUE ACID IN A MATRIX OF CELLULOSE TRIACETATE , AN ETHYL CELLULOSE AND THEIR CHARACTERIZATIONS	192
	Khaldia SEDIRI , Lamia BENNABI, Lahcene BELARBI	
P-82	A MILD AND EFFICIENT CATALYST USED FOR THE SYNTHESIS OF 5-ARYLIDINE BARBITURIC ACID DERIVATIVES	193
	Imene SEHOUT, Abdelmadjid DEBACHE	
P-83	CHROMIUM (VI) ADSORPTION BY A NEW VINE-WOOD AND CONDUCTIVE POLYMER COMPOSITE	194
	Rachida SOUIDI, Lahcene BELARBI	
P-84	MOLECULAR DYNAMICS OF PROTON DELOCALIZATION IN CROWN ETHER	195
	Wafaa TALEB BENDIAB, Abdelghani KRALLAFA	

P-85	SELECTIVE REMOVALS OF HEAVY METALS FROM AQUEOUS SOLUTIONS ONTO NATURAL MARNE CLAY (NMC) IN SINGLE, BINARY AND TERTIARY SYSTEMS	196
	Khadidja TIZAOUI, Belkacem BENGUELLA	
P-86	SYNTHESIS OF NEW THIAZOLOPYRIMIDINE DERIVATIVES	197
	Yousseuf TOUATI, Mohammed BENABDELLAH, Ridha HASSAINE, Nouredine CHOUKCHOU-BRAHAM	
P-87	ULTRASOUND PROMOTED SYNTHESIS OF A ROSIN BASED IONIC LIQUID MONOMER	198
	Aniss ZAOUI, Zakaria CHERIFI, Mohammed BELBACHIR	
P-88	SYNTHESIS OF BIOLOGICALLY ACTIVE A-AMINOPHOSPHONATES	199
	Samiya ZAOUT, A.Elkader HALLEL , Salah CHAFAA	
P-89	EFFICIENT SYNTHESIS OF ACRIDINES CATALYSED BY COPPER	200
	Sarah ZEGHADA, Ghenia BENTABED-ABABSA, Aïcha DERDOUR, Florence MONJIN	
P-90	AN EFFICIENT APPROACH FOR THE SYNTHESIS OF SOME NOVEL HYBRIDIZED QUINOLINE-PYRAN BASED TACRINE ANALOGUES	201
	Abd el karim SANDELI, Mohamed Abdenour Redouane, Saida BENZERKA and Naima KHIRI-MERIBOUT	

Lectures



NANOTECHNOLOGY ENABLES HOT GOLD NANORODS TO KILL CANCER CELLS AND TO STOP ALIVE CELLS FROM MIGRATING AND KILLING MORE SICK PEOPLE

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Abstract

Cancer kills many people after several years of suffering and after using a great deal of different unsuccessful treatments, like surgery, chemical and/or radiation treatments. The field of Nanotechnology showed us how different materials acquire so many different properties when their size is reduced to the nanometer scale. Gold nanoparticles having rod shape of nanometer size and a length: width ratio of 3:1 can absorb near infrared light (to which our body is transparent) and convert it into heat. If solution containing gold nanorods is injected into a cancer lump and exposed to near infrared light the hot solution (resulting from the gold nanorods upon absorbing the near infra-red light) melts the cancer cells leading to their death. This was demonstrated by our group in the photo-thermal destruction and destroying cancer cells in solution, in cancer lumps in small and in large animals (1,2,3,4).

Normally, some of the cancer cells that do not die are able to migrate to other parts of the body away from the location of their initial formation spot until they are located in a sensitive part of the body that leads to the cancer patient death. Thus most of the cancer patients die from cancer after the cancer cells migrate by a process called metastasis to more a very sensitive part of the body

Very recently, however, we discovered (5) that in our photo-thermal treatment, while treating cancer cells in the first cancer location with hot gold nano-rods, the cancer cell legs and arms and the motion proteins are photo-thermally destroyed. This makes it difficult for the cancer cells to migrate to new more important functional locations in the body. This treatment is thus effective in stopping cancer cell migration through the patient body and increases the success rate of the patient recovery.

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TRANSITION METALS: POWERFUL TOOLS FOR THE CONSTRUCTION AND FUNCTIONALIZATION OF HETEROCYCLES

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Abstract

In general, the synthesis of complex biologically active molecules are problematic, but the problems encountered during the syntheses can be a good source of inspiration to develop methods. One major challenge is the design of concise strategies as well as chemoselective and efficient methods that rapidly lead to the skeleton framework of natural and/or biologically active heterocyclic compound as well as to their functionalization.

In this context, we have explored the construction of heterocycles using catalytic reactions involving transition metal catalysts. Metal catalysts can induce cyclizations and functionalizations which can be highly diastereoselective and enantioselective if a chiral ligand is added in the reaction media. These reactions and their applications to the synthesis of heterocyclic natural and non-natural products will be presented.

Keywords: Heterocycles, construction, functionalization, transition metals.

THE ROLE OF SPECTROSCOPY IN UNDERSTANDING HETEROGENEOUS CATALYSTS

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Abstract

Spectroscopies are experimental techniques that investigate the interaction between matter and a particle beam (photons, neutrons, electrons, or ions), by monitoring the sample response as a function of the energy of the incoming or outgoing beam(s). Addressing the complexity of heterogeneous catalysts, light-based spectroscopies have raised relevance in the understanding of structural and functional properties, exploiting the full range of the electromagnetic spectrum, from microwaves (that probes the nuclear or electron spins), to the hard x-ray (that probe electronic transition from core to valence states). In general, in the study of a heterogeneous catalyst, there is never a single technique able to fully describe the system, disclosing the nature and the number of the active sites and how they evolve along the reaction, while a combination of different approaches is foreseen. Moreover, apart from the use of a variety of techniques, the possibility to perform an experiment in controlled atmosphere, at different temperature and using time and space resolved techniques, enlarge the possibilities thanks also to the enhanced sensitivity of the experimental apparatus and the combined availabilities of traditional laboratories and largescale facilities, like Synchrotron and neutron scattering sources. Finally, more and more molecular modelling approaches are extensively applied in combination with experiments, allowing a deeper understanding of the complex phenomena that occur at the surfaces of heterogeneous catalysts.

In this contribution, a short list of most common “*Frequently Asked Questions*” (FAQ) related to spectroscopies applied at the heterogeneous catalysts will be summarized. Moreover, a key study (Titanium Silicalite) will be described in more detail.

CYCLOADDITION AND CONJUGATE ADDITION REACTIONS ON CHROMONES LEADING TO OTHER OXYGEN AND NITROGEN HETEROCYCLIC COMPOUNDS

Artur M. S. Silva

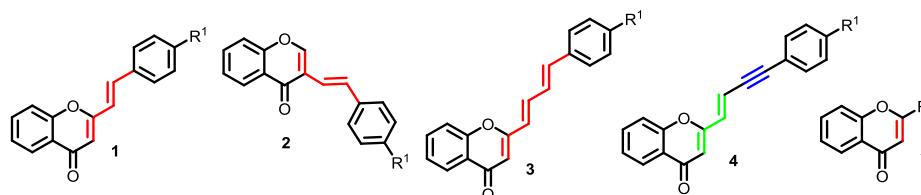
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Oxygen and nitrogen heterocyclic compounds constitute the largest and most varied families of organic compounds, comprising a great number of classes according to the size, number of heteroatoms and heterocyclic ring oxidation. The important industrial and biological applications of these types of compounds and also some problems associated with their application, such as multiple drug resistance to some nitrogen heterocycles and potential carcinogenesis of high doses of oxygen heterocyclic-based antioxidants, led us to develop new synthetic methods for novel biologically active derivatives of both referred families of heterocyclic compounds.

Taking into account our interest on the chemistry of chromones, namely on derivatives bearing unsaturated substituents at C-2 and C-3, we have established new synthetic routes for (*E*)-2- and 3-styrylchromones **1** and **2**, 2-[(1*E*,3*E*)-4-arylbuta-1,3-dien-1-yl]chromones **3** and (*E*)-2-(4-arylbut-1-en-3-yn-1-yl)chromones **4** and studied the reactivity of their unsaturated systems in cycloaddition and conjugate addition reactions [1,2].

In the last years, we have also been interested on the use of chromones bearing electron withdrawing substituents at position 3 **5** as building blocks for the synthesis of several other nitrogen and oxygen heterocycles. The key step of these syntheses involve conjugated and cycloaddition reactions and allowed us to prepare a plethora of different structures [3].

We will present and discuss the chemistry involved in each one of the referred synthetic routes and also some of the biological activities presented by some of those heterocycles.



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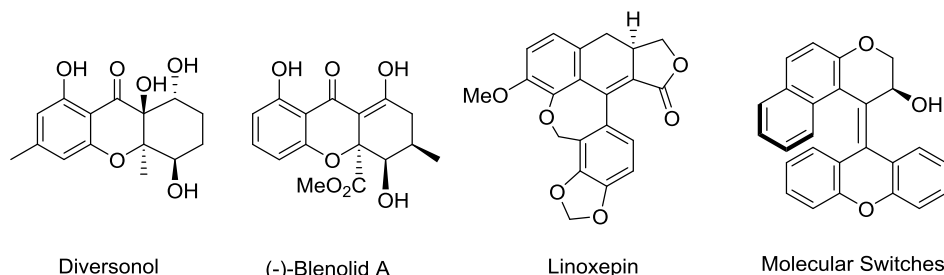
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CATALYTIC DOMINO REACTIONS FOR THE ECOLOGICALLY BENIGN SYNTHESIS OF NATURAL PRODUCTS AND MATERIALS

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The efficient synthesis of natural products, drugs, agrochemicals and materials is a very important aspect in academia and industry. To allow an ecologically and economically favourable approach in a green fashion the former stepwise procedures must be replaced by domino reactions which allow the preparation of complex molecules starting from simple substrates in a straight forward way. Domino reactions¹ allow the reduction of the amount of waste being formed and the preservation of our resources. Moreover, they are also favourable in an economical way since they consume less time.



The usefulness of the domino concept is demonstrated with the syntheses of fungal metabolites^{2,3,4} using an enantioselective domino-Wacker/carbonylation/methoxylation reaction as well as of natural aryldihydronaphthalene lignans⁵ employing a domino-carbopalladation/Heck reaction. The approach has also been applied for the synthesis of novel materials such as molecular switches⁶ and fluorescence dyes⁷ using a domino-Sonogashira/carbopalladation/CH-activation reaction.

Keywords: Organic synthesis / Natural products / Materials / C-H-activation / Pd-chemistry / Enantioselective reactions

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RECENT ADVANCES IN THE CHEMISTRY AND SYNTHETIC USES OF LITHIATED SMALL RING HETEROCYCLES

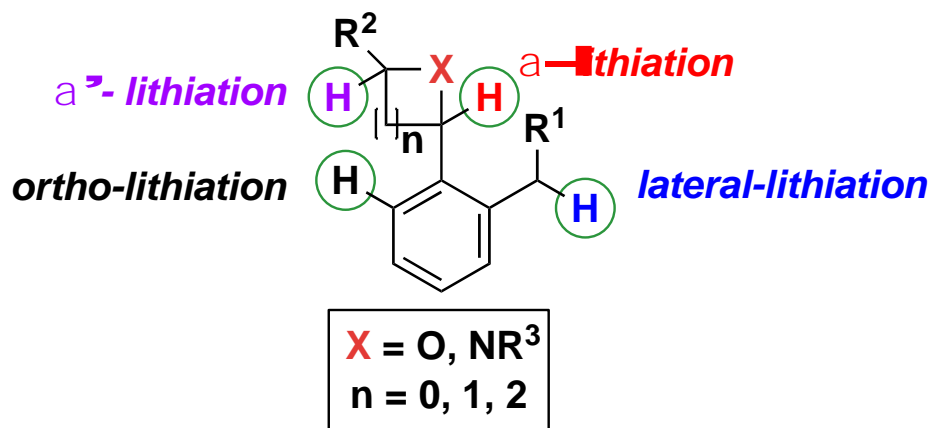
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Abstract

Over the past ten to fifteen years, α -lithiated small ring heterocycles from being just elusive intermediates have risen to the state of precious reactive intermediates and very useful synthons. Indeed, by fine-tuning the experimental conditions, they can be smoothly generated from commercially or promptly available precursors, mainly by deprotonation using strong bases (RLi, LiNR₂) or by transmetalation (Li-S, Li-Sn, Li-Si exchange), and successfully trapped with a variety of electrophiles. The carbenoid character of these species can be traced in their reactions including alkylation, allylation, arylation, eliminative dimerization, C-H and C-C insertion, isomerization, cyclopropanation. Moreover, the possibility of elaborating the heterocyclic moiety increases the synthetic potential of such intermediates.

This lecture will focus mainly on the lithiation of arylated small-ring systems and reactions of the resulting α -, α' -, *ortho*- and *lateral* metalated species. It will be shown that experimental conditions in terms of coordinating solvent ability, ring substitution, temperature, concentration, nature of the heteroatom, complexation and dynamic phenomena and presence of additives which may influence both aggregation and solvation, keep under control the regioselectivity of the lithiation-trapping sequence (α - vs *ortho*-, and *lateral*-lithiation) as well as the stereoselectivity of their reactions with electrophiles.



A selection of the most recent synthetic applications of the above lithiated species will be highlighted.

ALKANE PROTOLYTIC CRACKING BY ZEOLITES: IN SEARCH OF THE REACTANT STATE

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Abstract

The protolytic cracking of alkanes by acidic zeolites, discovered nearly 30 years ago by Dessau and Haag is archetypical of acid-catalyzed reactions and has been the subject of many experimental and theoretical studies. Still, the factors ruling this reaction are not understood because the catalytic activity is influenced by both adsorption thermodynamics and intrinsic cracking kinetics of the alkane adsorbed on the acid site. It is now generally admitted that the variations of apparent cracking rates are primarily governed by the intrinsic kinetics. However, strong discrepancies still persist as to the rate parameter which determines these changes, i.e. activation entropy vs. activation energy, as well as the magnitude of these parameters, in particular entropic factors. Overall, depending on the studies, the estimates of the coverage of active sites and of the intrinsic kinetics span several orders of magnitude. The origin of these huge discrepancies lies in the absence of experimental measurement of the coverage prevailing at reaction conditions, which up to now could only be assessed by extrapolation of low temperature measurements or state of the art simulations. In this talk, I will show how the use of IR *operando* spectroscopy allows for unambiguous determination the coverage of zeolites active sites by the alkane - the reactant state - and hence the intrinsic cracking kinetics of these alkane-acid sites complexes. The influence of the alkane confinement within the zeolite pores will be discussed.

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A JOURNEY IN CYCLOPEPTOIDS' CHEMISTRY

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Abstract

Macrocyclic peptoids, cyclic oligomers of *N*-substituted glycines, are an archetypal example of bioinspired peptidomimetics, which show interesting structural and physical properties and strong potentials as guest-binding receptor molecules.[1] A unique advantage of this class of compounds stems from their modular structure, enabling facile synthesis of large libraries of derivatives by modifying the backbone decoration or the ring size.[2] Moreover remarkable cation-binding ability has been demonstrated for differently cavity sized cyclic peptoids.[3] In this contribution our recent results on their glycosidase inhibition [4] and ionophoric activities and application in asymmetric phase-transfer catalysis [5] will be reported.

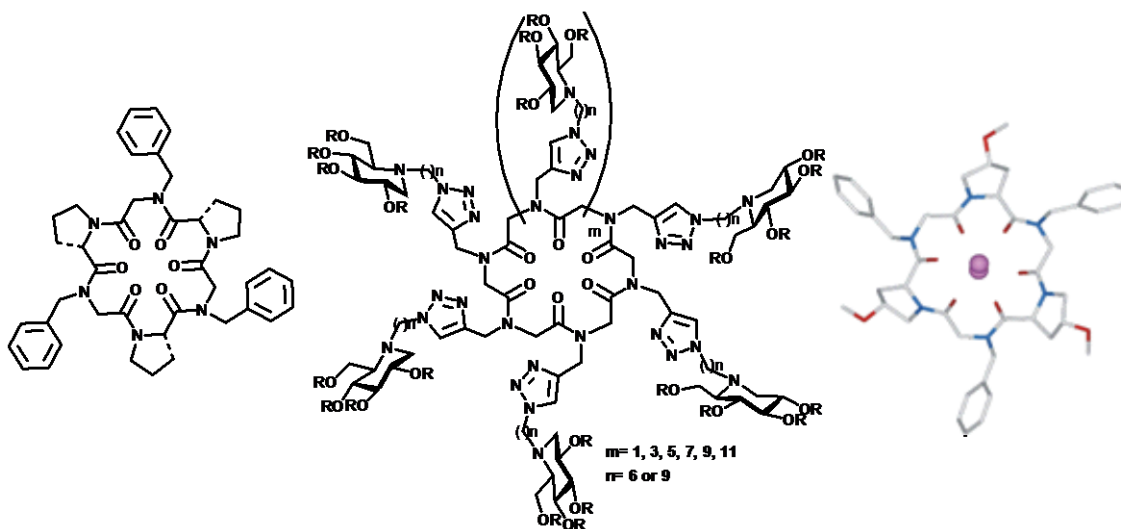


Figure 1. Cyclic peptoids

Keywords: Peptoids, asymmetric synthesis, bioactive compounds

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SYNTHESIS AND DESIGN OF FEW LAYER GRAPHENE-BASED COMPOSITES FOR CATALYSIS AND ENERGY RELATED APPLICATIONS

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Abstract

The accelerating societal requirements for energy together with a need of environmental respect calls for the development of energy efficient green approaches including technologies, materials and synthesis. It relates to the replacement of crucial, highly active but cost-deficient materials such as noble metal catalysts or some metal oxide electrodes (Pt, ITO). An important key to get optimum benefit from new crucial materials is the tailoring of their properties through the appropriate tailoring of their structures. In this context the tailoring of few layer graphene (FLG) and FLG-based metal NPs structures are discussed. The FLG is obtained by highly efficient and green synthesis [1]. The macroscopic bio-inspired assemblies of FLG such as fractal-like conductive networks for (transparent) electrodes are investigated [2]. Other fractal-like smaller architectures deals with tailoring of individual FLG sheets and fractal (re)active FLG edges, through catalytic etching [3]. Figure 1 illustrates the TEM micrographs of FLG with straight and fractal edges. On the other hand, the FLG sheets are also used to structurize metal NPs into high surface-to-volume ratio 2D NPs (Fe, Pd) [4]. The latter one, Pd/FLG hybrid, showed high electrocatalytic performance in oxygen reduction reaction [5].

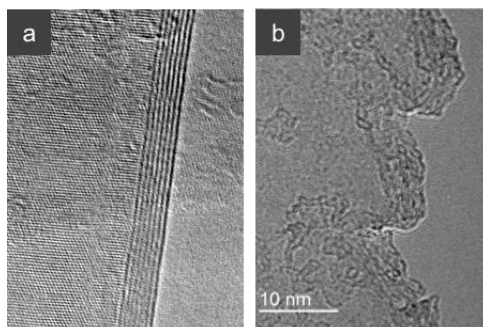


Figure 1. TEM micrographs of FLG with straight (a) and fractal (b) edges.

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N-HETEROCYCLIC CARBENES (NHCs) COMPLEXES OF Zn(II) AND GROUP 13 METALS: USE IN POLYMERIZATION CATALYSIS FOR BIOMATERIALS PRODUCTION AND FOR CO₂ FUNCTIONALIZATION

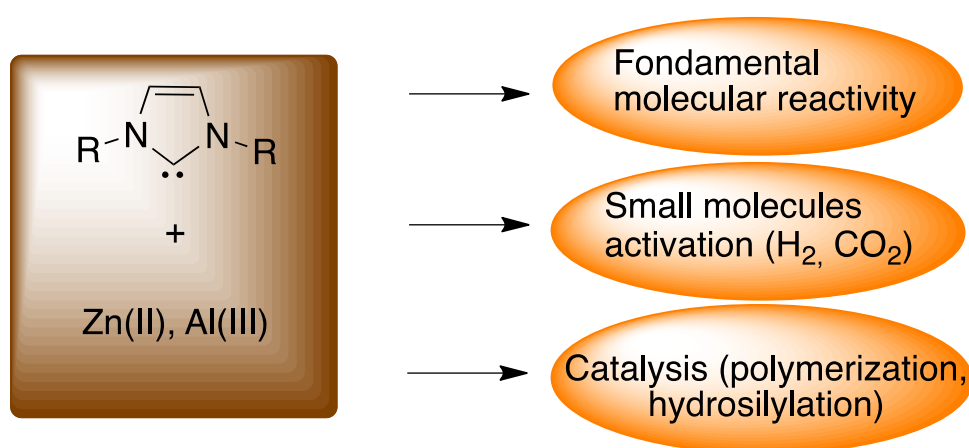
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Abstract

Over the past twenty years, *N*-heterocyclic carbenes (NHCs) have become an ubiquitous class of supporting ligands in coordination chemistry with countless successful applications of the derived NHC metal complexes in various areas. Due to their exceptional σ -donating properties and steric tunability, NHC-bonded metal complexes usually exhibit an inert NHC-M bond yielding enhanced stability: this has opened the way to the development of robust NHC-incorporating metal compounds that often feature an increased activity/selectivity in catalysis and may exhibit novel fundamental reactivity. Though less investigated thus far, combining NHCs with main group elements and/or oxophilic metal centers may also be the source of unusual reactivity and/or unprecedented activation chemistry.

We are interested in the NHC coordination chemistry of oxophilic and high-oxidation state metal centers (which are frequently cheap metal sources) for fundamental reactivity/activation studies and the use of the derived complexes in sustainable catalysis. In the present seminar, discrete NHC-Zn(II) and NHC-Al(III) species are described along with their reactivity trends. Such simple coordination complexes are shown to be of rather broad interest since they may be involved in various chemical transformations ranging from unusual fundamental reactivity (rearrangement processes), small molecules activation (H₂, CO₂) and homogeneous catalysis (cyclic esters/carbonates polymerization, hydrosilylation catalysis). All these aspects will be discussed.



Selected refs: *Chem. Commun.* **2010**, 46, 2480; *ChemCatChem* **2014**, 6, 1357; *Chem. Eur. J.* **2015**, 21, 17959; *Organometallics* **2016**, 35, 1726; *Chem. Eur. J.* **2017**, 23, 5509; *Chem. Eur. J.* **2017**, 23, 15908.

SYNTHESIS OF FIVE-MEMBERED AROMATIC HETEROCYCLES
APPLICATION TO THE PREPARATION OF POTENTIAL ACTIVE COMPOUNDS AND
NEW SCAFFOLDS

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The presentation will give an overview of methods developed over all the years for the synthesis of heterocyclic compounds like thiophenes, selenophenes, pyrroles, thiazoles, selenazoles containing different functions.

The common point of this method was the use of the reactivity of active methylene group present in very common starting material.

Application of the methodology was also to target compounds with specific activities in different domains.

SUPPORTED GOLD NANOPARTICLES. EFFICIENT HETEROGENEOUS CATALYSTS FOR OXIDATION REACTIONS AND C-C COUPLINGS IN LIQUID PHASE

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Abstract

Gold is a noble metal that is currently used in different technologies and industries (environmental protection, electronics, medicine, catalysis, nanotechnologies, ...) In reality, this has not always been the case since it has long been considered to be chemically inactive and particularly without any catalytic activity. It was used mainly for coinage, jewelry, and other arts.

However, in 1987 Haruta publication [1] triggered a breakthrough for gold to serve as an extraordinary heterogeneous catalyst in the low temperature oxidation of CO. It is even more active than other metals. This was absolutely not known before. Hutchings works [2, 3], in the same period, confirmed the catalytic effect of gold for the hydrochlorination reaction of ethyne in vinyl chloride and since then many researchers around the world have been interested in the use of gold as a heterogeneous catalyst for a large number of reactions.

Very quickly, the catalytic activity of gold was related to the size of its particles. Indeed, it is now established that gold has a catalytic activity if the particle size is less than 5 nm. Various reasons can explain the catalytic effect of gold at the nanoscale, but most of them are related to the plasmonic effects of gold at this scale.

This knowledge has made it possible to direct research towards the modes of preparation, dispersion and stabilization of nanoparticles on different supports and to use them as catalysts for a large number of chemical reactions.

This paper focuses on the different results obtained for gold as a supported metal for the catalysis of organic molecule oxidation reactions and organic molecule coupling reactions.

We will also discuss the different challenges for research in gold catalysis and particularly the development of sub nanometric catalysts (particle size less than 1nm)

Keywords: nano gold, oxidation reactions, coupling reactions

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RECENT PROGRESS IN THE SYNTHESIS OF AZAPHILONES AND ANALOGUES

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Abstract

This Azaphilones are a family of fungal polyketide metabolites possessing a highly oxygenated pyranoquinone bicyclic core, named isochromene, and a quaternary oxygenated carbon center at C-7. They belong to a large group of fungal pigments, turning red in the presence of primary amines due to an exchange of the pyran oxygen for nitrogen. With more than 170 members, they present an extremely large structural diversity associated with a wide biological activity spectrum.¹

In this azaphilone family, we picked-up two Natural Products named epicocconone and chaetoviridin-A, for their unusual structure, fluorescence properties when bound to proteins,² or biological activity.³

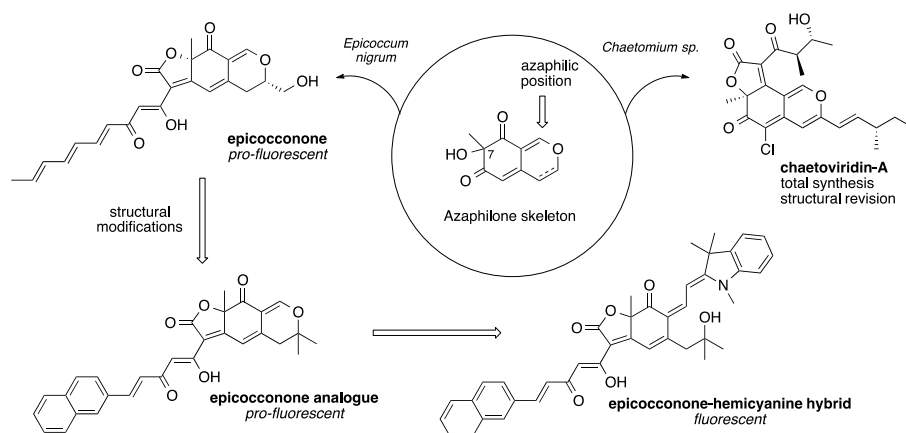


Figure 1. azaphilones synthesis

Keywords: Azaphilones, fluorescence, total synthesis

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PHOTOCATALYTIC PATHWAY TOWARD DEGRADATION OF VARIOUS PHARMACEUTICAL MOLECULES: KINETICS, MECHANISMS AND TOXICITY ASSESSMENT

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Abstract

Pharmaceutical residues have been found in municipal *wastewater* and have raised concerns about the potential risks they pose to both humans and our ecosystems. Among the different AOPs, heterogeneous TiO₂ photocatalysis is reported to be a promising technology for the degradation of such toxicants. The aim of this paper is to examine the degradation of various pharmaceutical compounds (Paracetamol, Naproxen and Ibuprofen) by the heterogeneous photocatalysis processes TiO₂ / UV, UV-LED and solar irradiations. The operating parameters, namely, pH, catalyst load and light intensity (number of LEDs) were optimized. The main parameter of degradation as a function of pH was the adsorption of the molecules studied on the TiO₂ entities. Thus, the rate of degradation increases as a function of the catalyst load, but the excess of catalyst load decreases the rate of degradation by opacity effect. The mineralization of the treated solutions was followed by the chemical oxidation demand (COD) measurement and/or the dissolved organic carbon (DOC). Significant mineralization was observed, however, no complete mineralization was observed in our study. Using different chromatographic techniques (HPLC) and ultra-high performance liquid chromatography coupled to tandem mass spectrometry (UHPLC-MS/MS), an attempt was made to identify some reaction intermediates formed during the degradation of our molecules studied. Reaction mechanisms have been established according to the results obtained in order to try to understand the degradation of the compounds studied and their mineralization. The effect of the matrix on the degradation was examined and the effect of the present ions was studied. In photocatalysis TiO₂ / solar, a study of the reusability of the supported TiO₂ film was carried out and the film maintained a stable catalytic activity after five reuses. The toxicity of the molecules studied and of the reaction intermediates was monitored by the avoidance test on *Eisenia andrei* earthworms and by the Microtox test on *Vibrio Fischeri* bacteria. The results showed that photocatalysis reduced the toxicity of the molecules studied.

Keywords: Photocatalysis ; pharmaceutical compounds ; Mineralization; Reaction mechanisms; Toxicity.

Topic 1

Catalysis

Oral Communications

X-ray crystal of aurate salt complex of di-protonated tetra (2-pyridyl) pyrazine (H_2TPPZ)

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ABSTRACT

The structure of a new ionic Au^{3+} crystal, $[H_2TPPZ][(AuCl_4)_2].H_2O$, was characterized by x-ray, elemental analyses, FTIR, electronic, 1H -NMR and cyclic voltammetry measurements. The results of anticancer studies of the Au^{3+} complex promised to be effective in MCF-7 (mammary gland breast cancer).

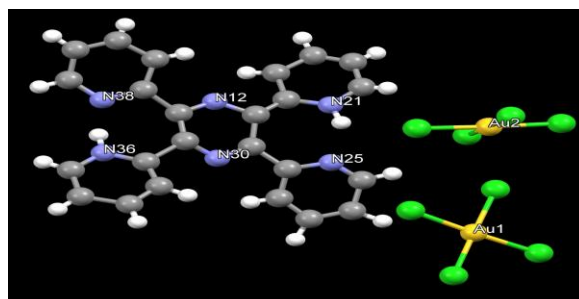


Figure 1. X-ray single crystal of $[H_2TPPZ][(AuCl_4)_2].H_2O$.

ACTIVITY OF MONO AND BIMETALLIC GOLD CATALYSTS ON ADIPIC ACID PRODUCTION

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Abstract

Adipic acid is an important bulk chemical product for different industrial chemistry process. It is largely used in the manufacture of polyurethanes, plasticizers and particularly nylon 6,6 polyamide.

The aim of this work is to study the activity of mono and bimetallic gold catalysts on cyclohexene oxidation by molecular oxygen. For this, we prepared Au/TiO₂ and Au/ZrO₂ by deposition precipitation with urea (DPU) with 1 wt% of gold, and the bimetallic catalysts (Au-Fe/TiO₂, Au-Fe/ZrO₂) are prepared by two different method: deposition under tension (UPD) and Co-deposition precipitation (Co-DPU). The catalysts are characterized by different techniques: TEM (EDX), XRD, DR/UV-Vis and ICP.

The catalytic test of the prepared materials shows an important production of adipic acid in all cases; but the conversion of cyclohexene is influenced by the presence of iron and the preparation method of the bimetallic catalysts.

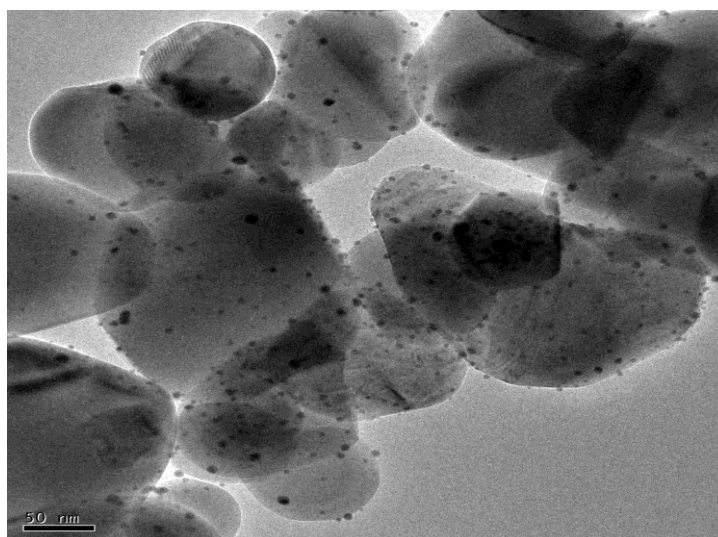


Figure 1. TEM image of bimetallic catalyst Au-Fe/TiO₂

Keywords: Bimetallic catalysts, gold, iron, oxidation, cyclohexene.

NOVEL CLEAN ROUTE OF ADIPIC ACID SYNTHESIS THROUGH CYCLOHEXANONE OXIDATION OVER METAL SUBSTITUTED POLYOXOMETALATES $H_{3-2x}Sn_xPMo_{12}O_{40}$ ($x = 0.25-1.5$)

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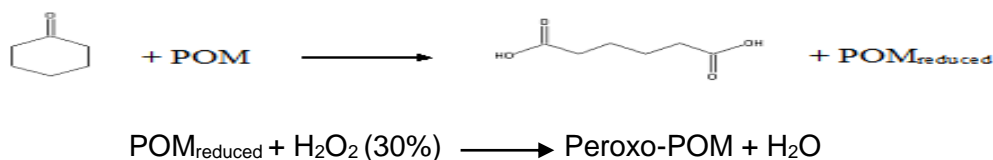
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Abstract

Hexane-1,6-dioic acid (named adipic acid) is a basic intermediate in the synthesis of nylon-6,6. Its industrial production uses concentrated nitric acid as oxidant. However, the major environmental concern for the reduction of nitric acid is the emission of nitrous oxide (N_2O) with a proportion of 0.3 ton for 1 ton of adipic acid produced, a greenhouse gas, which contributes to the destruction of the ozone layer.

Extensive researches have been devoted to the search for an environmentally friendly catalyst, that can lead to adipic acid with high yields conversion. In this context, Heteropoly acids and their related polyoxometalates (POMs) are a serious alternative due to their simultaneous properties of efficient oxidant and Bronsted strong acid approaching super acid region. Moreover, they are not toxic, not corrosive and they can operate in both homogenous and heterogeneous phases.

In this work, we have synthesized a series of tin substituted polyoxometalates $H_{3-2x}Sn_xPMo_{12}O_{40}$ ($x = 0.25 - 0.5 - 0.75 - 1 - 1.25 - 1.5$) in purpose to study the oxidation of cyclohexanone to adipic acid in presence of hydrogen peroxide H_2O_2 (See scheme below).



The prepared POMs are characterized by FT-IR and UV-Vis spectroscopies, by XRD, and elemental analysis. The study of the physicochemical characterization has shown that the cationic position of Sn^{2+} does not disturb the symmetry of the Keggin anion $[PMo_{12}O_{40}]^{3-}$. UV-visible spectroscopy revealed the molybdenum (VI) -oxygen transfer band.

Optimal reaction conditions such as temperature, stirring speed, H_2O_2 spiking and gainful molar ratio of catalyst/substrate have been achieved after several preliminary catalytic tests. Crystals of adipic acid obtained are characterized by the melting point (152°C) and FT-IR spectra.

The best rate of 44% conversion is reached with the catalyst $H_1Sn_1PMo_{12}O_{40}$ using a molar proportion of catalyst/substrate of 0.05%.

Keywords: Polyoxometalates, Green chemistry, Adipic acid

EFFECT OF REDUCING GAS ON THE HYDROGEN PRODUCTION BY THERMO-OXIDATION OF WATER OVER 1%Rh/Ce_{0.6}Zr_{0.4}O₂

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Abstract

We have studied the effect of the reducing gas (H₂, CO and CH₄) on the hydrogen production by thermo-oxidation of water over 1%Rh/Ce_{0.6}Zr_{0.4}O₂ catalyst prepared by impregnation. The catalyst is characterized by various techniques before and after catalytic decomposition of water. Catalyst is reduced in situ at 500°C for 4 h under H₂, CO, or CH₄ flow and flushed with argon. Then, pulses of water (1 μL/pulse) were injected at 500 °C under argon flow (30 mL/min). Figure 1 shows clearly that the reducing gas has an effect on the hydrogen production which follows the order: H₂ > CH₄ >> CO. H₂-chemisorption measurements at room temperature highlight a strong metal support interaction (SMSI) over fresh reduced catalysts which decreases after water decomposition.

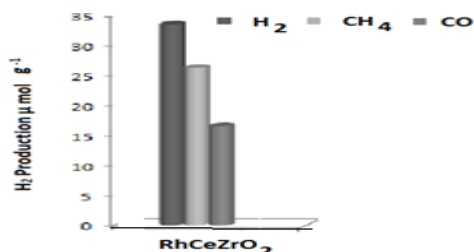


Figure 1. Effect of reducing gas on H₂-production over 1%Rh/Ce_{0.6}Zr_{0.4}O₂ catalyst.

Keywords: Hydrogen; Water splitting; Gas reducing.

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Ni AND/OR Co MOLYBDATE MATERIALS PREPARED BY MODIFIED CO-PRECIPIATION METHOD: STRUCTURAL, TEXTURAL APPLICATION IN ETHANE OXIDATIVE DEHYDROGENATION TO ETHYLENE

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Abstract

Because most intermediates in the petrochemical industry are produced from alkenes and aromatics, the demand of alkenes is increasing, and the existing capacity is becoming insufficient. Until recently, light alkenes have been obtained from steam cracking processes of natural gas and naphtha: these processes are very expensive due to the fact that the obtained products must be separated and purified. Nowadays, research pays attention to the development of new low-cost processes converting light alkanes to valuable products and alkenes by means of oxidation or dehydrogenation processes.

This work deals with the search for new materials formulations, which are known to be active in alkanes oxidative dehydrogenation, a process which could be replace in the near future the common steam cracking and pure dehydrogenation processes currently used for the production of alkenes.

The main purpose of this work is to investigate and try to rationalize the role played by Ni and/or Co when included in the formulation molybdates materials, and the effect that they have on the structure and the activity in the oxidative dehydrogenation of ethane.

A second part will be devoted to a study of the influence in the structural properties and the activity of Ni and/or Co molybdates when dispersed on different supports.

The Ni and/or Co molybdates supported and unsupported materials solids, show improved efficiency and activity in ODH of ethane (up to 55% of C₂H₆ conversion). They were very selective to ethylene. The highest activity is obtained on the material contain Ni which has the highest specific surface area and the lowest temperatures of reduction.

This material are active and selective in ODH of ethane in ethylene. The nature of the transition metal and the preparation method has a great effect on their activity.

Keywords: Ni and/or Co molybdate, ODH, Ethane.

SYNTHESIS OF A SERIES OF NEW PLATINUM ORGANOMETALLIC COMPLEXES DERIVED FROM BIDENTATE *SCHIFF* BASE LIGANDS AND THEIR CATALYTIC BEHAVIOUR TOWARDS THE HYDROSILYLATION OF ALKENE

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Abstract

The application of inorganic chemistry to catalysis and environmental chemistry is a rapidly developing field, and novel catalytic metal complexes are now having an impact on the industrial development practice.

The reaction of platinum(II) organometallic complexes with bidentate *Schiff* bases derived from 2-Hydroxynaphtalylideneaniline have been carried out. It concerns N,N'-naphtalidene para-nitroaniline (**1-a**), the, the N,N'-naphtalidene para-ethoxyaniline (**1-b**), the N,N'-naphtalideneaniline (**1-c**), the N,N'-naphtalidene para-chloroaniline (**1-d**) and the N,N'-naphtalidene para-methoxyaniline (**1-e**). The ligands were fully characterized by I.R., elemental analysis, ¹H-NMR, ¹³C-NMR, ESI Mass Spectrometry and X-Ray Diffraction. The resulting metal complexes were obtained as a cationic species [**1**], through a simple substitution reaction, leading to two geometric isomers (**2**, **3**), and characterized by IR, ¹H-NMR, ¹³C-NMR, LIFDI Mass Spectrometry and supported by Elemental Analysis and X-Ray diffraction. Furthermore, a bimetallic platinum complex was prepared from the same ligands and dichloro(1,5-cyclooctadiene)platinum and characterized by X-Ray diffraction (**4**). The catalytic properties of the prepared platinum complexes in the hydrogenative and dehydrogenative silylation of styrene were investigated, and reaction kinetics conversion to products was determined by ¹H-NMR and confirmed by GC-MS.

This presentation will details a comparison of the catalytic activity of five platinum organometallic complexes bearing different *Schiff* base ligands in the hydrosilylation of styrene, varying the experimental conditions of temperature, nature of the complex and the loading of the catalyst.

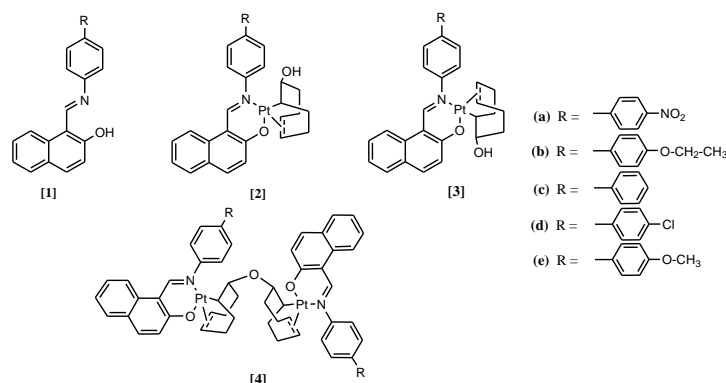


Figure 1. Structure of the platinum organometallic complexes

Keywords: *Schiff* base ligands, platinum organometallic complexes, hydrosilylation

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PHOTOCATALYTIC DEGRADATION OF METHYLENE BLUE THROUGH Au-Ag/TiO₂ AND Au-Ag/TiO₂/GRAPHENE

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Abstract

Bimetallic nanoparticles could show the combination of two different metals and the distribution modes and could be defined by random alloy and core-shell structure. The preparation method and the conditions affect the shape, size and the physicochemical properties of the final photocatalyst. In this study, bimetallic alloys nanoparticles Au-Ag (NPs) were prepared by mild impregnation method with a very small size at around 2 to 5 nm. The characterization of these nanoparticles was obtained by: X-ray diffraction, X-ray photoelectron spectroscopy, transmission electron microscope, diffuse reflectance spectroscopy, High angle annular dark field and Raman. These NPs combined with semiconductor TiO₂ nanoparticles allow the degradation of methylene blue using visible light through LED light and solar light.

We have prepared new plasmonic photocatalysts Au-Ag/TiO₂ through the impregnation and hydrothermal methods with varying the content of NPs Au-Ag with 1% and 2% for each method. The different materials were characterized also by different which finally the high activity found under visible light makes these nanomaterials good candidates as photocatalysts for organic pollutant degradation [1].

Keywords: Methylene blue, TiO₂, photocatalysis.

Reference

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REMOVAL OF POLLUTANTS FROM WASTEWATER USING SILVER DOPED MESOPOROUS TiO₂

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Abstract

The photocatalytic activity of Ag doped mesoporous TiO₂ was investigated under simulated solar light irradiation for wastewater pollutants degradation. Ag/ TiO₂ catalysts were prepared by impregnation-reduction with citrate with various Ag loadings (0.5wt%, 1.5wt% and 3wt%). Ag/ TiO₂ catalysts retain the physical properties of mesoporous TiO₂ and its hexagonal order. The degradation of methyl orange (MO) as a model organic pollutant and of a real wastewater effluent was carried out under simulated solar light irradiation and compare with P25 TiO₂. The mineralization of the organic pollutants was investigated by Total Organic Carbon (TOC) measurements. Mesoporous TiO₂ showed a better activity than P25 TiO₂ and photocatalytic performance of silver doped mesoporous TiO₂ was enhanced compare to undoped. The enhanced photocatalytic activity of Ag/TiO₂ can be ascribed to a strong inhibition of (e⁻ - h⁺) recombination [1]. 0.5wt%Ag/ TiO₂ exhibited the highest TOC abatement for both MO and wastewater effluent. The greatest activity of 0.5wt%Ag/TiO₂ could be ascribed to the smallest size of the Ag particles [2]. Moreover, this catalyst was very stable after three cycles of reuse and efficient under simulated solar light. Therefore, it is of much interest as a potential catalyst for the treatment of wastewater under solar.

Keywords: Mesoporous TiO₂, Silver, photocatalysis.

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PHOTOCATALYSIS WITH Fe/Ti-PILLARED CLAYS FOR THE OXIDATION OF ALKYLAROMATICS

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Abstract

A pillared montmorillonite containing iron doped titania (Fe/Ti-PILC) has been prepared from a natural clay. This material has been characterized by X-ray diffraction, nitrogen adsorption, temperature programmed desorption of ammonia, inductively coupled plasma atomic emission spectroscopy, atomic absorption, and diffuse reflectance UV-VIS spectroscopy. The layer structure of Fe/Ti-PILC resulted to be ordered with insertion of pillars, which caused a slight increase in the basal spacing of the clay. Its specific surface area was about three times larger than that of the parent Na-montmorillonite due principally to the creation of a remarkable microporous network. The doped material was a robust photocatalyst able to oxidize liquid alkylaromatics to the corresponding carbonylic derivatives, using O₂ as the oxidizing species, at mild pressure and temperature conditions. Accumulation of valuable carbonylic derivatives was possible since their over-oxidation to carbon dioxide was negligible. Fe/Ti-PILC was able to discriminate between toluene and cyclohexane in favour of the aromatic compound with an efficiency that is about three times higher than that of titanium pillared clays (Ti-PILC). It is likely that the addition of iron favoured the formation of new acid sites able to interact with the aromatic substrate. Iron doping caused a significant TiO₂ visible light induced activity ($\lambda > 400$ nm) with only minor negative effects on its performance under UV-light irradiation ($\lambda > 290$ nm).

Key words: Alkylaromatics oxidation, Heterogeneous photocatalysis, pillared clays.

