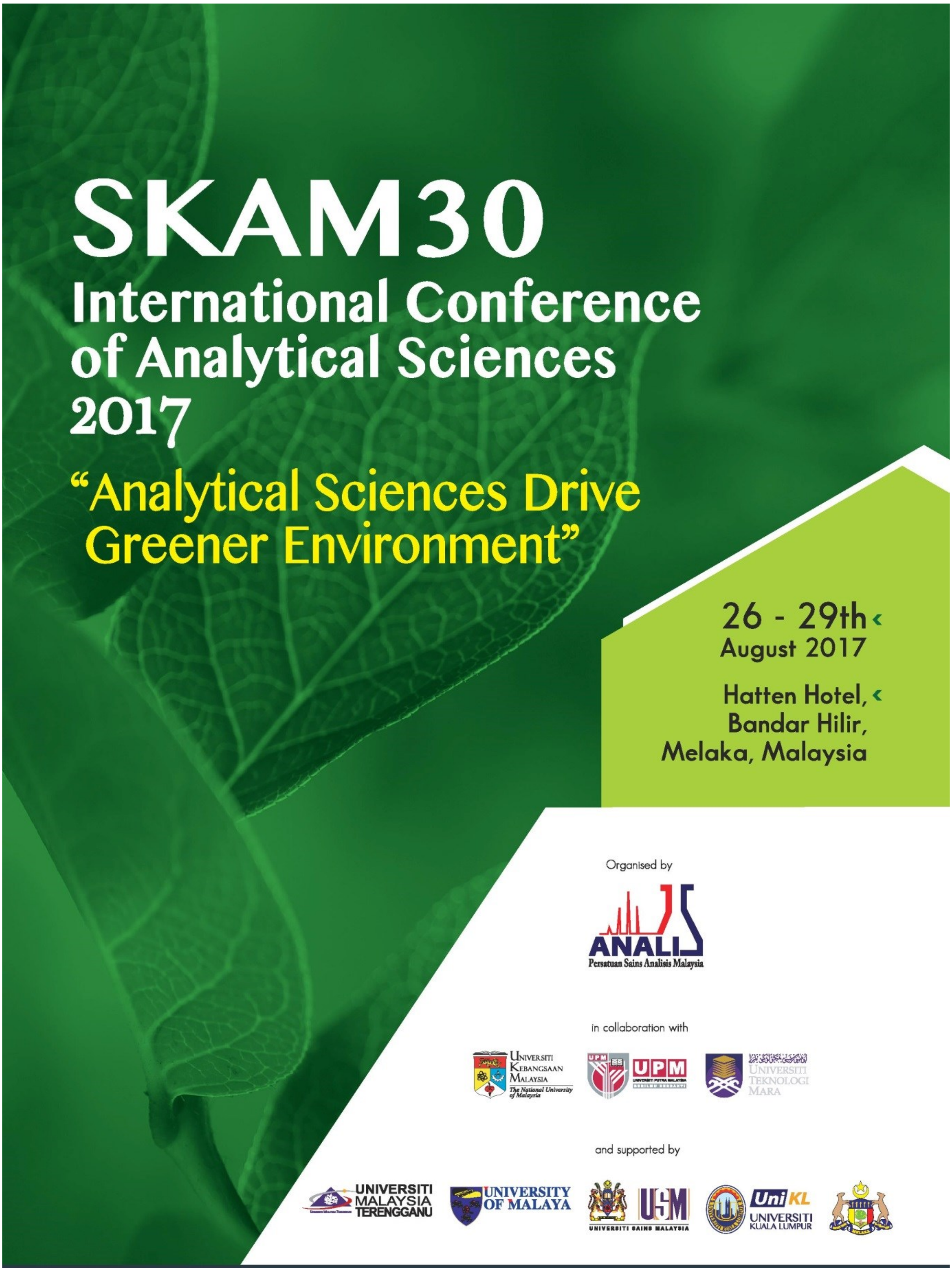

SKAM30

International Conference of Analytical Sciences 2017

“Analytical Sciences Drive Greener Environment”

26 - 29th
August 2017

Hatten Hotel,
Bandar Hilir,
Melaka, Malaysia

Organised by

ANALIS
Persatuan Sains Analisis Malaysia

in collaboration with

Universiti Kebangsaan Malaysia
The National University of Malaysia
UPM Universiti Putra Malaysia
Universiti Teknologi MARA

and supported by

Universiti Malaysia Terengganu
University of Malaya
USM Universiti Sains Malaysia
UniKL Universiti Kuala Lumpur

www.ukm.my/skam

International
Conference of Analytical Sciences
(SKAM30)

Organised by:

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Universiti Putra Malaysia
Universiti Teknologi MARA

Supported by:

Universiti Malaysia Terengganu
Universiti Malaya
Universiti Sains Malaysia
Universiti Kuala Lumpur
Kerajaan Negeri Melaka

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Foreword

Greetings from Malaysian Analytical Sciences Society!