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SYNTHESIS, CHARACTERIZATION AND OPTIMIZATION OF HETEROGENEOUS CATALYTIC CYCLOHEXENE OXIDATION VIA THE LATIN SQUARE DESIGN (TAGUACHI L9) METHODOLOGY

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Abstract

In this study, we report the synthesis of differrents keggin heteropolyanions (HPAs) with vanadium, ruthenium and tungsten. The synthesized heteropolyanions were supported on acid activated montmorillonite by impregnation method. The support and new catalysts have been characterized by FT-IR, XRD, particle size distribution and surface area measurement (BET method) and thermo gravimetric analyses (TGA). Their catalytic properties were evaluated for cyclohexene oxidation. This reaction is influenced by several qualitative factors such as type of catalyst (20%PRuW/Hmont, 20%PVW/Hmont and 20% Ru/Hmont), solvent (Acetonitrile, Heptane, Propal-2-ol), oxidant (H₂O₂, TBHP and O₂), etc... An important issue in the catalytic experimentation concerns the way to design experiments in order to explore and optimize the multidimensional parameter space, by minimizing the number of trials required to achieve a unique solution [1, 2]. The technique o experimental design is largely used in chemical industry [3-5]. For these reasons, the aim of our work is to optimize the conditions of the oxidation reaction of cyclohexene using heterpolyanions and ruthenium supported acid activated montmorillonite as catalyst following a Latin Square design, is looking to get a good conversion (Y₁) with good selectivity (Y₂) in cyclohexene oxide.

Characterization of synthesis catalysts showed that HPAS and Ru is well dispersed on the surface of montmorillonite. IR bands show the Keggin structure of HPA showed that the procedure used gives the expected materials. The prepared heterogeneous catalysts showed high catalytic activity (80 %) for cyclohexene oxidation. They are not expansive and can be used in heterogeneous media.

Keywords: Cyclohexene oxidation, Experimental design, Optimization

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Poster Communications

IN-SITU HYDROGASIFICATION/REGENERATION OF NiAl-HYDROTALCITE DERIVED CATALYST IN THE REACTION OF CO₂ REFORMING OF METHANE: A VERSATILE APPROACH TO CATALYST RECYCLING

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Abstract

A novel approach describing the in-situ regeneration of NiAl hydroalcite derived catalyst [1] between two cycle reaction systems of CO₂ reforming of methane, also known as dry reforming of methane (DRM) is described herein. The catalyst was initially prepared by co-precipitation method at pH = 11 and calcined at 450 °C for 6 h. The obtained material was characterized using X-ray diffraction (XRD) spectroscopy, Fourier transform infrared (FTIR) spectroscopy, Brunauer-Emmett-Teller (BET), atomic absorption spectroscopy (AAS), scanning electron microscopy (SEM), transmission electron microscopy (TEM), thermogravimetry (TG/ATD) and temperature programmed reduction (TPR-H₂) techniques. Following treatment of our catalyst under DRM conditions, the catalyst was subjected to in-situ hydrogasification conditions to promote regeneration followed by a second DRM cycle. An increase of 15.7% in the conversion of CH₄ and 17.3% in the conversion of CO₂ was attained, while the ratio of resulting H₂/CO augmented by 14%. The ratio of H₂ consumed over the course of two hours hydrogasification, to that generated over ten hours of DRM, was 9.6%. The small particle sizes of resulting Ni species as well as their high stability were both key factors contributing to the increase in the amount of H₂/CO produced prior to and after regeneration.

Keywords: Hydrotalcite, Dry reforming of methane, Hydrogasification catalyst regeneration

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GREEN SYNTHESIS OF Cu-Ni ALLOY NANOPARTICLES USING *ROSMARINUS OFFICINALIS* PLANT EXTRACT: APPLICATION TO ELECTROCATALYTIC ACTIVITY

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Abstract

There have been impressive developments in the field of nanotechnology in the recent past, with numerous methodologies formulated to synthesize nanoparticles of particular shape and size depending on specific requirements. Currently, there is a growing need to develop environmentally benign nanoparticle synthesis processes that do not use toxic chemicals in the synthesis protocol. As a result, researchers in the field of nanoparticle synthesis have turned to biological systems like plant extracts [1]. The easy availability and the protocol applicable at room temperature and pressure are the core advantages while using plant extract as the biogenic agents for the synthesis of metal nanoparticles [2]. In this study, we synthesized copper-nickel (Cu-Ni) alloy nanoparticles by green voice using *Rosmarinus officinalis* plant extract at room temperature. The formation of nanoparticles was confirmed using scanning electron microscopy (SEM) coupled with EDX and, using X-ray diffraction. Scanning electron microscopy (SEM) shows spherical shape of nanoparticles was obtained, X-Ray diffraction (XRD) pattern shows face cubic center (fcc) structure and the average crystallite size was calculated to be about 49 nm. These nanoparticles were used to modify carbon paste electrode for the study of the electrocatalytic determination of uric acid by cyclic voltammetry technique. The presence of this nanoparticles in carbon paste exhibit a higher electrocatalytic activity of uric acid oxidation in phosphate buffer (pH = 6) at 481 mV/ECS. Results obtained show an excellent electrocatalytic activity toward uric acid with the best sensitivity and good selectivity than the prepared electrode can be used as an electrochemical sensor for uric acid.

Keywords: Green synthesis, nanoparticles, uric acid.

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EFFECT OF ALKALINE TREATMENT OF A COMMERCIAL ZSM-5 ZEOLITE ON H₂ PRODUCTION FROM CH₄ DECOMPOSITION OVER Ni SUPPORTED HZSM-5 CATALYSTS

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Abstract

Hydrogen production via the catalytic decomposition of methane (CDM) has attracted a great attention since H₂ is considered as a green fuel for the future [1]. In addition, highly valuable by-products such as carbon nanotubes and carbon nanofibers can be formed. In this work, we have studied the effect of desilication's alkaline treatment of a commercial NH₄-ZSM-5 zeolite on the hydrogen formation during CDM reaction at 520°C over pre-reduced nickel supported over HZSM-5 and alkaline treated HZSM-5 (HZSM-5at) [2]. CDM reaction is carried out in a pulsed homemade set-up. Catalysts Ni/HZSM-5 and Ni/HZSM-5at with 20 wt.% of Ni are prepared by wet impregnation and characterized by ICP-OES, XRD, FTIR, SEM, N₂-adsorption, H₂-TPR, UV-Vis DR and TPO for coked samples. Figure 1 shows clearly that H₂ production is favored when using HZSM-5at support. This catalytic behavior of Ni/HZSM5at in CDM reaction could be related to modifications in both textural and physico-chemical properties provided by desilication treatment of the commercial ZSM-5. Further investigations are in course to correlate catalytic properties in CDM reaction with physico-chemicals properties of the Ni-catalysts.

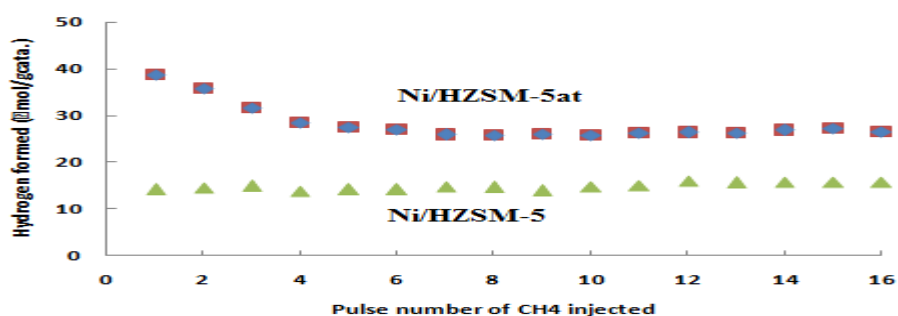


Figure 1. Hydrogen formed ($\mu\text{mol/gcata.}$) versus pulses number of injected CH₄

Keywords: CDM; ZSM-5 Desilication; Pure hydrogen.

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DEACTIVATION BY SINTERING OF COBALT CATALYST IN THE FISCHER-TROPSCH REACTION

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Abstract

Fischer-Tropsch synthesis is a catalytic process that allows the production of liquid hydrocarbons from a synthesis gas mixture essentially contains carbon monoxide and hydrogen. The catalysts most used in this reaction are cobalt-based catalysts because of their high activity. However, like all catalysts, they are ready for deactivation over time by several reasons such as oxidation of the active phase, poisoning by carbon deposition and sintering. During sintering a layer of CoO is formed and contributed to both reducing the surface energy and increasing the mobility of the small Co crystallites, which leads to the acceleration of the sintering rate. The growth of these crystallins is also accelerated by the water produced during the FT synthesis.

In the present work, we have studied the phenomenon of sintering a cobalt-based catalyst during the Fischer-Tropsch reaction under well-defined operating conditions of temperature, pressure and the molar ratio of the initial charge H₂/CO. The simulation results obtained show the effect of these parameters on the CO conversion rate and on the reaction rate before the sintering of the catalyst and after the appearance of this phenomenon. Generally, good performance of the process can be obtained prior to sintering where the increase in the initial total pressure leads to an increase in the rate of the reaction and thus the conversion of CO in the absence and in the presence of sintering. While the increase in temperature causes a decrease in speed and a slight increase in CO conversion. However, the increase in the H₂/CO molar ratio decreases the reaction rate, whereas the CO conversion increases in both cases before and after the sintering of the catalyst.

Keywords: Fischer-Tropsch, cobalt catalyst, deactivation, sintering.

HYDROGEN PRODUCTION FROM DRY METHANE REFORMING OVER RU SUPPORTED CATALYSTS

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Abstract

In this work the methane reforming with carbon dioxide ($\text{CH}_4 + \text{CO}_2 \rightarrow 2\text{CO} + 2\text{H}_2$) [1] performed on RuO_2 based catalysts. This route gained interest in the last years, due to the fast growth of natural gas extraction (methane is the main component of natural gas), and consequently of the high availability and low price of CH_4 .

In the methane reforming with carbon dioxide, two commercial RuO_2 containing catalysts prepared on different supports (silica and alumina), and RuO_2 bulk catalyst, were characterized and tested in a fixed bed reactor in the 450-600°C temperature range with a reactant ratio (CH_4/CO_2) of 1. The presence of RuO_2 increased the methane reforming, when compared to the reaction performed on the bare supports. As general trend, the activity was enhanced by increasing the reaction temperature, as expected for an endothermic reaction. At 450°C, the average conversions of CH_4 over $\text{RuO}_2/\text{SiO}_2$ and $\text{RuO}_2/\text{Al}_2\text{O}_3$ were respectively 16 and 15 %, and they increased to 86 and 73% at 600°C. The same behavior, but to a minor extent, was observed for the CO_2 conversion (21 and 17 % respectively for $\text{RuO}_2/\text{SiO}_2$ and $\text{RuO}_2/\text{Al}_2\text{O}_3$ at 450 °C, and 45 and 49 % at 600°C).

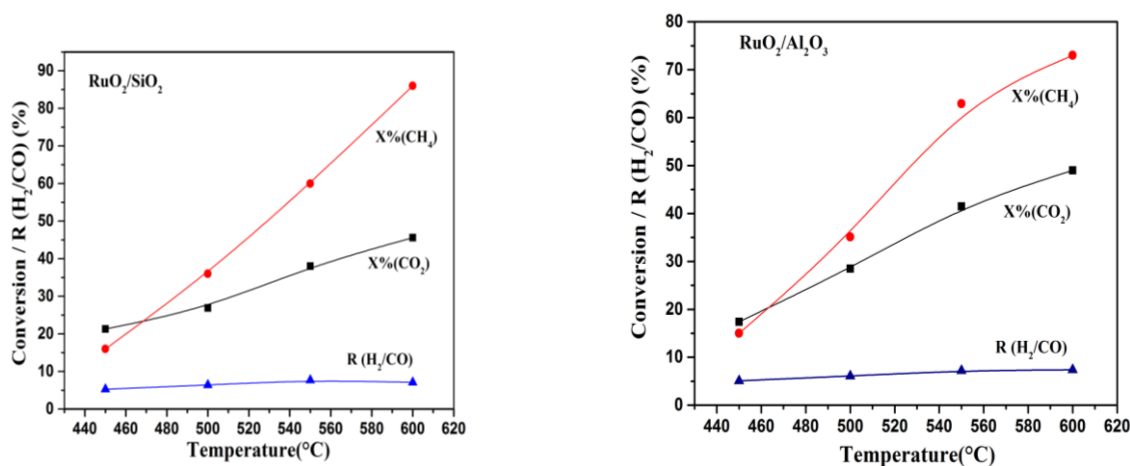


Figure 1. CO_2/CH_4 conversion and $R(\text{H}_2/\text{CO})$ at different reaction temperature, at atmospheric pressure CO_2/CH_4 was (1:1).

Keywords: methane, carbon dioxide, conversions.

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Cu-Zn BASED CATALYSTS, PREPARATION, CHARACTERIZATION AND ACTIVITY IN THE CATALYTIQUE HYDROGENATION OF CARBON DIOXIDE

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Abstract

Cu/ZnO-type catalysts are commonly used in the CO₂ hydrogenation to produce methanol due to their high activity and selectivity. The commercial Cu/ZnO catalysts [1] are generally synthesized by co-precipitation method, in the presence of sodium carbonate as a precipitating agent. More advanced methods, as sol-gel and improved co-precipitation synthesis in the presence of reducing agents (i.e. chitosane, NaBH₄), have also been developed with the aim to enhance the number of catalytic active sites. Polyol method is known to be particularly effective to control the size and the shape of crystallites by varying the reduction temperature, the pH, and the nucleation-protective agent concentration.

In the present work, the polyol method has been applied for synthesizing a new series of catalysts for methanol production. The influence of the chemical compositions of binary and ternary oxide catalysts (CuO-CeO₂, ZnO-CeO₂, CuO-ZnO-CeO₂ and CuO-ZnO-Al₂O₃) has been investigated. The catalysts were fully characterized by XRD, N₂-adsorption at -196°C, XRF, SEM, and XPS. Then, after a reducing pretreatment at 300°C under H₂ stream, the catalytic activity and selectivity have been tested at atmospheric pressure by feeding a CO₂:H₂= 1:9 molar mixture, at various reaction temperature, in a flow-reactor. The obtained products present in the out-streaming gas were analyzed by gas-chromatography.

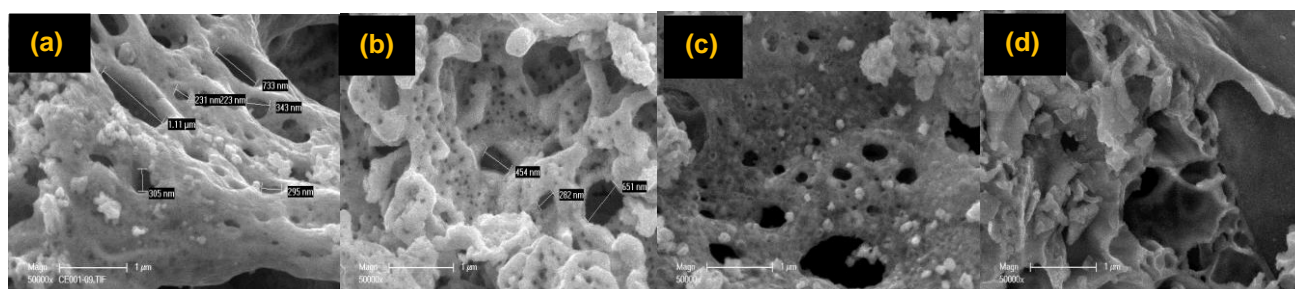


Figure.1. SEM-EDX images at different magnification of all catalysts (a-) CuO-ZnO-CeO₂, (b) CuO-CeO₂, (c) ZnO-CeO₂ and (d) CuO-ZnO-Al₂O₃.

Keywords: CO₂, hydrogenation, catalysts, polyol

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CATALYTIC DEHYDROGENATION OF ETHANOL TO ACETALDEHYDE AND ETHYL ACETATE

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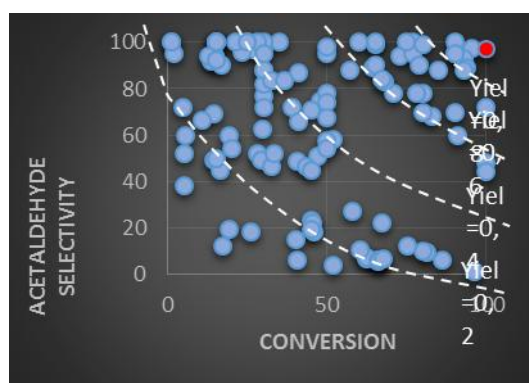
Abstract

The bioethanol production from biomass has gained a very important interest this last decade, because of its use as an alternative to fossil fuels. Its large availability and its biodegradability made it a building block for biorefineries and clean industrial technologies. For instance, the dehydrogenation of ethanol seems to be the most promising route, since it occurs at relatively mild conditions and generates value-added chemicals such as acetaldehyde which is a versatile chemical intermediate, ethyl acetate largely employed as a solvent in many industrial products, as a green alternative, replacing the harmful aromatic compounds and pure hydrogen used directly in many industrial processes.

Thus variety of heterogeneous catalytic systems for the reaction of ethanol dehydrogenation, have been intensively investigated. The most popular were based on copper, mainly because of its ability to dehydrogenate ethanol without C-C bond scission.

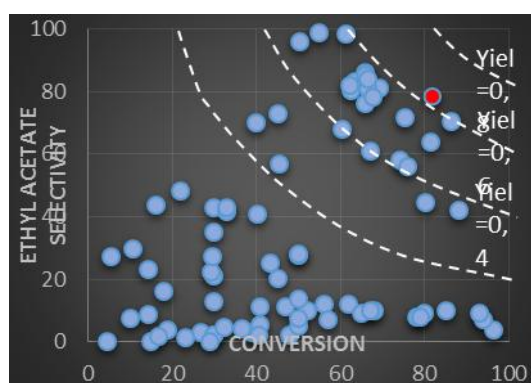
This study examines different catalytic systems used so far for the ethanol dehydrogenation (EDH) reaction which aims to produce either acetaldehyde or ethyl acetate. The following aspects are discussed: a) influence of metal used as an active phase, b) support effect, c) promoter effect d) metal loading effect and e) reaction conditions effect.

The results are summarized in Figures 1 and 2.



● Best catalyst CuO/ γ Al₂O₃ [1]

Figure 1. Selectivity vs conversion chart for EDH into acetaldehyde



● Best catalyst Cu/ZnO [2]

Figure 2. Selectivity vs conversion chart for EDH into ethyl acetate

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PHOTOCATALYTIC DEGRADATION OF ORGANIC POLLUANT USING HYDROTALCITE-LIKE COMPOUNDS

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Abstract

Due to the abundant use of anionic surfactants in our life and their disposal in the environment, the removal of surfactants from waste water becomes necessary.

The present study focused to the synthesis and application of layered double hydroxides and their derived oxides to remove the anionic surfactant, sodium dodecyl sulphate (SDS), from aqueous solution by adsorption and photocatalytic degradation under UV irradiation. The influencing of Zn²⁺/Al³⁺ molar ratio and initial SDS concentration on the adsorption and photocatalytic degradation of SDS are studied and optimized. The obtained adsorption data were correlated with Langmuir and Freundlich isotherms models. Equilibrium studies indicated that the SDS removal obeyed Langmuir type of adsorption. Kinetic data were better described by pseudo-second-order model. The photocatalytic degradation of SDS has been found to fit the first-order kinetics according to the Langmuir-Hinshelwood model.

Keywords: hydrotalcite, photocatalyst, removal

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PREPARATION OF GOLD NANOPARTICLES SUPPORTED ON NICKEL-CONTAINING HYDROTALCITE CATALYSTS AND THEIR ACTIVITY IN CO OXIDATION REACTION

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Abstract

The main goal of the present study was to investigate the influence of the Au/Ni-containing Hydrotalcite catalysts composition, prepared by direct anion exchange (DEA) [1,2], on these physical-chemical and catalytic properties. Various techniques were used such as chemical analysis, powder X-ray diffraction (DRX), surface area measurements (BET), transmission electron microscopy (TEM) and temperature programmed reduction (TPR). Concerning the total oxidation of CO, the catalytic tests showed that these catalysts has excellent catalytic properties in this reaction, and depends mainly on the composition of the support.

In summary, we have demonstrated through the chosen preparation method; DAE, it was possible to boost successfully active in oxidation gold species on a basic phase; known to be less active and unfavorable to deposit gold.

Au nanoparticles prepared were evenly well dispersed on the surface and we arrive under certain optimal conditions to form very small gold particles, more than 90% of the AuNPs having a size of less than 4 nm [3], satisfactory results.

Keywords: Gold nanoparticles ; Layered Double Hydroxides ; CO oxidation.

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HYDEOGEN PRODUCTION FROM ETHANOL STREAM REFORMING OVER Ni-BASED CATALYSTS

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Abstract

Ethanol steam reforming (ESR) is one of the possible industrial processes for hydrogen production. As a transition metal, Ni-based catalysts exhibit high activity in reforming processes and are cost-effective in comparison with noble metal-based catalysts. However, the catalyst deactivation by carbon deposition is a general problem for ESR over supported Ni catalysts. Cooking of Ni surface is an important technological problem and many experimental studies have been addressed to this process. The objective of present work was to explore the structure, surface properties and catalytic performance of Ni catalysts supported on lanthanide group in ESR.

Nickel catalysts supported on ZrO₂, CeO₂ and ZrO₂-CeO₂ were prepared by wet impregnation [1] method and evaluated in steam reforming of ethanol (ESR). The catalysts were characterized by chemical composition, textural analysis, crystalline structure and reducibility. It was observed that the interaction with the support and Ni reducibility depended on the support used. According to TPR results, NiO species supported on CeO₂ and ZrO₂ were the easiest to reduce. Possibly, this behavior combined to redox properties of CeO₂ inhibited side undesirable reactions and contributed to the higher selectivity for H₂ on the NiCe catalyst. The catalytic test at 500-650 °C on NiCe showed better H₂ selectivity than the test at 500 °C yield (above 54% from 500°C), suggesting that the higher temperature enhanced CO₂ conversion and hindered carbon deposition in dry reforming of ethanol reactions. The basic character of ceria-based support limits the formation of ethene product likely precursors of coke formation.

Keywords: hydrogen, energy renewable, energy storage

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DEEP OXIDATION OF TOLUENE OVER Pd AND/ OR Au SUPPORTED ON MESOPOROUS Co₃O₄

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Abstract

Volatile organic compounds (VOCs) are known as main contributors to air pollution, either directly, through their toxicity, or indirectly, as ozone precursors. Air pollution can cause serious health problems.

VOCs catalytic oxidation is a chemical process in which hydrocarbons are mixed with air at specific temperatures to produce carbon dioxide (CO₂) and water (H₂O). Catalytic oxidation is a better way for VOCs removal than thermal combustion because required temperatures are lower and selectivities are higher.

The use of zeolites and macro-mesoporous oxide supports for noble metal based catalysts in the total oxidation of VOCs is known to be very interesting.

In the present work, 1wt% Au and / or 1wt% Pd/ Co₃O₄ catalysts were synthesized using cobalt oxide prepared by the nanocasting pathway using the mesoporous SBA-15 silica as structure template and cobalt nitrate as precursor via a solid –liquid route. Catalysts were fully characterized using chemical analysis, X-ray Diffraction (XRD), Transmission Electron Microscopy (TEM) coupled with DTA (TEM/DTA), Infrared Spectroscopy (FTIR), Temperature-programmed reduction (TPR) and Diffuse Reflectance UV-Visible Spectroscopy. Total oxidation of toluene was investigated on noble metal/mesoporous Co₃O₄ catalysts; the catalysts are totally selective for CO₂ and H₂O. Pd-Au / Co₃O₄ is the most active catalyst; the highest activity exhibited by Pd-Au / Co₃O₄ was correlated to core-shell morphology with a gold-rich core and a palladium-rich surface. Pd-Au / mesoporous cobalt oxide catalyst is a potential candidate for the deep oxidation of VOCs.

Keywords: Mesoporous Co₃O₄; Palladium; Gold; total oxidation ; toluene.

NANOCAST ORDERED MESOPOROUS CERIA AS SUPPORT FOR HIGHLY ACTIVE SILVER CATALYSTS FOR PROPYLENE TOTAL OXIDATION

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Abstract

Volatile organic compounds (VOCs) are considered as an important class of air pollutant and are dangerous to human health. A great advantage of catalytic abatement of VOCs is that it could operate with diluted VOCs effluent streams (<1% VOCs) at a much lower temperature than thermal combustion, thus it has lower operating costs.

CeO₂ has an oxygen storage capacity and is often used as support material for noble metal-based catalysts in oxidation reactions [1, 2].

The catalytic properties of AuCeO₂ have been found to be highly dependent on the size and morphology of ceria particles. The particle size reduction down to the nanoscale will create a large number of more reactive sides or edges that would improve the reaction activity [3, 4]. Some reports indicate that CeO₂ in the form of nanocrystalline particles is a key factor for achieving high activity for complete oxidation of CO and VOCs [5, 6]. Therefore, the synthesis of nanostructured ceria, especially with ordered mesoporous structure has been explored [7]. In this study, mesoporous ceria was synthesized by the nanocasting pathway using the mesoporous SBA-15 silica as structure template, and doped with Ag. Ag/CeO₂ catalysts were prepared by three different methods: wetness impregnation (WI), deposition-precipitation with urea (DPU) and impregnation-reduction with citrate (IRC), with a loading of silver of 4 wt.%. They were characterized by elemental analysis, H₂-TPR, X-ray powder diffraction (XRD), BET surface area, diffuse reflectance and ultraviolet visible spectroscopy (DR/UV-Vis).

Ag/CeO₂ catalysts retain the physical properties of mesoporous ceria and its hexagonal order. Catalytic oxidation of propylene was investigated on Ag/mesoporous ceria catalysts. The silver/mesoporous ceria system toward the deep oxidation of propylene depends on the catalyst preparation method. Ag/CeO₂ (IRC) is the most active catalyst. The highest activity of Ag/CeO₂ (IRC) can be ascribed to higher surface lattice oxygen mobility over this catalyst and also to the strong interaction between silver and mesoporous ceria.

Keywords: Mesoporous ceria; silver; oxidation; propylene;

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SYNTHESIS, CHARACTERIZATIONS OF THE LAMELLATE DOUBLE HYDROXIDES AND THEIR APPLICATION CORROSION INHIBITOR

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Abstract

Clay's anion, so called lamellate double hydroxides (HDL) thanks to their facility of synthesis and their low costs of production constitute a very important family in chemical industry. Studied since nearly one half-century and considering their enormous potential to exchange anion, their behavior oxydo-reducer or acido-basic and their properties electric and thermal stability. This chemical malleability of the lamellate double hydroxides allows an adjustment of the properties opening the prospect for their applications in many fields: catalysis, environment, food industry, pharmaceutical industry, the industry of polymers and the electrochemistry of the Solid. In this work, we were interested in the preparation of the HDL containing Zn and Mg intercalated by benzoate of sodium and their study for inhibition of corrosion. The method of synthesis used is the Coprecipitation with constant pH and formed hydroxides Zn-AL-CL, Zn-AL-BZ, and MG-AI-CL, MG-AI-BZ of molar report $M_{2+}/M_{3+} = (1-2-2.5)$ are characterized by diffraction of X-rays (DRX), by spectroscopy will infra red (FTIR), these analyses showed the aptitude of these compounds exchanged the anions chlorides in their interfoliaceous space by sodium benzoates.

Keywords: lamellate double hydroxides, intercalation, benzoate of sodium

SYNTHESIS AND CHARACTERISATION OF SUPPORTED GOLD CATALYSTS FOR CATALYTIC APPLICATION

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Abstract

Supported gold catalysts have been found to be active for important selective (Partial) oxidation, such as selective oxidation of alcohols, hydrocarbons and sugars [1]. In particular, the number of papers on selective oxidation of amines on gold nanoparticle catalysts has exponentially flourished since 2008 [2]. Selective oxidation of amines may yield amides, secondary amines and imines. Imines also called Schiff base, are used as versatile components in nucleophilic addition. They have also potential for therapeutic applications such as lipoxygenase inhibitors, anti-inflammatory agents and anti-cancer agents. Among all the conventional methods for imine synthesis, the N-alkylation of amines is of fundamental importance in organic synthesis because the resulting higher amines have widely been used as synthetic intermediates for pharmaceutical, agrochemicals, fin chemicals, dyes, surfactants and functionalized materials [3].

In this work we have studied the oxidation of Benzyl alcohol and the N-alkylation of Benzyl amine and Benzyl alcohol in liquid phase on gold nanoparticles catalysts supported on hydrotalcites of Mg Al and Mg Al Ce type, prepared by deposition-precipitation. Different Mg / Al / Ce ratios were used for the preparation of the support.

After being assured of the support inactivity alone on the oxidation of benzyl alcohol and the coupling of benzyl alcohol and benzyl amine at 80 ° C, we studied the properties of the 2% Au / hydrotalcite catalysts. These catalysts allowed us to obtain excellent conversion and selectivity performances with better activity obtained on the hydrotalcite-supported gold catalyst prepared with an Mg / Al ratio of 2. The catalysts were characterized by DRX, BET, ATG

Keywords : Gold nanoparticles, Hydrotalcite, Imine

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ADVANCED METHANOL TRANSFORMATION INTO FORMALDEHYDE SENSIBILIZED BY RHODAMINE-B WITH THE PRESENCE OF CU AND ZN PHOTOCATALYSTS

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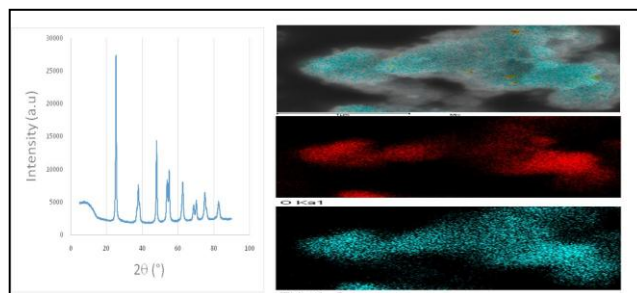
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Abstract

The photocatalytic transformation of methanol into formaldehyde under continuous visible light irradiation (100W, Phillips) is investigated in this work as a model reaction using of Rhodamine-B dye as sensitizer. This dye plays a role in the electrons mobility [1]. The Rhodamine-B solution sensitizer-dye ($2.087 \times 10^{-5} \text{M}$) was introduced in the aqueous solution of methanol ($2.1 \times 10^{-4} \text{M}$) and our experimental conditions have checked by UV-vis spectroscopy allowing to see the preservation of this dye all during the photocatalytic reaction. Commercial titania Millenium P500 was chosen in this study for its very high surface area superior to $300 \text{ m}^2/\text{g}$. Millenium P500 was calcined in air at 500°C to pre-agglomerate the grains and increases the size of crystallites. A mixture of anatase nanoparticles has been prepared by mixing 80% of non-calcined Millenium P500 powder and 20% of the calcined one and this support is noted "a-TiO₂" (a: anatase). To obtain new physicochemical and electron conduction properties, the surface of support has been loading by Cu and Zn nanoparticles. From $\text{Cu}(\text{NO}_3)_2 \cdot n\text{H}_2\text{O}$ and $\text{Zn}(\text{NO}_3)_2 \cdot n\text{H}_2\text{O}$ salts precursors, two samples containing 3wt% of Cu^{2+} and Zn^{2+} was synthesized by wet impregnation. The obtained photocatalysts were noted 3Cu/a-TiO₂ and 3Zn/a-TiO₂ and characterized by: XRD, UV-Vis spectroscopy, N₂ sorption by BET method with surface area applied between 0.05 and 0.30 and pore sizes obtained by BJH, FEG-SEM with EDS and cartography of the distribution of chemical elements. The X-rays diffraction pattern of copper catalyst is shown in Fig.1. The main diffraction peaks are consistent with the characteristic peaks of bare support, indicating that the anatase TiO₂ structure is preserved. It was obviously observed by XRD, the absence of copper peaks. This is highly evident strong interactions of Cu nanoparticles with titania (possibly inserted in the first layers). Cartography-X (Fig.1) of 3Cu/a-TiO₂ sample shows blue-zones of contrast which indicate a good and perfect homogeneously distribution of Ti at the surface and some interruptions indicating that Ti is consisting the bulk of catalyst in good agreement with XRD and EDS results. The red contrast is attributed to copper element. The obtained percentages values of sensibilization by Rhodamine-B indicate that the sensibilization is achieved approximately at 150min and reaches values of 67.8% with 3Cu/a-TiO₂ photocatalyst, while in the case of a-TiO₂ support, the degradation showed that only 23%.

Figure 1. XRD and Cartography X of 3Cu/a-TiO₂



Keywords: Photocatalysis, Rhodamine-B, Methanol

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SYNTHESIS AND CHARACTERIZATION OF IRON OXIDE NANOPARTICLES FOR HETEROGENEOUS PHOTOCATALYSIS APPLICATION

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Abstract

Alpha iron oxide ($\alpha\text{-Fe}_2\text{O}_3$) or hematite is a semiconductor widely present in nature, it has a sufficiently narrow band gap (2.1 eV) to absorb 45% of the incident solar radiation which makes it an advantageous material for heterogeneous photocatalysis. However, improving the efficiency of the photocatalysis process is necessary in order, among other things, to reduce the recombination of electron-hole pairs, to increase the pollutant / semiconductor surface and to develop new materials capable of being used in visible light. Recent research has taken a lead in this field by developing nanomaterials active in the visible light. In this context, two synthesis methods have been selected for the production of this oxide, in the form of nanoscale powder: co-precipitation and sol-gel. The variation of the synthesis method made it possible to obtain different morphologies of hematite. The crystalline structure and the particle size of the samples obtained were characterized by X-ray diffraction. The optical properties of the samples were studied by UV-visible absorption spectroscopy. The specific surfaces of the samples were calculated by the BET method. The photocatalytic activity of the synthesized nanoparticles was compared to a natural and commercial hematite for the degradation of an herbicide, monolinuron (MLN). A degradation percentage of the MLN of 75% was obtained during the irradiation of an iron oxide suspension obtained by the sol-gel method in the presence of the MLN at 365 nm. The addition of hydrogen peroxide in the suspensions of iron oxide / sol-gel (IOS) in the dark or under UV irradiation significantly improved the kinetics of disappearance of the MLN. The degradation of MLN is mainly carried out by the oxidizing action of hydroxyl radical produced in the reaction medium, this has been confirmed by the addition of 3% of isopropanol in the IOS / H_2O_2 system.

The work carried out during this study made it possible to synthesize nanoparticles based on iron oxide, which had good photochatic properties.

Keywords: synthesis, nanoparticles, photocatalysis.

REMOVAL OF REACTIVE BLACK 5 FROM AQUEOUS SOLUTIONS BY NANO-TiO₂ IMMOBILIZED ON ACTIVATED CARBON

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Abstract

Adsorption onto activated carbon (AC) and oxidation using photocatalyst are widely used technics to remove dyes from waste water. The aim of this work is to increase the removal capacity of activated carbon by combining AC and titanium dioxide (TiO₂) in a **Photocatalytically Regenerative Activated Carbon**.

Anatase titania were deposited on powder activated carbon made from grape seeds (AGS) by impregnation/mixing method and then the composite photocatalyst was employed for the removal of reactive black 5 from aqueous solution. The TiO₂/AGS was characterized by BET, MEB, XRD and optical absorption spectroscopy. The BET surface area and the pore structure of composite photocatalyst (TiO₂/AGS) and activated grape seeds (AGS) were evaluated from nitrogen adsorption data at 77 K in relation to process conditions. Our results indicate that the photocatalytic activity of TiO₂/AGS was much higher than single phase titania. The adsorption equilibrium of reactive black 5 from aqueous solutions on the examined materials was investigated. Langmuir, Freundlich, and Redlich–Petersen models are in good agreement with the experimental equilibrium data. The degradation kinetics fitted well to the Langmuir-Hinselwood pseudo first order rate law. The photocatalytic activity of TiO₂/AGS was much higher than virgin TiO₂. COD removal was measured at regular intervals to quantify the mineralization of the dye. Above 96% mineralization was observed. These results suggest that UV-irradiated TiO₂ immobilized on activated carbon may be considered as an adequate process for the treatment of diluted colored textile waste water.

Keywords: photocatalytic, activated carbon, composite.

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BIOINSPIRED SYNTHESIS OF SILVER NANOPARTICLES

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Abstract

In this work, silver nanoparticles (Ag-NP's) were synthesized at room temperature. using leaf extract (of a plant with high abundance in our local region) as a green reducing and stabilizing agents. The formation of AgNPs was approved by surface Plasmon resonance (SPR) absorption peaks, using UV-vis spectrophotometer

and transmission electron microscopy.

The X-ray diffraction pattern of the dried nanoparticles revealed the information about different phases and crystallinity of biogenic Ag nanoparticles. Fourier transform infrared spectroscopy was performed to identify organic molecules that reduced and capped the colloidal particles in the reacting medium.

The H₂O₂ detecting ability of these biogenic silver nanoparticles was explored and the Ag nanoparticles were found to detect H₂O₂ instantly.

Keywords: Bioinspired, silver nanoparticles

SYNTHESIS OF ZnO-BENTONITE AS PHOTOCATALYSIS TO DEGRADE CATIONIC YELLOW DYE

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Abstract

Over the years, a large number of semi-conductors have been used as photo catalysts: TiO₂, ZnO, Fe₂O₃,...etc. Therefore ZnO has been found to be suitable alternative to TiO₂ due to its interesting properties like wide direct band gap of 3.3eV and high excitation binding energy of 60meV. ZnO nanoparticles can be prepared via various methods,. Several studies have shown the toxic effects of this nanoparticles on human health and environment, to decrease this effects a suitable solid clay matrices are used to restrict nanoparticles movement without suppressing their properties.

In our study we fix ZnO nanoparticles on the surface of Bentonite clay to obtain nanocomposites, which have photocatalytic activity after exposure to UV light, in our study we prepare ZnO nanoparticles and ZnO-bentonite via precipitation method using Zinc nitrate Zn(NO₃) and sodium hydroxide NaOH as precursors. We have also studied the effect of pH, temperature, contact time on the degradation process of cationic yellow dye used in textile industry.

Keywords: ZnO, bentonite, photocatalysis

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EFFECT OF FLUORINATED MEDIUM ON THE SYNTHESIS OF AIPO-34

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Abstract:

Aluminophosphate (AIPO's) molecular sieves were first reported by Union Carbide in 1980s [1]. This new family of aluminophosphates has interesting applications in various fields. Recently, metal-substituted aluminophosphates (M-AIPOs) have been effectively used as unique catalysts for many environmental and industrial applications [2], methanol-to-olefins (MTO) process [3] and De-NO_x, therefore the synthesis of AIPO-34 is developed from several methods such as microwave crystallization or the hydrothermal method.

In the present study, we investigate the influence of fluorinated medium on the synthesis of AIPO-34, chabazite is crystallized with fluorinated medium in the presence of silica but the absence of both element Fluor and silica the synthesis does not lead to formation of ALPO-34 where the introduction of the fluorine agent is required for crystallization, investigation of the various synthesis parameters was done, different reagents have been studied such as the aluminum source: aluminum hydroxide, aluminum isopropoxide and catapal, , in addition the effect of director the amine derivated are tested morpholine, DEA, TEA and piperidine. For a structural and textural study, the samples were analyzed by different characterization techniques such as DRX, BET, scanning electron microscope SEM, infra-red FTIR, thermal analysis ATG / ATD.

Keywords: Aluminophosphate, synthesis, Characterization.

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CO₂/CH₄ HYDRATES FORMATION KINETICS INFLUENCED BY Al₂O₃ AND SiO₂ NANOPARTICLES

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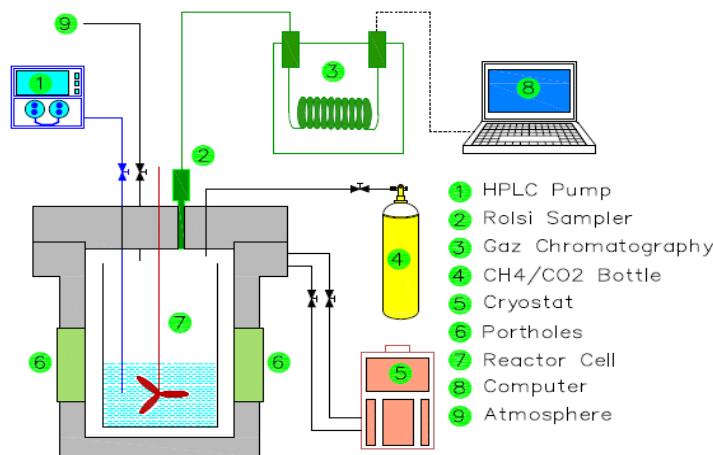
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Abstract

The process of CO₂ capture by hydrate formation is a promising alternative compared to the technologies available for carbon trapping and sequestration. Applying this technique to gas fields aims to achieve a selective separation of CO₂ under pressure. In this work we report the effect of oxide nanoparticles Al₂O₃, SiO₂ on the kinetic process of gas hydrates formation in a mixture (CO₂/CH₄). We measured the amount and the rate of gas consumed during the process of hydrates formation in the presence of suspensions (0,1-0,3% wt.) of nanoparticles, in pure water under 40bars at 1°C. It was observed that, the SiO₂ nanoparticle in suspension at 0,3% wt. showed the most significant influence. The average amount of consumed gas was higher than that in pure water at about 45% during the dissolution and nearly 77% during the crystallization step. The results were explained by the important increase in the transfer phenomena of heat and mass, allowed by the important increase in the surface area brought to the system at the nanoscale.

Figure 1. Schematic of the experimental setup.



Keywords: Gas hydrates, nanoparticles, kinetics.

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PHOTOCATALYTIC ACTIVITIES FOR HYDROGEN EVOLUTION OF NEW LAYERED COMPOUND $\text{Cu}_{0.5}\text{Nb}_3\text{O}_8$

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Abstract

$\text{Cu}_{0.5}\text{Nb}_3\text{O}_8$ was synthesized by successive reactions of conventional solid-state reaction followed by ion exchange reaction. X-ray diffraction, UV-vis diffusive reflectance and thermal analysis were used to characterize the photocatalyst. A tentative structural formula has been proposed on the basis of chemical composition, pH titration and thermogravimetric analysis. The obtained compounds had a layered structure similar to that of the original KNb_3O_8 powder. The thermal stability of these oxides and their parameters of orthorhombic cell were determined. Its physico-chemical characterisation reveals a semiconducting behaviour with a band gap energy of 1.89 eV showing efficient visible light absorption. The photocatalytic activity was evaluated for hydrogen generation under visible irradiation.

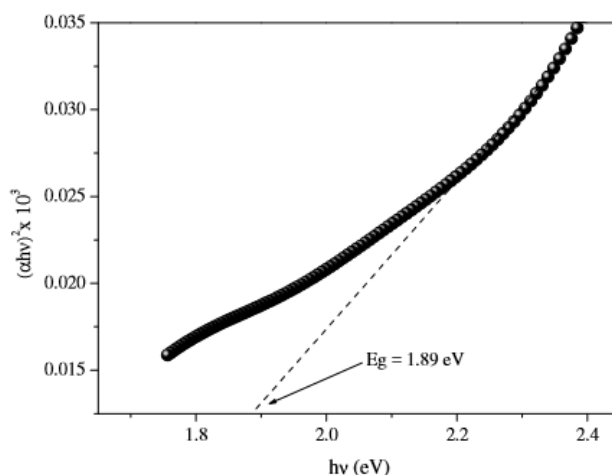


Figure 1. The direct optical transition

Keywords: Layered niobate; Hydrogen, Visible light.

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SYNTHESIS AND CHARACTERIZATION OF THE [Zr] ZSM-5 COMPOSITE FROM Ludox SILICA AND USE AS A CATALYST FOR OXIDATION OF 2-MERCAPTOBENZOTIAZOLE

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Abstract

Catalytic studies suggest the incorporation of zirconium into the MFI network. Indeed, previous studies concerning the properties of [Zr] ZSM-5, have shown that this material has very interesting applications, in particular, catalytic oxidation reactions in the field of industry. In this study, we present : the oxidation of 2-mercaptobenzotiazole to disulfide, catalyzed by [Zr] ZSM-5, in the presence of hydrogen peroxide as oxidizing agent.

Identification of the reaction products obtained, performed using spectroscopy, FTIR, elemental analysis (Sulfur) and melting points.

The thiol compounds disulfide readily with a weak oxidizing agent and presence of [Zr] ZSM-5, according to the literature, the thiol provides a binding energy (SH) 80 Kcal / mol.

The conversion of 2-mercaptobenzotiazole to disulfide was observed for a period of 15 minutes and the use of [Zr] ZSM-5 catalyst resulted in a yield of 61%. and the important conclusion from our results is that disulfide is a stable product because there are many C = S

From this study, we conclude that [Zr] ZM-5, obtained by incorporation into Zirconium in zeolite ZSM-5 can be reused in catalytic reaction. The [Zr] ZSM-5 as a catalyst offers several advantages, short reaction times, potential green chemistry, environmentally friendly and good returns

Keywords: [Zr] ZSM-5 MFI ,2-mercaptobenzotiazole, catalyse, oxidation

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THE EFFICIENT REMOVAL OF XYLENOL ORANGE DYE IN AN AQUEOUS SOLUTION BY MAGADIITE

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Abstract

Magadiite is a layered silicate in which solid layers are formed by the arrangement of silicon tetrahedra that contain terminal oxygen atoms neutralized by interlayer sodium ions and silanol groups (Si–OH). It has some specific properties, such as a high capacity for ion exchange, interlamellar adsorption of water and polar organic molecules [1], organosilane grafting, and transformation into crystalline layered silicic acids by proton exchange [2]. These specific properties could promote its application as cation exchanger, adsorbents for environmental pollutants [3] and supports for catalyst [4].

In the present work, a synthetic Na-magadiite material is organically modified by lactoserum. The materials obtained are characterized by X-ray diffraction and infrared spectroscopy and used in xylenol orange dye adsorption which is carried out as a function of pH, contact time and initial concentration of OX dye. The Langmuir and Freundlich adsorption isotherm models are applied in order to fit the experimental data in linear regression. The process of dye removal followed a pseudo second-order kinetics rather than pseudo first order.

Keywords: lamellar polysilicate – magadiite- adsorbent material

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H₄SiMo₁₂O₄₀ AS A CATALYST FOR THE SYNTHESIS OF 4-HYDROXY-4-METHYL COUMARIN

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Abstract

Coumarins are an important class of heterocyclic compounds of great structural diversity. They are generally used in the perfume and cosmetics sector, in pharmaceuticals [1], as well as in the preparation of insecticides, fluorescent brighteners and dye lasers [2]. They have potential biological interests such as anti-bacterial [3], anti-cancer [4], protease inhibitors of HIV-1 [5] and serve as anticoagulants.

The Pechman reaction [6] is the most widely used method for the synthesis of coumarin and its derivatives. Indeed, based on simple starting materials, phenols and β -keto esters, in the presence of an acid catalyst, the Pechman process leads in a relatively short reaction time to high yields.

In the present study, we propose to examine the efficacy of high-solids, non-toxic, non-polluting and very easy to synthesize heparopolyacides (HPA), as catalysts in the synthesis of 7-hydroxy-4-methyl coumarin via the condensation of Pechman.

The results of the variation of different parameters of the reaction show that the optimum conditions are: a 3/2 mmol ratio of resorcinol / ethyl acetoacetate, a reaction temperature of 80°C., 0.5%mol of catalyst (H₄SiMo₁₂O₄₀) 30 minutes of time, without use of solvent. Thus, the yield reaches 97% in these conditions, quite economical in terms of energy and environmentally friendly.

Keywords: Coumarin, Pechman Reaction, , Heteropolyacid,.

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FENTON AND FENTON-LIKE PROCESSES FOR INDUSTRIAL EFFLUENTS TREATMENT- A REVIEW

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Abstract

The removal of chemical pollutants and especially organic contaminants from industrial effluents requires the use of different technologies to achieve good effluent quality consistent with the discharge guidelines.

Alone or in combination with other techniques, Advanced Oxidation Processes (AOP) are promising technologies for the degradation of this type of pollution.

The most oxidation processes are based on the generation of hydroxyl radical (OH•) in sufficient quantity to effect the degradation of the organic matter. It is the most reactive oxidizing agent in wastewater treatment. Hydroxyl radicals are produced through different methods, including a combination of oxidizing agents such as H₂O₂ and O₃ and catalysts such as Fe²⁺ / Fe³⁺ used in the so-called Fenton and Fenton-like reaction.

This paper reviews the main advances in the application of Heterogeneous and homogeneous Fenton and Fenton-like processes for the treatment of industrial liquid effluent. Important parameters are discussed, particularly the dose and ratio effects of Iron and hydrogen peroxide.

Keywords: Fenton, Fenton-like processes, Industrial effluent

GOLD/CHITOSAN CATALYZED THREE MULTICOMPONENT COUPLING OF ALKYNE, CH₂CL₂ AND AMINES

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Abstract

The synthesis of gold nanoparticles of desired shape and size with uniform distribution within the polymer matrix remains highly challenging. This challenge can be made more meaningful by employing simpler and easier synthetic routes. Chitosan (CS), a natural biopolymer, has emerged as an attractive biomaterial for drug-delivery systems because of its biocompatible, biodegradable, and nontoxic^[1].

The multicomponent coupling reactions occurring between amines, haloalkane and terminal alkynes (AHA coupling reaction) are another useful transformation catalyzed by the homogenous and heterogeneous systems bearing gold^[2].

The reactions are a synthetic source of propargylamines which are also the important intermediates for the synthesis of biological active compounds used as the components for pharmaceuticals and plant protectants^[3].

In this study gold/chitosan was prepared by deposition precipitation with urea and characterized by IR, DRX, MEB and BET. This catalyst was used in AHA coupling with good yield of propargylamine.

Keywords: Chitosan, propargylamine, gold nanoparticle.

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BOROSILICATE ZSM-5 ZEOLITE SYNTHESIS IN THE PRESENCE OF *N,N*-DIMETHYLANILINE AND ITS PERFORMANCE IN CO₂ ADSORPTION

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Abstract

ZSM-5 zeolite has a great importance in hazardous substances adsorption and catalytic process. In this study, a borosilicate ZSM-5 (B-ZSM-5) zeolite was carried out using *N,N*-dimethylaniline (NNDMA) as a novel structure-directing agent. The synthesized material was characterized by X-ray diffraction (XRD), N₂ physical adsorption (BET), scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). CO₂ adsorption at different temperatures was evaluated by a volumetric method, and both Langmuir and Freundlich adsorption models were applied. According to the experimental results, B-ZSM-5 zeolite has a favorable adsorption behavior at low temperatures. Different kinetic models were used to describe the adsorption of CO₂ over B-ZSM-5. A good agreement with experimental data was found for pseudo-n order.

Keywords: Borosilicate ZSM-5; *N,N*-dimethylaniline; CO₂ adsorption.

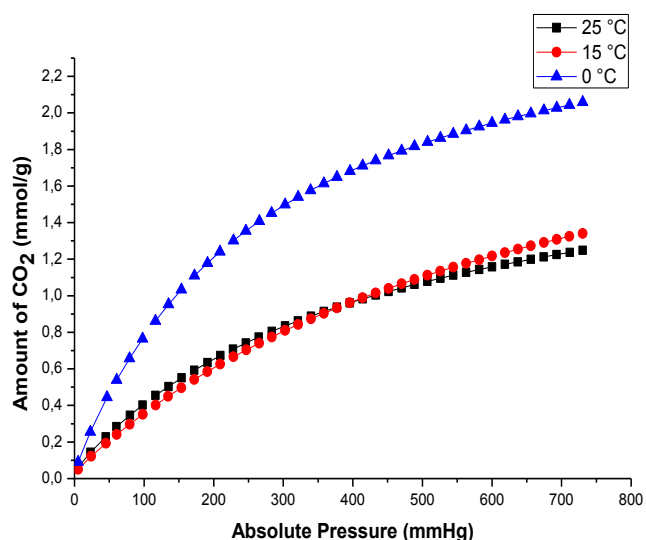


Figure 1. CO₂ adsorption at different temperatures.

SYNTHESIS OF TITANIUM-BASED HETEROGENEOUS CATALYSTS FOR PHOTOCATALYSIS APPLICATION

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Abstract:

A new form of pollution threatens the environment which is pharmaceutical pollution. In recent years several scientific studies have confirmed the presence of pharmaceutical and cosmetics traces in the aquatic environment, especially antibiotics and anti-inflammatory drugs [1], mainly rejected by the pharmaceutical industry, and this pollution causes dangers on the health and the ecology [2]. In addition, the presence of these pollutants continues to increase and their degradation presents a challenge to overcome.

Photocatalytic treatment is a technology of choice for the degradation of these pollutants because it is a powerful, simple and economical system [3]. The photocatalysis belongs to the wider field of heterogeneous catalysis; it is essentially a surface phenomenon using catalysts containing titanium oxide TiO₂, known by these oxidizing properties under ultraviolet light (UV). The free radicals released by the titanium allow oxidation-reduction: the organic molecules present on the treated surface are adsorbed and decomposed by the catalyst. Titanium dioxide, an inert compound, remains the raw material chosen by most manufacturers. It has also been proved that the oxides of semiconducting metals are very effective catalysts in the field of photocatalysis [3].

The aim of this work is to prepare a novel composite abrasive ZrO₂ particles coated by TiO₂ according the protocol described by Zhao et al [4] and Sadek et al [5] and apply in photodegradation of pharmaceutical rejections. These materials are also investigated by X-rays diffraction (XRD), N₂ adsorption-desorption at 77 K, thermal analysis (TGA/DTA) and Fourier-Transformed Infrared Spectroscopy (FTIR) technologies. The results of this study will be presented.

Keywords: Photocatalysis, pharmaceutical releases, semiconductors.

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VALORIZATION OF THE BIOMASS-BASED PLATFORM MOLECULE ON CLAY

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Abstract :

The depletion of fossil fuels and the degradation of the environment raise concerns about the sustainability of our society and the environment.

In the recent decades, various resources have been studied and developed to meet growing energy needs. Biomass remains the most available and the richest on what is suitable to replace its fossil energy.

The transformation of lignocellulosic biomass into target molecules allows the synthesis of a large number of products called "platform molecules", among these compounds we find furfural, which is an important constitutive element in bio-refinery.

Several catalytic processes have been studied to optimize this platform molecule such as: hydrogenation, condensation and oxidation.

Catalytic oxidation is used for the transformation of furfural into various high value-added molecules such as furoic acid, fumaric acid and succinic acid.

In this sense, we were interested in studying the oxidation of furfural with hydrogen peroxide in the presence of lamellar double hydroxides as catalysts, we prepared the catalysts Mg_RAlCO_3 (R = 2,3,4) by direct coprecipitation and precipitation by urea. These materials will be analyzed by DRX, FTIR, BET and SEM to confirm their formation, and their catalytic capacities will be tested in the oxidation of furfural, and the reaction products will be analyzed by HPLC.

Keywords: Biomass, recovery, double lamellar hydroxide, furfural, oxidation.

EFFECT OF SBA-15/SH MOLAR RATIO ON Pb(II) and Cd(II) IONS ADSORPTION FROM AQUEOUS SOLUTION

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Abstract

High levels of heavy metals in wastewater represent a serious threat to human health and ecological systems. Adsorption was shown to be one of the most promising techniques for heavy metals remediation. Utilization of functionalized mesoporous materials known as hybrid materials as adsorbent is widely reported in the literature [1-4]. Organic functional groups can be anchored to the silica backbone by post-synthesis reactions with residual Si–OH groups (post-synthesis grafting) or by a direct synthesis process based on co-condensation of silica precursors with organoalkoxysilanes. The purposes of the current study were to investigate the adsorption of Pb(II) and Cd(II) from aqueous solution by thiol-functionalized SBA-15(x) where x designed SBA-15/SH molar ratio. These materials were synthesized using 3-Mercaptopropyltrimethoxysilane (MPTMS) as organosilane precursor via post-grafting approach. SBA-15/SH(x) materials were characterized by XRD, nitrogen adsorption-desorption and FT-IR. The incorporation of SH fragments was proved by FT-IR Spectroscopy. The effects of the contact time, initial metals concentrations, pH, temperature and SBA-15/SH molar ratio on Pb (II) and Cd(II) uptake from aqueous solution have been studied.

The removal of lead ions is more important than that of cadmium ions. Furthermore, the lead removal on SBA-15/SH(x) increased with the increase of SBA-15/SH molar ratio while the removal of cadmium was not affected. The lead and cadmium removal on SBA-15/SH was pH dependent; it increased with the increase of pH showing a maximum at pH 5.5 and 6.1, respectively.

The equilibrium data fitted well with Langmuir model, while the adsorption kinetics followed the pseudo-second-order.

Keywords: Hybrid materials, adsorption, lead, cadmium.

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PRELIMINARY INVESTIGATION OF M-DOPED PILARED CLAY FOR VOCs TOTAL OXIDATION

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Abstract

Volatile organic compounds (VOCs) are recognized as the main sources of air pollution. In order to eliminate the VOCs, several studies have been made. Among the various methods employed for VOCs removal, catalytic oxidation is considered as one of the most efficient methods, which can destroy pollutants totally to carbon dioxide and water at relatively low temperatures [1-4]

In the present work, we report the preparation, characterization of M-doped pillared clay (M=Ni, Co, Cr and Fe) and their potential application in total oxidation of acetic acid. The catalysts were characterized by various techniques including N₂ physical adsorption, TG-DTA, XRD and TEM.

In acetic acid total oxidation, the results have shown that the CO₂ yield depends on the nature of the support, the nature of the active phase and the addition of lanthanum.

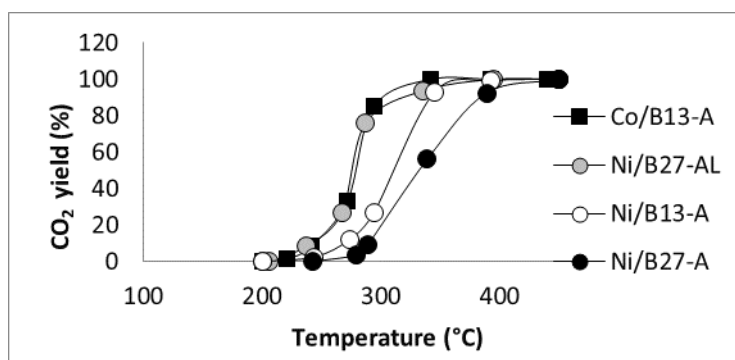


Figure 1. CO₂ conversion curves

Keywords: Catalytic oxidation, COVs, Pillared Clay

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PHOTOCATALYTIC ELIMINATION OF MALACHITE GREEN BY MATERIALS BASED ON TRANSITION METALS

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Abstract :

Advanced oxidation processes (POA) allow the total degradation (mineralization) in aqueous medium of persistent and / or toxic textile dyes to humans and to environment.

Malachite Green "MG" is a commercial synthetic dye that is used for various applications, e.g :

- to color fabrics (silk, wool, jute, leather, cotton) and paper,
- as an additive and food coloring,
- as a pH indicator,
- in the acrylic industry.

MG triphenyl methane dye has been used in aquacultures around the world for the treatment of fungal and parasitic fish infections. The presence of MGs in farmed fish could be a consequence of environmental pollution, they are considered as genotoxic and / or carcinogenic dyes.

The aim of this thesis is to photocatalytically degrade heterogeneous phase MG by new photocatalysts based on transition metals (Fe, Ag, Zn and W).

These photocatalysts will be prepared by different methods : Hydrothermal; Sol-Gel, co-precipitation.

The monitoring of the degradation will be carried out by UV spectrophotometry, GC / MS and by the measurement of the chemical oxygen demand (COD)

Keywords: Malachite green; heterogeneous photocatalysis, materials; COD; degradation ; mineralization

HYDROGEN GENERATION OVER COBALT CONTAINING MICROPOROUS ALUMINOPHOSPHATES PHOTO-CATALYST

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Abstract

Novel photo-catalytic materials based on cobalt type aluminophosphate molecular sieves have been elaborated in AEL and AFI structure by hydrothermal method. The photo-catalytic activity of CoAlPO₄-5 and CoAlPO₄-11 were evaluated for hydrogen production via water reduction under visible irradiation. The synthesized materials were characterized thoroughly with respect to elemental analysis, X-Ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), diffuse reflectance spectroscopy (DR), N₂ adsorption (BET) and NH₃ desorption measurement. Electrical conductivity, electrochemical and photo-electrochemical measurements such as cyclic voltammetry, currents-potential, Mott-Schottky and Nyquist were also studied. Since AlPO₄ materials possess a unique chemical composition as well as structural and micro-morphological characteristics. The photo-catalysts have shown a more negative flat band potential ($V_{fb} = 0.31$ eV) and a remarkable surface area ~ 183 and 258 m² g⁻¹ which lead to a better acceleration of the charge (e⁻/h⁺) transfer by increasing the number of surface reaction sites. In this approach, hydrogen was produced at atmospheric pressure and a temperature of 50 °C. Under visible light, the photo-catalysts exhibited a good photo-catalytic activity for hydrogen production with a yield of 64 and 1260 μ mol g⁻¹min⁻¹ for CoAlPO₄-5 and CoAlPO₄-11, respectively.

Keywords: CoAlPO₄-5, CoAlPO₄-11, nano-cobalt oxide, photo-electrochemical, photo-catalysis, hydrogen evolution.

ACIDO BASIC CATALYTIC HYDROLYSIS OF N,N-DIMETHYL-4-((4-NITROPHENYLIMINO) METHYL)ANILINE IN THE PRESENCE OF CYCLODEXTRIN : EXPERIMENTAL AND THEORETICAL STUDIES

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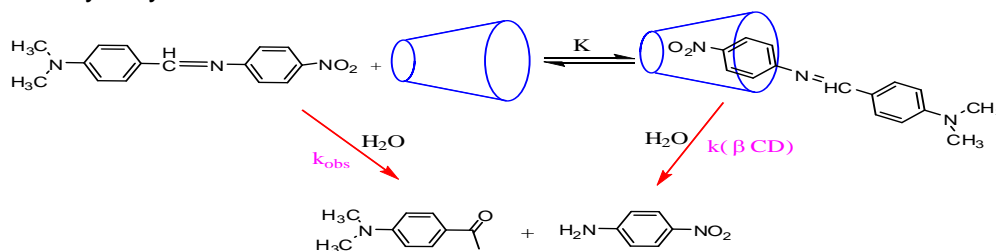
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Abstract

The kinetics of the generalized acido-basic hydrolysis of N,N-dimethyl-4-((4-nitrophenylimino) methyl)aniline was studied using UV-Vis spectrophotometry at 25 °C with respect of pH in absence and presence of cyclodextrine in methanol at ionic strength $\mu=0,1\text{M}$ KCl.

The PH – rate profile of the hydrolysis which is represented by the dependence between the logarithm of the observed pseudo-first order rate constants and the pH, shows that the decomposition kinetic behavior of the title compound alone is quite different from the one obtained in the presence of the cyclodextrin.

These results indicate that the guest molecule inclusion in the macromolecular cavity favors the stabilization of the N,N-dimethyl-4-((4-nitrophenylimino) methyl)aniline in the pH range interval where the formation conditions of the Host-Guest complex are combined, which could explain the reduction of the hydrolysis rate.



To justify this assumption the inclusion complex formation between N,N-dimethyl-4-((4-nitrophenylimino) methyl)aniline and the cyclodextrin was studied experimentally and with the help of molecular modeling. The non covalent interaction was carried out with UV-Vis spectrophotometry at 20 °C. The stability constant of the 1:1 complex and the thermodynamic quantities (ΔH , ΔS and ΔG) were obtained.

To get better insights, the work was complemented with DFT study at B3LYP level with the 6-311G basis set taking into account Grimme dispersion corrections.

The inclusion reaction in vacuum is exothermic and enthalpy driven process, while in water the inclusion is endothermic and enthalpy-entropy co-driven.

The molecular electrostatic potential (MEP), frequency calculations, HOMO-LUMO, energy gap, then the various intermolecular interactions that are responsible for the stabilization of the obtained complex were carried out by MEP, natural bond orbital (NBO) and QTAIM analysis

Keywords: catalytic hydrolysis – stabilization – interaction modeling

PHOTODEGRADATION OF PHENOL USING mont-La (6%)-Cu_{0.6}Cd_{0.4}S CATALYST UNDER NUV-VISIBLE IRRADIATION

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Abstract

Among persistent organic pollutants (POPs), phenol and its derivatives are generally considered as some of the most relevant organic pollutants discharged into the environment. Phenols are harmful to living organisms even at low concentrations [1] and recognized as carcinogenic compound [2]. They are easily absorbed through the skin and mucous membranes, and toxic to different organs and tissues: lungs, liver, kidneys, and genitourinary system [3]. For these reasons, it has become a challenge to achieve an effective removal of this POP from wastewater. A mont-La (6%)-Cu_{0.6}Cd_{0.4}S nanocomposite based on montmorillonite clay, cadmium sulfide, copper sulfide and lanthanum was prepared by a simple cation exchange and impregnation method and characterized by: XRD, FTIR and UV-vis DRS. Phenol in aqueous solution was used as a model compound for the photocatalytic activity evaluation of the synthesised catalyst under near UV-vis. We have studied the following parameters: load of photocatalyst, load of phenol, initial pH of solution and dissolved oxygen concentration (O₂). The obtained results show that within 240 min, heterogeneous suspensions of 1.0 g.L⁻¹ of mont-La (6 %)-Cu_{0.6}Cd_{0.4}S nanocomposite allowed removal of approximately 86 % of 20 mg.L⁻¹ solution of phenol at natural pH (pH = 5.44), with dissolved oxygen from air (21 % O₂). The kinetics of photocatalytic transformation followed the Langmuir-Hinshelwood kinetic model. Reaction intermediates produced during the degradation process were detected, and a suitable mechanism is proposed for the degradation of phenol based on high performance liquid chromatography coupled with mass spectrometry (HPLC/MS) analysis.

Keywords: photocatalysis, Phenol, NUV-Visible.

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ACETYLENE HYDROGENATION OVER Ni-Cu BIMETALLIC CATALYSTS: CHARACTERISATION AND REACTIVITY

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Abstract

A great deal of attention has been paid to Ni–Cu bimetallic catalysts for several decades by a number of groups [1-3]. The reason is that addition of a second metal is a way to modify the structural and electronic properties of the first one. Alloying could affect both the number of metallic atoms involved in the active sites and their reactivity [4]. Generally speaking, the addition of copper to a nickel supported catalyst decreases the activity of the nickel phase [3,5]. This is the case of low temperature hydrogenation reactions: benzene, ethylene [3] acetylene, 1-3 butadiene, unsaturated nitriles. In the present study we report the results of a study on the effect of a metal copper additive on the surface and catalytic properties of silica supported nickel nanoparticles prepared by reduction of nickel acetate by hydrazine in aqueous media [6].

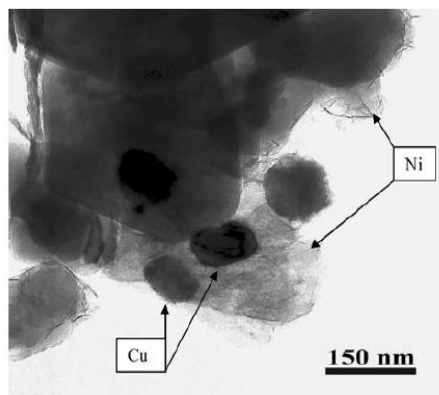


Figure 1. TEM image of the Ni-Cu bimetallic catalyst

Keywords: Bimetallic Catalysts, Acetylene, Hydrogenation

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SYNTHESIS OF FUNCTIONALIZED CARBON BASED MATERIALS FOR HMF PRODUCTION WITHOUT SOLVENT

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Abstract:

In the last decades, 5-hydroxymethylfurfural (HMF) captured great research interests driven by the economic and environmental incentives. A furanic derivative has been considered as a molecule platform with high exiting commercial and technical opportunities. It has a structure that can be accessed to versatile compounds. Among its very interesting valuable products there is an 2,5-furandicarboxylic acid, classified in the second row of the synthons valuable, in the report of the United States department of Energy (DOE), considered as one of the twelve most potentially useful biomass derived chemicals, and might be used as a renewable intermediate for polymers, fine chemicals, pharmaceuticals and agrochemicals. The HMF can be also converted by selective hydrogenolysis towards 2, 5-dimethylfuran (DMF), a biofuel which could have a superior energy efficiency of 40 % in that of some bioethanol. It is also the precursor of Levulinic acid used as to produce a series of biochemicals through esterification, halogenation, hydrogenation, condensation and other chemical reactions, and these chemicals are widely applied in solvents, medicine, food, biodegradable surfactants, agriculture, and biofuels.

As far as HMF is concerned, it can be produced by dehydration of C6 sugars in the presence of an acid catalyst. Synthesis of HMF from various sugar sources has traversed a long span of time and has witnessed various modifications viz., use of heterogeneous catalysts over homogeneous catalysts, employment of organic phase over aqueous, introduction of biphasic systems to overcome side reaction limitations and still counting.

Heterogeneous catalysts elaborations, such as new mesoporous materials have been presented the object of intense searches. Catalysts based mesoporous carbon materials, especially, activated carbon, carbon nanotubes, supported carbons and carbon sphere are widely studied in reason that they possess high stability, strong acid sites on the surface and a well determined pore size. Functionalized carbon materials by Brønsted and Lewis sites have excellent catalytic performances, we compare by activated, amorphous carbon and natural graphite; because of the structure chain compact carbon of these precursors and the lack of functional groups, particularly acid sites on surfaces. Therefore, this study has been devoted to the development of a series of catalysts based carbon material chemically functionalized by different agents precursors to produce HMF from fructose and/or glucose in absence of organic solvents to check the possibility of using these materials with simple procedure based "green" chemistry on the beginning step.

Keywords: Carbon, HMF, biomass.

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EFFECT OF EXCHANGEABLE CATION IN MONTMORILLONITE ON THE MULTICOMPONENT REACTION OF BIGINELLI FOR THE SYNTHESIS OF DIHYDROPYRIMIDONES

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Abstract

This work is part of green chemistry, in which we are interested in the use of montmorillonite; a natural product, to catalyze the multicomponent reaction of Biginelli for the synthesis of dihydropyrimidinones which have a very interesting application potential in the pharmaceutical industry [1-5].

The aim of the work is to study the effect of the exchangeable cation on the yield and duration of the Biginelli reaction, we chose the cations: Cu^{2+} , Ni^{2+} , Cr^{3+} , Co^{2+} , Fe^{2+} , Fe^{3+} known for their catalytic properties in homogeneous media.

After the cationic exchange operation, the Cationic Exchange Capacity (CEC) was determined by atomic absorption. The catalytic tests were carried out under reflux at a temperature of 85 ° C, the products of the reaction were characterized by Fourier Transform InfraRed (FTIR) and Nuclear Magnetic Resonance (NMR).

The obtained results have shown that the effect of the cation on the reaction yield and its kinetics is remarkable, we have found that the clay- Fe^{3+} has the highest yield (60.07%) followed by the clay- Co^{2+} (57.20%), the clay- Fe^{2+} is in third position (34, 15%), then come the other three exchanged with Cr^{3+} , Ni^{2+} and Cu^{2+} with approximately the same yield, the clays exchanged with the Fe^{3+} cation and the Co^{2+} cation have even competed with the homogeneous catalysis in the presence of HCl, a very good yield and faster reaction kinetics (88.33% and 9h) were obtained by optimizing some parameters of the reaction such as the amount of the catalyst, the type of solvent and the temperature.

Keywords: heterogeneous catalysis, montmorillonite, dihydropyrimidinones

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MESOPOROUS Al-MCM-41 MOLECULAR SIEVE OR $TiCl_4$ AS CATALYST FOR THE ALLYLATION OF AROMATIC ALDEHYDES WITH ALLYLTRIMETHYLSILANE

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Abstract

Mesoporous Al-MCM-41 molecular sieve with Si/Al ratio equal to 12.5 was synthesized under hydrothermal condition using cetyltrimethylammonium bromide (CTAB) as surfactant. The structural and textural properties of this catalyst were measured using various analytical instruments. The catalytic performance of Al-MCM-41 catalyst as Lewis acid was used without treatment and was compared with $TiCl_4$ in the allylation of aromatic aldehydes with allyltrimethylsilane. Pyridine coupled FT-IR spectroscopy analysis on Al-MCM-41 showed the existence of a high concentration of acid sites, with both Brønsted and Lewis sites. The site acids of Lewis of Al-MCM-41 are highly efficient for the allylation of various aldehydes with allyltrimethylsilane. Similar to $TiCl_4$ the Lewis acid promotes the formation of the diallylation [2], whereas in the presence of Al-MCM-41, the single allylation was obtained using mild conditions. To explain this different allylation in the presence of the MCM-41 or $TiCl_4$, a reaction mechanism is proposed [1]. The Al-MCM-41 was used in four consecutive experiments without important loss of activity, confirming its stability.

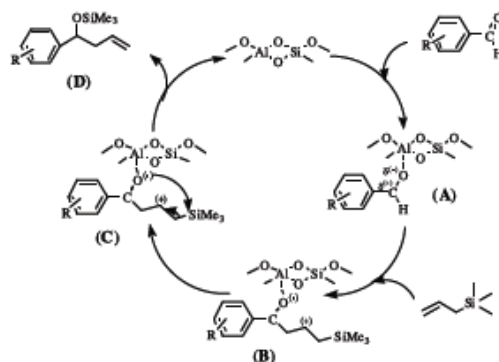


Figure 1. Plausible mechanism of role of Al-MCM-41 as a Lewis acid catalyst in allylation of various aldehydes with allyltrimethylsilane

Keywords: Al-MCM-41, $TiCl_4$, Allyltrimethylsilane

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VISIBLE-LIGHT PHOTOCATALYTIC PERFORMANCES OF TiO₂ NANOBELTS DECORATED WITH IRON OXIDE NANOCRYSTALS

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Abstract

A TiO₂-Fe₂O₃ hetero-nanostructure was fabricated via a three-step method and evaluated for its visible-light photocatalytic performances. TiO₂ nanobelts (NBs) were synthesized by controlled hydrothermal oxidation of a Ti sheet (Figure 1) while Fe₂O₃ nanocrystals (NCs) were prepared by forced hydrolysis in polyol. NBs were then decorated with NCs using direct impregnation [1]. The structure, morphology and chemical composition of the resulting hybrids were investigated by X-Ray Diffraction (XRD), Field Emission Gun Scanning Electron Microscopy (FEG-SEM), Transmission Electron Microscopy (TEM), X-ray Photoelectron Spectroscopy (XPS), Raman and UV-visible diffuse reflectance spectroscopy. Its photocatalytic activity was assessed by following methylene blue photodegradation in an aqueous solution under visible light irradiation and compared to that of bare TiO₂ NBs. Its photoelectrochemical properties were determined measuring its ability to oxidize water under mimetic sunlight irradiation. The significant observed improvement was attributed to sensitization of the wide-gap titania semiconductor by the narrow-gap hematite one, namely faster interfacial charge transfer and wider spectral light absorption (Figure. 1).

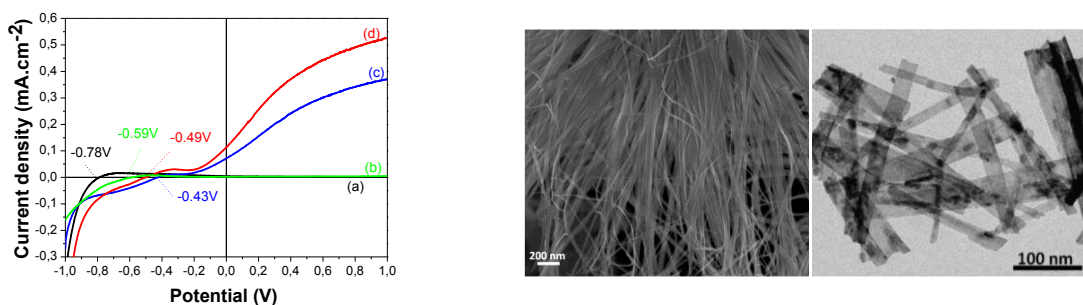


Figure 1. Photocurrent density J_p versus voltage E (against Ag/AgCl) of TiO₂/Ti (a), TiO₂-Fe₂O₃/Ti (b) in the dark and under xenon lamp illumination (c) and (d) respectively (left). SEM top view of TiO₂ NB array and TEM micrograph of some representative NBs peeled from Ti sheet (right).

Keywords: Photocatalytic performance, TiO₂ NBS, Fe₂O₃ NCS.

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GOLD CATALYSTS AU/C IN BASE-FREE AEROBIC OXIDATION OF GLUCOSE TO GLUCONIC ACID

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Abstract

Gluconic acid is an important compound, it is an important additive for the food and pharmaceutical industry. Until now, gluconic acid has been mainly produced by an enzyme catalyzed oxidation procedure, which is marked by numerous preparation steps and lower productivity [1]. There is therefore an industrial interest for an alternative glucose oxidation method using air on a heterogeneous catalyst. So far, gold-based catalysts have been mainly used for partial and complete gas phase oxidation (eg oxidation of CO [2,3]) and for selective hydrogenation [4]. An important requirement for catalytic activity is the presence of enough small gold particles in the range of 3-6 nm. In this study, a series of gold colloids were prepared and immobilized on commercial activated carbon. The influence of the colloidal preparation and stability has been studied and related to the size of the gold particles in the final catalyst. The catalysts show significant activity in the glucose oxidation reaction to gluconic acid, resulting in a gluconic acid yield close to 90% under mild conditions without base (0.1 MPa O₂ and 40°C). Size-activity correlation and probable mechanism were also discussed. Finally, the viability of the catalyst was tested by recycling it up to four times.

Keywords: Au, Carbon, Glucose.

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NICKEL CATALYSTS FOR HYDRODEOXYGENATION OF FURFURAL-ACETONE CONDENSATION ADDUCTS TO BIO LIQUID ALKANES

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Abstract

Biomass conversion into transportation fuels, biofuels, has become more important because of rising prices and the shortage of oil and global warming [1],[2]. Transportation fuels such as gasoline, diesel, and jet fuel are the main targets of biofuels.

These “first-generation” biofuels such as Bioethanol and fatty acid alkyl esters as biodiesel are now commercialized on a large scale. However, they have problems as low energy density, low stability, and conflict with food production. To resolve these problems intense work has been undertaken to develop next-generation biofuels that are produced from nonfood biomass and are totally compatible with petroleum-based transportation fuels composed of hydrocarbons. Since biomass usually contains a large amount of oxygen and low energy density, hydrocarbon production for next-generation biofuels requires a hydrodeoxygenation step (removal of oxygen with hydrogen).

However, hydrodeoxygenation is rarely used in industrial chemistry, and the development of effective catalytic systems has been conducted only in recent years. many research focus on the conversions of carbohydrates and carbohydrate-derived compounds to alkanes because carbohydrates are the main components of the most abundant and accessible resources: crops and lignocellulose [3] . Whereby lignocellulosic biomass is first converted through biotechnological processes to well-identified platform molecules that can be employed as building blocks for chemical synthesis by catalytic routes such as Furfural [4].

Our goal is the production of liquid bioalkanes starting from a precursor molecule (1,5-bis(2-Furanyl)-1,4-pentadien-3-one), the path we will follow involves two major steps:

1) The organic step: is a condensation reaction for Furfural in liquid phase allows the production of a larger molecule (precursor molecule).

2) The catalytic step consists of converting the precursor molecule into a liquid alkane by a hydrodeoxygenation reaction in the presence of Nickel supported oxides catalysts.

Keywords: Furfural , Hydrodeoxygenation , Bioalkanes

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EFFECT OF THE METAL LOADING ON THE CATALYTIC OXIDATION OF CYCLOHEXENE OVER RUTHENIUM SUPPORTED ON TITANIUM-PILLARED CLAY

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Abstract

The aim of this work is the valorization of bentonite-type clay from Maghnia (Algeria) as green catalyst for the production of added value fine chemicals. The modification of the clay structure by acid activation (H-Mont) and intercalation of transition metals such as titanium (Ti-PILC) and finally by impregnation of ruthenium, enhanced redox properties and increased Lewis and Bronsted acidity. Various x%Ru/Ti-PILC were prepared and characterized by diffuse-reflectance UV–vis spectroscopy, surface acidity followed by FT-IR, chemical analysis, FT-IR, X-ray diffraction and nitrogen adsorption–desorption isotherms. The catalysts prepared were studied in the cyclohexene oxidation, using tert-butyl hydroperoxide as oxidant and heptane as solvent. Both catalysts can selectively oxidize cyclohexene through allylic oxidation to give 2-cyclohexene-1-one (Enone) as the major product, and 2-cyclohexene-1-ol (Enol) as the minor product. 5%Ru/Ti-PILC was employed as catalyst, 59 % cyclohexene conversion, 87 % selectivity for 2-cyclohexene-1-one and 13 % selectivity for 2-cyclohexene-1-ol were obtained under ambient pressure, at 70 °C, for a 6 h reaction time. The materials were reused in four consecutive runs.

Cyclohexene oxidation in presence of catalysts and supports

Catalyst	Conversion (%)	TBHP Consumption (%)	Selectivity (%)	
			Enol	Enone
Blank	0	0	0	0
H-Mont	0	3	0	0
5% Ru/H-Mont	22	31	33	67
Ti-PILC	26	42	89	11
1%Ru/Ti-PILC	18	43	61	39
2%Ru/Ti-PILC	42	44	24	75
3%Ru/Ti-PILC	44	40	39	60
5%Ru/Ti-PILC	59	73	13	87

Reaction conditions: 29 mmol cyclohexene, 58 mmol TBHP, 20 mL heptane, 0.1 g catalyst, time: 6 h, reaction temperature 70 °C.

Keywords : Titanium, Ruthenium, Bentonite, Oxidation and cyclohexene.

COORDINATION OF MODELING AND EXPERIMENTAL RESEARCH OF NANOMATERIAL CATALYSTS FOR THE ADSORPTION AND OXIDATION REACTIONS OF ORGANIC MOLECULES

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Abstract

The purpose of this work is to study the oxidation of organic molecules with the use of nanomaterials of type zeolites and more particularly the ZSM-5 by the combination of experimental techniques and molecular modeling. This type of zeolite is generally used in separation and catalysis processes; This is due to the relatively medium pore size, its high specific volume and its low Si/Al ratio which makes it possible to prepare materials rich in metal ions, and also its low cost of synthesis.

An experimental study was carried out by syntheses of ZSM-5 zeolites with low transition metal contents, as we have done a series of catalytic tests on the oxidation of organic molecules by these zeolite products; The various physico-chemical characterization techniques (SEM, BET, EDX, DRX, IR, UV) give us information on the structure and texture of the products obtained and confirm the effectiveness of the catalytic tests. As part of this research, we also used molecular modeling, which is an essential tool for predicting the reactivity of these materials. Molecular modeling techniques have been carried out using Gaussian software 09 used in specific computer programs to evaluate physicochemical, energy properties, determine reaction pathways, identify reaction intermediates and transition states of molecules and clusters (Nanomaterials, Oxidant, Substrate).

All the results obtained showed the bearing capacity of the experimental study (in vitro) with a computational study (in silico) to predict all the scientific information on the zeolite-substrate system.

Keywords: Nanomaterials catalysts, Experimental synthesis, Molecular modeling.

ANDERSON-TYPE POLYOXOMETALLATES AS CATALYSTS IN ADIPIC ACID SYNTHESIS

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Abstract

Polyoxometalates have proven their efficiency as catalysts in adipic acid synthesis [1,2]. This later is one of a basic intermediate for the nylon production [3], however, its industrial production [4], involves the use of concentrated nitric acid that contributes to atmospheric pollution by release harmful gases (N₂O..)[5].

The aim of this study is to substitute the polluting and corrosive nitric acid by hydrogen peroxide and develop an ecologic process for adipic acid (AA) synthesis, using Anderson-type polyoxometalates as catalysts. For this purpose, (NH₄)₃[H₆SbMo₆O₂₄], (NH₄)₄[H₆SnMo₆O₂₄] and (NH₄)₄[H₆CoMo₆O₂₄] were prepared, characterized and tested in the AA synthesis from cyclohexanone and mixture of cyclohexanone and cyclohexanol in the presence of hydrogen peroxide, free solvent. The effects of reaction temperature and substrate nature on adipic acid formation were examined. The highest AA yield (69 %) was obtained at 90°C from cyclohexanone and cyclohexanol mixture with (NH₄)₃[H₆SbMo₆O₂₄] catalyst and reaction mixture stirring rate of 1000rpm during 20h.

The use of both Anderson type polyoxometalate and hydrogen peroxide in solvent free media can be an efficient and green strategy for adipic acid production where hydrogen peroxide is only reduced to water, compared to industrial process that leads to the N₂O formation, atmosphere pollutant product.

Keywords: Anderson, polyoxometalates, Adipic acid

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EFFECT OF POSITION OF NICKEL ON THE Ni/Mg/Al HYDROTALCITE STRUCTURE FOR THE DRY REFORMING OF METHANE

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Abstract: CO₂ reforming of methane shows a growing interest from both industrial and environmental viewpoint. From an environmental perspective, CO₂ and CH₄ are undesirable greenhouse gases and both are consumed by the proposed reaction. The main purpose of this work is to study the effect of Ni/Mg/Al synthesis methods. Ni/Mg/Al solids were prepared via three ways: (i) Common impregnation of Ni salts on MgAl hydrotalcite and was noted Ni/MgAl. (ii) Coprecipitation at basic pH of Ni²⁺, Mg²⁺ and Al³⁺ to obtain NiMgAl sample. (iii) The final way was based on the ability of Ni²⁺ to react with an anionic chelating agent of EDTA⁴⁻ (ethylene diamine tetraacetate) forming the highly stable [Ni(EDTA)]²⁻. It was suggested that coprecipitation of Mg²⁺ and Al³⁺ with pre-formed [Ni(EDTA)]²⁻ gave rise to Mg–Al intercalated with Ni chelate [1] to obtain Ni(EDTA)MgAl. The synthesized samples were calcined at 800°C. The calcined and non calcined samples were characterized by XRD, FTIR, SEM, BET and method H₂-TPR measurements. The XRD patterns for the non calcined samples exhibit the characteristic diffractions of hydrotalcite-like layered double hydroxide materials. BET surface area results after calcination for NiMgAl and Ni(EDTA)MgAl presented a relatively high surface areas. The results of H₂-TPR measurements of the calcined samples showed a single reduction peak in the case of NiMgAl and Ni/MgAl at higher temperature reduction (around 800°C), indicating the existence of species of NiO with strong interaction resulting from the formation of the NiO–MgO solid solution, characterized by its high stability [2]. However Ni (EDTA)MgAl present two reduction Peaks. The CH₄/CO₂ reaction was carried out in a fixed-bed tubular reactor at 750°C. A reactant mixture CH₄ and CO₂ were mixed at a ratio of diluted in He (10:10:80). Reactants and products were analyzed using a gas chromatograph (Varian, GC-3800) with a thermal conductivity detector (TCD) and a column Porapak. The catalysts show a significant CH₄ conversions such as 94,0%, 80,0 % and 60,0% respectively for NiMgAl, Ni(EDTA)MgAl and Ni/MgAl versus respectively 95,0%, 85,0% and 75,0% for CO₂ conversions. CO₂ conversion as given were found to be higher than the one for CH₄ conversion in the case of Ni(EDTA)MgAl and Ni/MgAl which indicate that probably the CO₂ is consumed in parallel in the water gas shift reaction (RWGS) [3]. Thus, it's clear that synthesis methods of catalysts have a clear effect on catalytic performances.

Key words : *Hydrotalcite materials, Dry reforming of methane.*

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REMOVAL OF 4- (2-PYRIDYLAZO) -RESORCINOL FROM AQUEOUS SOLUTIONS BY AN ADSORPTION TECHNIQUE USING AN ALGERIAN KAOLIN

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Abstract

In recent years, a large amount of dyes used in the textile industries are azo compounds, which are characterized by the chromophoric group -N = N- linked to aromatic cycles as (Congo red, orange methyl ...). These dyes have become one of the most toxic compounds because of their adverse effect on the environment, causing damage to the gills of aquatic organisms and disrupting their spawning sites.

The toxicity of these compounds is enhanced by bioaccumulation and can be transported and released elsewhere in water sources, sediments and food. Several physicochemical techniques have been used to remove azo compounds from aquatic environments, including advanced oxidation, microbial biodegradation and photo-degradation. However, many of these techniques are too expensive, especially when used to treat large waste streams. Therefore, adsorption onto natural solid substrates such as clays, activated carbon and natural fibers are among the most practical and economical techniques used to remove azo compounds from aqueous media.

In the present work, we were interested in the removal of 4- (2-pyridylazo) -resorcinol from an aqueous solution by an adsorption technique using an Algerian kaolin extracted from Jebel Dbagh in Guelma. In order to achieve proper removal of this compound, we activated natural kaolin using nitric acid to improve its adsorption properties. The effects on the adsorption of certain physicochemical parameters such as the contact time, the amount of kaolin, the initial dye concentration, the pH of the medium and the bath temperature were carried out.

Thermodynamic parameters (ΔH , ΔG and ΔS) at different temperatures were determined.

Keywords: 4- (2-pyridylazo) -resorcinol, Adsorption, Algerian kaolin.

BIOCONVERSION OF LIGNOCELLULOSIC BIOMASS FOR THE PRODUCTION OF BIOETHANOL FROM AGRICULTURAL WASTE

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Abstract

In this study, the valorization of agricultural waste in order to produce bioethanol fuel of the second generation have been made from lignocellulosic biomass represented in peelings of pomegranate (*Punica granatum*). Two processes are carried out in this study: Separate Hydrolysis and Fermentation (SHF) and Simultaneous Saccharification and Fermentation (SSF). Two different methods of pretreatment were used in order to depolymerize the lignocellulosic structure. As well as, saccharification has been carried out using two different enzymes cellulase and hemicellulase in order to have fermentable sugars. After this stage, the hydrolysate was fermented using *Saccharomyces cerevisiae* under anaerobic conditions (pH=4.7-5, T=30-37°C, 250 rpm). The concentration of bioethanol was evaluated after 48 and 72 hours of fermentation.

Keywords: bioethanol, lignocellulosic biomass, *Saccharomyces cerevisiae*

SYNTHESIS OF METAL-CONTAINING MCM-41 AND ITS APPLICATION FOR CATALYTIC OXIDATION OF PRIMARY ALCOHOL WITH H₂O₂

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Abstract

In this work, Metal-containing MCM-41 (Fe/Al-MCM-41 and Cr/Al-MCM-41) materials have been synthesized by direct synthesis and dry impregnation methods, for testing in the oxidation reaction of a primary alcohol under a pressure inert. The catalysts were characterised by XRD, FTIR, N₂ adsorption-desorption isotherms and TGA-DTA. The hexagonal structure of mesoporous material modified by iron or chromium, either by direct synthesis or by dry impregnation, was preserved, which assigned the incorporation of Fe and Cr species do not destroy the pristine hexagonal symmetry. The BET surface area measurements revealed the reduction in the surface area and pore volume due to the incorporation of the metal species into the MCM-41 framework. All prepared catalysts have been tested in the oxidation reaction of 1-hexanol with hydrogen peroxide as oxidant. The results showed that Cr-containing Al-MCM-41, prepared by direct synthesis, was the most active catalyst among the different catalysts investigated in this study, which could be attributed to the Lewis acid sites.

Keywords: mesostructured silica, aluminosilicates, oxidation of 1-hexanol.

DEVELOPMENTS IN HETEROGENEOUS CATALYSIS FOR THE SUSTAINABLE PRODUCTION OF BIODIESEL

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Abstract

Heterogeneous catalysis has a rich history of facilitating energy efficient selective molecular transformations and contributes to 90% of chemical manufacturing processes and to more than 20% of all industrial products [1]. Biodiesel has become beguiling nowadays for its environmental benefits and it seems an opposite alternative fuels for future. It is made from renewable biological sources such as vegetable oils and animal fats [2].

Transestérification is one of the methods for biodiesel production in which oil or fat is reacted with alcohol. Therefore, this review paper is aimed to give an overview on the recent trends of catalyzed transesterification and the advantages and disadvantages of heterogeneous. The castor oil composition was determined by FTIR, HPLC, and their physicochemical properties in order to evaluate their effectiveness in the synthesis.

The solid catalyst was prepared with bentonite modified by metals. The optimal conditions of the reaction are determined to end in a good conversion (report molar oil/alcohol, temperature and quantity of the catalyst). The EEHV (vegetable oil ethyl esters) are characterized and compared with the fossil diesel fuel.

Keywords : transesterification, biodiesel, heterogeneous catalyst, modified clay.

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SYNTHESIS, CHARACTERIZATION OF CeO₂/SBA-15 MESOPOROUS MATERIAL AND ITS PHOTOCATALYTIC PERFORMANCE IN THE 2,2'-BIPYRIDINE DEGRADATION UNDER VISIBLE-LIGHT IRRADIATION

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Abstract

In this paper, we describe the synthesis of CeO₂ nanoparticles (NPs) by nanocasting route on the mesoporous silica support. Firstly, CeO₂ NPs was prepared in alkaline medium via the homogeneous precipitation method while the mesoporous silica (SBA-15) was prepared using soft templates by hydrothermal technique. In the second step, CeO₂/SBA-15 mesoporous assembled material was synthesized by impregnating cerium salt on SBA-15 support and followed by calcination at 550 °C (denoted as CeO₂/SBA-15). All the samples are characterized by XRD, FTIR, SEM-EDX, UV–VIS DRS and N₂ adsorption/desorption analysis. Photocatalytic efficiency of these materials was assessed by the degradation of 2,2'-Bipyridine (BPy) in the simulated waste, under visible light irradiation. Results from characterizations showed that CeO₂ had a small crystalline size and highly dispersed on SBA-15 and it is confirmed by XRD. CeO₂/SBA-15, material maintains its ordered hexagonal mesoporous structure and exhibits a red shift (2.16 eV vs 3.20 eV) compared to the bare CeO₂. The inclusion of SBA-15 significantly enhanced the photocatalytic efficiency of the catalyst. CeO₂/SBA-15 catalyst yielded the highest degradation (η' = 96.54 %) of BPy under visible-irradiation within 180 min, in comparison with CeO₂ (η' = 10.32 %). The photodegradation of BPy substrate by CeO₂/SBA-15 catalyst followed a pseudo first-order rate law. From the above physicochemical and photocatalytic performances point of view, the enhanced photocatalytic efficiency of CeO₂/SBA-15 catalyst was attributed to the synergetic effect of mesoporosity and the presence of Ce³⁺ species along with oxygen vacancies.

Key word: 2,2'-Bipyridine, CeO₂/SBA-15, photocatalytic efficiency.

PHOTODEGRADATION OF ORGANIC POLLUTANTS IN WATER BY MAGNETIC HETEROGENEOUS FENTON CATALYSTS UNDER VISIBLE IRRADIATION

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Abstract

The incapability of the conventional wastewater treatment methods to effectively remove many toxic pollutants stands for the development of new treatment systems. In this field, Advanced Oxidation Processes (AOPs) are becoming increasingly important, especially for the degradation of a large number of hazardous and recalcitrant organic pollutants. Among them, the Photo-Fenton process is considered as one of the most efficient AOPs. Although the photo-Fenton process is also efficient in homogeneous phase, the use of a heterogeneous catalyst provides the possibility to recover and reuse the catalyst and to operate in a broader pH range [1]. One of the challenges in this field is to develop heterogeneous catalysts which efficiently use visible light irradiation instead of UV light. Iron oxides are suitable candidates to be used as visible-light photo-Fenton catalysts for water treatment [2]. In this work, we report the degradation of various model organic pollutants, especially dyes, by the photo-Fenton process under the visible light emitted by a simple halogen lamp, in presence of heterogeneous catalysts based on maghemite ($\gamma\text{-Fe}_2\text{O}_3$) nanoparticles. The strong magnetic susceptibility of these materials implies that they can be easily recovered by a simple application of an external magnetic field given by a magnet or an electromagnet. This can be used to remove the catalyst from the effluent, with a better efficiency than conventional methods such as filtration or settling [3]. The catalysts were characterized by various methods (electronic microscopies, adsorption volumetry, XRD, magnetometry...). Two catalysts were tested, depending on whether the $\gamma\text{-Fe}_2\text{O}_3$ nanoparticles were used dispersed in water, or supported on the internal surface of silica microspheres. The degradation of the pollutants was always followed both by UV-visible spectroscopy, and NPOC analysis. Experiments of long-term stability showed that the MS catalyst, although generally less active than the NP catalyst, retained almost all of its activity after five repeated experiments under visible light. The good stability of this catalyst was also confirmed by the low level of iron leaching, making it suitable candidate for an application as photo-Fenton catalyst in industrial wastewater treatment.

Keywords: Photo-Fenton process, magnetic heterogeneous catalysts, wastewater treatment

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EFFICIENT CATALYTIC DEGRADATION OF 4-NITROPHENOL USING SiO₂@Cu CORE@SHELL MATERIALS

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Abstract

In recent years, various techniques have been developed for the removal of toxic dyes and nitro-aromatic pollutants. However, catalytic reduction has been found to be superior to other methods owing to its low cost, simplicity of design, and ease of operation [1]. So, numerous toxic organic pollutants can be removed by catalytic reduction in the presence of NaBH₄ in a short reaction time. Because of its high stability and solubility in water, 4-NP is not removed and effectively purified under a conventional water purification treatment [2]. An environment friendly, simple, and cost effective method for the removal of the 4-NP compound is to convert it into useful 4-aminophenol (4-AP) using catalytic reduction in the presence of NaBH₄ as the reducing agent [3]. The reduction of the 4-NP compound towards 4-AP is industrially significant and a vital method for environmental remediation. Recently, chemists have increasingly focused on the development of new, cheap, and more efficient catalysts that can support the treatment of wastewaters laden with these harmful organic pollutants. Accordingly, a variety of heterogeneous catalysts comprising noble metals have been developed and used extensively for the reduction of 4-NP in the presence of sodium borohydride. Nevertheless, their high price and low accessibility limit their utilization in practical applications. For this reason, it is very crucial to develop highly efficient, low-cost, environmentally friendly and non-noble metal catalysts to facilitate the catalytic reduction of 4-NP by NaBH₄.

In the present work, we focused on the synthesis of copper particles supported on spherical silica SiO₂@Cu type core-shell by hydrothermal method with a copper weight of 5 and 10% followed by drying, and reduction in hydrogen flow at 300 °C. The materials were characterized by different techniques as X-ray diffraction XRD, scanning electron microscopy SEM/XDE, Raman, temperature programmed reduction TPR, Fourier transform infrared spectroscopy FTIR, surface analysis by BET method, ATG/DTG analysis and X-ray Fluorescence spectroscopy XRF. The results have shown the apparition of copper particles and the spherical silica with a mean diameter of 300 nm. The applicability of SiO₂@Cu core-shell catalyst for the degradation of 4-NP was investigated. The conversion yield of 4-nitrophenol to 4-aminophenol in the reaction mixture was determined to be 100 % .The influence of various parameters on the degradation efficiency was studied.

Keywords: core@shell materials, 4-nitrophenol degradation, catalytic activity.

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EFFECT OF THE ELECTROLYTE CONCENTRATION FOR CO₂ PHOTO-ELECTROCHEMICAL REDUCTION BY CuO PHOTO CATALYST: A CYCLIC VOLTAMMETRY STUDY

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Abstract

As it is the most important of the greenhouse gases, the utilization and reduction of carbon dioxide have attracted a great attention. In this research, CuO as a photocatalyst is used to achieve the carbon dioxide (CO₂) photo electrochemical reduction (PEC) into acid formic and fuels such as methanol and ethanol under visible light illumination. Copper oxide is a well-known p-type semiconductor with a narrow band gap of 1.2 eV; the material was characterized by X-ray diffraction, Fourier transform infrared spectroscopy, ultraviolet–visible,

We focus to study the effect of concentration of electrolyte (NaHCO₃) by the cyclic voltammetry method on dark and illumination conditions. The properties of reaction medium play a key role in determining the efficiency of CO₂ photoreduction and modulating the distribution of reaction products. The results show an interesting reduction of CO₂ to useful carbon-based chemicals.

Keywords: greenhouse gases, CO₂, photo electrochemical reduction, concentration of electrolyte.

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ULTRASOUND-ASSISTED BIOSORPTION OF BASIC BLUE 9 FROM AQUEOUS MEDIA

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Abstract

In this study, the novel material was evaluated as biosorbent for the removal of basic blue 9 from aqueous solutions in the absence and presence of ultrasound. The influence of acoustic power, initial concentration of the dye, biosorbent dose and temperature has been verified in order to explain the influence of ultrasonic irradiation on biosorption kinetics. The acoustic power was an important factor for the enhancement of the removal of dye. Equilibrium isotherms were determined by stirring and sonication. These equilibrium biosorption data were analysed with five different forms of Langmuir, Flory – Huggins and Freundlich isotherms models. This study showed that the combination ultrasound – biosorption can be an alternative technique to conventional method.

Additionally, the biosorbent was characterized by isoelectric potential (pH_{ZPC}) and the acidic and basic sites were calculated by the Boehm titration method.

Keywords: Ultrasound; Biosorption; basic blue 9.

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QUANTITATIVE STUDY OF HYDROPHOBICITY OF MATERIALS IN CATALYSIS

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Abstract

In catalysis, the property of hydrophobicity is of great importance to understand the adsorption phenomena of catalysts and to quantify the impact of this property on its activity and/or its selectivity in a given reaction [1, 2]. Like any surface property of a catalyst, hydrophobicity is also important for understanding, improving, and directing a given reaction. Moreover, the increased hydrophobicity does not affect the diffusion of non-polar hydrocarbons in the micropores, but drastically increases the mobility of water and polar molecules [3, 4]. But, unlike what happens with the determination of texture parameters, for which specific methods exist, the evaluation of hydrophobicity does not have a well-established methodology. Different methods have already been used to characterize this property. Several research studies have focused on determinations based on water adsorption and porosity measurements under static conditions [5]. The evaluation of the hydrophobicity of a material requires exact values. Weitkamp [6] has established a quantitative study of the hydrophobic/hydrophilic properties of materials by determining their hydrophobicity index. This was the most recently used method for this type of study [7, 8]. The monitoring of the evolution of hydrophobic/hydrophilic adsorptions is also diverse, there is mainly gravimetry [9], or by gas chromatography [10]. Our work aims to determine the hydrophobicity index by IR spectroscopy by adsorbing different molecules probes by developing a cell that can be installed on an IR device coupled to an adsorption assembly equipped with primary and secondary vacuum.

Keywords: adsorption, hydrophobicity, infrared spectroscopy

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SYNTHESIS OF CYCLIC ENAMINES CATALYZED BY POROUS MATERIALS

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Abstract

Enamines represent one of the most important classes of reaction intermediates in organic synthesis and construction of bioactive molecules such as alkaloids, terpenes, heterocycles and some drugs.

Various methods have been reported in the literature for their synthesis. The usual conditions to prepare enamines involve either strong Lewis acids (TiCl₄, AlCl₃, SnCl₄, etc.) or Bronsted acids (AcOH, PTSA) in refluxing solvent (benzene, toluene, or xylene).

In continuation of our ongoing program to develop environmentally benign synthetic protocols, our attention has been focused on the preparation of these products using heterogeneous catalysts that can easily be recovered and reused. Herein, we wish to report a simple synthetic procedure of cyclic enamines from cyclic ketones and secondary amines using micro and mesoporous materials as catalysts.

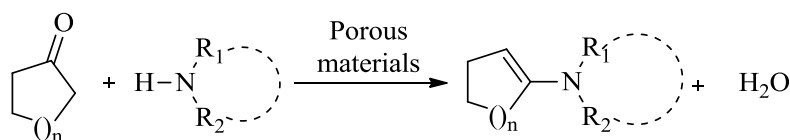


Figure 1. Synthesis of cyclic enamines catalyzed by micro and mesoporous materials

Our materials and enamines were characterized by different techniques : (DRX, BET, IR, ¹H and ¹³C NMR).

Keywords: Cyclic enamines, catalysis, porous materials.

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SYNTHESIS, CHARACTERIZATION AND REACTIVITY OF MESOPOROUS MATERIALS FE-TUD-1

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Abstract

With increasingly stringent environmental and economic regulations, the main challenge faced by all industrial sectors is to find processes that are more environmentally friendly and less resource intensive [1]. In particular, the petrochemical industry has focused on the search for naphthenic ring-opening reactions on noble metal catalysts to improve the cetane number of diesel fuels and reduce harmful emissions [2-4]. A practical solution is to find a "clean" and efficient catalyst to replace noble metal catalysts. Thus, the use of metal oxide catalysts can offer an alternative because of the environmental and economic advantages they provide, their good catalytic performance and their competitiveness with noble metals. In this context, the TUD-1 (Technische Universiteit Delft) mesoporous materials [5] appear to be good candidates for hydrocarbon conversion in view of their large internal surface, high thermal stability and Which create new opportunities for heterogeneous catalysis [6]. Therefore, in this study a research was conducted on Fe-TUD-1, to synthesize, characterize and test it in order to evaluate its catalytic capacity to convert the selected MCP as a model molecule.

Keywords: cetane number, decyclization, mesoporous materials.

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GREEN OXIDATION OF CYCLOHEXENE WITH HYDROGEN PEROXIDE OVER GOLD SUPPORTED ON CLAY

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Abstract

A series of monometallic catalysts which is based on gold supported on clay: the purified Bentonite (Na-B), pillared by aluminum (Al-B) and iron (Fe-B) is prepared by deposition-precipitation method, characterized by means of X-ray diffraction (XRD), S_{BET} , UV-vis diffuse reflectance (UV-vis-DR), FTIR and testing in the free cyclohexene oxydation reaction. The finding of characterization showed that the pillaring process of the natural Bentonite results in an increase in the porosity and surface area of the solid, the size and distribution of gold particles are not the same on the supports Na-B, Al-B and Fe-B. The catalytic test of Au /support reveals that the activity and selectivity of catalysts increase with decreasing particle size of gold.

Keywords: Gold nanoparticles, Bentonite, Cyclohexene.

REMOVAL OF TEXTILE DYES BY ADSORPTION ON ACTIVATED CARBON AND ADVANCED OXIDATION PROCESSES

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Abstract

Industrial waste resulting from textile activities of the tannery often have a significant coloring pollutants mass that is hardly to biodegrade in the environment. The decontamination by conventional techniques is sometimes ineffective in some cases [1,2]. In this work, we studied the elimination of synthetic dyes by two distinct processes. The study was conducted on dyes that are used by the tanning industry in Algeria. In the first part of this work, we are interested in studying the adsorption of the various dyes on activated carbon powder. A systematic study allowed us to evaluate the influence of certain parameters on the adsorption capacity of these dyes, such as the contact time, the mass of the adsorbent and the initial dye concentration. According to the results obtained, the adsorption is favorable for Carbon Derma and NT-Black. On the other hand, Derma 135%, Bunzun Derma and Vert Derma dyes showed no affinity for activated carbon. In the second part, we studied the elimination of these pollutants by an advanced oxidation method which is the Renton reagent. The results obtained suggest that these techniques may be of great interest in the field of treatment of liquid effluents.

Keywords: Textile dyes, wastewater, advanced oxidation processes

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COBALT ALUMINATE NANOPARTICLES SUPPORTED ON MIL-101 STRUCTURE: CATALYTIC PERFORMANCE INVESTIGATION

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Abstract

MOF's, especially MIL-101, are interesting materials for a wide range of catalysis applications including cyclo addition of epichlorohydrin and oxidation of alcohols to aldehydes. Recently, non-aqueous sol-gel routes have proven to be versatile for the synthesis of several highly crystalline and pure bimetal, metal oxide and multi-metal oxide nanoparticles (NPs) and hybrid materials which were previously impossible to synthesize. A novel, facile method based on a non-aqueous sol-gel solvothermal process has been developed to synthesize different size and shape NPs including CoPt_3 ,^[1] CoAl_2O_4 , TiO_2 , In_2O_3 , Fe_3O_4 , CoAl_2O_4 , SrHfO_3 , Ln_2O_3 ($\text{Ln} = \text{RE}$) in one pot at low temperature. They were showing interesting structural, catalytic, photocatalytic, optical, electric and magnetic properties. Detailed XRD and HR-TEM studies prove that the pure phases of different NPs and other oxides were formed with particle sizes in the range of between 1.9-8 nm.^[2] We are now making composites of CoAl_2O_4 @MOF's materials with these NPs to introduce catalytic activity. A remarkable catalytic performance was found for thioanisole oxidation under sustainable conditions and the catalytic application of the most active composite was extended to styrene oxidation.^[3] The recyclability and the stability of composites after catalytic use were investigated.

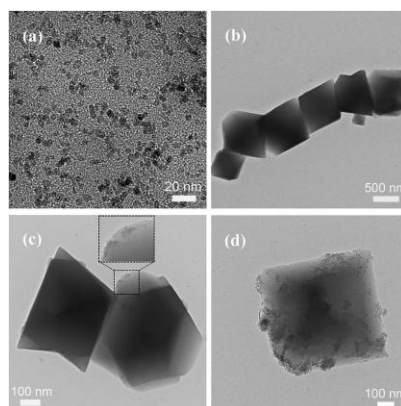


Figure 1. HRTEM images of (a) CoAl_2O_4 nanoparticles, (b) solid support MIL-101(Cr), (c) and (d) CoAl_2O_4 @MIL(Cr) composite material

Keywords: CoAl_2O_4 nanoparticles, MOF's, catalysis

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HETEROGENEOUS PHOTODEGRADATION OF METHYL ORANGE BY PHOTO-FENTON USING A NATURAL CLAY POWDER

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Abstract

This work aims to study and evaluate the efficiency of a photo-catalytic process photo-Fenton-type for the removal from aqueous medium of an azo dye (orange methyl). It was for objective to study (or to investigate) the photocatalytic activity of a local clay rich in iron oxide for the degradation of certain kind of organic compounds.

The first part of this work has been devoted to the acquisition of some local clay and the obtaining of their characteristic properties by a few characterization methods such as: thermal analysis (ATD and ATG), scanning electron microscope, X-ray diffraction, UV-visible spectroscopy and X-ray fluorescence.

The results indicated that the photodegradation rate of pollutants was clearly increased in the presence of clay compared with the direct photolysis. The introduction of oxalic acid considerably improved the photocatalytic process (about 90% removal). The optimal conditions for a better degradation were obtained for: [clay] = 1 g/L and acidic pH (between 3 and 4) in the presence of [oxalic acid] = 10^{-2} M.

Keywords: Advanced oxidation processes, heterogeneous Photo-Fenton, Photodegradation, Pollutants, Photocatalyst.

ELIMINATION OF HEAVY METALS FROM AQUEOUS SOLUTION USING NATURAL SORBENT (CHITIN AND CHITOSAN)

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Abstract:

With the rapid development and the growth of industry, water contamination has become major environmental problem. The control of water pollution has become of increasing importance in recent years. The aim of our study is to test the effectiveness of natural sorbent (chitin and chitosan) for the removal of heavy metals (cobalt, manganese and nickel) from water. The adsorption was studied using the batch technique. The effect of different parameters such as, contact time, adsorbate concentration, temperature and pH was investigated. The selectivity order of the two adsorbents is $Co > Ni > Mn$. From these results, it can be concluded that the chitin and chitosane could be good adsorbents for the metal ions from aqueous solution.

Keywords: Wastewater, heavy metals, adsorption.

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DEGRADATION OF AZO DYE IN AQUEOUS SOLUTION BY SEVERAL ADVANCED OXIDATION PROCESSES

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Abstract

Advanced oxidation processes (AOPs) utilizing Homogenous photocatalysis (Fenton and photo-Fenton reactions), and Heterogeneous photocatalyse (TiO₂ and ZnO) were investigated for the degradation of commercial azo dye "Orange G" wastewater. The degradation is estimated using chemical oxygen demand (COD) reduction and color reduction using spectrophotometric methods.

Fenton experimental conditions: Hydrogen peroxide concentration: (10⁻² M), Ferrous ions concentration (5.10⁻⁴ M), pH (2.8 – 3), UV lamp power (6 watt). Adding more ferrous ions enhanced the oxidation rate for the H₂O₂/Fe²⁺ and UV/H₂O₂/Fe²⁺ processes.

The optimum catalyst loading was found 2.0 g/L in our case for both catalysts TiO₂ and ZnO.

A comparative study of the photocatalytic degradation showed that these two catalysts have a comparable reactivity; it follows a pseudo-first-order kinetics

The degradation trends followed the order: UV₃₆₅/Fenton > UV₃₆₅/TiO₂ > Solar Fenton > Solar TiO₂ > Fenton ~UV₃₆₅/ZnO .

Among AOPs, processes using Fenton type reagent are relatively cheap and easy to operate and maintain. Moreover, UV₃₆₅/Fenton process has been shown as effective in the treatment of OG dye.

Dye was degraded following second-order kinetics. The rate constants was 0,041 .10⁺⁶ L.M⁻¹.min⁻¹.

The degradation was followed by spectrophotometric method, chemical oxygen demand (COD) measures and high performance liquid chromatography analyses (HPLC). Some aromatic and aliphatic degradation compounds were identified. Degradation of Orange G by UV Fenton mechanism was also proposed.

Keywords: AOPs, Homogenous catalysis, Heterogeneous catalysis, Orange G; hydroxyl radical

**PHENOL CATALYTIC OXIDATION BY NEW
Bi_{1.495}Sb_{1.415}Co_{0.44}Fe_{0.52}O₇ PYROCHLORE COMPOUND**

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Abstract

We report here the synthesis of new bismuth antimony cobalt iron pyrochlore oxide compound by solid state reaction method. The structural, morphological and compositional properties of Bi_{1.495}Sb_{1.415}Co_{0.44}Fe_{0.52}O₇ ceramic have been investigated by means of X-ray diffraction (XRD), scanning electron microscopy (SEM) and X-ray energy dispersion spectroscopy (EDAX). The catalytic activity of the material has been tested until the decomposition of a phenol. The X-ray powder diffraction results have showed that the new pyrochlore compound crystallizes in a cubic system with Fd-3m space group. The calculated lattice parameter by Dicvol Program is $a = 10,4330 \text{ \AA}$. Any other peaks had observed as impurities in the pyrochlore pattern. The SEM image was revealing large grains with quasi-spherical shape. The EDAX analysis had confirmed the presence of all elements into this new structure. The catalytic activity of as-prepared material has been followed by high phase liquid chromatography (HPLC). The new catalyst was highly active and reached the 46% for the oxidation of phenol to ketone acid 39% and carboxylic acid 7%.

Keywords: catalytic oxidation, phenol, New pyrochlore compound.

HYDROGEN STORAGE OF POROUS SILICON

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Abstract

Hydrogen presents itself as an interesting candidate because its combustion only generates water. However; there are two serious disadvantages to be solved: mass production and storage. Solid state storage is under study for forty years. Some materials may react reversibly with hydrogen and have interesting hydrogen densities. Porous silicon (PS) is a candidate that could be used as a hydrogen storage material. The structure of pores and defects lead to a large area (200-600 m²/cm³). In addition, porous silicon is obtained by different Methods such as electrochemical anodizing which allows obtaining thick layers of this material, which gives rise to our interest in choosing it to study and improve its properties and storage behavior[1].

In this study, we have prepared porous silicon (PS) layers by electrochemical anodization. The elaborated (PS) layers were characterized by Infrared Spectroscopy (FTIR), Contact Angle and Scanning Electron Microscopy (SEM). The electrochemical characterization and hydrogen storage were carried out in a three-electrode cell, using sulfuric acid 3M H₂SO₄ as electrolyte by cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and galvanostatic charge/discharge. The results indicate the presence of a single oxidation peak at 0.4 V on the anode side corresponding to hydrogen desorption and a reduction peak at -0.2 V corresponding to the adsorption of hydrogen. Moreover, the EIS studies show that the PS electrode behaves electrochemically better than silicon.

The highest hydrogen storage was 101 mAh / g. This storage capacity storage decreases by only 7% of the value of the initial capacity, after 40 cycles.

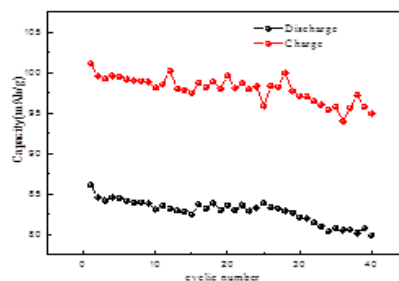


Figure 1 . Cycle life performances of PS sample

Keywords: Hydrogen storage, Porous silicon, electrochemical

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EFFECT OF DEPOSIT POTENTIAL OF CUXOY NANOCOMPOSITE ON PHOTOCATALYTIC DEGRADATION OF PHENOL

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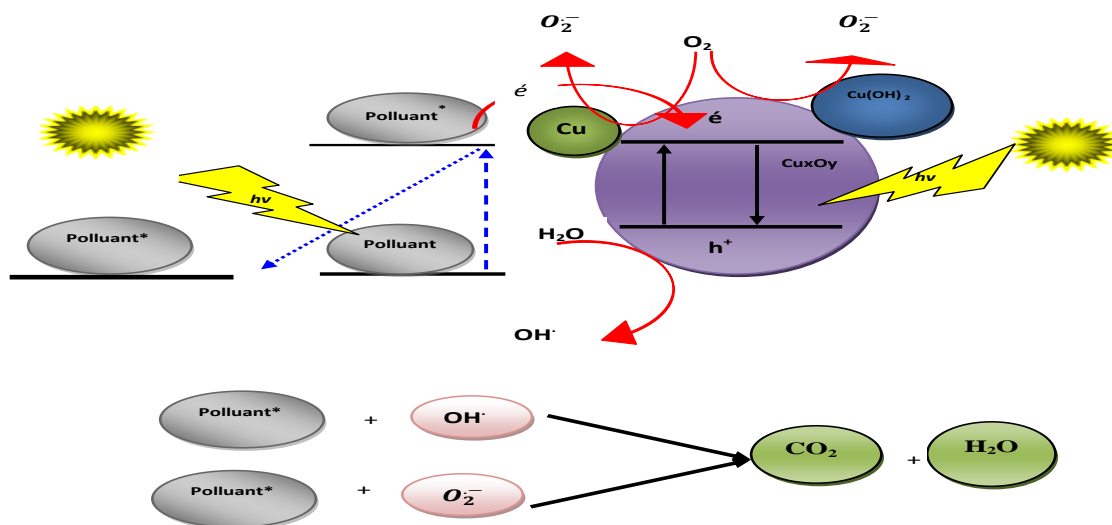
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Abstract

Phenol is one of the most refractory pollutants with high stability and solubility in water. It can be present in industrial and agricultural wastewater, which poses a significant environmental pollution problem. In general, these contaminants can be effectively decomposed by several advanced oxidation processes (POAs), such as microwave-assisted oxidation, electro-catalytic oxidation, photocatalytic oxidation [1]. Otherwise nanomaterials as photocatalysts are the subject of numerous works, mainly because of their high surface area. This work fits into this context. Indeed, photolysis of electrodeposited Cu_xO_y thin films immersed into an aqueous system containing phenol results in the degradation of the contaminant. The influence of the deposit potential of the nanocomposite is pictured out. This activity shows a degradation rate over than 85% during 105min in the presence of H_2O_2 as a precursor of OH^\cdot .



Scheme.1. Illustration mechanism of photocatalytic degradation of phenol

Keywords: Cu_xO_y , phenolic compound, depollution

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LA-PROMOTED TUNGSTATED ZIRCONIA CATALYST FOR N-BUTANE ISOMERIZATION

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Abstract

Promoted tungstated zirconia (WZ) catalysts are active and selective for isomerization of light alkanes, offering good prospects for industrial application. This account is an abbreviated summary of what these catalysts are and how they work. La-promoted Tungstated zirconia (LWZ) was prepared by a slurry impregnation method. The textural properties as well as the acidities of the La-promoted catalysts were characterized by, X-ray powder diffraction (XRD), N₂ adsorption, NH₃ temperature-programmed desorption (NH₃ TPD) and temperature-programmed reduction (TPR). The catalytic behavior of LWZ for n-butane Isomerization was studied in the presence of hydrogen at 300°C, WHSV=0.47h⁻¹ and nC₄/H₂=6. In comparison to Tungstated zirconia (WZ), the catalytic activity of the La-promoted catalyst was greatly improved.

Keywords: Tungstated zirconia, lanthanum, nbutan Isomerization.

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THE NANOSTRUCTURED γ -MnO₂ FILM ELECTROCHEMICALLY PREPARED FOR USED AS NEW PHOTOCATALYST FOR THE DEGRADATION OF AN ORGANIC POLLUTANT

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Abstract

This work focused on the synthesis of nanostructured MnO₂ for application as a new photocatalyst for the degradation of an organic pollutant (Rhodamine B dye). The nanostructured MnO₂ was successfully synthesized by electrodeposition process. The analysis of the result electrodeposited MnO₂ by different techniques: SEM, EDS, TEM, XRD and BET and UV-visible diffuse reflectance spectroscopy (UV-vis DRS) revealed the presence of nanostructured γ -MnO₂ with high specific surface area of 139.59 m².g⁻¹ with the particles size is less than 20 nm. The electrochemical study of the sample conducted by linear voltammetry, electrochemical impedance spectroscopy (EIS) measurements are showed that the performance of the synthesized nanostructured MnO₂ was higher than that of the commercial MnO₂. In addition, the diffuse reflectance measurements show that the electrodeposited nanostructured γ -MnO₂ film presents a direct band gap of about 1.41 eV. The Mott-schottky plot confirms the n-type semiconductor character of the γ -MnO₂ film, the potential value of the flat bande $V_{FB} = 0.016$ V vs. Ag/AgCl and the concentration of the electron donor charge carriers $ND = 0.8 \cdot 10^{20}$ cm⁻³. The conduction and valence energy band values were estimated at $E_c = 3.498$ eV and $E_v = 5.908$ eV, respectively. It was shown also that these films exhibit good ability for the degradation of Rhodamine B especially under visible light irradiation. Indeed, degradation rates of about 90% and 60% were obtained after 60 min of visible and UV light irradiation, respectively. Finally, the degradation process mechanism of RhB is discussed.

Keywords: MnO₂, nanostructures, photocatalysis, Pollutant, Rhodamine-B.

PHOTOCATALYTIC DEGRADATION OF DYES BY MESOPOROUS MgO UNDER SOLAR LIGHT

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Abstract

Nanomaterials have attracted much consideration owing to their unique properties making them suitable for various applications. Photocatalysis is considered one of the most effective methods for wastewater pollutants degradation particularly using nanosized metal oxides as catalysts.

Mesoporous MgO is an interesting catalyst due to its chemical inertness, optical transparency, high thermal stability and high surface area.

In this study, mesoporous MgO was synthesized by the nanocasting pathway using the mesoporous SBA-15 silica as structure template and magnesium nitrate as the MgO precursor via a solid-liquid route. This catalyst has been characterized by means: X-ray powder diffraction (XRD), BET surface area, diffuse reflectance ultra-violet visible spectroscopy (DR/UV-vis), Fourier-transform infrared spectroscopy (FTIR).

Photocatalytic activity of mesoporous MgO was evaluated both under UV and solar light irradiation for Congo red, methylene blue and methyl orange degradation as model pollutants. Performances of this new mesoporous catalyst were compared to commercial P25 TiO₂. Degradation of dyes was followed by UV-vis spectroscopy and mineralization of the organic pollutants by total organic carbon (TOC) measurements.

Keywords : Mesoporous magnesium oxide, Hard template, photocatalysis, Dyes

KINETIC STUDY OF THE SPIRAMYCIN REMOVAL BY SUPPORTED TiO₂/ACTIVATED CARBON

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Abstract

The aim of this work is to study the effectiveness of combined activated carbon adsorption to heterogeneous photocatalysis process for the removal of a macrolide antibiotic (spiramycin) in water. The experiments were carried out following a recirculation mode with supported TiO₂ catalyst/activated carbon adsorbent. The study of process on supported media required the design of a new tubular reactor, where the hydrodynamic study showed that the liquid flow nature is similar to that of at least 4 or 5 stirred reactors in series (equivalent to plug reactor).

The kinetics of degradation and influence of main process parameters, such as flow rate, initial substrate concentration and pH has been studied. Maximum degradation (over than 75%) was achieved within 300 min photoperiod under 0,3gL⁻¹ equivalent catalyst dosage, 2,4 mW.cm⁻² light intensity and 0,35 L.min⁻¹ flow rate. The kinetic constants by application of Langmuir-Hinshelwood model were determined.

Keywords: Photocatalysis, kinetics, spiramycin

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DEGRADATION OF CONGO-RED BY TI PILLARED CLAYS

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Abstract

To remedy the problem of pollution caused by organic dyes, a series of procedures known as Advanced Oxidation Processes (AOPs), based on the generation of hydroxyl ($\cdot\text{OH}$) and perhydroxyl ($\text{HO}_2\cdot$) free-radicals and the use of their high oxidative capacity, have been developed. In this work, we looked at three types of POAs that are, the heterogeneous Fenton process, the photocatalysis and a process combining the two so called photo-Fenton in order to have a synergistic effect. In our studies we chose to work in heterogeneous environment using titanium intercalated clay in order to degrade an azo dye that is Congo Red by the three heterogeneous processes Fenton, photocatalysis and photo-Fenton.

Table: Yields obtained with different catalysts

* 50 mL Congo-Red [10^{-4} M], 0.033 g catalyst, 0.025 mL H_2O_2 , 1h. ** 100 mL Congo-Red [10^{-4} M], 0.067 g catalyst, 0.05 mL H_2O_2 , 1h.

Catalysts	Fenton*	Photocatalysis**	Photo-Fenton**
5mmol Ti-PILK10	14	53	71
10 mmol Ti-PILK10	0	48	90
5mmol Ti-PILH-Mont	0	36	76
10mmol Ti-PILH-Mont	0	38	78
K10 (commercial clay)	10	39	86
H-Mont (natural acidified clay)	17	27	68

CATALYTIC OXIDATION OF AROMATICS COMPOUNDS OVER CU-CONTAINING LAYERED DOUDLE HYDROXIDES

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Abstract

Layered double hydroxides (LDH), $[M_{1-x}^{II}M_x^{III}(OH)_2]^{x+}[X^{n-}]_{x/n} \cdot mH_2O$, are known as hydrotalcite (HT)-like anionic clay materials [1]. The structure of these compounds are very similar to that of brucite, $Mg(OH)_2$, where some of Mg^{2+} represented as $[M^{(II)}]$ are isomorphously replaced by Al^{3+} represented as $[M^{(III)}]$ and the net positive charge is compensated by the inter-layered exchangeable anion (A^{n-}) [2].

These last years, the LDH are the subject of an interest growing for their properties of anion exchange [3], their properties magnetic and electrochemical, their use in heterogeneous catalysis [4] and their applications in the liquid waste processing by using them like adsorbents [5, 6] or catalysts of reaction of oxidation of aromatics compounds.

In this work, carbonated phases LDH of type $[M_{(2-x)}-Cu_{(x)}-Al-CO_3]$ with x from 0 to 2 and calcinated phases $[M_{(2-x)}-Cu_{(x)}-Al-T_{cal}]$ with x = 0; 1 and 2 and T_{cal} from 250 to 900°C were used like catalysts in the catalytic oxidation of the aromatics compounds "phenol and 2-naphtol" in the presence of hydrogen peroxide. The carbonated phases are obtained by co precipitation with constant pH [7] with a molar ratio $R=M^{2+}/M^{3+}$ equal to 2.

The elaborate "carbonated" and "calcinated" materials are characterized by XRD, SEM-EDS, ATD/TG and IRTF, before their implementation in the catalytic oxidation of aromatics compounds. Several studied parameters as the mass of catalyst, the concentration of oxidant H_2O_2 , the temperature of reaction, the pH of solution, and the time of contact. The oxidation products are identified by HPLC.

Keywords: LDH, aromatics compounds, Catalytic oxidation.

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EFFECT OF CALCINATION ON COORDINATION AND REACTIVITY OF MOLYBDENUM SUPPORTED BY SULFATED ZIRCONIA

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Abstract

Sol-gel method for the preparation of solids is an easy way to obtain homogenous mixed oxides with good arrangement of the constituents. However, it depends significantly on the preparation conditions. Our work consists on the preparation of aerogel sulfated zirconia doped with molybdenum in order to use it as a catalyst for the reaction of n-hexane isomerization. The scope of this work is the study of calcination temperature influence on the properties of solids and the stabilization of their active sites. The preparation of sulfated zirconia doped with molybdenum is the same as it is reported in the protocol of Raissi et al. [1]. It consists on the preparation of aerogels calcined at temperatures varying from 673 to 973K.

XRD patterns of all the solids are characterized by the development of ZrO₂ tetragonal only. The size of nanocrystallites is slightly affected by temperature. Nevertheless, the study of the reactivity of the obtained solids showed that increasing calcination temperature decreases catalytic performances even the presence of a crystalline favorable phase. In the case of our catalysts the coordination of the dopant metal seems to play a key role in the determination of the catalytic performances. We note that the solid calcined at 673 K, the lowest temperature, is the most stable and presents the best activity. In fact, the presence of a characteristic peak on UV-Visible spectra at 245 nm revealed that molybdenum is on tetrahedral coordination. However, the geometry of the metal changes to the octahedral one by increasing the calcination temperature which seems to affect the catalytic performances of the catalysts. A similar effect of the oxidation state and the coordination of the dopant metal was noted in the case of use of cobalt as dopant of sulfated zirconia [1].

Keywords: molybdenum IV octahedral, molybdenum IV tetrahedral, n-hexane isomerization.

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AN EFFICIENT MICROFLUIDIC REACTOR NANO-ENABLED FOR WATER PURIFICATION FROM VOLATILE ORGANIC COMPOUNDS (VOCs)

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Abstract

This study reports a fast and efficient chemical decontamination of water within a tree-branched centimetre-scale microfluidic reactor enabling Zinc oxide nanowires (ZnO NWs) acting as a photocatalytic nanomaterial (Figure 1). Direct growth of ZnO NWs within the microfluidic chamber brings this photocatalytic medium at the very close vicinity of the water flow path, hence minimizing the required interaction time to produce efficient purification performance. We demonstrate a degradation efficiency of 95% in less than 5 seconds of residence time in one pass only [1,2]. To conduct our experiments, we have chosen a case study addressing a health concern related to water contamination by Volatile Organic Compounds (VOCs); the contaminated water sample contains mixture of six pollutants Benzene, Toluene, Ethylbenzene and m-p-o Xylenes (BTEX) diluted in water at 10 ppm concentration of each. Degradation was analytically monitored in a selective manner until it falls below the maximum contaminant level (MCL) of 1 ppm established by the US Environmental Protection Agency (Figure 1).

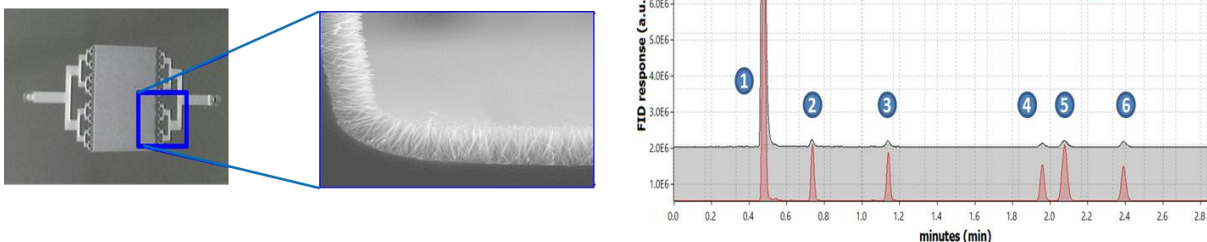


Figure 1. **Left:** Hierarchical micro-nano structuration combining microscale structures obtained by Deep Reactive Ion Etching of silicon and further growth of ZnO NWs using the static method, **Right:** Comparative analysis by gas chromatography of a water sample before and after a one-pass photocatalysis within the micro-reactor. A 10x reduction of the concentration for each of the 5 VOCs can be noticed after purification. (1) Solvent: methanol, (2) benzene, (3) toluene, (4) ethylbenzene, (5) m-p xylene, (6) o-xylene

Keywords: Zinc-oxide nanowires, Photocatalysis, BTEX, Water purification, Microreactor

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SYNTHESIS OF HIGHLY ORDERED NONMATERIAL FROM ALGERIAN DIATOMITE MCM-41 AND SBA-15 CATALYSTS FOR ESTERIFICATION OF LAURIC ACID

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Abstract

Mesoporous materials SBA-15 and MCM-41 were synthesized by a hydrothermal method from Algerian diatomite by using alkali fusion to extract silica and aluminum sources from Algerian diatomite [1]. The prepared materials were characterized by XRD, FTIR and N₂ adsorption-desorption. The mesoporous materials were applied for the biodiesel production by esterification of lauric acid with methanol (Fig.1). The reaction was carried out at 70°C with 5wt% of catalyst for 1h.

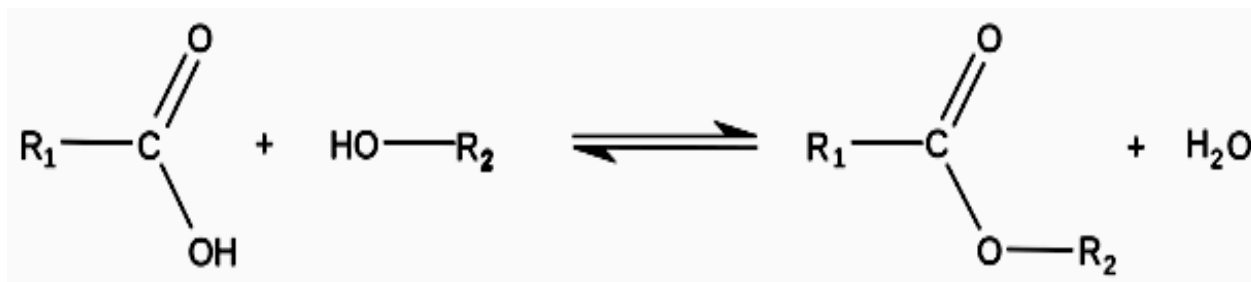


Figure 1. Esterification reaction.

Keywords: MCM-41; SBA-15, Esterification

References:

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THE USE OF ADVANCED OXIDATION PROCESSES FOR THE DEGRADATION OF DYES: CASE OF METHYL VIOLET 2B

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Abstract:

Methyl violet 2B (MV2B) is a basic cationic dye used in the fields of paints, textiles, food industry and cosmetics, and is also used as pH indicator. However, MV2B may be toxic to the environment and therefore to humans. The latter can cause severe skin and eyes irritation as well as respiratory and gastrointestinal tract damage. This work consists in studying the elimination of MV2B by the following advanced oxidation processes (POA): direct photolysis, homogeneous photocatalysis (photo-Fenton), heterogeneous photocatalysis, and sonolysis. The removal of MV2B was followed by UV/Vis spectrophotometry and the homogeneous photocatalysis process (photo-Fenton) presents the best result with a degradation rate of 100%.

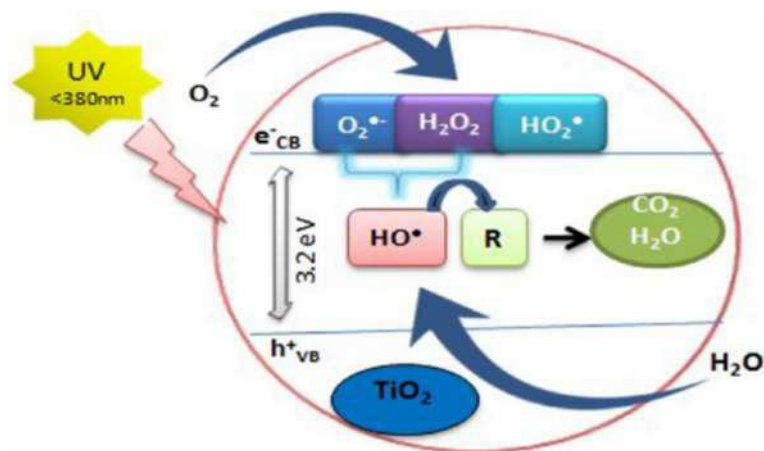


Figure 1: Photocatalysis Oxidation Mechanism of TiO_2 [1]

Keywords: Violet methyl 2B, degradation, photocatalysis.

References:

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CaTiO₃-TiO₂ NANOCOMPOSITE PRODUCED BY CLASSICAL AND MICROWAVE HYDROTHERMAL METHOD

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Abstract

In this work TiO₂ nanotubes were prepared by anodization of pure titanium sheets that were subsequently covered with CaTiO₃ using microwaves hydrothermal method. The TiO₂ nanotubes were first prepared using 2wt. % HF in water under constant voltage (20 V) for 5 hours. Thereafter, the prepared samples were hydrothermally processed in a solution of 0.007 M Ca(OH)₂ in a microwave oven. Different techniques of analysis such as X-ray Diffraction (XRD), X-ray Photoelectron Spectroscopy (XPS), Fourier Transform Infrared Spectroscopy (FTIR), Electron Probe Microanalyses (EPMA) and Scanning Electron Microscopy (SEM) were used to characterize the obtained layers. The XRD analysis shows the formation of TiO₂ and CaTiO₃ composite after hydrothermal process. The formation of CaTiO₃ was also confirmed by XPS and FTIR analysis. Optical image analysis of these layers showed that TiO₂ nanotubes were almost entirely covered with Ca nano-particles of approximately 100 to 200 nm in diameter. The EPMA analysis show that the titanium dioxide (anatase and rutile) present the main phase, while the calcium component present a very small amount.

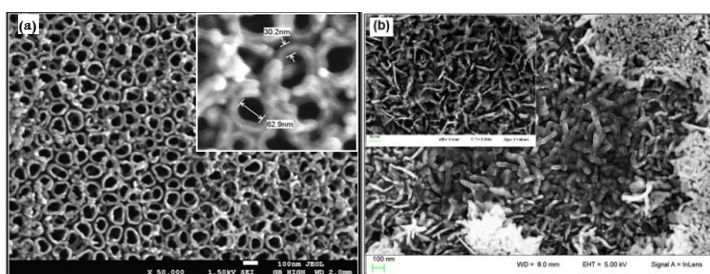


Figure 1. SEM images of the top view of TiO₂ nanotubes array formed at 20 V for 5 h in 2 wt% HF acid electrolyte (a) and CaTiO₃ nano-composite prepared by hydrothermal method at 200°C for 2 hours in 0.007 M Ca(OH)₂ solution (b).

Keywords: TiO₂ nanotubes, hydrothermal method, CaTiO₃-TiO₂ nanocomposite.

NATURE OF HEAT TREATMENT EFFECT ON NANO-GOLD SUPPORTED CATALYSTS IN THE SELECTIVE OXIDATION OF ORGANIC MOLECULES – SOLVENT INFLUENCE

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Abstract

Gold, which is a noble metal, has long been regarded as catalytically inactive. In 1987 the group of Dr Haruta discovered the catalytic properties quite remarkable of gold nano-particles at low temperature [1].

In our work, gold particles of nanometer size, catalytically active, are deposited on Fe and Al pillared montmorillonite - Na⁺, by deposition - precipitation. The objective is to study the Influence of the nature of heat treatment on these catalysts and consequently on their activity and selectivity in the oxidation of cyclohexane [2].

The characterization by XRD shows the expansion of the interlamellar distance of clay after intercalation by Fe or Al. Characterization by UV-Vis shows that most gold particles are smaller; the maximum absorbance band is shifted to smaller wavelengths [3].

The catalytic activity of these materials was tested in the oxidation reaction of cyclohexane by tert-butyl hydro-peroxide. Several parameters have been studied: the temperature and nature of heat treatment (H₂, O₂ or H₂ + O₂) of the catalysts and also the solvent effect.

The materials studied in this work lead to the selective production of cyclohexanone, compared to cyclohexanol: the catalysts reduced under H₂, which seems to have the smallest particles, leads to the highest cyclohexanone production. A yield of 38% has been observed with the catalysts reduced at 200°C compared to those reduced at 500°C (18%).

Keywords: Nano-gold, plasmon resonance, oxidation

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A DFT STUDY OF THE PROTOLYTIC CATALYTIC CRACKING REACTION OF *n*-PENTANE ON ALUMINOSILICATE ZEOLITE

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Abstract

Hydrocarbon conversion processes are essential for the modern oil and chemical industries. In these processes, zeolites are extensively used as catalysts. The H-ZSM-5 zeolite is broadly used in the petrochemical industry for catalytic cracking of hydrocarbons because of its interesting catalytic properties, including high acid strength. Rapid progresses in the development of computer technology promote the application of quantum chemistry to chemical processes for investigating large systems, including hydrocarbon reactions catalyzed by zeolites.

In this work, a theoretical study is realised on the protolytic cracking reaction of *n*-pentane on zeolite. The aluminosilicate zeolite H-ZSM5 (T3) model cluster is applied to investigate mechanism, kinetic and thermodynamic properties regarding this reaction.

All calculations are performed with Gaussian 09 software package. The reactants, transition states and products were optimized by DFT-B3LYP/6-31G(d) method and the accurate energies are calculated with complete basis set composite method CBS-QB3.



Keywords: Protolytic cracking reaction, zeolite, DFT calculations keywords

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IMPROVEMENT CATALYTIC PROPERTIES OF BaCuO₂ DELAFOSSITE IN THE GAS PHASE HYDROGENATION OF BENZALDEHYDE . SYNERGY WITH PARTIAL SUBSTITUTION OF Cu by Ni,Co AND Fe

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Abstract

Hydrogenations of aromatic aldehydes to aromatic alcohols are important processes in synthesis of pesticides, flavouring, cosmetics and pharmaceuticals industries. These heterogeneous catalytic hydrogenation processes are conducted usually in vapor or liquid phase. The vapor phase hydrogenation reactions are more industrially favorable, because they have higher conversions and ease in product separation compared to liquid phase reactions [18].

Mixed oxide delafossite-type of the form BaCuO₂ and BaCu₁M_{1-x}O₂ (M=Ni, Fe and Co) were successfully synthesized following sol-gel citrate method. The products obtained after heat treatment under N₂ at 800°C, were characterized by several techniques such as: Thermogravimetric analysis (TG), inductively coupled plasma atomic emission spectroscopy (ICP-AES), X-ray diffraction (XRD), Brunauer-Emmett-Teller method (BET), Fourier transform infrared spectroscopy (FTIR) and Temperature programmed reduction (TPR). Catalytic performance of the catalysts was evaluated in the vapour phase catalytic hydrogenation of benzaldehyde. Benzyl alcohol and toluene were obtained through consecutive hydrogenation/ hydrogenolysis reactions while benzene was formed by a parallel hydrogenolysis reaction. BaCu_{0.47}Ni_{0.51}O₂ catalyst showed better activity at 250 °C with 98% benzaldehyde conversion. Under the same conditions, the BaCu_{0.52}Fe_{0.48}O₂ catalyst showed the highest selectivity for benzyl alcohol (84%).

Keywords: mixed oxide; Hydrogenation; Benzyl alcohol

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TEXTILE DYES REMOVAL FROM AQUEOUS SOLUTION USING KAOLIN-BASED ZEOLITE A

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Abstract:

The discharge of dye into the receiving water constitutes big problem of water pollution, Dyes are important pollutants, causing environmental and health problems to human being and ecosystem because they have toxic, mutagenic and/or carcinogenic effect.

Removal of dyes from wastewater can be realized by several techniques such as precipitation, flocculation, adsorption, ions exchange, and membrane separation. Each of the above processes has their own benefits and limitations. Adsorption is one of the most promising techniques in dyeing wastewater treatment.

Zeolite A was synthesized by hydrothermal transformation of natural Algerian kaolin by mixing 4g of metakaolin with 3M NaOH solution and gently stirred for 10 min at room temperature and then crystallized in Teflon bottle at 100 °C for 20 h; the synthesized product was washed with distilled water three times and then dried at 80 °C for 24 h, result microporeux was confirmed by XRD and FT-IR spectroscopy

The present work shows a limited color removal from textile dyes solutions using synthetic zeolite A with high degrees of purity and structural order, our raw material was DD3 Kaolin waste from the Guelma regions in the Algerian east that was successfully valorized for the synthesis of zeolitic material that have a high economic value.

Keywords: Adsorption, Dye, Clay, Synthetic zeolite

KINETIC STUDY OF MOLYBDENUM BASED KEGGIN TYPE CATALYST IN THE CYCLOHEXANONE OXIDATION

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Abstract

Mono- and dicarboxylic acids are part of the most important building blocks for the synthesis of compounds that find their use in a wide variety of applications such as fibers, pharmaceuticals, cosmetics or adhesives. Adipic acid (AA), an aliphatic diacid, is one of the foremost and crucial elementary chemicals for various industrial processes [1, 2]. In the current chemical industry, adipic acid is obtained from oxidation of a mixture of cyclohexanone and cyclohexanol (KA oil) by nitric acid [3, 4]. The chief disadvantage of this method is generation of large amount of nitrous oxide, a well-known greenhouse gas responsible for global warming.

The aim of this project is to develop a new system based on the use of Mo-based polyoxometalates for a sustainable catalytic liquid phase oxidation of cyclohexanone to adipic acid, alternative to the conventional process using aqueous hydrogen peroxide 30% as oxidant, in free solvent. Keggin-type ammonium salts namely $\text{NH}_4\text{APMo}_{12}\text{O}_{40}$ (A^{n+} : Sn^{2+} , Sb^{3+} or Bi^{3+}) were successfully synthesized. All compounds were characterized by IR, XRD, ICP, BET, TGA-DTA and ^{31}P NMR. In this study, kinetics over time have been done to follow the evolution of the reaction products formation, and we considered the effect(s) of oxidant/substrate molar ratio on the catalytic response.

The proposed green process is eco-friendly and pure adipic acid is produced in high yield (57 %) with $\text{NH}_4\text{SnPMo}_{12}\text{O}_{40}$ as catalyst. Identification of functional group of AA was characterized by using FTIR and ^1H NMR spectrophotometer. Glutaric and succinic acid, ϵ -caprolactone and 1, 3 butanediol its the main secondary products of the reaction.

Keywords: cyclohexanone, Keggin, adipic acid

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VALORIZATION OF MOLECULAR SIEVE'S END-OF-LIFETIME IN THE REMOVAL OF PHENAZIN DYE IN THE SIMULATED WASTE WATER

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Abstract

Frames molecular sieve-based dehydration technology is widely used for the simultaneous removal of water and hydrocarbons from both gas and liquid feed streams in order to achieve clean gas streams ready for gas compression or even liquefaction (LNG/LPG). Molecular sieve's end-of-lifetime compounds from **LNG2 Liquefaction Plant** of Arzew, Algeria (denoted as WMS) was modified by a two-step procedure. In the first step, waste molecular sieve (WMS) was heat treated at 450°C (denoted as WMS-450) and in the second step, this material was subsequently used for impregnation with sodium salt (denoted as WMS-450-Na). The bare molecular sieve (denoted as MS) was employed for comparison raison. The resulting materials were systematically characterized by X-ray fluorescence, TG-TD, XRD and FTIR technique. The potential ability of these material to remove phenazin dye (Neutral red; NR), as probe pollutant, in the simulated waste water was investigated at various experimental key parameters such as reaction time and temperature of system, using UV-visible spectrophotometers. Results revealed that all molecular sieves were belonging to the LTA-4A zeolite-type. The use of heating in combination with a wetness impregnation technique significantly enhanced the removal efficiency of the catalyst. Among the four types of molecular sieves in this study (MS, WMS-450, WMS-Na and WMS-450-Na), WMS-450-Na yielded the highest sorption (62.5 mg/g) of NR within 60 min at 25°C and pH 7. The sorption reaction of NR was perfectly described by pseudo-second-order kinetic model. The experimental data fit well to Langmuir isotherm. In addition, the thermodynamic parameters suggest that the sorption process was endothermic in nature. The numerical value of the mean free energy $E_a = 9.7$ kJ/mol indicated physical sorption. The work enables the development of valorization strategies for molecular sieve wastes and reduces their environmental impact.

Keywords: Molecular sieve, removal efficiency, phenazin dye

Topic 2

SPECIALTY CHEMICALS

Oral Communications

ENANTIOSELECTIVE ORGANOCATALYTIC CONJUGATE ADDITION OF NUCLEOPHILIC IMINES TO α,β -UNSATURATED ALDEHYDES

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Abstract:

The field of organocatalysis is a rapidly progressing area and a large number of new asymmetric reactions have been developed [1]. Since the reappearance of L-proline at the forefront of organocatalysis, tremendous efforts have been made to devise new catalysts based on a proline core structure [2]. Proline can react as a nucleophile with carbonyl groups or Michael acceptors to form iminium ions or enamines.

The Michael addition to α, β -unsaturated systems is an important carbon-carbon bond-forming reaction in organic synthesis.

In this work, an organocatalytic highly enantioselective conjugated addition reaction of nucleophilic imines to α,β -unsaturated aldehydes is presented; where the catalyst activates the substrate through the iminium-ion mechanism, thereby facilitating the addition of the nucleophile to the β -carbon atom.

A screening of catalysts, solvents, concentrations, temperature and reaction time was performed to optimize the reaction conditions. With the optimized conditions in hand, the scope of the reaction for different α,β -unsaturated aldehydes was investigated where the reaction tolerates different lengths in the alkyl chain with very high enantioselectivities.

Final and intermediate products were characterized by different methods: ^1H and ^{13}C NMR; HRMS; optical rotations.

Keywords: Asymmetric synthesis, organocatalysis, Michael addition reaction.

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A NEW STRATEGY FOR THE SYNTHESIS OF NOVEL CHROMENO[2,3-*d*]PYRIMIDINE DERIVATIVES

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Abstract

5*H*-chromeno[2,3-*d*]pyrimidines constitute an important class of heterocyclic compounds having diverse biological activities such as antifungal¹, anti-tumor², antibacterial³, antihypertensive⁴ and antiallergic activities⁵. 5*H*-chromeno[2,3-*d*]pyrimidines structure are constituted from two rings, 4*H*-Chromene and pyrimidine (Fig 1).

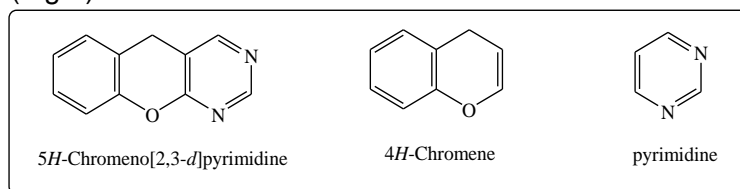


Figure 1. Structure of 5*H*-chromeno[2,3-*d*]pyrimidines

In the continuation of our research aimed at developing new methods for the preparation of fused chromenopyrimidinederivatives and in view of the important biological properties of the chromenes and pyrimidines, we have developed, in this work a simpler, effective and more universal synthesis to prepare this type of heterocycles “5*H*-chromeno[2,3-*d*]pyrimidines”, while trying to respect the criteria of the green chemistry, in which we employed, as a key step, the synthesis of 2-amino-3-cyano-4*H*-chromenes under microwave irradiation and solvent-free conditions.

Keywords: chromeno[2,3-*d*]pyrimidines, solvent-free, microwave irradiation.

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PHENYLBORONIC ACID CATALYZED FACILE ONE-POT SYNTHESIS OF 1-AMIDOALKYL-2-NAPHTHOL

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Abstract

Amidoalkyl Naphthols as synthetic intermediates play an important role in medicinal chemistry [1]. They are an important synthetic building blocks [2] and used as precursors for the synthesis of many important derivatives which have attracted strong interest to their potentially numerous biological and pharmacological activities [1] including antirheumatic, antibiotic, antitumor, antipsychotic, antimalarial, antianginal, analgesic, anticonvulsant, antihypertensive and antibacterial properties.

The preparation of amidoalkyl naphthols can be carried out by multicomponent condensation of aldehydes, 2-naphthol and amide or urea in the presence of Lewis or Brønsted acid catalysts such as chlorosulfonic acid [3]. Heterogeneous catalysts like cation-exchange resins [4], and the use of an additional microwave oven [5] or ultrasonic irradiation [6].

In this work we report a simple and efficient green protocol for one-pot preparation of novel Amidoalkyl Naphthols derivatives, using ecofriendly and green catalyst by a three-component condensation reaction of β naphthol, aryl aldehyde, amide under thermal solvent was described.

The operational simplicity of the procedure, shorter reaction times, simple workup, environmental friendliness, good to excellent yield and costeffective recovery are also attractive.

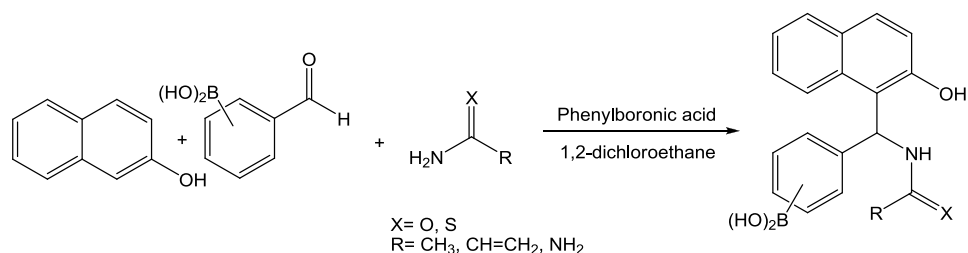


Figure 1.

Keywords: Betti base, Amidoalkyl naphthol, multicomponent reaction.

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LIPASE-CATALYSED DIRECT C–P BOND FORMATION IN ONE POT SYNTHESIS VIA KABACHNIK-FIELDS REACTION

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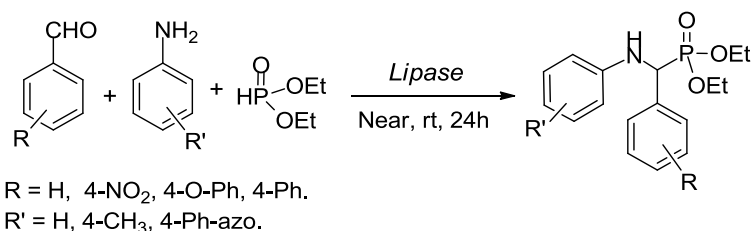
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Abstract

This study extends the applicability of lipase in organic synthesis by catalytic promiscuity. Enzymatic promiscuity is the ability of an enzyme active site to catalyze several different chemical transformations [1]. Especially, lipases are the most used enzymes in synthetic organic chemistry due to its broad specificity and excellent stability in various conditions. Recently, catalytic promiscuity of lipases has attracted considerable attention for the C-C bond formation [2]. The α -aminophosphonates are considered as analogues of amino acids have been found a wide range of applications in chemistry and biological [3].

In this work and in the continuation of our previous work for the preparation of α -aminophosphonates derivatives according to the Kabachnik-Fields reaction [4], we prepared these molecules type by catalytic promiscuity using immobilized *Candida Antarctica* lipase-B in solvent-free conditions and at room temperature [5]. This novel, efficient and eco-friendly method proceeds the new α -aminophosphonates synthesized from good to excellent yields. The immobilized lipase is reusable several times. All the synthesized compounds are characterized by NMR ¹H, ¹³C, ³¹P and HRMS spectra.



Key-Words: α -aminophosphonates, catalytic promiscuity, Kabachnik-Fields reaction.

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SYNTHESIS OF 1,10-DICHLOROBENZO[C]CINNOLINE, AND ITS N-OXIDE WITH SOME STRAINED MOLECULES RELATED TO BIPHENYLENE

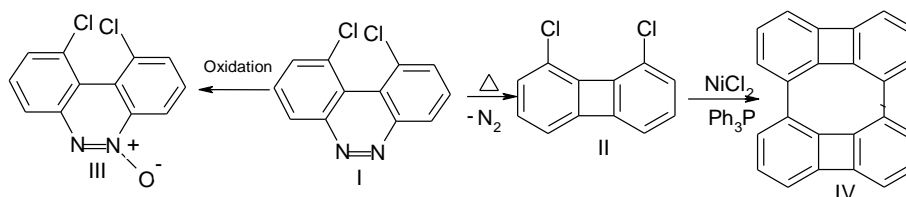
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Abstract

The 1, 10- dichlorobenzo[c]cinnoline ,was synthesised in three steps, from the commercially available 1,2- dichloro-3-nitrobenzene, the oxidation of (I) with hydrogen peroxide in acetic acid gave the 1, 10- dichlorobenzo [c] cinnoline –5- oxide (II). Vacuum pyrolysis of the cinnoline (II) at 800 °C, gave pure 1,8- dichlorobiphenylene (III) in high yield, and the latter hydrocarbon (5) is an intermediate molecule in order to synthesise some polycyclic aromatic hydrocarbons related to biphenylene such as cyclo-octatetraene derivative (IV).



Keywords: Dichlorobenzo[c]cinnoline, biphenylene, cyclooctatetraene derivative.

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VISIBLE LIGHT-MEDIATED SYNTHESIS OF ORGANOPHOSPHORUS COMPOUNDS: SYNTHETIC AND MECHANISTIC INVESTIGATIONS

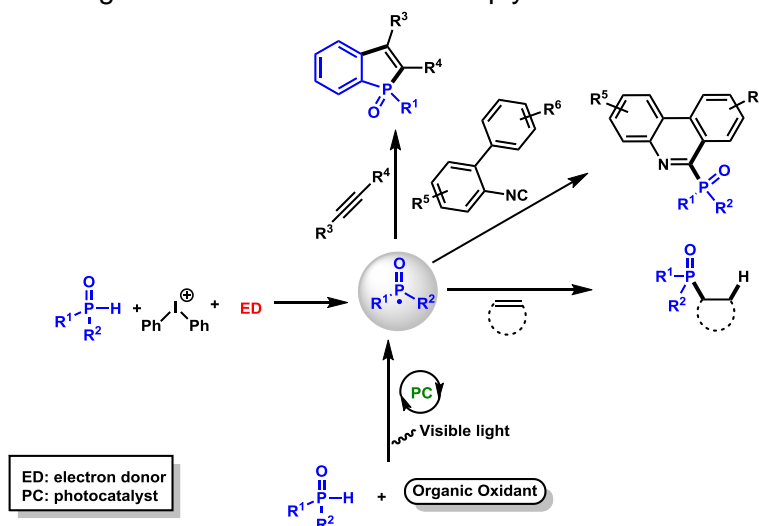
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Abstract

Reactions involving phosphorus centered radicals play a pivotal role in the construction of organophosphorus ligands, biologically active molecules and π -conjugated molecules.¹ Common synthetic approaches for the generation of such radicals imply the use of harsh reaction conditions.



In this communication, we show that access to a large variety of organophosphorus compounds can be achieved under mild reaction conditions through: *i*) visible light photoredox catalysis or *ii*) visible light irradiation of Electron–Donor–Acceptor complexes (EDA). The scope and limitations of these methods will be discussed with a special focus on their mechanistic aspects.²

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ADVANCED SYNTHESIS OF PHARMACEUTICALS CATENA VIA MACROCYCLIC CYCLONES

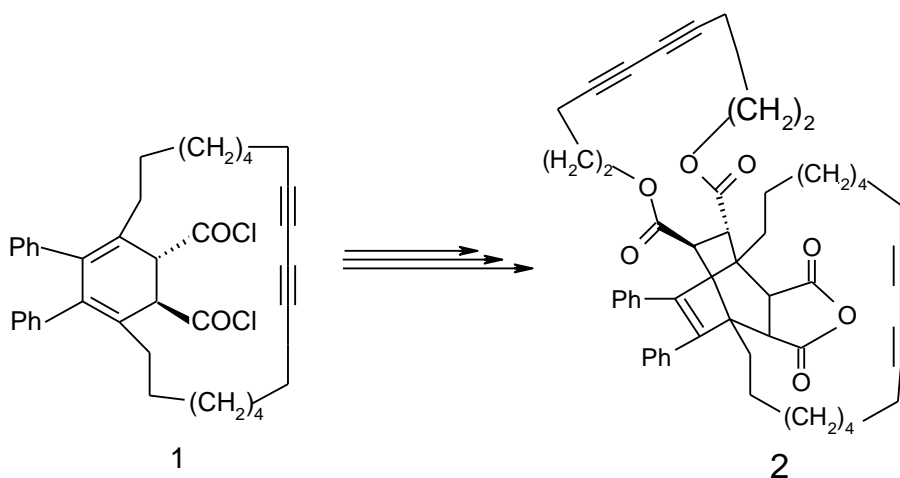
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Abstract

Diels-Alder cycloaddition of macrocyclic cyclopentadienone to fumaryl chloride to give carbonyl bridged product, than followed by decarbonylation to give (1) which was reacted with Maleik anhydride to produce the trans anhydride adduct attachment of the medium chain of acetylenic alcohol to the trans anhydride adduct followed in many steps to obtain the required compound (2) in successful yield, as shown below.



Keywords: Cycloaddition, Carbonylbridged, Catena.

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CANDIDA ANTARCTICA LIPASE-B CATALYZED ALKALINE-HYDROLYSIS OF A SET OF ACETATES IN NON AQUEOUS MEDIA

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Abstract

Lipases (EC.3.1.1.3) are an efficient biotools widely used for their remarkable *chemo*-, *regio*- and *enantio*-selectivity, especially, in kinetic resolution of racemates. They offer access to a large panel of enantiopure building blocks, such as secondary alcohols, commonly used as synthetic intermediates in pharmaceutical and agrochemical industries. ^[1] Lipases are highly active and stable in water and organic solvents, they are able to catalyze both transesterification of arylalkylcarbinols and hydrolysis of their corresponding acetates. ^[2] The use of enzymatic hydrolysis in aqueous media remains limited because of two main disadvantages, the first reside on the possibility of perturbation on the lipase's enantioselectivity due to the co-solvent nature, and the second, is the difficulty of controlling the pH value of the aqueous solution overcome the hydrolysis procedure.

In this presentation, we expose a practical methodology for the preparation of a series of various optically enriched acetates using a *candida antarctica* lipase B-catalyzed hydrolysis in organic media in the presence of alkaline carbonate salts. ^[3] The influence of several parameters which can intervene on the enzymatic efficiency such as the impact of the introduction of the carbonates salts, its amount and the nature of the alkaline earth metal are discussed. The obtained results show that the use of sodium carbonate with *CAL-B* in hydrolysis reaction enhances drastically both reactivity and selectivity of this immobilized lipase. In all cases, the resulting alcohols and remaining acetates are obtained in high ee values (up to >99 %) and optimal selectivities ($E > 500$).

Keywords: Alkaline-Hydrolysis, Enzymatic kinetic resolution, Non-aqueous media.

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A MILD AND EFFICIENT CATALYST USED FOR THE SYNTHESIS OF 5-ARYLIDINE BARBITURIC ACID DERIVATIVES

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Abstract

Barbituric acid is an organic compound based on a pyrimidine heterocyclic structure. It was synthesized in 1864 by a German chemist: Adolf von Baeyer (1835-1975) [1]. It's used as a precursor in the synthesis of several compounds that called "barbiturates". These compounds have special place in pharmaceutical chemistry, especially as a central nervous system depressants, hypnotic and local anesthetic drugs [2], they have been also found useful in anti-tumor and anti-cancer treatments [3]

Barbituric acid can be involved in many reactions. With an active methylene group at position 5, it can be involved in Knoevenagel type condensation reaction.

Different synthetic protocols have been reported for the synthesis of 5-arylidine barbituric acid derivatives, by using: Ni nanoparticles [4], microwave irradiation [5], ionic liquid mediated condensation [6], and a variety of catalysts such as $\text{NH}_2\text{SO}_3\text{H}$ [7]. However, these methods are suffering of the longer reaction time, the lower yields, and the employ of large amounts of hazardous and toxic catalysts. The choice of using natural or mild catalysts in a reaction is becoming more and more required, due to its environmental impact and cost of chemicals.

For our part, we have synthesized with good yields a variety of barbituric acid derivatives (Barbitones) that results from a Knoevenagel condensation of aromatic aldehydes with barbituric acid. We have used as catalyst in this condensation the pyridinium p-toluene sulfonate. (**Figure.1**)

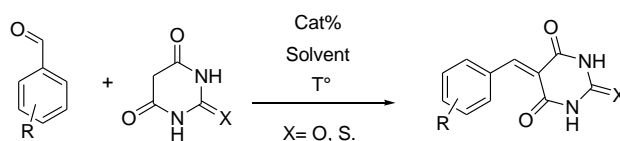


Figure 1. Synthesis Of 5-Arylidine Barbituric Acid Derivatives.

Keywords: Barbituric acid, Arylidine barbituric acid, pyridinium p-toluene sulfonate.

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CATECHOLASE ACTIVITY OF IN SITU TRANSITION METALS COMPLEXES OF A THIOSEMICARBAZIDE DERIVATIVE LIGAND

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Abstract

((E)-2-[(E)-(4-hydroxynaphthalen-1-yl)methylidene]hydrazin-1-yl)(methylsulfanyl)(methylidene)azanium hydrogen sulfate L1, has been synthesized and characterized by single-crystal X-ray diffraction, FT-IR. *In situ* catecholase activity towards 3,5-Di-tert-butylcatechol (3,5-DTBC) was investigated with a series of transition metals, Cu(II), Zn(II), Co(II) and Fe(III) under ambient condition in the presence of atmospheric oxygen. The progress of the oxidation reactions was closely monitored over time following the strong peak of 3,5-DTBC using UV-Vis. Oxidation rates were determined from the initial slope of absorbance vs. time plots, then analyzed by Michaelis-Menten equations¹. The results show that all complexes were able to catalyze the oxidation of 3,5-DTBC, however we distinguish a difference in the catalytic models between the *in situ* complexes, some were observed to follow the simple Michaelis-Menten model while others follow that of inhibition by excess of substrate. The results suggest that the rates and kinetic models of the oxidation reactions were strongly influenced by the nature of metal ion².

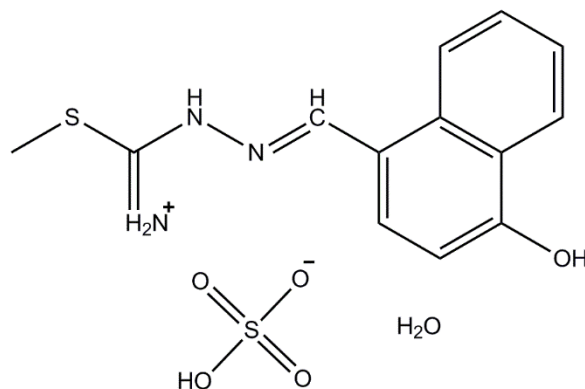


Figure 1. Structure of ligand L1

Keywords: Catechol, Oxidation, Catalysis

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Poster Communications

FORCED DEGRADATION STUDY OF AN ANTIHISTAMINE SYRUP BY HPLC

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Abstract:

Antihistamine syrup manufactured by SAIDAL was exposed to a forced degradation study under different stress conditions: acid hydrolysis, base, oxidation, heat and light in order to check its stability. The doxylamine succinate, sodium benzoate and methyl parahydroxybenzoate were separated by high performance liquid chromatography using a reverse phase Kinetex C₈ column and a mobile phase of 0.05M phosphate buffer (pH=3.0) / Water / Methanol / Acetonitrile. The detection was carried out at 250 nm with a flow rate of 1.0 ml/min and an injection volume of 20µl.

The analytical procedure used is considered as indicating stability and the loss in mass of active ingredient and preservatives not exceeding 0.2% allowed us to conclude that the syrup remained stable under stress conditions.

Key Word : HPLC, Forced degradation, stress, antihistamine

REMOVAL OF PHENOL FROM AQUEAS SOLUTION WIYH ADSORPTION MUDDY SEDIMENTS: EQUILIBRIUM, KINETIC AND THERMODYNAMIC STUDY

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Abstract

Muddy sediment from a dam Algerians was used as adsorbent of phenol, to test their ability to fix the organic pollutants. The adsorbent material is characterized by X-ray diffraction (PANalytical PW-3710). The results show that the mud contains impurities and is made in large proportion of halloysite-7A. The chemical composition of the mud is determined, it shows that the mud contains largely SiO₂ (39.64%) and Al₂O₃ (14.39 %).

The widely used in the industry phenol has a chemical structure containing functions that may interact with the surface of the solid adsorbent.

The adsorption of phenol on the mud dam was studied in batch system in an aqueous medium. The study is followed by the influence of the experimental parameters such as: the mass of adsorbent, contact time adsorbent / adsorbate, the medium temperature, pH and the initial concentration of adsorbate. The adsorption isotherms of phenol by the mud are described satisfactorily by the Langmuir model. Experimental results show that the adsorption kinetics is very fast; equilibrium was attained after contact of 1 h only. It follows the kinetic model pseudo second order. Adsorption is favorable at room temperature and at an acid pH. Thermodynamic parameters including the Gibbs free energy (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) were also calculated. These parameters indicated that adsorption of phenol onto mud was feasible, spontaneous and exothermic.

Keywords: sediment, phenol, adsorption.

A NEW 1,2,3-TRIAZOLE-BASED LIGAND CONTAINING AN ARYLSULFONAMIDE : SYNTHESIS, CRYSTAL STRUCTURE AND COMPUTATIONAL STUDIES

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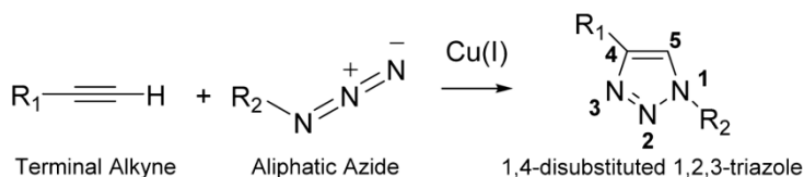
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Abstract

Recently, 1,2,3-triazole containing ligands have been extensively studied for various applications because of their excellent ability to form a stable complexes, and their easy synthesis through click chemistry.^[1] Among click reactions, the CuAAC, is found to be an excellent synthetic approach to generate a large number of this important class of heterocyclic compounds (Scheme. 1).^[2]



Scheme 1. *Copper-catalyzed Azide-Alkyne cycloadditions (CuAAC)*

In this context, we focused in click chemistry as a synthesis strategy to design a new 2-pyridyl-1,2,3-triazole ligand containing arylsulfonamide arm. This organic ligand was synthesized starting from commercial 2-ethynylpyridine and azido compound. Characterization was accomplished by the spectroscopic methods and crystallographic study. Density functional theory (DFT) and time-dependent density functional theory (TDDFT) calculations were performed to explore and explain the electronic structures and bonding properties, as well as to understand the electronic transitions character in the click ligand.^[3]

Keywords: *Click chemistry, triazole, DFT calculations*

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ORTHO-LITHIUM/MAGNESIUM CARBOXYLATE-DRIVEN AROMATIC NUCLEOPHILIC SUBSTITUTION REACTIONS ON UNPROTECTED NAPHTHOIC ACIDS

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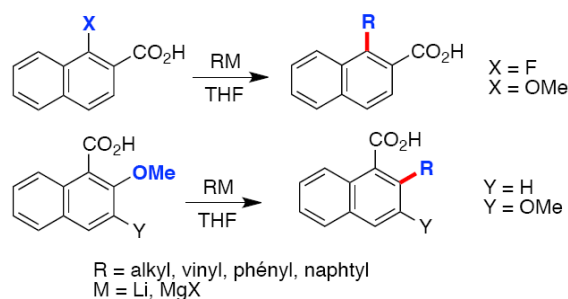
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Abstract

Substitution of an ortho-fluoro or methoxy group in 1-and 2-naphthoic acids furnishing substituted naphthoic acids occurs in good to excellent yields upon reaction with alkyl/vinyl/aryl organolithium and Grignard reagents, in the absence of a metal catalyst

without the need to protect the carboxyl (CO₂H) group. This novel nucleophilic aromatic substitution is presumed to proceed via a precoordination of the organometallic with the substrate, followed by an addition/elimination.



Keywords: Substitution nucléophile aromatique, Organolithiens, Organoagnésiens

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ACETALIZATION OF BENZALDYHEDA AND ACETONE CATALYZED BY ACID ACTIVATED CLAY

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Abstract

In this work , we treated benzaldehyde and propanone with glycerol under the same operating conditions to obtain the corresponding cyclic acetals. The raw clay used in this work comes from Maghnia in the Tlemcen region of northeastern Algeria. We prepared the clay AL1 by activating with hydrochloric acid of purified clay ALC.

The catalytic performance of acid activated clay shows dioxolane production selectivities in the case of acetalization of acetone whereas acetalization with benzaldehyde leads to dioxolanes and dioxanes. The products are six-member cyclic isomers of 2-phenyl-1,3-dioxan-5-ol and isomers of the five-membered (2-phenyl-1,3-dioxolan-4-yl) methanol cyclic and (2,2-dimethyl-1,3-dioxolan-4-yl) methanol. These isomeric compounds are identified by NMR spectroscopy

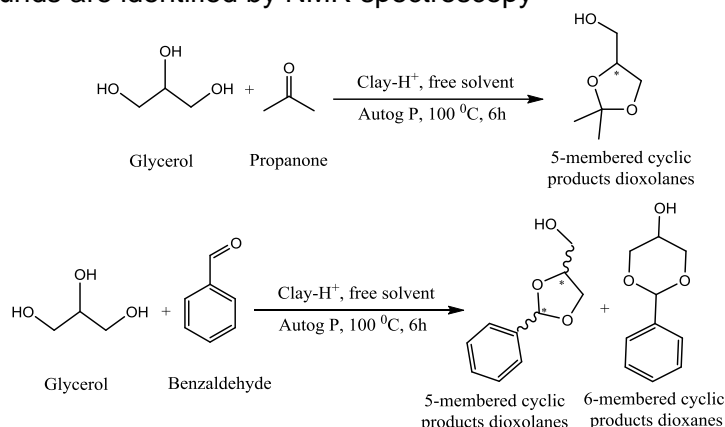


Figure 1: réactions d'acétalisation du Propanone et du benzaldéhyde

Keywords: acetalization ,dioxolane, dioxane

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PHOSPHOTUNGSTIC ACID TYPE DAWSON AS GREEN CATALYST FOR THE SYNTHESIS OF NEW CYCLIC IMIDES

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Abstract

The application of heteropolyacids HPAs, as catalytic materials is growing continuously in the catalytic field.

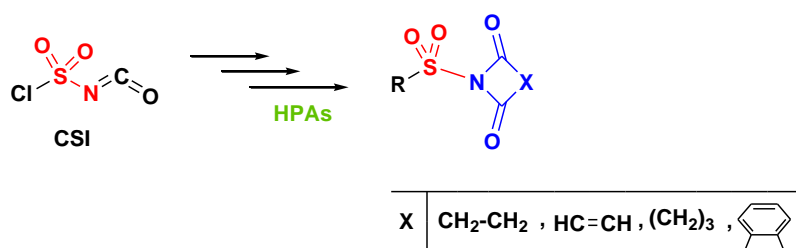
These compounds possess unique properties such as: Bronsted acidity, possibility to modify their acid-base and redox properties by changing their chemical composition (substituted HPAs), high proton mobility, being environmentally benign and presenting fewer disposal problems^[1, 2].

Cyclic imides are an important class of molecules known for their diverse array of bioactivities^[3-5]. These biological activities and various pharmacological uses of cyclic imides have been attributed to their unique structural features, which encourage us to look for newer and more advantageous methods for their efficient synthesis.

Moreover, the incorporation of sulfonamide group into organic molecules has a potential to modify the bioactivities^[6].

Considering the importance of sulfonamides, cyclic imides and their derivatives as bioactive compounds, we report herein, a convenient, clean and environmentally friendly procedure for the synthesis of new cyclic imides derivatives containing sulfonamide moiety in the presence of a catalytic amount of heteropolyacids (HPAs) including, $H_6P_2W_{18}O_{62}$, $H_6P_2W_{12}Mo_6O_{62}$, $H_3PW_{12}O_{40}$ and other acidic solids.

Figure 1



Keywords: Heteropolyacid Dawson type (HPA), cyclic imides, sulfonamides.

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SYNTHESIS OF SOME GLYCOSTEROIDAL BOLAPHILES

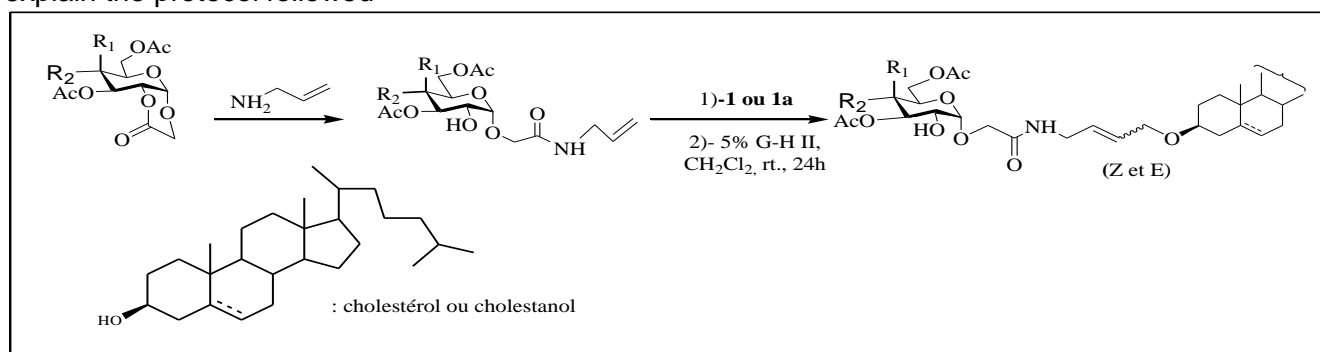
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Abstract:

This work describe the synthesis of bolaphile biomimics composed based on sacharids head groups and steroidal units, the two entities are linked together by a methylene chain of varying length. Many compounds in this family are natural products, which possess important biological properties[1] This family of compounds has been prepared by the CMGL-synthon strategy[2] and reporting our preliminary results on the thermotropic behaviour of acyl steroid glycosides (ASG), namely cholesteryl 6-O-acyl- β -gluco- and -galacto-pyranosides. These systems can exhibit several types of structures depending on their polarity pattern based on the number of polar and non-polar moieties and their resulting formed by self-organization of the products as a function of temperature were characterized by differential scanning calorimetry and thermal polarized light microscopy. The diagram below can explain the protocol followed



Scheme 1: Diagram explained the protocol followed for the synthesis of bolaphils compounds

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DEPROTO-METALLATION OF *N*-ARYLATED PYRROLES AND INDOLES USING A MIXED LITHIUM–ZINC BASE

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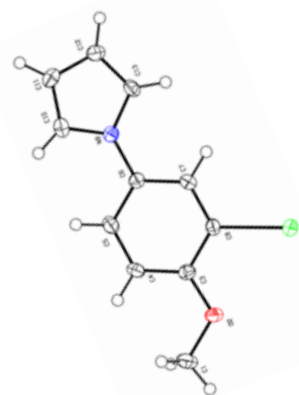
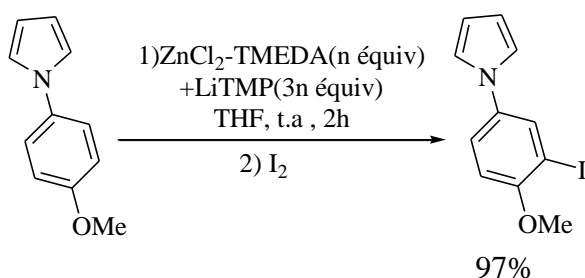
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Abstract

Pyrrole occurs in very important natural products such as tetrapyrrolic (linear) bilirubinoids, and (cyclic) porphyrins and corrins, as well as in pharmaceuticals (e.g., pyrrolnitrin) and polymers. Indole is similarly present in numerous natural products (e.g., tryptophan, melanin) including bioactive products (e.g., strychnine, brucine, lysergic acid), agrochemicals (e.g., auxins, pyroquilon), and pigments (e.g., indigo, indocyanines)².

The deprotonative metallation³ is a valuable tool for the regioselective functionalization of aromatic heterocycles such as pyrroles and indoles⁴. A few examples concern the reaction of *N*-arylprrroles and -indoles.

The synthesis of *N*-arylated pyrroles and indoles is documented, as well as their functionalization by deprotonative metallation using the base in situ prepared from LiTMP and ZnCl₂·TMEDA (1/3 equiv). With *N*-phenylpyrrole and -indole, the reactions were carried out in hexane containing TMEDA which regioselectively afforded the 2-iodo derivatives after subsequent iodolysis.



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SYNTHESIS OF SULFONYL ANALOGUES OF FOTEMUSTINE

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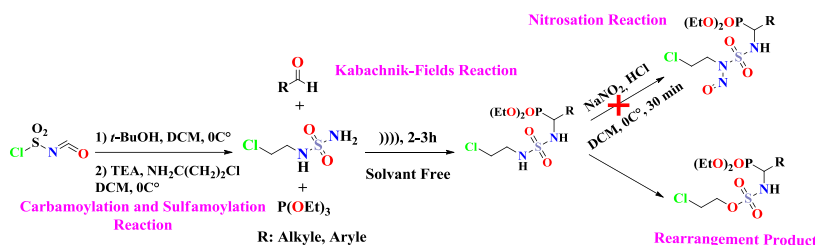
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Abstract

Cancer is a disease whose effect is unfortunately still tragic in many cases. This is one of the leading causes of death according to OMS data. In 2012, melanoma represents between 2-3% of all cancers. By definition, melanoma is a disease of skin cells called melanocytes. It develops from an initially normal cell that transforms and multiplies anarchically to form a lesion called malignant tumor. [1] Among the treatments used against melanoma, **Fotemustine** is a cytostatic anticancer agent of the **nitrosoourea** family. This treatment is very interested because of the broad spectrum of activity. [2] However, its clinical application is somewhat limited by the toxicity of isocyanate during his decomposition in physiological medium. [3] Our work aims at the synthesis of new **sulfonyl analogues** molecules of **Fotemustine**. The synthesis can be done in four steps according to the reaction scheme above. Unfortunately we did not achieve to optimize the best conditions to isolate the desired products, the spectral analyzes and the crystallographic study confirms the obtaining of the new products of rearrangements.



Scheme 1: Synthesis of sulfonyl analogues of Fotemustine

The details of the synthesis and the structural study will be presented.

Keywords: Fotemustine, nitrosoourea, sulfonyl analogues.

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COMPOSITION AND SYNERGISTIC EFFECT OF ESSENTIAL OILS OF *THYMUS FONTANESII* AND *SYZIGIUM AROMATICUM* AGAINST *CANDIDA ALBICANS*

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Abstract

The insertion in the mouth of a removable dental prosthesis influences the oral physiology. It is at the origin of the formation, on the surface of the apparatus, of a microbial biofilm of structure and complex composition. He thus participates in the establishment of stomatitis denture which is the most common form of oral candidiasis, of which *Candida albicans* is the most commonly detected yeast [1,2].

In the face of the problem raised for several years by the resistance of microbes to pharmaceutical products, the only reliable alternative to the use of antimicrobial agents to be that of essential oils whose anti-infectious efficacy has been scientifically demonstrated in vitro [3]. Indeed, the terpene compounds of essential oils and more specifically their functional groups such as phenols react with membrane enzymes and degrade the plasma membrane of yeasts.

In this context, we are interested in studying the antifungal activity of the essential oils of *Thymus fontanesii* and *Syzgium aromaticum* and especially of their synergistic effect. The analysis of these essential oils by CG and GC/MS reveals their richness in phenolic compounds (carvacrol and eugenol, respectively).

These essential oils displayed the highest activity against all strains of *Candida albicans* (taken from prosthetic intrados) of oral origin tested with MICs values 0.125 µl/ml for *Thymus* and 0.500 µl/ml for *Syzgium*. Moreover, the synergistic effect of these two oils was more effective with very low MICs values 0.036 µl/ml and 0.125 µl/ml, respectively in combination.

In conclusion, these essential oils therefore have potential for the treatment or prevention against *C. albicans* oral fungal infections.

Keywords: Essential oil, Composition, Antifungal activity.

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SYNTHESIS OF NOVEL TETRAHYDROBENZO[b][1,8]NAPHTHYRIDINES DERIVATIVES

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Abstract

Pyridines derivatives are molecules that have recently attracted a lot of interest because of their potential wide use in many areas of research and everyday life. Their potential utilization is oriented to medicine, nuclear medicine and nuclear waste reprocessing, biochemical research (nucleic acid, ATP..)^{1,2}. Nowadays, lots of efforts were carried out to prepare tetrahydrobenzo[b][1,8]naphthyridine Tacrine analogs to overcome the related adverse effects.³

This work describes the synthesis of new tetrahydrobenzo[b][1,8]naphthyridine (Figure 1) derivatives of and related heterocycles using the 3-cyano-2-aminopyridines⁴ as a starting material, following compounds, and others, will be presented and discussed.

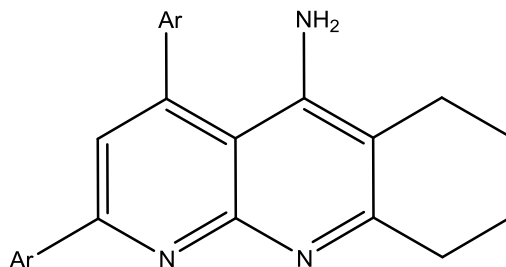


Figure 1. Structure of tetrahydrobenzo[b][1,8]naphthyridine derivatives

:

Keywords: aminopyridines, Tacrine, tetrahydrobenzo[b][1,8]naphthyridine.

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SYNTHESIS AND ELECTROCHEMICAL PROPERTIES OF 2,5-BIS(2-PYRIDYL)-1,3,4-THIADIAZOLE AND ITS NICKEL COMPLEX

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Abstract

2,5-Bis(2-pyridyl)-1,3,4-thiadiazole (L1) was synthesized by Hydrazine and 2-Pyridinecarboxaldehyde [1]. The electrochemical study of 2,5-Bis(2-pyridyl)-1,3,4-thiadiazole (L1) and their Ni(II) complexes were studied by cyclic voltammetry on the hanging mercury drop electrode (HMDE) using DMSO as solvent in the presence of tetrabutylammonium perchlorate (TBAP) 0,1 M as the supporting electrolyte, in order to compare them with those of the corresponding complexes under the same conditions [2]. Indeed, this comparison allowed us to distinguish the electrochemical phenomenon relative to the metal ion Ni²⁺ from that of the ligand, in the considered complex. Subsequently, the electrochemical study is particularly focused on the redox behavior of the metal ions of the complexes in solution in DMSO.

Keywords: nickel complex, cyclic voltammetry, HMDE

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SYNTHESIS AND CHARACTERIZATION OF NEW POLY (4', 4 METHYLENEDIANILINE) NANOCOMPOSITE USING MAGHNITE-Co AS CLAY CATALYST

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Abstract:

The synthesis and characterization of a poly (4', 4 methylenedianiline) is described. This compound is synthesized by Maghnite-Co as catalyst. Here we report the synthesis of a poly (4', 4 methylenedianiline) obtained. The effect of amount of catalyst and time on the polymerization yield of the polymers was studied. Structure was confirmed by elemental analysis, ¹H-NMR, ¹³C-NMR, UVvis, FTIR, and DRX spectroscopies.

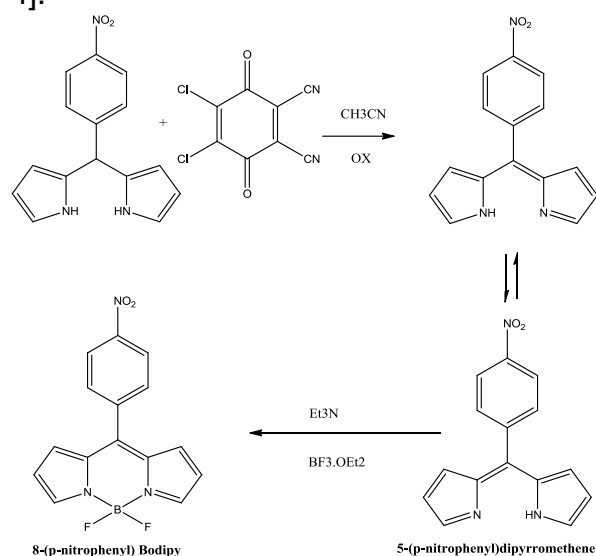
Keywords: conjugated polymer, poly (4', 4 methylene dianiline), Maghnite-Co, synthesis, characterization.

A GREEN METHOD FOR THE SYNTHESIS OF BODIPY FROM ARYLDIPYRRROMÉTHANENawal BAYOU-KHIER^{a*}, Mohamed AMARI^b, Mokhtar FODILI^c^a Département de chimie, Faculté des sciences, UMBB, Siège (Ex INIL), 3500 Boumerdes, Algérie^b Faculté de chimie, USTHB, BP 32, El-Alia, Bab-Ezzouar, Alger, Algérie^c Laboratoire de Chimie Organique et Substances Naturelles. Université Ziane Achour, Djelfa

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Abstract

In this work, we succeeded in synthesizing 5-aryldipyrromethane products in an aqueous medium with excellent yields. The water possesses the advantage to be not expensive, not toxic and friendly solvent of the environment, we think that this way of synthesis of (green chemistry) is essential within the hour current and is part of the innovative efforts of the synthesis of oligopyrroliques macromolecules. The aryldipyrromethanes [1] obtained will be used as a precursor in the synthesis of Bodipy compounds which have remarkable fluorescence properties[2-4].



All derivatives were characterized by ¹H NMR and mass spectrometry

Keywords: Dipyrrométhane, Bodipy, dipyrrométhéne.

References:

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SYNTHETIC STUDIES AND ANTIBACTERIAL ACTIVITIES OF CADIOLIDES ANALOGUES

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Abstract

Functionalized acylfuranones are present in many natural and synthetic products, cadiolides secondary metabolites from marine ascidians and tunicates present fascinating and similar antibacterial activities¹ or better than those of marketed drugs such as Vancomycin² and Platensimycin¹, the biomedical importance of these compounds prompted us to search for other bioactive cadiolides.

However, the structure activity relationship of these natural compounds has not been established and may be function of the number and position of bromine atoms on the phenolic rings, recently a one multicomponent procedure developed for the preparation of substituted acylfuranones by condensation of hydroxyketone, a functionalized dioxinone and an aldehyde and now report on the application of this methodology to the synthesis of various cadiolide analogues and their antibacterial activities³.

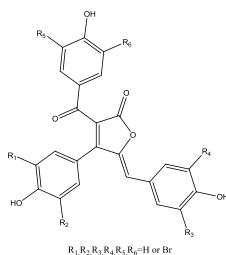


Figure 1. General structures of cadiolides.

Keywords: Acylfuranones, cadiolides, antibacterial.

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SYNTHESIS OF THE 1,2,4-TRIAZOLES. BIOLOGIC ACTIVITY

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Abstract

Triazoles are cyclic compounds with five members. Known for their many properties because their structures. They are diversified and opens to them new application in particular in chemical industry, pharmaceutical and agricultural as weed killers, fungicides, antiviral, agent antibacterial, anti-inflammatory drugs, and anti-cancer. Our study is based on the effect of the modification of the succinate of hydrazide in heterocyclic compounds with five members. The synthesis of 1,2, 4-triazoles was obtained by the cyclization of the acid thiosemicarbazide A2. The latter were synthesized by condensation of the acid hydrazide A1 correspondent with the isothiocyanate alkyl.

The products obtained were characterized by IR, RMN¹H and RMN ¹³C and we tested biologic activity and antifungal. [1, 2, 3]

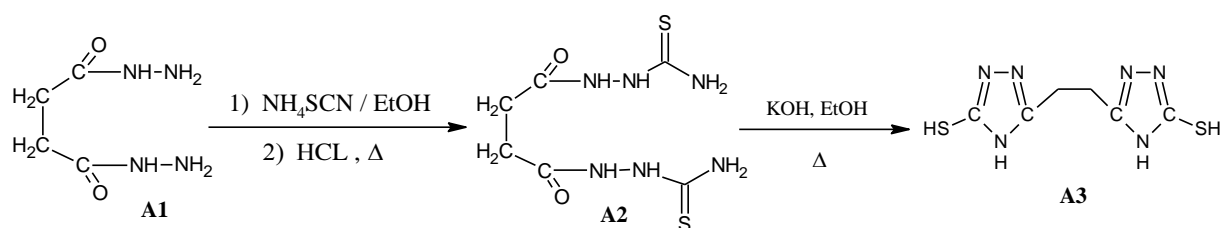


Figure 1. Synthesis of the di- (4H-1,2,4-triazole -3-thiole) succinate.

Keywords: heterocyclic, 1,2,4-triazole, biologic activity.

References:

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CHEMICALLY & ENZYMATICALLY SYNTHESIS OF SOME CARBOXYLIC AMIDES

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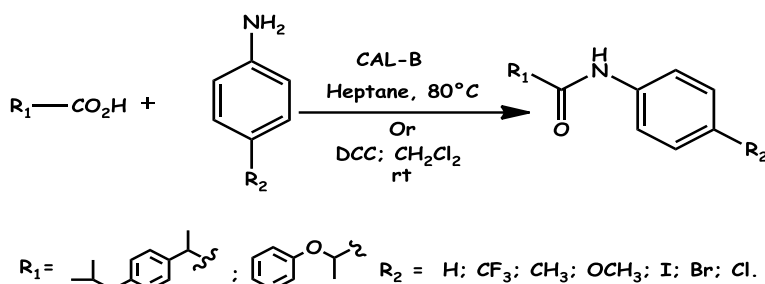
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Abstract

One of the major preoccupations of the pharmaceutical industry is the generation of amide bond ^[1]. In general, the mostly used methodologies for the N-acylation from carboxylic acid and amine are based on acid chloride intermediate, or by using coupling reagents ^[2]. But these methods show various inconveniences such as toxic by-products, for thus the development of novel environmentally benign pathways are tolerant, especially for the pharmaceutical industry ^[3]. One of the most interesting and clean pathways to the amide structures are those using lipases as biocatalyst. The lipase catalyzed aminolysis of carboxylic acid is well studied by Gotor and al, through kinetic resolution of chiral amines ^[4]. Generally, few described works, used the biocatalytic way to synthesize simply amide structure, the exclusive structures are in all circumstance bulky acid and aliphatic or cyclic amine ^[5].



In the following investigation, we show our study on the enzymatic aminolysis of carboxylic acids, acryloxypropionic acid and arylpropionic acids (profens) using substituted anilines (bulkier structures). The selected lipase is the candida Antarctica lipase immobilized on acrylic resin (CAL-B). The biocatalytic pathway is compared to a chemical way using DCC as coupling reagent. An efficient synthesis of a set of carboxylic amide is accomplished, with quantitative isolated yields. Some of the obtained amides are original.

Key words: Aminolysis, CAL-B, carboxylic acid.

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ANTI-INFLAMMATORY DRUG ACTIVITY OF THE SEEDS EXTRACTS OF ALGERIAN PRICKLY PEAR

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Abstract

The prickly pear cactus "*Opuntia ficus-indica*" (OFI) or Nopal is a plant that is well adapted to arid and semi-arid climates like that of Algeria. It has been used in traditional folk medicine because of its role in treating a number of diseases including diabetes, hypertension, gastric mucosa pain, cardiovascular and anti-inflammatory diseases in many countries over the world.

The objective of this work is to evaluate in vivo the anti-inflammatory activity of opuntia seed extracts. The anti-inflammatory activity of OFI was tested in rats weighting (200-300) g. Physiological saline (0.09%) control group, the both extracts (500mg/kg), and brexin (12 mg/kg) was administered (p.o.) for 30min before an edema was induced in the rat paw by subcutaneous injection of carrageenan. The rat-paw volume was measured 1 h30min, 3h and 6 h after injection.

The OFI showed significant reduction of oedema in carrageenan induced rat paw oedema model for 44% and 50% at 1 h30 min and 3 h respectively for the hydro-ethanolic extract. Compared to it, the aqueous extract reduces the increase in paw volume with the same percentage of 25%, for the reference brexin (12mg/kg) exhibited % reduction in paw volume 72.71%, 71.32%, and 28.72% after 1h 30min, 3h, 6h respectively in the control group. Furthermore, the phytochemical tests detect the presence of flavonoids, tannins, sterols, and alkaloids in the seeds of prickly pear cactus (OFI).

The obtained results of the present investigation revealed that the extract of OFI seeds (aqueous, hydro-ethanolic) has significant anti-inflammatory activity. It can be deduced that this species of plant is a promising source as therapeutic agents without undesirable effects for the investigation of new bioactive compounds.

Keywords: Anti-inflammatory activity, Nopal, Seeds.

DEVELOPMENT OF STUDY ON THE EFFECT OF INHIBITION CORROSION IN A CORROSIVE ENVIRONMENT BY A NEUTRAL POLYMER

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Abstract

Corrosion results from a chemical or electrochemical action of an environment on metals and alloys. The consequences are significant in various fields and in particular in industry: production stoppage, replacement of corroded parts, accidents and risks of pollution are frequent events with sometimes heavy economic impact.

The effect of synthetic polymers on the inhibition of corrosion in an acid environment is great economic and scientific interest.

We performed a study on the effect of the poly (N-vinylpyrrolidone) inhibitor on steel rounds. So a gravimetric study was done. Tests on the different concentration such as perchloric acids. Inhibited efficacy was perchloric acid 80.55%.

Keywords: Inhibitor, Corrosion, Acid, polymers, inhibitory efficacy

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ELECTROCHEMICAL CATHALISIS OF INHIBITING EFFECT OF NATURAL PRODECT ON HARD GROWTH WATER

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Abstract

Hard water form compact and adherent deposits to the walls of pipes and industrial or domestic installations causing enormous technical and economic problems, in the factory constructions, agronomic and the domestic equipment (valves, taps and boilers). Therefore, it is very important to establish a treatment of Algerian hard ground waters in order to reduce their hardness and exploit them. Bounouara drilling is one of the important water resources that feeds the south east of city of Constantine with drinking water and even water for industrial and agronomic settlements. But the great inconvenience of Bounourara water is very high hardness which is in the order of 60°F.

Several works have been done to inhibit the hardness but all the inhibitors that were used are chemicals that have an adverse effect on health, that's why all the chemical inhibitors are used only for industrial water treatment. In this work, we used citrus bark as a natural and ecological inhibitors to reduce the scaling power of Bounourara water and to prepare drinking water on the one hand, and valorize waste of two citrus bark on the other hand. The catalisis and evaluation of chemical treatment effect of hard water of Bounouara was carried out by electrochemical methods of chronoamperometric and impedancemetric measurement of the deposits of calcium carbonate electrolyte interface.

Keywords: Electrochemical cathalisis, hard water natural inhibitor.

CHARACTERIZATION AND TREATMENT OF USED LUBRICATING OILS

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Abstract

The treatment of waste oils is a difficult phase for the fight against pollution. Indeed, the purifier has a difficult problem to solve for multiple reasons: problems collecting these toxic products. Non-availability of the oil processing company in Algeria, necessity and environmental requirements Moreover, the economic importance of this problem is illustrated by the high cost, both in investment and in operation it can represent. We can recover 85% base oils from used motor oils. The impact of its treatment method. The main objective of this study is to find a method to maintain the environment and good yield to treat this oil, we selected a type of oil (10 W40 NAFTILIA), and then to characterize these oils (new, used And after treatment) by: Analysis of the physical and chemical properties of the oil (flash points, Viscosity index, total acid number, total base number and Density ...), Fourier transform infrared spectroscopy (FTIR-RTA) for determining the functional groups, for example: OH, CH, C-Cl, C-Br, Cl, CN, NH, etc.); And finally detection of heavy metals by atomic absorption spectroscopy (SAA).

Keywords: used oils, oil treatment, lubricating oil.

References:

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MICROWAVE-ASSISTED SYNTHESIS OF N-BENZYLAMINES CATALYZED BY COPPERMohamed El Hadi BENHALOUCHE^a, Abdellah MILOUDI^{a,b}^a *Laboratoire de Chimie Fine- Université d'Oran1- Ahmed BENBELLA, BP1524 El M'Naouer Oran, 31000, Algérie*^b *Ecole Nationale Polytechnique d'Oran- Maurice AUDIN, BP1523 El M'Naouer Oran, 31000, Algérie**Corresponding author: benhalouche.mohamed@edu.univ-oran1.dz, elhadi_31@hotmail.fr**Abstract**

Since first Microwave Assisted Organic Synthesis (MAOS)^[1], microwaves have got great interest in chemistry and actually dedicated reactors are proposed for laboratory use.

This technique responds of actual challenges that are: environment, economy and efficiency in organic synthesis^[2].

The synthesis of amines is an important reaction in organic synthesis and more particularly the secondary and tertiary amines which find scopes in various fields such as in medicine^[3], catalysis^[4] and industry.

In our work, we will expose the microwave-assisted N-benylation of different amines catalyzed by copper derivatives in a mixture of ethanol/water solvent.

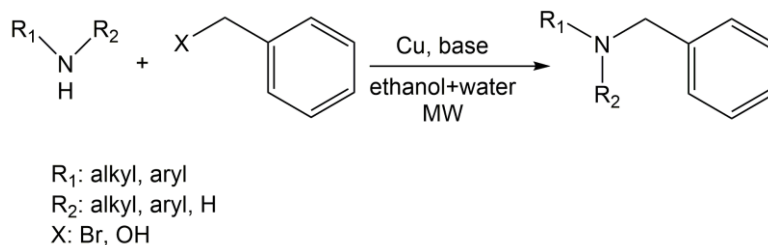


Figure 1. General scheme of N-benylation reaction assisted by microwave

Keywords: amines, microwaves, copper

References:

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SYNTHESIS OF A NEW FAMILY OF GLYCOLIGANDS BY A SIMPLE AND EFFICIENT REACTION

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Abstract

Many pyranoses and furanoses with the ring oxygen replaced by an amino group, known as imino sugars or azasugars, have been found to inhibit specific enzymes such as glycosidases [1]. Inhibition of these enzymes which catalyze the hydrolysis of the glycosidic bond to residues of oligosaccharides or saccharides is a potentially useful target of a wide range of important biological processes, such as intestinal digestion and posttranslational processing of glycoproteins, cancer and diabetes and other metabolic disorders.

In this theme we are interested in the synthesis of new nitrogen ligands derived from sugars in order to test them as glycosidase inhibitors. [2]. **Figure 1**

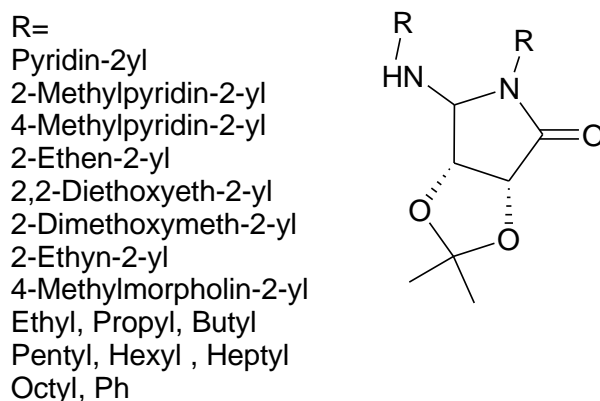


Figure 1.

Keywords: Iminosugars, glycoligands, glycosidase inhibitors.

References:

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ELIMINATION OF CHROMIUM (VI) WITH NEW CATALYTS MATERIALS: POLY(N-VINYLEPYRROLIDONE-SODIUM BENTONITE

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Abstract

The key of success of clays as catalysts is their ionic character and consequently their surface acidity, which allowed it the possibility to interact with polymers and form new catalyst materials.

The industry uses heavy metals such as chromium (VI) in different fields; these metals are toxic for human beings and the environment. The clays and the poly (N-vinylpyrrolidone) could form a new range of adsorbents which are more economic and less polluting.

The work reported here deals with investigation into the use of locally available clay from the fields of Hammam-Bouhrara-Maghnia-Algeria and poly (N-vinylpyrrolidone) as adsorbent for the removal of chromium (VI) from an aqueous solution. The Kinetic adsorption studies of chromium (VI) on sodium bentonite modified by poly (N-vinylpyrrolidone) BNa-PVP show that maximum chromium removal was achieved within 40 min of the start of every experiment at most conditions. The adsorption of chromium (VI) follows with removal more than 75 % on BNa-PVP at all conditions. Equilibrium data fitted well with the Langmuir and Freundlich models, while maximum adsorption capacity was 2.17 mg.g⁻¹ for only 0.01 g of BNa-PVP.

Keywords: Chromium (VI), adsorption, clay, poly(N-vinylpyrrolidone).

ZIRCONIUM COMPLEX OF THE ANTIBIOTIC SULFANILAMIDE, THE X-RAY SINGLE CRYSTAL STRUCTURE OF $[\text{Zr}(\text{C}_{12}\text{H}_{17}\text{N}_4\text{O}_4\text{S}_2)] \cdot \text{NO}_3$

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Abstract

Sulfanilamides were successfully employed as effective chemotherapeutic agents for the prevention and cure of bacterial infections in human biological systems. Moreover, sulfadugs and their complexes, have applications as diuretic, antiglaucoma or antiepileptic drugs, antifungal, antiviral, antitumor, and anti-inflammatory ones among others.

The new complex of zirconium of sulfanilamide formulated as $[\text{Zr}(\text{C}_{12}\text{H}_{17}\text{N}_4\text{O}_4\text{S}_2)] \cdot \text{NO}_3$ is synthesized by the hydrothermal route. The crystal structure of complex zirconium was identified by X-ray. The complex of zirconium crystallize in the orthorhombic space group *Pbca* with crystal data $a=13.904(5)$, $b=13.2572(7)$, $c=18.7436(8)$, $V=3455(3)$, $Z=4$, with sulfanilamide acting as bidentate ligand. Zirconium is coordinated to the nitrogen and oxygen from the sulfanilamide group. In the crystal structure of the title compound, $[\text{Zr}(\text{C}_{12}\text{H}_{17}\text{N}_4\text{O}_4\text{S}_2)]^+ \cdot \text{NO}_3^-$, the cations and anions are connected by N—H...O hydrogen bonds into a three-dimensional network.

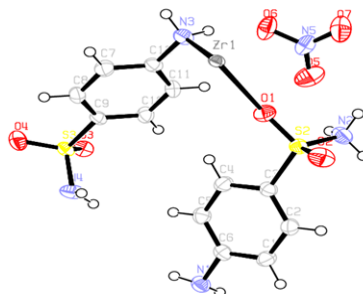


Fig. 1. Molecular structure of $[\text{Zr}(\text{C}_{12}\text{H}_{17}\text{N}_4\text{O}_4\text{S}_2)]^+ \cdot \text{NO}_3^-$ with the numbering scheme.

Key indicators: Single-crystal ,X-ray study; Hydrothermal synthesis, Zirconium sulfanilamid.

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AZA-DIELS-ALDER REACTIONS OF CYCLOPENTADIENONES

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Abstract

Multi-component reactions (MCRs) have increasing importance in synthetic organic chemistry. Among them, Povarov which is known as the aza-Diels-Alder reaction is a powerful means for the construction of nitrogen-containing six-membered heterocycles [1-3]. This reaction can be carried out in one pot using an aniline, an aldehyde and an electron rich dienophile catalyzed by either protic [4], lanthanide [5] or Lewis acid [6].

We reported herein the one-pot reactions of 2-nitroaniline, 4-methyl-2-nitroaniline and 1-aminoanthraquinone with formaldehyde and tetrasubstituted cyclopentadienones in the presence of trifluoroacetic acid. The X-ray structures of the new quinolines were reported.

Keywords: Multicomponent reaction, aza-Diels-Alder cycloaddition, tetraquinoline systems.

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AN EFFICIENT SYNTHESIS OF QUINOLINES AND NAPHTHYRIDINES USING HETEROPOLYACIDE CATALYSTS

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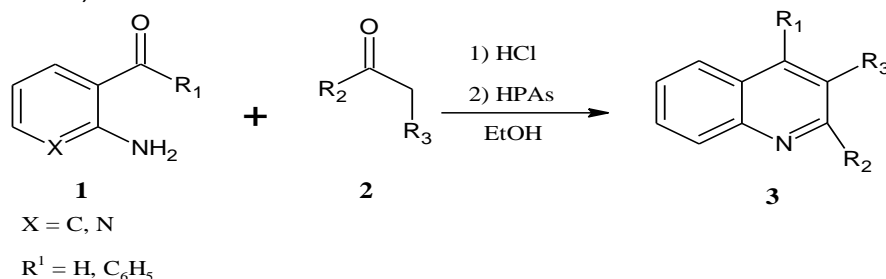
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Abstract

The synthesis of quinolines and 1,8-naphthyridines was carried out in the presence of HCl and a series of eco-friendly solids including Keggin type heteropolyacids (HPAs) $H_4SiW_{12}O_{40} \cdot 14H_2O$, $H_4SiMo_{12}O_{40} \cdot 14H_2O$, $H_3PW_{12}O_{40} \cdot 13H_2O$ and $H_3PMo_{12}O_{40} \cdot 15H_2O$ used as catalysts. HPAs exhibit high yields in particular the silicates, SiW_{12} and $SiMo_{12}$ with 73-93% of yield. Quinolines and Naphthyridines derivatives are valuable materials related to their important biological activities [1]. Due to their broad application domain, the development of effective methodologies for the synthesis of these compounds has expanded.

The Friedlander reaction, well-known as method for the preparations of quinolines and 1,8-naphthyridines involves a thermal condensation between a 2-aminoaryl ketone **1** and another carbonyl compound **2** possessing a reactive methylene group [2] under acidic or basic conditions [3].

In this work, we report for the first time the synthesis of quinolines and 1,8-naphthyridines in the presence of HCl and a series of Keggin type HPAs as catalysts $H_4SiW_{12}O_{40} \cdot 14H_2O$, $H_4SiMo_{12}O_{40} \cdot 14H_2O$, $H_3PW_{12}O_{40} \cdot 13H_2O$ and $H_3PMo_{12}O_{40} \cdot 15H_2O$ noted SiW_{12} , $SiMo_{12}$, PW_{12} and PMo_{12} , respectively (**Scheme1**).



Scheme1. Synthesis of quinolines and 1,8-naphthyridines derivatives

This study showed that the Keggin type heteropolyacids (HPAs) are excellent catalysts for the synthesis of quinolines and 1,8-naphthyridines via Friedlander condensation of 2-aminoaryl Ketones with α -methylene Ketones in particular the silicates (SiW_{12} , $SiMo_{12}$) by exhibiting high yields (72-93%) compared to the conventional mineral acid.

Keywords: Quinolines, Naphthyridines, Catalysis

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SYNTHESIS OF NOVEL CHIRAL INTERMEDIATES THROUGH REGIOSELECTIVE REDUCTIVE CLEAVAGE OF BIS-BENZYLIDENE ACETAL: A NEW ACCESS TO CARBASUGARS

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Abstract

Protection and deprotection of hydroxyl functional groups are an integral part of organic synthesis, especially in compounds containing more than one hydroxyl groups¹. The benzylidene acetal is one such protecting group widely used in organic synthesis due to its tolerance to a variety of chemical conditions. Selective cleavage of benzylidene acetals is crucial as it leads to synthetically useful mono-protected diols².

D-Mannitol plays an important role as a readily available chiral building block in organic synthesis. In addition, mannitol and its derivatives are widely used as chiral reagents and chiral auxiliaries. Our continuing interest in the stereoselective transformation of D-mannitol to biologically active and pharmaceutically important compounds recently resulted in the development of a regioselective reductive cleavage of *bis*-benzylidene acetal derivatives of D-mannitol to give novel chiral intermediates using $\text{BF}_3 \cdot \text{Et}_2\text{O} \cdot \text{Et}_3\text{SiH}^3$.

Keywords: Regioselective; Reductive cleavage; chiral intermediates; Benzylidene acetals

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ISOXAZOLIDINE'S SYNTHESIS BY INTRAMOLECULAR REACTION

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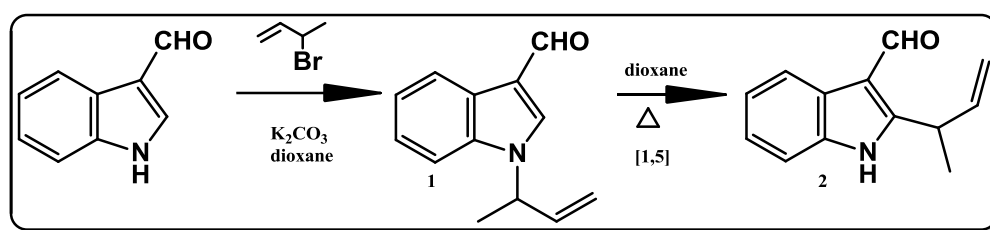
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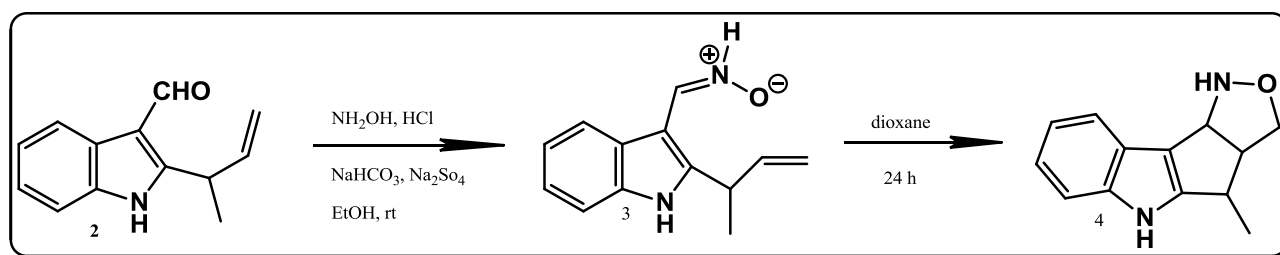
Abstract

The 3-heterosubstituted indoles have recently attracted a considerable interest because of their many biological applications ¹. For that reason, the reactions of indolyl 1,3-dipoles as convenient routes for the synthesis of heterosubstituted indoles was investigated. N-alkylation with 3-bromo-but-1-ene led to compound 1 after heat. N-alkylation with 3-bromo-but-1-ene led to compound 2 after heat.



Scheme 1

The reaction of 2 with hydroxylamine gave nitron 3. The 1,3-dipolar cycloaddition intramolecular reaction led to 4.



Scheme 2

K

Keywords: Nitron - N-alkylation- intramolecular reaction

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POMEGRANATE (*PUNICA GRANATUM L.*) SEED OIL: COMPOSITION AND PHYSICOCHEMICAL PROPERTIES

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Abstract

Pomegranate (*Punica granatum L.*) is one of the oldest known species among edible fruit trees. Pomegranate cultivation is very old in Algeria. This fruit tree is widely grown in many parts of the country. Our attention has focused on the pomegranate "*Atmi*" which is widely spread in the region of Tlemcen (northwestern Algeria).

Due to its antioxidant properties, pomegranate is currently considered as one of the most consumed fruits for its health benefits. It has been proven to be particularly effective against hormone-dependent cancers [1] (breast and prostate cancer) and cardiovascular diseases (hypertension) [2].

The pomegranate parts that are most suitable for human consumption are the arils, which represent about 52% of the total weight of the fruit. The arils are mainly composed of 78% juice and 22% seeds. After drying and grinding the seeds, we obtained the pomegranate seed oil (PSO) which represents 12- 20% of the total weight of the pomegranate seeds.

The physicochemical properties (density, viscosity, refractive index) of pomegranate seed oil (PSO) of the *Atmi* variety as well as the various indices used to characterize that oil (acid value, peroxide value, saponification number and iodine value) were highlighted.

A more accurate composition of the pomegranate seed oil (PSO) could be obtained using fine analyses (CPG and HPTLC). It was found that this oil consists mainly of fatty acids (about 95%) of which 99% are in the form of triglycerides. The main fatty acids obtained, after analysis, were octadecatrienic acids, whose content in Punic Acid (cis-9, trans-11, cis-13 C18: 3, PA) is high [3].

The analysis of pomegranate seed oil (PSO) has indicated the existence of other compounds, such as polyphenols, tocopherols and estrogenic compounds, in smaller quantities, but equally important. These compounds help to give PSO anti-inflammatory, antioxidant and antitumor properties [4].

Keywords: *Punica granatum L*; Fatty acids; Punicic acid.

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INFLUENCE OF LIPIDS IN FORMULATION OF NANOPARTICLES BY HOT MELTED SONICATION TECHNIQUE

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Abstract

In order to study the influence of lipids in the preparation of Solid Lipid Nanoparticles (SLN) by the ultrasonication method ⁽¹⁾, we realized different formulas with three lipid-matrix: Stearic Acid (S-LP), Palmitic Acid (P-LP) and Compritol ATO 888 (C-LP). These matrix were stabilized by the soy lecithin / Poloxamer 407 as pair surfactant / Co surfactant with a ratio (1:1). Sonication was performed at a frequency of 60 Hertz at 30 and 60 seconds, followed by high speed homogenization at 9500 rpm in 10% mannitol solution at 4°C. Measurements of the Zeta potential were favorable for a good stability ⁽²⁾ of the dispersions for the three formulas (S-LP, P-LP, C-LP) with respective values of -26.5, -20.9, -26.8 mV. The best results were obtained with the Compritol ATO 888, an average diameter of 390 nm and Polydisperse Index of 0.627.

Keywords: SLN- Sonication-Compritol

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INDIUM-MEDIATED ALLYLATION OF CARBONYL COMPOUNDS WITH FUNCTIONALIZED ALLYLIC HALIDES AND DEHYDRATIVE CYCLISATION TO TETRAHYDROPYRANS DERIVATIVES

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Abstract

Substituted tetrahydropyrans are the common structural motif of many natural products [1,2] Such as Avermectins, Althoyrtin A, Bryostatin, Halichondrin B, Lasalocide A, Milbemycine, Monensine, Phorboxazole B, Salinomycin and Talaromycins. Because of their potential importance to construct structurally complex molecules, several methods are reported [3], many of these procedures involve extended reaction timings and the use of expensive reagents.

Tetrahydropyrans in subject have been efficiently synthesized in High yields via allylation of aromatic aldehydes with functionalized allylic halides, using metallic indium as catalyst in anhydrous DMF as solvent [4]. After silyl ether cleavage of obtained homoallylic alcohols, dehydrative cyclisation in mild conditions led to substituted tetrahydropyrans [5]. Structures of the synthesized compounds have been confirmed by spectral analysis.

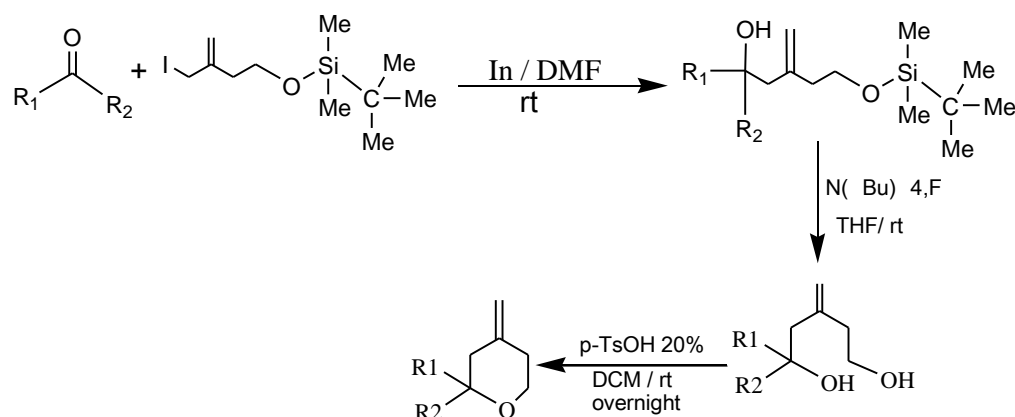


Figure 1. Synthesis of substituted tetrahydropyrans

Keywords: Indium, allylation, functionalized allylic halides, dehydrative cyclisation.

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SYNTHESIS OF 1, 3, 4- OXADIAZOLE DERIVATIVE AND STUDY OF ITS SURFACTANT PROPERTIES

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Abstract :

Heterocycles are a class of chemical compounds where an atom or more of carbon is replaced by a heteroatom such as oxygen; nitrogen, phosphorus, sulfur, etc. The most common heterocycles contain nitrogen and oxygen.

In the field of oleochemistry, fatty acids are considered as the main constituents of vegetable oils. The physical and chemical properties of fatty acids affect those of vegetable oils% [1].

Long-chain fatty acids are particularly studied as precursors of surfactant platform molecules. They are used as surfactants and have a high economic potential since agro-surfactants, derived from renewable materials, have a very good reputation, due to their low toxicity and their low safety for human health.

In this work, oxygen and nitrogen containing heterocycle is synthesized having surfactant properties employing a fatty acid. This synthetic pathway has been done through several steps (esterification, amination then cyclization). The final product 5- (dec-9-enyl)-1, 3, 4- oxadiazole-2-thiol as well as the intermediate products have been characterized using diverse methods as : Infrared spectroscopy, Thin-Layer Chromatography, Melting and refractometry. Surfactant properties of the final product have been investigated by measuring its surface tension and que sa critical micellar concentration (cmc) using tensiometry method.

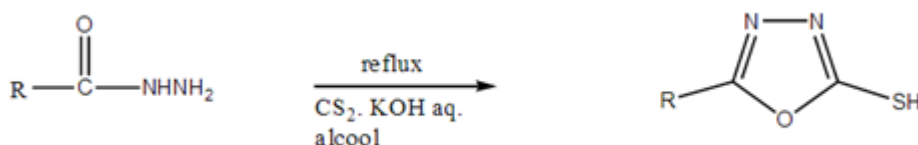


Figure 1 : Synthesis of 1, 3,4-oxadiazole by cyclization of hydrazide

Keywords: 1, 3, 4- oxadiazole, Surfactant properties, oleic acid

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SELECTIVE SYNTHESIS AND POLYMORPHISM OF A NEW FERROCENYL ALLYLPHOSPHONIUM SALT

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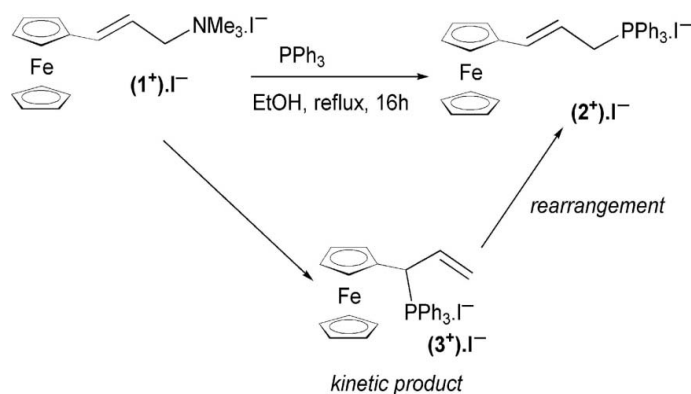
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Abstract

We recently developed an efficient and straightforward synthesis of ferrocenyl allyl compounds by reaction of various nucleophiles with a new ferrocenyl allylammonium salt, [3-(trimethylazaniumyl) prop-1-en-1-yl] ferrocene iodide, (1⁺)I⁻. With nitrogen-based nucleophiles, different ferrocenyl allylamine isomers have been synthesized successfully in good yield. Optimization of the basicity of the reaction medium has allowed selection of the best operating conditions to obtain the targeted isomer. In a similar way and in order to introduce phosphorus-containing functional groups, the reaction of ammonium salt (1⁺)I⁻ with a phosphorus nucleophile, namely triphenylphosphine, was attempted. It was then possible to isolate single crystals of (3-ferrocenylprop-2-en-1-yl)triphenylphosphonium iodide, [2⁺]I⁻, which is shown to crystallize in two concomitant polymorphic forms.



Keywords: Phosphonium; ferrocenyl allylic compounds; polymorphs; crystal structure.

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H₆P₂W₁₈O₆₂.14H₂O CATALYZED SYNTHESIS, SPECTRAL CHARACTERIZATION OF α -AMINOPHOSPHONATES CONTAINING AMINOTHIAZOLE MOIETY

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Abstract:

In the recent years, the α -aminophosphonates have received considerable attention in organic and medicinal chemistry [1] because of their structural resemblance with α -amino acids. They are used as antitumor agents, antivirals, anti-inflammatory and antibiotics. As a result, a number of procedures have been developed for their synthesis. However, many of these methods suffer from some disadvantages such as long reaction times, environmental pollution caused by utilization of organic solvents and expensive catalyst.

The heteropolyacids have received great attention as environmentally benign catalysts for organic synthetic processes [2].

In this context and in continuation of our interest toward developing novel biologically important organophosphorus compounds [3], we report the heteropolyacid-catalyzed three-component reaction between 2-aminothiazole, triethylphosphite and various aldehydes under solvent-free conditions, the synthesis of α -aminophosphonates was completed with excellent yields and shorter reaction times, the newly synthesized compounds were systematically characterized by IR, ¹HNMR, ¹³C NMR and SM. The experimental results will be presented.

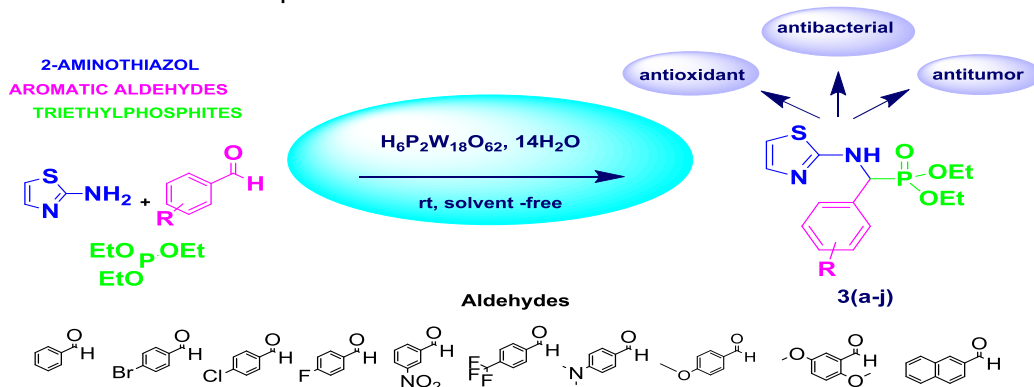


Figure 1. Graphic abstract

Keywords: 2-Aminothiazole, α -Aminophosphonates, $H_6P_2W_{18}O_{62} \cdot 14H_2O$ catalyst.

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MICROWAVE-ASSISTED ONE-POT SYNTHESIS OF NEW SULFONAMIDES CONTAINING BENZOTHAIAZOLE MOIETY CATALYZED BY HETEROPOLYACIDS

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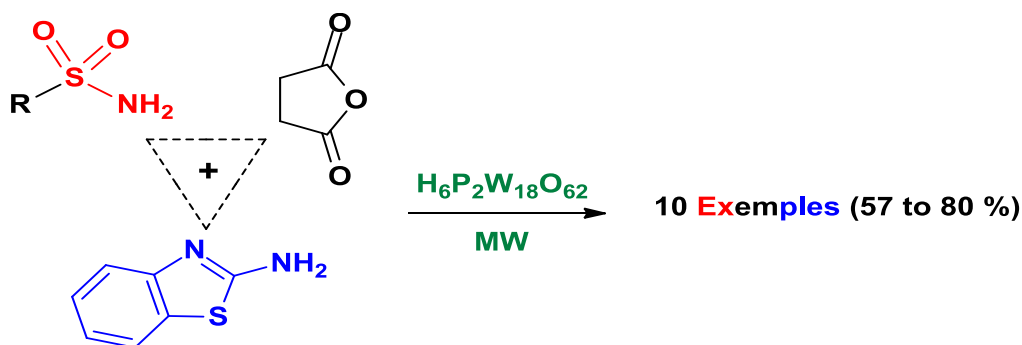
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Abstract

Green chemistry is an approach to the synthesis, processing, and use of chemicals that reduces risks to humans and the environment. Thus the development of methods using heteropolyacids (HPAs) as solid and green catalysts for fine organic synthetic processes related to fine chemicals, such as flavors, pharmaceuticals and food industries have been under attention in the last decade (1).

As part of an ongoing development of efficient benign protocols for the preparation of biologically active heterocycles from common intermediates (2-4), herein, $H_6P_2W_{18}O_{62}$ was utilized as an efficient and reusable catalyst for the one-pot synthesis of sulfonamide derivatives containing benzothiazole moiety from sulfonamides, succinic anhydride and 2-amino benzothiazole under microwave irradiation. The present approach offers the advantages of a mild reaction conditions, simple and eco-friendly method proceeds.



Keywords: Heteropolyacids, one pot, sulfonamides, benzothiazole derivatives.

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SYNTHESIS AND EVALUATION OF THE ANTIOXIDANT ACTIVITY OF TRIPODAL STRUCTURES BASED ON PYRAZOLE

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Abstract

The present work is located at the interface between Chemistry and Biology. More specifically, it consists the synthesis of tripodal compounds based on pyrazole substituted by a functionalized group and the evaluation of their antioxidant activity. This work presents the synthesis of two different series of tripodal compounds based on (3,5-dimethylpyrazol-1-yl) methanol, and 1- (hydroxymethyl) - 5-methyl-1H-pyrazole-3-ethyl carboxylate as basic precursors with a series of suitable amine.

Keywords: Anti oxidant, pyrazole, tripod.

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ORGANOCATALYTIC 1, 4-CONJUGATE ADDITION OF N-SUBSTITUTED BARBITURIC ACID ON α,β -UNSATURATED KETONES: SYNTHESIS, NMR AND SINGLE-CRYSTAL X-RAY DIFFRACTION STUDIES OF NOVEL 2,4-DIMETHYL- 7,11-DIARYL-2,4-DIAZASPIRO[5.5]UNDECANE-1,3,5,9-TETRAONE DYADS

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Abstract

Spiro compounds have been reported to possess biological activities that include hypotensive, analgesic [1], anti-inflammatory activity [2], anticonvulsant, narcotic [3]. Several works have been reported for the synthesis of other spiro barbiturates [4-7]. In this study, we report a new and facile route for the synthesis substituted spiro-barbiturates by the Michael addition reaction of N-substituted barbituric acid with 1,5-diaryl penta-1,4-dien-3-one derivatives.

This reaction of α,β -unsaturated ketones with N- methyl barbituric acids lead to synthesis new series of "2,4-dimethyl- 7,11-diaryl-2,4-diazaspiro[5.5]undecane-1,3,5,9-tetraone" in good yield.

Structure elucidation is carried out by ¹H NMR, ¹³C NMR, 2D NMR (HSQC and HMBC), Mass spectroscopy HRMS and X-ray crystallography techniques. A possible mechanism of the formation is discussed.

The structural conformation also demonstrated by coupling constants derived from dihedral angles between vicinal and geminal protons.

Keywords: Michael addition, Barbituric acid, Spiro barbiturate.

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SYNTHESIS AND REACTIVITY OF FUNCTIONALIZED 5-ARYLIDENE-2-THIOXOTHAZOLIDIN-4-ONE

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Abstract

Heterocyclic compounds containing nitrogen, sulfur and oxygen have been studied for a long time for their medicinal properties. Of these, 4-thiazolidinones possess various biological activities such as antibacterial^[1,2], antifungal^[1], anticancer^[3], anti-HIV^[4], anti-inflammatory^[5] and analgesic properties^[6]. In addition, 4-thiazolidinones have potent antituberculous properties^[7].

In view of the above facts and in the pursuit of our search for a new class of molecule of biological interest, we report the synthesis of 5-arylidene-2-thioxothiazolidin-4-one derivatives functionalized by a Knoevenagel condensation of various aromatic and heteroaromatic aldehydes with rhodanine in ethanol and potassium acetate. We have also been able to study some of their transformations, such as the synthesis of mercaptoacetic acid.

Keywords: 5-arylidene-2-thioxothiazolidin-4-one, Knoevenagel condensation.

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EFFECT OF 5 METHYL 1H, BENZOTRIAZOL ON THE CORROSION INHIBITION OF API N 80 STEEL IN ALBIAN WATER

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Abstract

In the present work, the corrosion behavior of API N80 in albian water, in the presence of inhibitor namely 5-methyl benzotriazole (MBTA) is studied. The inhibition efficiency of MBTA is evaluated from electrochemical techniques. Experimental studies showed that MBTA reduces markedly the corrosion, this reduction in corrosion rates enhances with increasing concentration of MBTA. potentiodynamic polarization study revealed that the formulation consisting of 30 ppm of MBTA showed around 50 % inhibition efficiency. Polarization studies also revealed that the inhibitor act as mixed type for albian water. The adsorption of MBTA on the steel surface followed the Langmuir isotherm. The effect of gradient temperature is also studied in range between 0- 6°C.

Keywords: API N80, albian water, 5-methyl benzotriazole, polarization study

QUANTITATIVE CONFORMATIONAL STABILITY HOST-GUEST COMPLEX OF METHIONINE WITH B-CYCLODEXTRIN: A THEORETICAL INVESTIGATION

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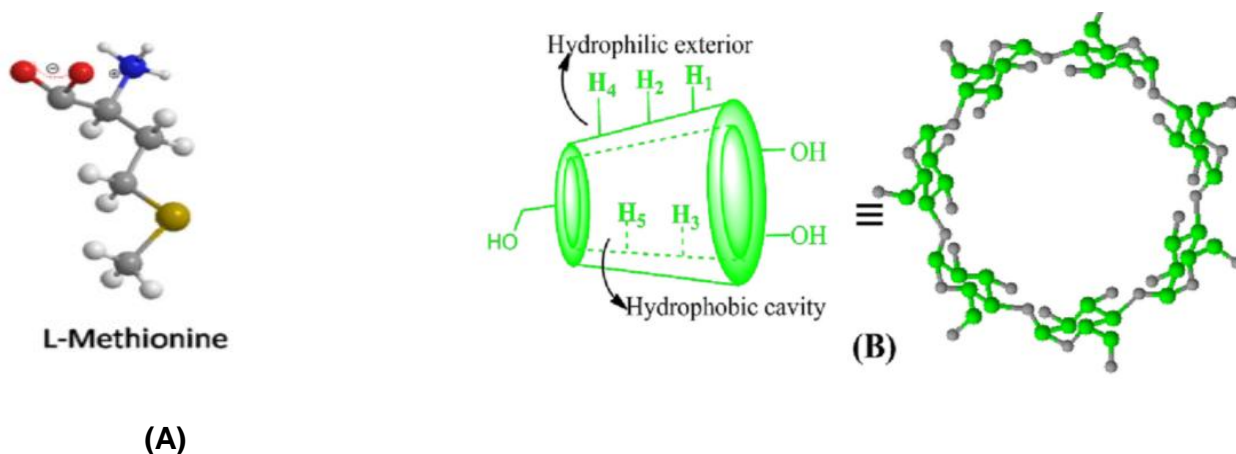
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Abstract

We simulated the docking of L -Methionine in beta-cyclodextrin (β - CD) considered in this study complexe formed by 1:1 host–guest stoichiometry in vacuo and in aqueous phase, using quantum chemical calculations. all DFT calculations have been performed with the 6-31G and 6-31+G(d) basis sets. Two modes of complexation were taken into consideration ‘orientation’. The obtained results clearly indicate that the formed complexes are energetically favored. The most reactive sites in the complexes were identified by molecular electrostatic potential map. Finally, charge transfer between the donor and acceptor orbital of L -Methionine and beta-cyclodextrin plays an important role to stabilize the inclusion complexes.



Scheme 1. The molecular structure of the selected amino acid(A) and the structure of β - CD (B) (hostmolecule).

Keywords: inclusion complex ; DFT ; charge transfer

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SYNTHESIS OF A PHOTOCROSSLIKABLE MACROMONOMER PROMOTED BY A GREEN CATALYSIS

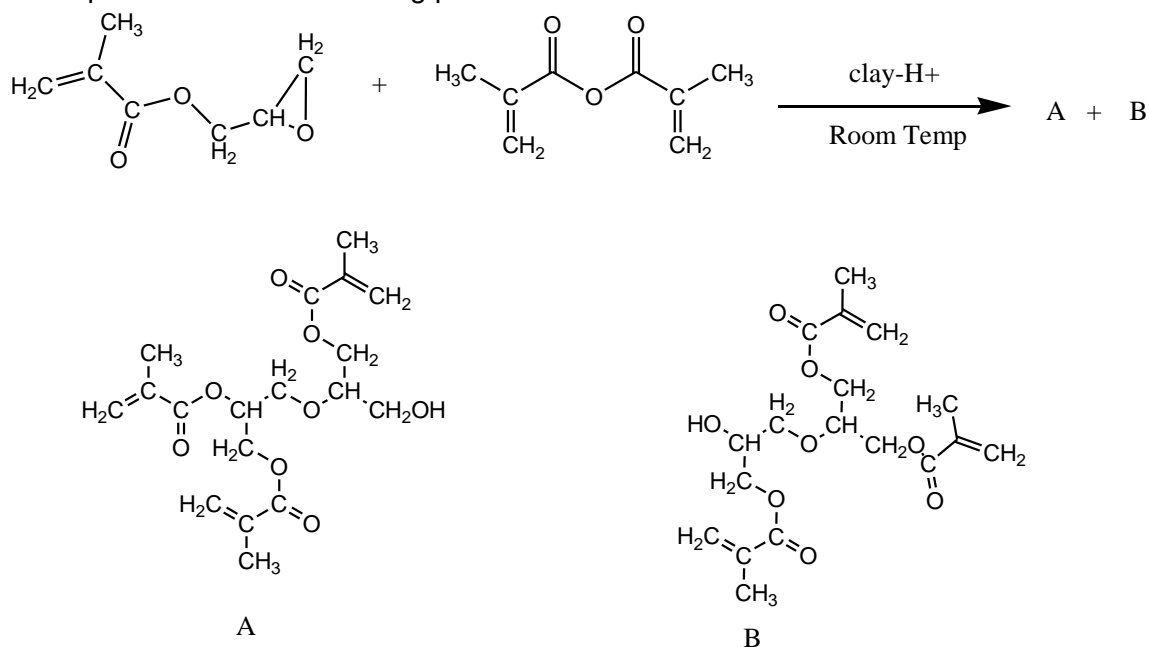
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Abstract

Glycidyl methacrylate (GMA) is a monomer that possesses two polymerisable functions. The epoxy group which can be initiated by a cationic ring opening process and the vinyl group that may be activated using a radical initiator. The GMA can be used in the synthesis of a wide range of macromonomers which can be further crosslinked to form complex networks [1] or be used to synthesise macromolecules [2], such as grafted copolymers, that can't be accessed using other methods. This work describes the synthesis of a crosslinkable macromonomer produced by reacting the GMA with the methacrylic anhydride and catalysed by an acidic clay (scheme). The structure of the obtained product was verified using proton NMR and FT-IR.



Keywords: Glycidyl methacrylate, green catalysis, macromonomer

References:

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SYNTHESIS OF NEW TRIAZINE DERIVATIVES BY CYCLIZATION OF 2-PYRIDYL HYDRAZONES

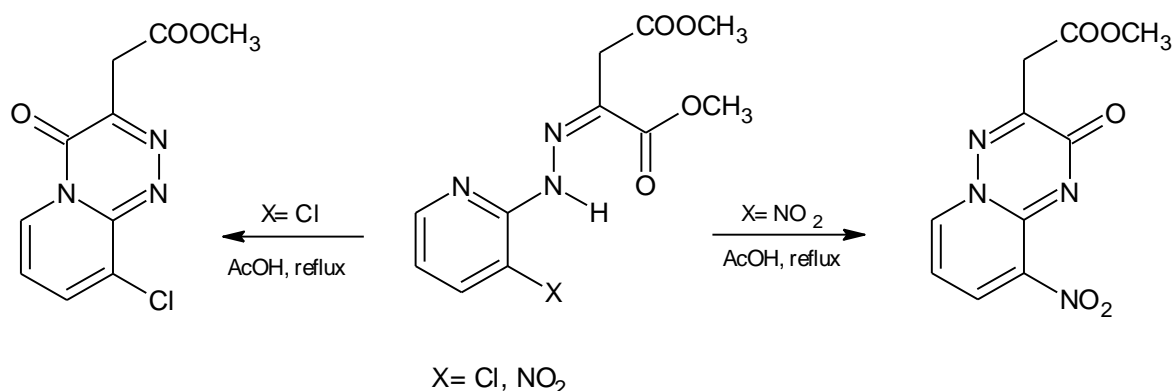
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Abstract

Homologous hydrazones formed by addition of substituted 2-hydrazinylpyridines to dimethyl acetylenedicarboxylate or dimethyl-2-oxoglutarate undergo under reflux in acetic acid different types of cyclizations depending on the kind of substitution. Whereas 3-chlorohydrazinylpyridine leads finally to derivatives of the pyrido[2,1-c][1,2,4]triazinone skeleton, starting from 3-nitro-2-hydrazinylpyridine gives rise to regioisomeric pyrido[1,2-b][1,2,4]triazinone derivatives. The finding is discussed in terms of substituent effects on the reaction behaviour which requires an *N-N* cleavage to give access to these heterocycles.



Keywords: Cyclizations, Hydrazones, Isomers Pyrido[1,2,4]triazinones.

References:

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A CASCADE SYNTHESIS, BIOLOGICAL EVALUATION AND MOLECULAR MODELING OF SOME NOVEL TACRINE-PYRANOPYRAZOLE DERIVATIVES AS CHOLINESTERASE INHIBITORS

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Abstract

Alzheimer's disease (AD) is a progressive neurological trouble leading to impairment in memory, language skills, judgment, and orientation.^[1] Currently, one possible treatment is inhibition of acetylcholinesterase (AChE) and butyrylcholinesterase (BChE) (to some extent) to maintain the ACh neurotransmission as long as possible which is hindered by the formation of β -amyloid plaques and the enhancement of the levels of the neurotransmitter acetylcholine (ACh) through the administration of acetylcholinesterase inhibitors. Tacrine (Cognex®) is an AChE inhibitor that proved to have a beneficial effect in patients with mild to moderate dementia of AD.^[2]

In this work, we describe the preparation of some new Tacrine analogs modified with an pyranopyrazole moiety. A *one-pot* multicomponent reaction of 3-methyl-1H-pyrazol-5(4H)-one, aryl(or hetero)aldehydes, malononitrile and cyclohexanone involving a Friedländer condensation led to the title compounds. Through synthesizing heterocyclic analogs of this molecule to evaluated *in vitro* for their AChE and BChE inhibitory activities in search for potent cholinesterase enzyme inhibitors. Molecular modeling simulation on AChE and BChE receptors, showed good correlation between IC₅₀ values and binding interaction template of the most active inhibitors docked into the active site of their relevant enzymes.

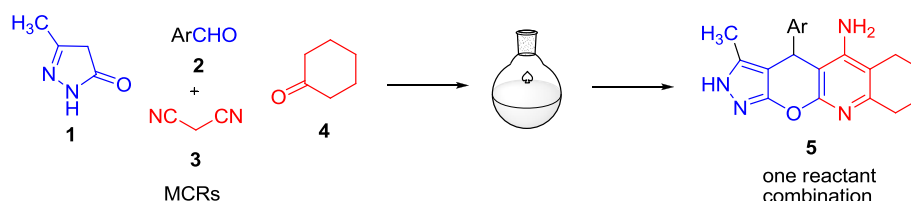


Figure 1. Possible functionalization of the Tacrine–Pyranopyrazoles scaffold.

Keywords: Tacrine-pyranopyrazole derivatives, Alzheimer's disease, Docking study

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A GREEN SYNTHESIS AND ANIONIC POLYMERIZATION OF METHACRYLIC MONOMERS N-ALKYL METHACRYLAMIDE CATALYZED BY AN ECOLOGIC AND FRIENDLY CATALYST MAGHNITE H⁺

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Abstract

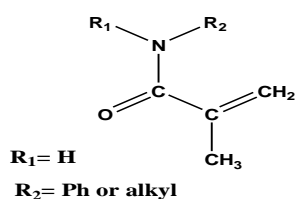
Green chemistry is focused to the use of new chemical approaches to reduce waste and toxic effluents. Therefore, it can minimize damage to the environment and helps us to improve the quality of life. That is why the use of new inflammable and recyclable solvents and initiators is the concept of several researches and syntheses [1-2]. Acrylic/ methacrylic monomers are an important class of functional polymers for their wide range of applications. They can be used to prepare polymers with rigid, flexible, ionic, non ionic, hydrophobic, or hydrophilic properties [3].

There are various studies on the synthesis of N- alkyl acrylamide or methacrylamide by using methacryloyl chloride or methacrylic acid with amine and triethylamine in CH₂Cl₂ or Chloroform was cooled using an ice bath followed by stirring at room temperature for 60 min.

In the present work, we have developed a novel procedure to synthesis and polymerized a new monomers 1,4-Pipérazine dimethacrylamide (NPDM), N-Methacryloyl morpholine (NMM), N-Phenyl methacrylamide (NPM) by the reaction of derived amine (heterocyclic and aryl amine) respectively (Piperazine, Morpholine and aniline) with methacrylic anhydride catalyzed by Maghnite H⁺ a green catalyst and recyclable in one-step with the aim to respect the principles of green chemistry. This new approach is even more interesting since these synthesis are carried out in bulk (Without solvent) and at room temperature during 4 hours, obtaining a better yield around 70%.

The polymerization of the monomers was catalyzed by Maghnite Na⁺. (Anionic polymerization) These monomers and polymers are characterized and confirmed by Infrared Spectroscopy (FTIR), ¹H and ¹³C nuclear magnetic resonance (NMR) spectroscopy.

Keywords: Green chemistry; Maghnite-H⁺; N-alkyl methacrylamide



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IODINE-PROMOTED SYNTHESIS OF AROYLBISINDOLIZINES FROM N-PHENACYL PYRIDINIUM SALTS AND ACTIVATED ALKINES

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Abstract

The indolizine ring is a key structural unit of molecules with a wide variety of applications as versatile building blocks for the synthesis of dyes, biological markers and pharmaceuticals [1].

In accordance with our interest in developing indolizine chemistry [2], a molecular iodine-catalyzed [3] 1,3-dipolar cycloaddition/oxidation/aromatization one-pot process with hydrogen peroxide as the terminal oxidant for the construction of indolizines under mild conditions is described. The reaction afforded a variety of products in good yields.

This strategy has overcome the inherent shortcomings of the 1,3-dipolar cycloadditions using conventional methods by taking advantage of the novel dehydrogenation reaction of the primary cycloadducts under the action of the mild oxidant molecular iodine.

In comparison with other reported methods using metal catalyst, this approach is less expensive and more practical.

Keywords: indolizine, 1,3-cycloaddition, iodine - catalysis

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SYNTHESIS AND STUDY OF FIELD HETEROCYCLIC DERIVATIVES CONTAINING NITROGEN ATOM

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Abstract

Metallic multi-link intramolecular transition reactions are one of the most effective approaches for preparing oxygenated heterocycles, which are important constituents in many biologically active compounds [1].

In this work, we synthesized halo-oxazolidinone from propargyl carbamates by intramolecular reaction using CuCl_2 as catalyst and N-halosuccinimide acid. These reactions can be performed using only amounts of N-halosuccinimide acid and only stoichiometric amounts of CuCl_2 .

We also discussed the synthesis of some derivatives of thiohydantoines which has a great biological interest and which goes into the synthesis of drugs.

All compounds were monitored by TLC, purified by the chromatographic column and identified by proton NMR

The objective we set for ourselves was to access, by simple, efficient and inexpensive synthesis methods, heterocyclic nitrogen compounds.

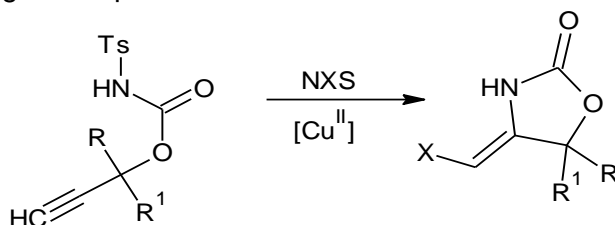


Figure 1. General procedure for the synthesis of halo-oxazolidinones by copper

Keywords: oxazolidinones, intramoléculaire catalytic reaction, thiohydantoines.

References:

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ACTIVATED CARBON FROM JUJUBE STONES FOR THE REMOVAL OF RHODAMINE-B FROM AQUEOUS SOLUTION

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Abstract

Activated carbon is widely used in industrial fields as an adsorbent for water treatment. Jujube stones are local agricultural by-product they were used in this study as a cheap precursor to produce activated carbon using a chemical activating agent H_3PO_4 (impregnation ratio of 1/2). Then, the impregnated precursor was carbonized at 500 °C under nitrogen atmosphere during 2 hours. the resulting activated carbon (JSh) was characterized by scanning electron micrograph (SEM), N_2 adsorption/desorption isotherms and Fourier transform infrared spectroscopy (FTIR). The results obtained showed a high BET surface area of 845.96 m^2/g and total pore volume of 0.828 cm^3/g . The adsorption capacity of prepared carbon was investigated by the removal of rhodamine B (RhB) dye from aqueous solution. RhB was chosen for the adsorption experiments due to its presence in the wastewaters of textile, leather, jute and food industries. The effect of different parameters such as pH, temperature and initial dye concentration on adsorption process were performed in batches.

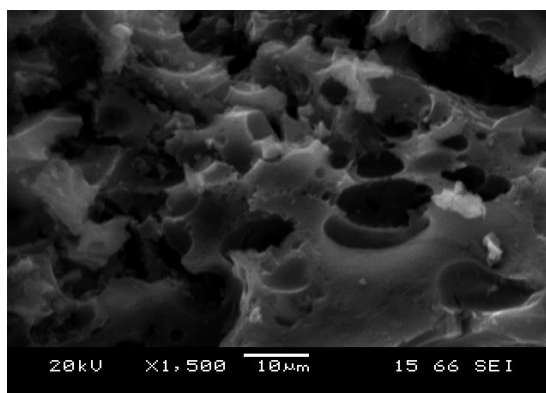


Figure 1. SEM image of JSh

Keywords: Activated carbon, Adsorption, Rhodamine-B

SYNTHESIS, CHARACTERIZATION, BIOLOGICAL ACTIVITY AND DOCKING STUDY OF NOVEL SCHIFF BASE

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Abstract

Schiff bases are some of the most widely used organic compounds. They are used as pigments and dyes, catalysts, intermediates in organic synthesis, and as polymer stabilisers. Schiff bases are aldehyde- or ketone-like compounds in which the carbonyl group is replaced by an imine or azomethine group. They are widely used for industrial purposes and also exhibit a broad range of biological activities [1].

In the present investigation, A Biologically active [2] of novel title compounds 2-[(2-hydroxy-4-nitrophenyl)methylidene]amino-3-methylbutanoic acid and [(2-hydroxyphenyl)imino]methylphenol Schiff bases were synthesized through Schiff reaction with high yields. Their structures were confirmed by means of ultraviolet/visible, infra-red spectroscopies, ¹H NMR, ¹³C NMR and elemental analysis.

In addition, these novel compounds were screened for their in vitro antioxidant activity employing 2,2'-diphenyl-1-picrylhydrazyl radical (DPPH) and ferric reducing antioxidant power (FRAP) scavenging assays. The inhibitory activities against *Escherichia coli* were investigated in vitro and molecular docking simulation also surveyed.

Keywords: Synthesis; Schiff base; biological activities.

References:

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SELECTIVE SYNTHESIS OXAZABOROLIDINE–BORANE REDUCTION OF INDANE-1,3-DIONE

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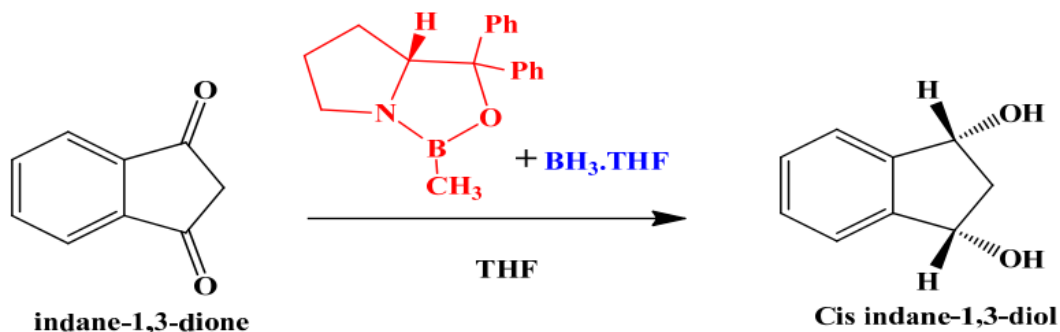
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Abstract

Cis-indan-1,3-diol was selectively synthesized in high efficiency via reduction of indane-1,3-dione using the reducing agent the borane tetrahydrofuran complex (BH₃-THF) in the presence of chiral oxazaborolidine as catalyst. The employed catalyst is (R)-2-methyl-CBS-oxazaborolidine. The reduction was carried out with a ratio of 1:2 diketone: borane in the presence of 60 mol% (R)-oxazaborolidine (molar solution) in toluene. The obtained isomer was confirmed by different spectroscopic methods (FTIR, ¹H NMR and ¹³C NMR) and elemental analysis. Furthermore, we have carried out a conformational analysis of cis-indan-1,3-diol and evaluated the role of the intramolecular hydrogen bond in the stabilization of various conformers using quantum mechanical DFT (B3LYP).



Key words: Asymmetric catalysis, Oxazaborolidine, Cis 1,3-indanediol.

A NOVEL AND GREEN PROCEDURE FOR THE SYNTHESIS OF SULFAHYDANTOINS UNDER ULTRASOUND IRRADIATION

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Abstract

Sulfahydantoins (3-oxo-1,2,5-thiadiazole-1,1-dioxides)¹ constitute as an emergent class of heterocycles, claimed as protease inhibitors (especially matrix proteinases) and ligand of MHC class II,^[1] aglycones in pseudonucleoside analogues,^[2] phosphate mimetic,^[3] or substructure in the constrained peptides.^[4]

In this work we have developed a highly efficient synthesis of new series of sulfahydantoins via intramolecular cyclisation starting from carboxylsulfamides derived from aminoesters, using ethanol as mild solvent, under ultrasonic irradiation.

The desired products were obtained in good yields, this method provided several advantages such as shorter reaction time.

The newly synthesized compounds were systematically characterized by IR, 1H NMR, and MS.

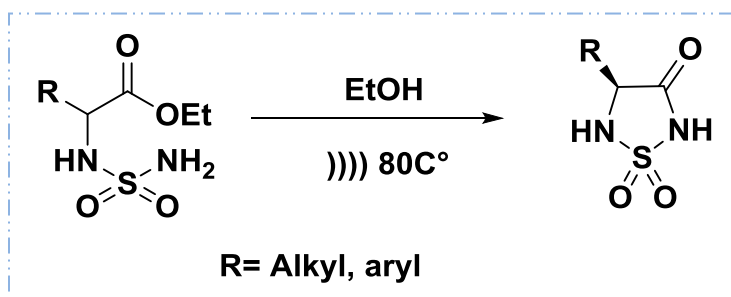


Figure 1. Synthesis of sulfahydantoins.

Keywords: Carboxylsulfonamides, ultrasound irradiation, intramolecular cyclisation.

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CHEMICAL SYNTHESIS AND STUDY OF H-BONDING INTERACTIONS IN FUNCTIONALIZED O/P-XYLYL IMIDAZOLIUM DICATIONIC IMIDAZOLIUM IONIC LIQUIDS

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Abstract:

Very recently; the chemistry of ionic liquids has garnered spectacular interest from the scientific community and have attracted considerable attention due to their attractive properties in various fields. Currently, however, only a few of Algerian laboratories are working on these compounds, most of them using conventional methods.

In this work, through an introduction of Xylyl group into the imidazolium cation, we changing the position (para or metha), four functionalized dicationic imidazolium ionic liquids eg; [p-C₆H₄(CH₂ImMe)₂⁺] containing four different anions as; [Cl⁻] [(CF₃SO₂)₂N⁻], [BF₄⁻] and [PF₆⁻] are synthesized. In order to avoid the presence of any impurities and to check the occurrence of the expected chemical reactions, the obtained functionalized dicationic imidazolium ionic liquids are characterized by ¹H-NMR, ¹³C-NMR, ¹⁹F, ³¹P-NMR and FT-IR spectroscopy.

The thermal analysis of these ILs indicated that the thermal decomposition occurs in the temperature range ≈ 300–450 °C, also, the changes in the chemical structure by functionalization and interactions lead to an enhanced stability of these compounds.

To obtain information on the inter molecular H-bonding interactions between anions and cations and explore conformational stability at molecular level, vibrational spectroscopy properties of our DILs were investigated by both techniques; infrared (FTIR-ATR) and FT- Raman spectroscopy.

Finally; a combination of computational study with experimental results of these DILs were performed using DFT calculations to localize these interactions and for better understanding role of H-bonding intermolecular interactions.

Key words: ionic liquid; hydrogen bonding; α,α' diimidazolium-p-xylene; conformers; imidazolium; Ft- Raman spectroscopy.

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NOVEL MULTI-COMPONENT SYNTHESIS OF AMRINONE ANALOGUES

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Abstract:

Development of new approaches to nitrogen Heterocycles ring is a topical area of continuous interest due to the high number of biologically active molecules containing these moieties.

The 2-pyridone¹ structure (figure 1) is an important heterocyclic framework that can be found in numerous biologically active compounds.

Recent interest in the 2-pyridone ring system has led to several new procedures for its preparation. Herein, we describe an efficient synthesis of functionalized 2-pyridones a new Amrinone² analogues via multi-component reaction under solvent-free conditions³⁻⁴.

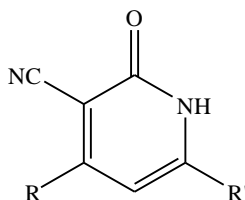


Figure 1. Structure of 2-pyridones

Keywords: Nitrogen heterocycles, multi-component reaction, 2-pyridone.

References:

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REACTION OPTIMIZATION OF THIOALKYLATION AND THIOARYLATION BY CLASSICAL WAY AND MICROWAVE

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^a Laboratoire de Chimie Fine, Université Ahmed Ben Bella, Oran1 Algérie

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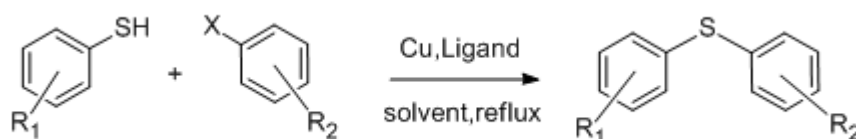
*Corresponding author 221halimasalem.adnane@gmail.com

Abstract

Our main focus is to optimize the yield of an alkylation and arylation reaction on a heteroatom, by classical way and microwave. This optimization is particularly fixed on the influence of certain reaction factors that are most often carried out in organic synthesis, namely physical and chemical parameters.

For our part, we have been interested in the reaction of S-alkylation and S-arylation that we can subject them under optimal conditions of clean reaction environment to promote a better yield. [1]

So, our choice is kinetically focused on the variation of different polar and non-polar solvents, different transition metal catalysts and different mineral and organic bases as a function of time and the temperature of the reaction.



X= I, Br, Cl
R= H, alkyl, aryl

Figure 1: general scheme of S-Arylation

Keywords: Optimization, classical way, microwave, S-alkylation, S-arylation

References:

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SIMULATION STUDY ON H₂S REMOVAL FROM ALGERIAN NATURAL GAS ON HIGH DENSITY POLYETHYLENE MEMBRANE

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Abstract:

A simulation study was carried out on the use of tubular membrane for the capture of H₂S from Algerian liquefied natural gas. A bed composed of dense microporous membranar particles of high density polyethylene was considered. Almeesoft simulator integrating Hysis membrane calculation was employed. A clogging time of 70 minutes, adsorption capacity of 2778 mg/g and a transmembranar pressure of 2.76 bars were calculated. Furthermore, the selectivity, the maximal gas pressure, the maximal natural gas concentration and the adsorption models were considered. Particular focus was put on the permeability between the membrane skin and the natural gas.

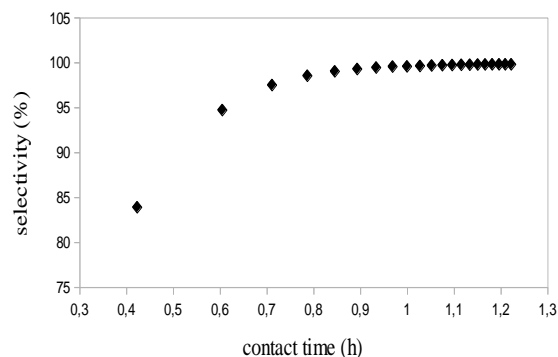


Figure 1. Effect of contact time on the selectivity of the HDPE membrane.

Keywords: Simulation, Desulfurization, Algerian natural gas.

References:

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SYNTHESIS, CHARACTERIZATION, CONFORMATIONAL AND COMPLEXATION STUDIES OF DI-SUBSTITUTED CALIX[4]ARENE DERIVATIVES BEARING N-ACYLHYDRAZONE UNITS

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Abstract

In the last years an increasing interest in calixarenes as potential complexing ligands metal cations, is observed, due to the fact that they can be pre-organized and tuned to complex metal ions. It is supported by several reviews [1-3].

A novel calix[4]arene derivatives with two N-acylhydrazone groups at the lower rim has been synthesized from di-acetohydrazone of *p-tert*-butylcalix[4]arene and α,β -unsaturated aldehydes via condensation reaction.

The macrocyclic N-acylhydrazone-calixarenes synthesized have been characterized by ¹H-NMR, ¹³C-NMR, FT-IR, Mass and UV-vis spectral data. The conformational properties of these compounds have been investigated in solution by variable-temperature ¹H-NMR spectroscopy.

Also complexation properties of previous compound towards divalent transition and heavy metal cations (Mn²⁺, Fe²⁺, Co²⁺, Ni²⁺, Cu²⁺, Zn²⁺, Cd²⁺, Hg²⁺) have been studied in solution using UV-Visible absorption spectrophotometry. The stability constants ($\text{Log } \beta_{ML}$), the molar extinction coefficients (ϵ) and stoichiometries of species formed in solution, have been studied and discussed. It has been observed that N-acylhydrazone-calixarenes have a good selectivity for heavy metal cations, especially for cadmium(II).

Keywords: Calixarene, N-acylhydrazone, Complexation.

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SOLVENT-FREE SYNTHESIS OF BIOLOGICALLY ACTIVE AMINOPHOSPHONATES UNDER MICROWAVE IRRADIATION

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Abstract

α -Aminophosphonates are probably the most important substitutes for the corresponding amino acids in biological systems [1]. The utility of the α -aminophosphonates as peptidomimetics, pharmacogenetic agents, antitumoral enzymatic inhibitors, antitumoral, antibiotics and pharmacologic agents are well documented [2,3]. The use of microwave irradiation has become a common heat source in organic chemistry. Inspired by this enormous success, the use of microwave irradiation is also increasingly studied for aminophosphonates synthesis. In the present study, an eco-friendly direct solvent-free synthesis of aminophosphonates is achieved by microwave irradiation of aminophenols derivatives, aromatic aldehydes and triethylphosphite via Kabachnik-Fields reaction. Heating with microwaves versus under classical conditions was shown to be higher yielding, cleaner, and faster. The title compounds were tested for in vitro antibacterial and antifungal activities at concentrations of 100-200 $\mu\text{g/ml}$.

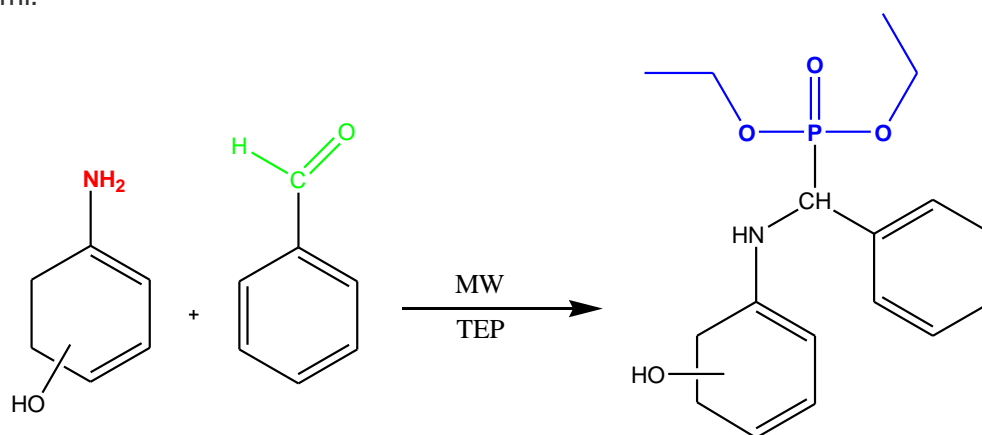


Figure 1. Synthesis of Aminophosphonates from Aminophenols.

Keywords: aminophosphonates, aminophenols, microwave irradiation, antibacterial activity, antioxidant activity.

References:

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VALORIZATION OF AN AGRO-FOOD WASTE FROM N'GAOUS ORANGE JUICE FACTORY IN WATER DEPOLLUTION

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Abstract

N'GAOUS factory of orange juice produced every year tones of orange pith and peel which represents a great potential of agro-food waste, so given the availability of this waste of low market value the valorization of this biomass in several areas including water depollution is indispensable.

To do this, we proceeded with the physicochemical characterization of the studied biomass, and then the accent was placed on the study of the adsorption of two dyes Methylene Blue (cationic dye) and Indigo Carmine (anionic dye) on the waste of oranges. The biosorption process was evaluated under the influence of various parameters, pH, biosorbent content and initial concentration of the solution.

The results obtained show that our biosorbent has an acidic character with a pH value of about 5.60 and a zero point of charge with a value of 4.94 and that it is rich in functional groups. The adsorption capacity of Methylene Blue (BM) reached its maximum at the pH range 6-10 after 90 minutes of treatment, however Indigo Carmine (IC) has a good adsorption capacity at acidic solution with elimination after 60 minutes. For the initial concentration ranges examined, the experimental data are well interpreted by the Langmuir model for Methylene Blue; however in the case of Indigo Carmine the Freundlich model describes better the data. The kinetics of adsorption of the two dyes was also studied using the pseudo-first-order and pseudo-second-order equations.

It should be noted that this suitability has also been an attempt to understand how organic pollutants are bound by this biomaterial in aqueous synthetic solution by the adsorption process and in light of the results obtained, it appears that the natural abundance of this food waste could offer a new and cheaper bio-adsorbent support and help to clean up colored water.

Keywords: valorization, agro-food waste, adsorption dyes.

SYNTHESIS, PHARMACEUTICAL FORMULATION and EVALUATION OF ANTI-ADDICTION OF NEW ANALOGUES OF BACLOFENE[®] AS ANTI-CRAVING AGENTS

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Abstract

Baclofen[®] is an old drug marketed for over 40 years. It is used as antispastic agent and muscle relaxant. The main problem of baclofen[®] that is administered as a racemate. However, since the R-(–) enantiomer is more active and toxic than the S-(+) enantiomer, also can cross (weakly) the blood-brain barrier. In our work, our main challenge is to synthesis The R-(–) enantiomer and optimize bioavailability, vectorization of baclofen[®], this by synthesizing new analogs (Figure 1). Later, our synthesis analogs are formulated with, native and amphiphilic cyclodextrin (CDs). Our choices is justified by the fact that the formulations with CDs proved to be efficient in providing a way to increase the solubility, the stability and/or other relevant physico-chemical properties of drugs at the same time. For evaluation of anti-addiction activity, we have developed new model in vivo, and the result are that our synthesized baclofen[®] analogues, showed an anxiolytic effect. Regarding the toxicity, our results showed that our analogues have less toxic effect than baclofen, it reduces the activity of TGO enzymes, TGP. From a histological point of view has no effect on the liver structure in addition to having a protective effect against lesions liverworts induced by alcohol [1].

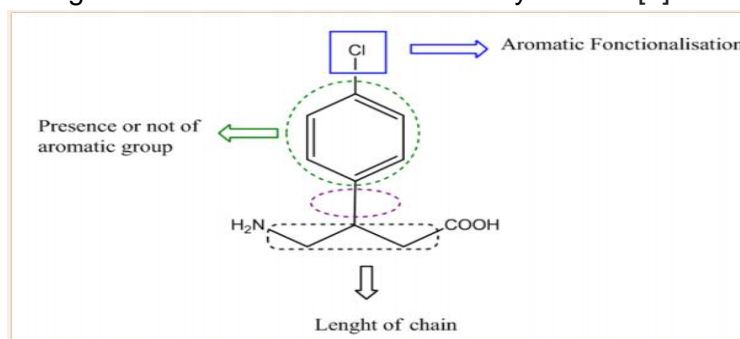


Figure 1. The targets modifications on the structure of baclofen[®].

Keywords: Baclofen[®], Synthesis analogs, Cyclodextrin.

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SYNTHESIS AND BIOLOGICAL EVALUATION OF MONO AND DISUBSTITUTED KETOAMIDES

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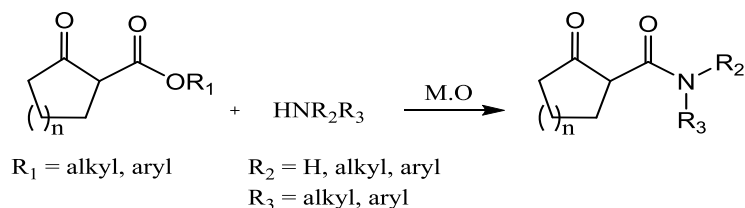
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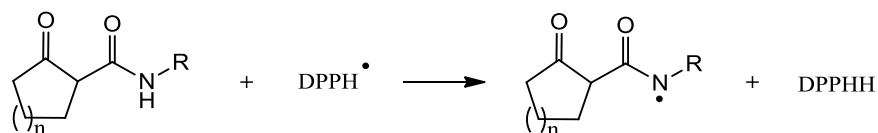
Abstract

The 1,3-dicarbonyl derivatives constitute a family of compounds grouping the 1,3- ketoesters, the 1,3-diketones, 1,3-diester, 1,3-ketoamides and 1,3-amidoesters. They have attracted considerable interest because of their recurring structural pattern in many natural products and biologically active molecules, without forgetting their synthetic utility in various transformations. Due to their amide function, 1,3-ketoamides are interesting nucleophiles because they have an additional site that can be involved in the formation of C-N bonds.

-In the context of the discovery of new antioxidant synthetic non-phenolic compounds, we are interested in studying the biological activity of keto-amide compounds. Thus, we first developed a new method of amidation of 1,3-ketoesters very practical and economical. The reaction is performed in dry conditions, activated by microwave radiation and it requires no catalyst. Our sequence has a major advantage over other classical reactions: it allows access to β -ketoamides with a relatively short reaction time.



-Currently, we are also interested in studying the antioxidant activity of these prepared ketoamides, since no study of their biological activity has been revealed in the literature. The method used to measure antioxidant activity is that of free radical scavenging using DPPH •.



Keywords: Ketoamide, Amidation, Antioxidant.

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ONE-POT SYNTHESIS OF IMIDAZO[1,5-a]IMIDAZOLE DERIVATIVES

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Abstract

One of the biggest challenges of contemporary organic chemistry is to develop green, efficient and economical synthesis methodologies to produce the desired molecule with quantitative yield and total selectivity.

In this work, we have adopted a new approach for the synthesis of Imidazo[1,5-a]imidazoles from a single reaction between 1,3-Oxazol-5(4H)-one derivatives and nitrogen nucleophiles [1,2,3], in order to participate in the emergence of this new concept of organic chemistry

Keywords: 1,3-oxazol-5-one, imidazol, one pot synthesis

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SYNTHESIS, CHARACTERIZATION AND ANTIBACTERIAL ACTIVITY STUDIES OF POLY(ESTER-AMIDE)

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Abstract

Antibacterial polymer is one of the important research orientations in polymer science for protection against bacterial infections. In the present work, we investigated the antibacterial activity of the synthesized poly(ester-amide) PEA. This polymer was prepared through the reaction of Poly(Ethylene Adipate) with bisoxazoline as chain extender. Spectral studies (FTIR, ¹H-NMR and ¹³C-NMR) magnetic susceptibility confirm the final compositions that were very similar to the initial feed compositions.

The in vitro antimicrobial activity of PEA was screened with the agar well diffusion method against bacterial strains such as *Staphylococcus aureus*, *Escherichia coli* and *Pseudomonas aeruginosa*.

From the results it was indicated that the prepared poly(ester-amide) possess antimicrobial activity against both gram-positive and –negative bacteria, The activity was varied according to the tested microorganism.

Keywords: poly(ester-amides); Characterization; antibacterial activity.

PHASE EQUILIBRIUM PROPERTIES OF BINARY SYSTEMS CONTAINING 1-BUTYL-3-METHYLIMIDAZOLIUM THIOCYANATE [BMIM][SCN], WATER, 1-PROPANOL, OR 1-BUTANOL) AT SEVERAL TEMPERATURES

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Abstract:

The growing awareness of the pressing need for greener, more sustainable technologies has focused attention on the use of atom efficient catalytic methodologies for the manufacture of fine chemicals and pharmaceuticals. Another aspect which is receiving increasing attention is the use of alternative reaction media that circumvent the problems associated with many of the traditional volatile organic solvents.

Green solvents are environmentally friendly solvents or biosolvents, which are derived from the processing of agricultural crops.

The use of petrochemical solvents is the key to the majority of chemical processes but not without severe implications on the environment. The Montreal Protocol identified the need to re-evaluate chemical processes with regard to their use of volatile organic compounds of VOCs and the impact these VOCs has on the environment. Green solvents were developed as a more environmentally friendly alternative to petrochemical solvents.

Alternative promising areas of research in the replacement of the use of VOCs in industry include the application of supercritical carbon dioxide and ionic liquids as alternative solvents.

Vapor-liquid equilibria (VLE) have been measured for water, 1-propanol and 1-butanol with the ionic liquids 1-butyl-3-methylimidazolium thiocyanate [BMIM][SCN] at temperatures between 273 and 363 K with the means of a static device.

The experimental VLE data were correlated using the PC-SAFT EoS. The binary interaction parameters k_{ij} were optimized on experimental VLE data. The results obtained for the all binary mixtures studied in this paper indicate that the PC-SAFT EoS can be used to represent systems containing ionic liquids. Additionally, NRTL model has been applied successfully to correlate the experimental vapor data for the three investigated systems.

Keywords: Vapor-liquid-liquid equilibria, Ionic liquids, PC-SAFT, NRTL, Correlation.

TOTAL SYNTHESIS OF MACROCYCLIC MARINE ALKALOIDS, HALICLAMINE A

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Abstract

The bis-3-alkylpiperidine^[1] is an alkaloid having one unsaturated carbon chain which was isolated for the first time from the sponge Chimney Haliclona Genus by Fusetani and Coll in 1989. Biological tests have shown that it has very interesting biological activities such as: cytotoxicity, antifungicity and antimicrobial against Gram-Escherichia Coli, or Gram + Staphylococcus Aureus^[2, 3].

In this work we have been interested in the total synthesis of an analogue of bis-3-alkylpiperidine by simple and effective reactions which are already done in our laboratory.

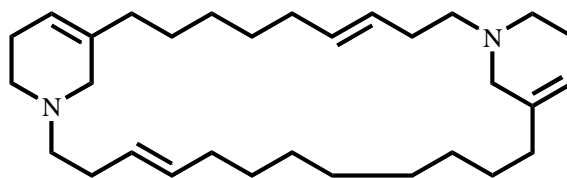


Figure 1. bis-3-alkylpiperidine

Keywords: bis-3-alkylpiperidine, Total synthesis, Antimicrobial, Antifungicity.

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SYNTHESIS AND PHARMACOMODULATION ALDIMINES: ALDIMINES 2-METHYL IMIDAZO [1,2-a] PYRIDINE-3-CARBOXALDEHYDE

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Abstract

The first preparation of aldimine from primary amines on 2-formylimidazo [1, 2-a] pyridine has been described since 1993. [1]

For our part, we chose to study the reactivity of 3-formyl-2-methylimidazo [1, 2-a] pyridine in the primary amines in order to obtain the corresponding imines.

The aldimine derivatives obtained were prepared under standard conditions by the action of the aromatic amine on the 3-formyl-2-methylimidazo [1, 2-a] pyridine obtained and hexamethylenetetramine in an acidic medium. [2]

The results of proton and carbon 13 NMR analyzes confirm the structure of the aldimine compounds obtained.

Keywords: aldimine, formylmethylimidazopyridine, hexamethylenetetramine.

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1,3,4-OXADIAZOLE, 1,2,4-TRIAZOLE and 1,3,4-THIADIAZOLE SYNTHESIZED FROM 2-FLUOROBENZOIC ACID: PHYSICO-CHEMICAL CHARACTERIZATION AND PRELIMINARY EVALUATION OF ANTIBACTERIAL PROPERTIES

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Abstract

Heterocyclic derivatives are now an essential family in antiviral and antibacterial chemotherapy. Their gradual development over the last 50 years has made it possible to stem many epidemics and provide effective treatments against bacterial and viral infections, despite these continuing to represent a major public health problem due to emergence of resistant strains. As such, the development of heterocyclic remains more relevant than ever. Among them, the five-membered nitrogen heterocyclic, 1,3,4-oxadiazoles and 1,2,4-triazole and 1,3,4-thiadiazole exhibit remarkable biological and pharmacological activity as antibacterial activities, antifungals, analgesics, anti-inflammatories and hypoglycemic and anti-HIV(1-3). In light of this, our objective is to synthesize new heterocyclic such as: 1,3,4-oxadiazole, 1,2,4-triazole and 1,3,4 thiadiazole, derivatives of 2-fluorobenzoic acid, to which it has undergone modifications in the carboxylic function and then a cyclization. The structures of the synthesized products have been elucidated thanks to the spectral data IR, H-NMR C-NMR, UV-Visible.

Finally our synthesized products are valued by the study of their antibacterial activity on pathogenic bacterial strains.

Keywords: 2-fluorobenzoic acid, heterocyclic derivatives, antibacterial activity.

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SHORT AND EFFICIENT SYNTHESIS OF PEPTIDOMIMETICS WITH SATURATED HETEROCYCLICS

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Abstract

Peptidomimetics and analogs have found wide application as selective and potent mimetics of natural peptides. Many studies have reported the development of linear and cyclic peptidomimetics with several important advantages compared to natural peptides such as increased bio-stability and affinity for the natural biological target. The preparation of peptidomimetics analogs is the subject of several researches which have been focused on changing the nature of the peptide skeleton with different modifications. Those modifications are used, for example to incorporate heterocyclic moiety or other building blocks with similar properties of peptide ligands ^{[1],[2],[3]}.

In this study, we describe an effective synthesis of new peptidomimetics molecules with a very good stereoselectivity which involves the incorporation of a heterocyclic unit into the chemical structure using various amino acids as precursors. Oxazolidinone has given rise to a considerable attempt because it belongs to a large class of saturated heterocyclic compounds; those derivatives are used as antibiotic agents such as Linezolid ^[4].

On the other hand, oxazolidinone heterocycles are also used as chiral auxiliaries in asymmetric synthesis ^[5].

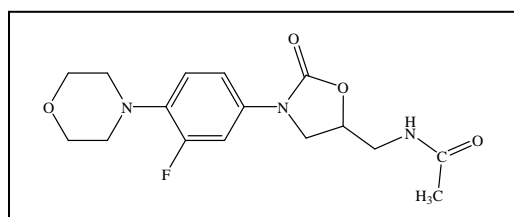


Figure: Structure of Linezolid

Keywords: peptidomimetics, oxazolidinone, amino acids.

References:

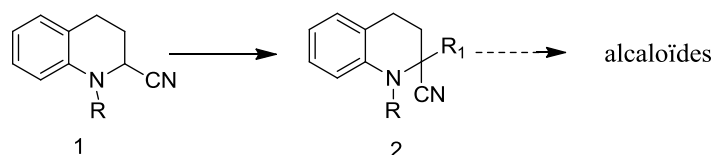
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SYNTHESE DES TETRAHYDROQUINOLEINES 2- SUBSTITUEES *via* L'-AMINONITRILEFadila LOUAFI,^a Saurabh SHAHANE^b, Jean-Pierre HURVOIS^b^a UR CHEMS, Université des Frères Mentouri Constantine, Route Ain El Bey, 25000 Constantine, Algérie^b Sciences chimiques de Rennes, UMR 6226 : CNRS–Université de Rennes 1, 12, rue Léon Bernard 35042 Rennes Cedex, France

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Abstract

Les quinoléines possédant un substituant alkyle en position 2 permettent la synthèse de nombreux produits dérivés comme l'angustueine, la galipinine, la cuspareine. Ainsi, la synthèse de ces composés est généralement effectuée par des méthodes de première génération utilisant comme étape clef la réduction diastéréosélective d'iminium ou d'acyliminium prochiraux.[1] A l'inverse, les voies de seconde génération (également appelées approches C₂-C₁) utilisent la condensation d' α -amino-carbanions et d'un agent électrophile.

Figure 1. Alkylation de l' α -aminonitrile

Dans ce contexte, l' α -aminonitrile **1**, constitue un précurseur commun à diverses quinoléines substituées en position 2. En effet, les α -aminonitriles sont généralement considérés comme des équivalents synthétiques d'ions iminium et à ce titre, peuvent être utilisés comme agents électrophiles dans les stratégies de première génération.[2] La présence d'un groupement nitrile permet également la formation d' α -aminocarbanions stabilisés susceptibles d'être condensés avec des agents électrophiles comme des halogénures d'alkyle.

La préparation par voie électrochimique de l' α -aminonitrile **1** sera détaillée au cours de la présentation et l'intérêt synthétique de la méthode sera illustré par la synthèse de divers alcaloïdes.

Keywords: α -aminonitrile, tetrahydroquinoline, alkaloids

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DETERMINATION OF THE MICROBIAL ACTIVITY OF A GLYCOSIDE ANALOGUE DERIVATIVE OF A CARBOXYLIC ACID (LACTIC ACID)

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Abstract

Lactic acid is the most widely existing carboxylic acid in nature. This acid has a very important role in many biochemical processes. It is not only found in milk and dairy products, but also used in food and pharmaceutical industries. This encouraged us to synthesize bioactive compounds derivatives of this well known carboxylic acid. The synthesis of new bioproducts is one of the greatest research challenges required in several areas. This work consists of the synthesis of new molecules, analogues of seco-acyclo glycosides. Starting with the synthesis of a five-membered heterocyclic compound, derivative of lactic acid, such as 3-(1-hydroxyéthyl)-4-amino-1,2,4-triazole-5-thiol. To achieve the aim of this work, it has been necessary to link the latest five-membered heterocyclic compound to a synthetic carbohydrate. Glycerol was taken as a seco-acyclo sugar moiety. The structures of all compounds have been characterized by infrared spectroscopy and nuclear magnetic resonance. The antimicrobial test of our products has shown an interesting activity against Gram-positive bacteria.

Keywords: glycoside analogue, lactic acid, amino-triazole.

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EFFECT OF PYRIDO [2,3-D] PYRIMIDINES ON T CELL PROLIFERATION

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Abstract

The pyrido [2,3-d] pyrimidines constitute the most described group of pyrimidines in the literature. This system is associated with a large biological effects such as kinase inhibition , anti-tumoral , anti-viral , anti-bacterial, anti-hypertensive , anti-inflammatory.

In this study, we examined the in vitro effects of these new pyrido[2,3-d] pyrimidines on the proliferative responses of human lymphocytes stimulated by mitogens at different concentrations (1–100 μ M) and on the intracellular oxidative status. Peripheral blood was obtained from five healthy non-smoking male (aged 25 years) donors, under no medication or food supplements intake and free of any known exposure to genotoxic agents. Fasting venous blood samples were collected in heparinized tubes. These samples were used for immediate lymphocyte isolation. The purpose of the study was explained to the volunteer subjects and their consent was obtained. The protocol was approved by the ethical com-mittee of the Tlemcen-University Hospital.

To determine the effects of the compounds synthesized, lymphocytes were incu-bated with different concentrations of P1. These compound was initially dissolved in DMSO (final solvent concentration 1%) and prepared immediately before use. The concentrations of each compound were adjusted in complete RPMI 1640 culture medium to yield the appro-priate final concentration (1–100 μ M). After incubation, cells were harvested by washing with RPMI 1640 medium. Cell viability was controlled by using a trypan blue exclusion test, and was unaffected by the compound con-centrations used in our experiments (greater than 80%).

The results showed that P1 at low concentrations (1–25 μ M) resulted in activation of ConA stimulated lymphocyte proliferation. However, at high concentrations (50–100 μ M), P1 induced a dose-dependent inhibition of lymphocyte proliferation .Changes in intracellular levels of reduced Glutathione, hydroperoxides and carbonyl proteins and in the activities of catalase and SOD were observed after P1 exposure reflecting a diminution oxidative stress specially at high concentrations.

In conclusion, P1 had significant immunomodulatory properties with diminution of oxidative stress at low concentrations.

Keywords: lymphocytes, oxidative stress, pyrimidines.

SYNTHESIS OF NEW SUBSTITUTED BIS-2-PYRIDONES DERIVATIVES FROM ENAMINONITRILES UNDER FREE-MILD CONDITIONS

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Abstract:

Heteroaromatic rings play an important role as the scaffolds of bioactive substances. It is well-known that the pyridone [1] and its derivatives are among the most popular *N*-heteroaromatic compounds integrated into the structures of many pharmaceutical compounds and the structural units occur in various molecules exhibiting diverse biological activities [2-4].

Solvent-free reactions represent very powerful green chemical technology procedures from both the economic and synthetic point. In our work, we developed a new method for an easier, simpler and more universal synthesis to prepare this type of heterocycles “bis-2-pyridones” derivatives, in which we employed, as a key step, the synthesis of enaminonitriles.

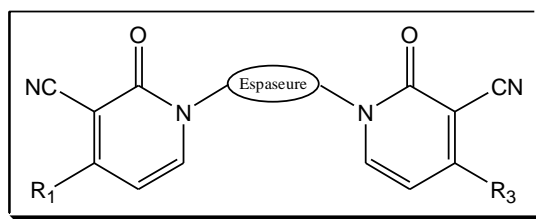


Figure 1. Structure of bis-2-pyridones

Keywords: 2-pyridones, enaminonitriles, free-mild conditions.

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KEY FACTOR AFFECTING THE BASICITY OF MESOPOROUS SILICAS MCM-41: EFFECT OF SURFACTANT EXTRACTION TIME AND SI/AL RATIO

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Abstract

Mesoporous silica Si-MCM-41 was prepared by hydrothermal method using TEOS and CTAB as the source of silica and structuring agent, respectively. The surface of the as-synthesized material was treated using HCl/ETOH solvent to remove the CTA surfactant instead of using the calcination. Characterization of the catalysts was performed using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), nitrogen sorption at 77 K, scanning and transmission electronic microscopy (SEM,TEM), and thermogravimetric analysis TGA. The catalytic properties of the prepared materials in the condensation of acetophenone with ethyl cyanoacetate were studied. The effects of the catalyst type, Si/Al ratio, reaction kinetics, and reaction temperature were also investigated to find an optimal parameter. The results show that an interesting yield was obtained (about 96%) in a short reaction time; it is found that the yields of products depend not only on the amount of surfactant inside the mesopores but also on the Si/Al ratio. The catalyst reuse shows that this catalyst can be used up to five cycles, and at temperatures higher than 50 °C, the yield of products decreases due to the slight destruction of the catalyst as confirmed by the XRD analysis. Based on the results obtained, a possible mechanism of the condensation reaction of acetophenone was proposed.

Keywords: Mesoporous silica, As-synthesized Si-MCM-41, Condensation of acetophenone.

EMULSION IONIC LIQUID MEMBRANE FOR RECOVERY PROCESS OF LEAD. COMPARATIVE STUDY OF EXPERIMENTAL AND RESPONSE SURFACE DESIGN

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Abstract

The technique of emulsion ionic liquid membrane (EILM) was used as chemical process for Pb(II) recovery, from nitrate medium, using Aliquat336 as ionic liquid carrier. The Tween20 as a dispersive non-ionic surfactant was used for the emulsion formation. The results showed that the lead ions were extracted at 82.61% by Aliquat336 and recovered at 82.16%, in aqueous solution of the nitric acid, from a feed phase of lead(II) nitrate of 207.2 ppm at pH equal to 5.5, in presence of 1% w/w Aliquat336 and 0.5% w/w of Tween20 under 30 min of stirring at 210 rpm. The tests of separation experiments of Pb(II) and Cu(II) were carried on the basis of the optimal conditions of lead (II) recovery. Thus, the separation factor of lead over copper was equal to 1.30, obtained from their equimolar synthetic mixture. Indeed, the recovery of Cu(II) can be advantageous towards of Pb(II) if the molar composition of Cu/Pb in mixture was of 0.65. Response surface methodology (RSM) using Box–Benheken Design (BBD) was used for the statistical study. So, the reduced cubic of the quadratic model showed that the predicted values were in good agreement with those found experimentally and the parameter of ionic liquid concentration has an important individual effect on the response. Therefore, the recovery of Pb(II) can be predicted at 82.14% with the best desirability of the chosen model under our experimental conditions.

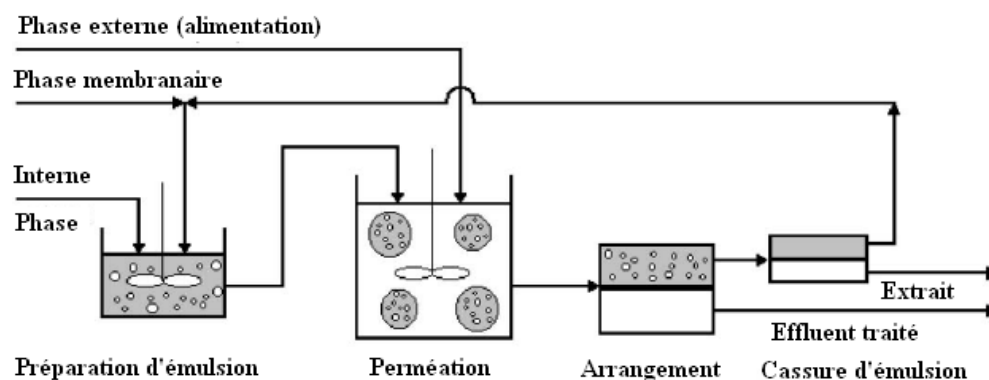


Figure 1. Representation of EILM

Keywords: Recovery, Lead, Aliquat336

SYNTHESIS OF ARYLIDENES PYRROLIDI-3-YL)-1,2,4-TRIAZOL-4-AMINE AS POSSIBLE CDK'S KINASES INHIBITORS

N. METAHRI ; A.SAAD EL HACHEMI AMAR ; A.SAFER

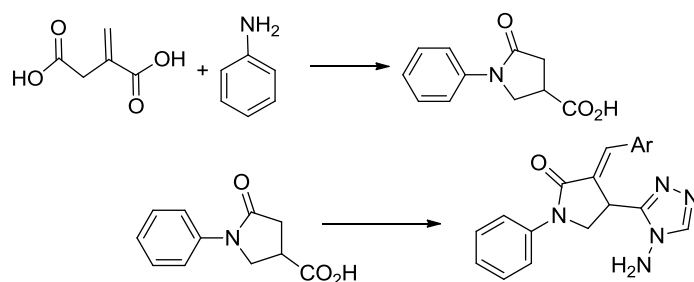
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Abstract

1,2,4-Triazoles nucleus and their derivatives have emerged rapidly with the advance of modern heterocyclic chemistry, promising a variety of medical applications such as antibacterial, antifungal, anticancer, antitumor, anticonvulsant, anti-inflammatory and analgesic properties¹ and are known for their activities as clinically used drugs, for example, fluconazole, itraconazole, terconazole. On the other hand, some studies on pyrrolidinone nucleus showed anti-HIV-1², inhibitors of carbonic anhydrase³ antimicrobial and anticoagulant activity⁴. Thereby, a series of novel compounds bearing substituted arylidene pyrrolidinone moieties and 1,2,4-triazole nucleus was synthesized as shown in scheme 1 in order to study their possible kinase inhibitor activity. The first results are very promising as kinase inhibitors for this kind of structures.



Keywords: 1,2,4-Triazoles, pyrrolidinone, kinase inhibitors.

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SYNTHETIC OF NEW POLYMERS FROM POLYSTYRENE MERRIFIELD VIA COOPER IODIDE CATALYZED CLICK CHEMISTRY AND APPLICATION IN TREATMENT OF WASTEWATER : EXTRACTION OF NITRATE AND METALS

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Abstract :

In order to remove metals and ions nitrate located in wastewater, a new series of clickable polystyrene Merrifield grafted with azide and alkynes were synthesized and implicated in this domain, the click chemistry based on the Huygens's reaction catalyzed by copper (I) was used in this synthesis. The new obtained polymers were containing 1,4-disubstituted triazole. The structure of these polymers was confirmed by infrared spectroscopy (ATR-FTIR), UV-visible spectroscopy and elemental analysis only for nitrogen element. Differential Thermal Analysis (ATD) and Thermo Gravimetric Analysis (TGA) were used to study crosslinking behavior of the polymers. These polymers were subsequently used in the extraction of metals and nitrate ions found in wastewater of Beja station (Tunisia). A comparative study of extraction of metal and nitrate ions by these supported triazoles was made.

Keywords : Polymers with 1,2,3-triazoles, Extraction of metals and nitrate ions, Wastewater.

ANTI TUMOR MOLECULES: SELECTIVE DESIGN FOR SYNTHESIS OF NEW HETEROCYCLIC COMPOUNDS CALLED FROG AND FOX CATALYZED BY MAGHNITE (ALGERIAN MMT)

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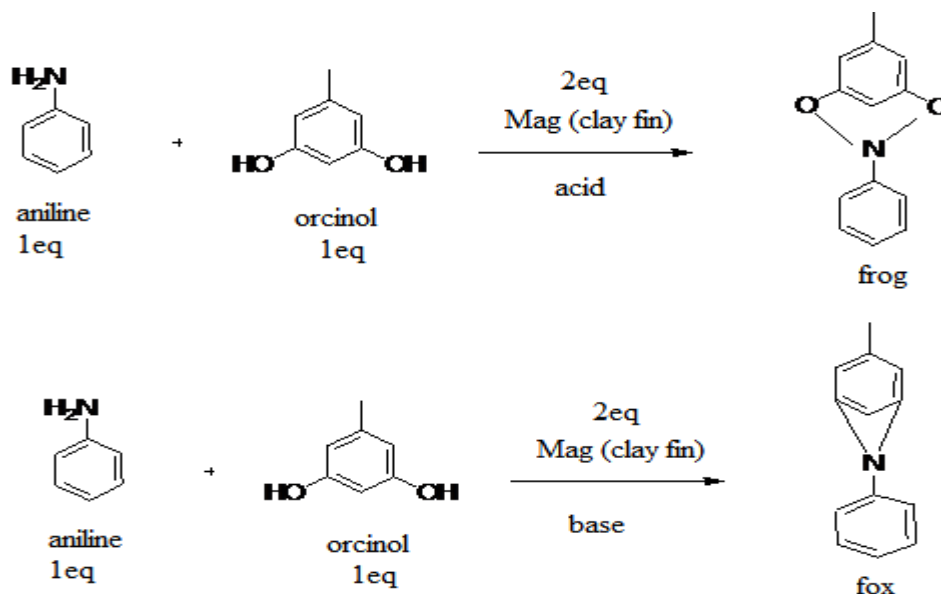
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Abstract

Phenolic compounds can be considered as important parameters of organoleptic (color, taste, aroma) [1] and nutritional qualities of food products of plant origin [2]. The use of phenolic compounds as natural antioxidants in food is in progress in relation to their role in prevention of some cancers [3] and cardiovascular diseases [4]. The aim of this work describes new and efficient strategy for synthesis of new heterocyclic compounds called frog and fox using Maghnite (Algerian MMT) as catalyst [5].

Keywords: Maghnite (Algerian MMT) ; heterocyclic ; design



Scheme 1. Design for new heterocyclic compounds called frog and fox catalyzed by maghnite (Algerian MMT)

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SYNTHESIS OF NEW PYRAZOLONE AND FUSED PYRAZOLONE HETEROCYCLIC DERIVATIVES

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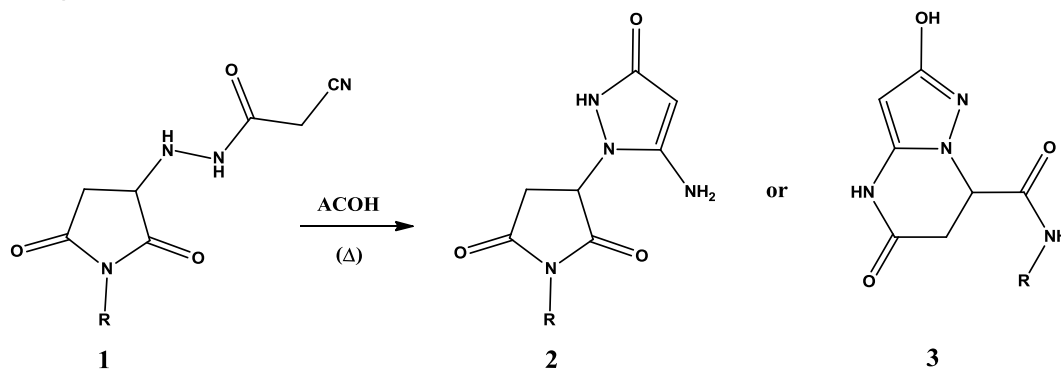
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Abstract

Compounds containing pyrazolone and fused pyrazolone heterocyclic moieties have attracted the attention of organic chemists due to their chemical structures as well as their biological activities, such as antimicrobial [1], anti-inflammatory [2] and anticancer activity [3].

As part of our ongoing research on the synthesis of bioactive compounds, herein we report the reactions of 2-cyano-N'-(2,5-dioxopyrrolidin-3-yl) acetohydrazide derivatives **1** toward acetic acid to lead to two different series of heterocyclic compounds, pyrazolones **2** and pyrazolopyrimidinones **3**, according to the experimental parameters and the structure of the precursor used. In this communication, the experimental procedures, the reaction mechanisms and the structures of compounds **2** and **3** will be described and discussed.



Scheme1. Synthesis of new pyrazolone and fused pyrazolone heterocyclic derivatives

Keywords: pyrazolone, pyrazolopyrimidinone, fused pyrazolone heterocyclic.

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CYCLIZATION REACTION OF THIOSEMICARBAZONE FOR FUNCTIONALYZED 4-THIAZOLIDINONES. EVALUATION OF ANTIOXYDANT ACTIVITY

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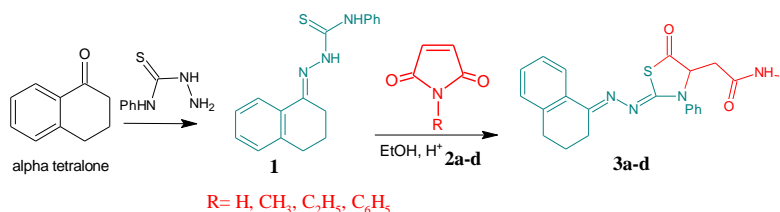
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Abstract

The increasing diversity of small molecule libraries is an important source for the discovery of new drug candidates. In this context, thiazolidinone are of great importance in modern medicinal chemistry due to their interesting biological activities.¹

In the present work, we report a new synthetic approach of the formation of 4-thiazolidinone compounds **3** involving the reaction of alpha tetralone with phenyl thiosemicarbazide and maleimide derivatives in the presence of catalyst. The thiosemicarbazone **1** was synthesized by refluxing a mixture of α -tetralone with phenyl thiosemicarbazide in acid medium.² Compound **1** react with maleimide substituted **2a-d** to give 4-thiazolidinones **3** in low yields as described in Scheme 1.



Scheme 1. Synthetic pathway to generate 4-thiazolidinone structures

The free radical scavenging effect of the 4-thiazolidinones **3** were evaluated in DPPH essay.

Keywords: maleimide, cyclisation, 4-thiazolidinone.

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SYNTHESIS, CHARACTERIZATION AND ANTIOXIDANT PROPERTIES OF NEW 2-AMINOPYRIDINE DERIVATIVES.

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Abstract

A series of novel substituted 2-aminopyridine and bis-2-aminopyridines delta-lactones derivatives were synthesized via condensation between delta-lactone enamionitrile and various aliphatic, aromatic, heterocyclic primary amines and diamines, the reaction was catalyzed by antimony trifluoride SbF₃. The structures of the new compounds obtained were characterized by various spectroscopic methods (IR, ¹H NMR, ¹³C NMR and MS). The antioxidant property of the products against DPPH radicals (DPPH•) were evaluated in vitro, in terms of their efficient concentration IC₅₀, as well as their reduction kinetics. Results showed a great antioxydant capacity for the most of compounds tested with a variation of IC₅₀ between 1.30- 3.61 mg/ml and times of reaction of 30 minutes. Besides, The scavenging activity of 7a to DPPH• was excellent among all of the tested samples, at 1.30 mg/mL. These data indicate that bis -2 aminopyridines derivatives have better antioxidant activities than 2-aminopyridines, and the scavenging effect indices are affected by the number and position of the amino group on pyridine grafted to the bis -2 aminopyridines derivatives.

Keywords: 2-aminopyridine, bis-2-aminopyridine, enamionitrile, antioxidant.

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SEM AND FTIR STUDY OF ADSORPTION CHARACTERISTICS USING XANTHATE (PIPX) SYNTHESISED COLLECTORS ON GALENA

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Abstract

Background: The interaction between collectors and surfaces plays an important role and the understanding of the interaction mechanisms of different reagents with mineral surface is significant in achieving selective flotation.

It is now widely accepted that there are two separate mechanisms by which collectors adsorb on sulphide minerals. Firstly, there is the chemisorptions mechanism where the adsorbed xanthate molecule forms a chemical bond with metal atoms at the sulphide surface. The other mechanism is electrochemical, and involves the electrochemical oxidation of the adsorbed collector molecules to give oxidation product species, which renders the sulphide surface hydrophobic. Andreev.G.N

Materials/Methods: The galena sample was crushed and the selected grains ground in an agate mortar. The galena fraction of -208+108 μ m was used in adsorption tests. The final grinding product (-208 +108 μ m) was used for the SEM microscopy examination. The pH was adjusted using HCl and NaOH. Xanthate (PIPX) solution prepared by dissolving the chemical grade PIPX collector in purified water. The purification of xanthate includes dissolving commercial grade xanthate in acetone and its crystallization.

Results: The interaction between collectors and surfaces plays an important role in understanding of interaction mechanisms of different reagents with the mineral surface. It is now widely accepted that there are two separate mechanisms by which collectors adsorb on the sulfide minerals. Firstly, there is the chemisorption mechanism where the adsorbed xanthate molecule forms chemical bond with metal atoms (Pb) at the sulfide (PbS) surface.

The other mechanism is electrochemical and involves electrochemical oxidation of the adsorbed collector molecules to give oxidation product species, which renders the galena surface hydrophobic. Results of galena surface oxidation to form hydrophobic and hydrophilic species depends strongly on potential. The rest potential measurement of galena activated with CuSO₄ 10⁻⁴M in 3.10⁻³M PIPX at pH 9

Conclusion: Using the SEM technique action of 3.10⁻³M potassium isopropyl xanthate has been identified (adsorption to specific surface sites and colloidal precipitation from solution).

The FTIR spectra revealed the presence of copper on the surface of galena and this is confirmed the adsorption of PIPX onto surface (Pb-PIX, 1123.08Cm⁻¹, (IPX)₂, 1273.56Cm⁻¹).

Keywords: Xanthate (PIPX), Adsorption, FTIR.

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ENCAPSULATION OF THE ASCORBIC ACID IN A MATRIX OF CELLULOSE TRIACETATE , AN ETHYL CELLULOSE AND THEIR CHARACTERIZATIONS

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Abstract

Biocompatible and biodegradable polymers are among the most used materials in several fields such as the health given their characteristics and chemical and physical properties which are a major asset for the sustained release of certain active substances.

In this work, the cellulose triacetate CTA was prepared from a natural polymer that is cotton cellulose to use in the release of ascorbic acid that is used as a model molecule. The medium use dis a synthetic medium recobstituted to simulate the gastric and intestinal environment. In the seconde part of this work, we prepared microspheres using commercially available Polycaprolactone PCL, ethyl cellulose EC, then associated with CTA in order to show the effect of these polymymers on the mass distribution of microsphers as well as on the kinetics of the dissolution of the antioxidant : ascorbic acid, which is very used in the medical and agri-food field.

The results obtained lead us to conclude that the medium pH =1.2 acid (gastric medium) is not appropriate for the release of ascorbic acid, against pH =7.4 of the colon(intestinal medium) is better adapted and the use of ethyl cellulose with CTA in microspheres gives a percentage of active ingredient more important and a prolonged release that is the goal. The microspheres were characterized by IR spectroscopic, DRX and SEM.

Keywords: cellulose triacetate, microsphere, prolonged release, ascorbic acid.

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NEW SYNTHESIS OF PYRAZOL-BARBITURIC AND THIOBARBITURIC ACID DERIVATIVES

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Abstract

Multicomponent reactions (MCRs) are one of the most powerful research protocols for generation of complex polyfunctionalized molecules using convergent one-pot transformations [1]. Nitrogen-containing compounds have been known to have a tremendous potential application in chemistry.

Pyrimidine-2,4,6-trione derivatives are an important class of nitrogen heterocycles that have attracted more attention in the last decade, which is due to their use as a precursor for the construction of condensed heterocyclic systems. They represent an interesting pharmacophore for pharmaceutical products [2].

Recently, Barbituric acid who can be involved in many reactions is used as a precursor in the synthesis of several compounds [3]. Pyrazole derivatives in the other hand are of particular interest, because of their pharmacological profile. They may possess some promising pharmacological activities once fused with other privileged structures.

In this work, we have synthesized with good yields a variety of pyrazole-thiopyrimidine-trione derivatives via one-pot multi-component reactions by a condensation of aromatic aldehydes with barbituric acid and 3-methyl-1-phenyl-1*H*-pyrazol-4(5*H*)-one. We have used as catalyst in this condensation the ascorbic acid. (**Figure.1**)

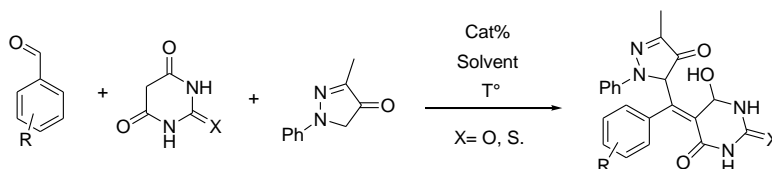


Figure 1. Synthesis Of 5-Arylidine Barbituric Acid Derivatives.

Keywords: Barbituric acid, Pyrazol-(thio)barbituric acid, Ascorbic acid.

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CHROMIUM (VI) ADSORPTION BY A NEW VINE-WOOD AND CONDUCTIVE POLYMER COMPOSITE

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Abstract:

Pollution caused by industrial waste water has become a common problem for most countries by the presence of certain heavy metals which can be carcinogenic and toxic., and may cause allergic dermatitis, skin irritation, liver cancer,

The aim of this survey is to test the efficiency of the adsorption of hexavalent chromium in an aqueous solution by using a local lignocellulosic biomass based on vine wood. To achieve this objective, a BV/ PANI lead composite was synthesized.

A series of infrared characterization was performed to determine the surface functions and scanning electron microscopy.

This adsorption has been studied by the kinetics of some influencing parameters, the necessary contact time to reach the reaction equilibrium, the mass of adsorbent BV / PANI and the determination of the adsorption isotherms so as to identify the mechanisms of retention .

Based on the results obtained, it's possible to conclude that the composite can be used for the removal of mineral pollutants such as chromium in water with moderate capital costs.

Keywords: Adsorption – Chromium (VI) - Composite

MOLECULAR DYNAMICS OF PROTON DELOCALIZATION IN CROWN ETHER

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Abstract

Crown ethers are macrocyclic polyethers with a cavity characterized to a large extent by the relative positions of the oxygen atoms in particular conformations. The flexibility of the backbone of crown ethers offers to a wide variety of guest molecules the possibility to penetrate and accommodate to form inclusion complexes. The complexation with atomic and molecular cations has been a central issue since the first synthesis of crown ethers by Pedersen more than four decades ago [1], and has remained a field of major importance both in fundamental and applied research areas.

In the present work, ab initio molecular dynamics calculations are performed to characterize the host-guest interactions and conformational dynamics of the complexes formed by the H_3O^+ and NH_4^+ cations with the native crown ethers 15-crown-5 and 18-crown-6 [2]. A particular focus is set on the proton delocalization effects unveiled in recent spectroscopy experiments for some of these complexes under well-defined isolated (solvent-less) conditions.

The calculations confirm that the infrared signature of these benchmark crown ether complexes provides a sensitive probe for proton delocalization [3]. The ab initio study also shows that proton delocalization occurs for the H_3O^+ host (and not for NH_4^+), provided that the size of the crown ring allows for tight H-bonding interactions between the cationic guest and the crown ether.

Keywords: Ab initio molecular dynamics, IRMPD spectra, Host-guest interactions

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SELECTIVE REMOVALS OF HEAVY METALS FROM AQUEOUS SOLUTIONS ONTO NATURAL MARNE CLAY (NMC) IN SINGLE, BINARY AND TERTIARY SYSTEMS

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Abstract

Natural Marne clay (NMC) is a new clay was used for the removal of heavy metal ions (Co(II), Ni(II) and Cr(III)) in aqueous solutions. Batch method was employed to study the influential parameters such as concentration of metal ions, pH, temperature and mixing. The adsorption isotherms and kinetics studies were carried out in heavy metal systems, respectively. The Langmuir isotherm model fitted to the equilibrium data better than the Freundlich model did, and the kinetics of the adsorption were well described by the pseudo-second order model. The selectivity sequence of the adsorption found as Co(II)>Cr(III)>Ni(II). For a cleaver charactering of the results, the clay have been using Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), X-ray diffraction (XRD) and BET. The results demonstrate the practical and economic advantages of using NMC in the decontamination of wastewater.

Keywords: Selectivity, Kinetic, Marne clay.

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SYNTHESIS OF NEW THIAZOLOPYRIMIDINE DERIVATIVES

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Abstract

The heterocyclic chemistry has made considerable progress, particularly the chemical structures that contain nitrogen and sulfur atom widely exist in nature. They play a vital role in the metabolism of all living cells, and present great interest in the theoretical and practical plan¹.

Among then, the skeleton which contains a thiazole ring, is associated with particularly wide range of biological properties such as anticonvulsant, antidiabetics, antihelminthics and also has depressant effect on the central nervous system².

In this present study, we are interested in the preparation of a new series of 7H-thiazolo[3,2-a]pyrimidine derivatives (Figure 1) in only one step and the use of the multicomponent reaction in green chemistry conditions.

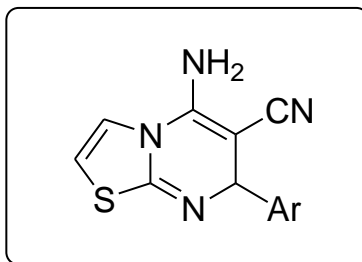


Figure 1. Thiazolo[3,2-a]pyrimidine derivatives structure

Keywords: Pyrimidines, Thiazolopyrimidines, Green chemistry.

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ULTRASOUND PROMOTED SYNTHESIS OF A ROSIN BASED IONIC LIQUID MONOMER

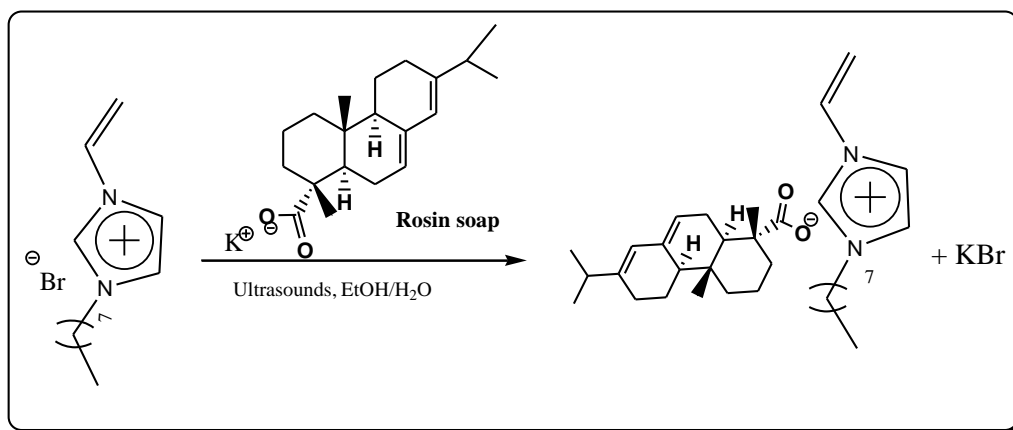
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Abstract

Rosin is a yellow-brownish solid form of resins obtained from pines and similar types of plants belonging to the conifer family. It is composed of a mixture of resin acids (mainly abietic acid) having the same functional groups (namely a carboxylic acid and carbone double bonds) [1]. Ionic liquids (ILs) are a class of ionic species which possess a melting point that is close to room temperature (up to 100°C). Their particular physicochemical properties have brought a growing interest through the last two decades [2]. They are non-volatile, highly thermally and chemically stable and possess a remarkable electrical conductivity. Those properties opened up a path to a broad range of applications as solvents, catalysts, extracting agents in separation processes and also as electrolytes to mention a few [3]. In this work we describe the ultrasound promoted synthesis of a polymerisable rosin based ionic liquid by an anion exchange methathesis reaction using a saponified rosin (**Scheme**). The FT-IR, ¹H and ¹³C NMR analysis confirmed the structures of the ILs. The thermogravimetric analysis (TGA) of the prepared ILs shows an improvement of the thermal stability with a raise of the degradation temperature (up to 50°C).



Scheme : Ultrasound promoted anion exchange methathesis reaction

Keywords: Ionic liquids, Rosin, Sonochemistry

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SYNTHESIS OF BIOLOGICALLY ACTIVE α -AMINOPHOSPHONATES

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Abstract

Organophosphorus compounds have a wide application in the industry, medicinal chemistry. In recent years, the synthesis of phosphonates compounds has attracted significant attention, due to their multiple uses in many vital areas.

α -aminophosphonates are the analogs of natural amino acid and some of them exhibit broad-spectrum biological activities, such as anti-HIV, antibiotic, antibacterial, and antiviral agents, anticancer, inhibition of activities of enzymes and anti-virus activities. Furthermore, α -aminophosphonates are used in agricultural industry as fungicidal, herbicidal agents and plant growth regulators.

A three-component synthesis of α -aminophosphonates by reaction of aromatic-substituted aldehyde, aromatic amine and triethylphosphite at 76 ° C catalyzed by HCl as organocatalyst with excellent yield and short reaction time. The structures of the synthesized derivatives were characterized and confirmed by the FT-IR and ¹H NMR.

Keywords: α -aminophosphonates, Synthesis, Biologically Active

EFFICIENT SYNTHESIS OF ACRIDINES CATALYSED BY COPPER

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Heterocycles possessing an aromatic core are key entities in numerous molecules of pharmaceutical interest, as well as in organic materials for a wide range of applications. Among them, acridins are of the highest interest¹.

Acridines are present in compounds having versatile pharmacological activities (anticancer, antibacterial, etc) including alkaloids from natural plants and marine organisms. Moreover, they are used as pigments and dyes, as well as in fluorescent materials².

A new method for preparing acridines in short reaction times was developed. The synthesis of these heterocycles was performed by combine copper-catalyzed N-arylation of 2-aminobenzaldehyd with various iodoaryls. Separate cyclization was also performed under acidic conditions to obtain acridins and related compounds.

Finally, some of the acidins such synthesized were evaluated for their antiproliferative activity

Keywords: Acidine; N-arylation; copper

AN EFFICIENT APPROACH FOR THE SYNTHESIS OF SOME NOVEL HYBRIDIZED QUINOLINE-PYRAN BASED TACRINE ANALOGUES

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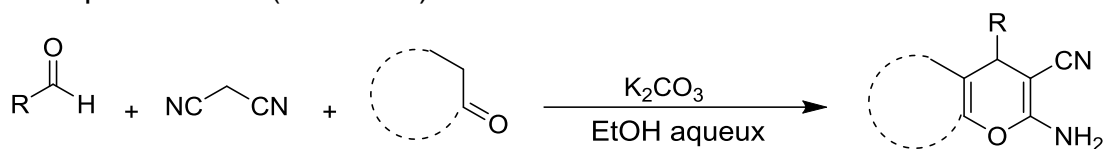
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Abstract

Heterocycles, an important class of organic compounds, constitute more than 70% of promising bioactive and drug molecules presently available in literature. Among these widespread heterocyclic compounds, Quinolines are an important class of heterocyclic compounds. Several compounds of this class have been screened for biological activities; among these are 2-chloroquinoline-3-carbaldehydes which occupy an important position, as the latter are key intermediates for further annulations of a wide variety of ring and for various functional group interconversions[1]. The synthesis of quinolines and their derivatives has been of considerable interest to organic and medicinal chemists for many years as a large number of natural products and drugs contain this heterocyclic nucleus.

In addition, the synthesis of 4*H*-pyran derivatives has attracted strong interest due to their different applications in medicinal chemistry because of their biological and pharmaceutical activities [2]. They bearing a free amine group at the 2-position and a nitrile group at the 3-position are very effective precursors for accessing Tacrine analogue derivatives[3] on one hand and pyranopyrimidine-4 (5*H*) derivatives on the other hand.

In this work, novel hybridized quinoline-4*H*-pyran compounds, highly functionalized and differently substituted, have been synthesized. The use of various activated heterocyclic methylene compounds has made it possible to introduce different groups of the 4*H*-pyran ring linked to the quinoleic unit (scheme 1).



Scheme 1

Keywords: Quinolines, One-pot synthesis, Tetrahydrobenzo[*b*]pyrans,

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