On behalf of The Malaysian Analytical Science Society (ANALIS) and the organizing committee, it is an honour and a great pleasure to welcome all delegates to the International Conference of Analytical Science 2017 in conjunction with the 30th Symposium of Malaysian Analytical Sciences (SKAM30), being held at the Hatten Hotel, Malacca, Malaysia from 26th – 29th August 2017.

The 30th Symposium of Malaysian Analytical Sciences (SKAM 30) is jointly organized by ANALIS in collaboration with seven local universities namely Universiti Kebangsaan Malaysia, Universiti Putra Malaysia, Universiti Teknologi MARA, Universiti Malaysia Terengganu, Universiti Malaya, Universiti Sains Malaysia and Universiti Kuala Lumpur. This year, ANALIS is commemorating the 30th year of the SKAM event organization since its inauguration in 1987. This series of conference was initially known as the National Symposium of Analytical Chemistry (*Simposium Kimia Analisis Kebangsaan*, SKAK) until 1994 when SKAK was rebranded to SKAM in recognition of the rapid development in the field of analytical sciences in Malaysia. This year, echoing the spirit embraced by the State of Melaka where this conference is being held, the theme chosen for the conference is '*Analytical Sciences Drive Greener Environment*'.

The SKAM30 conference brings together industrial professionals, academic experts, researchers and graduate students to meet, interact and share their latest research findings in analytical sciences, environment and related technologies. It is by working collectively and collaboratively that the SKAM30 outcome will facilitate the move towards the sustainable development of the nation. The organizing committee has also provided ample opportunities for delegates to exchange ideas and application experiences face to face and develop networking among them throughout this conference.

Lastly, I would like to thank everyone (keynote and invited speakers, presenters, participants, sponsors, and the organizing committee members) who has contributed, directly or indirectly, to the success of the International Conference of Analytical Science 2017. Wishing you all a fruitful conference and a very enjoyable stay in Malacca.

Thank you

PROFESSOR DR. NORHAYATI MOHD TAHIR
Acting President of ANALIS

Welcoming Message

Greetings and Salam 1 Malaysia

It is my great pleasure to welcome all delegates and participants to the International Conference of Analytical Sciences (SKAM30) held in Melaka, Malaysia. This year's conference event will be a special occasion for the ANALIS in celebration of its 30 years achievement since its first event held back in 1987.

This year's conference theme is '*Analytical Sciences Drive Greener Environment*'. A diverse range of topics from analytical biotechnology, environmental analysis, radiochemistry and nuclear science, as well as green chemistry and technology studies are featured in the conference program. With more than 100 papers presented by local and international participants, I believe this conference will broadening your knowledge through excellent and fruitful discussion.

I would like to convey my special thanks to Melaka State Government, our sponsors and exhibitors for their strong support. My special thanks also to our organizing committee members for their hard work to ensure this conference a success. It has been a great honor for me to serve as the Chairman of SKAM30 and I would like to thank all participants for your participation. I wish you an enjoyable and memorable experience in this beautiful historical city of Melaka, Malaysia.

Thank you.

PROFESSOR DR. AMRAN AB MAJID
Chairman of SKAM30

Conference Background

The 30th Symposium of Malaysia Analytical Sciences (SKAM 30) is organised by ANALIS with Universiti Kebangsaan Malaysia (UKM), Universiti Putra Malaysia (UPM), Universiti Teknologi MARA (UiTM), as the main collaborators and supported by Universiti Malaysia Terengganu (UMT), Universiti Malaya (UM), Universiti Sains Malaysia (USM) and Universiti Kuala Lumpur (UniKL). SKAM 30 will be held on 26 – 29 August 2017 in Melaka, Malaysia. SKAM 30 also known as International Conference of Analytical Sciences 2017 commemorates the magic number of 30, being the number of times SKAM has been organised since its inauguration in 1987. This event brings together academics, scientist, researchers and students, to present and discuss the latest development and findings of their research in areas related to analytical sciences.

Scope of Conference

The symposium comprises of plenary, parallel and poster sessions. The topics to be covered are:

- Green Chemistry and Technology
- Environmental Analysis
- Extraction and Separation Sciences
- Spectroscopic Techniques
- Organic and Inorganic Synthesis
- Analytical Biotechnology
- Nano and Advanced Materials
- Catalysis and Industrial Applications
- Biomedical and Pharmaceutical Analysis
- Foods and Trace Analysis
- Natural Products Analysis
- Radiochemistry and Nuclear Sciences
- Oleochemistry and Polymers
- Chemical Sensors and Biosensors

Previous host of "Simposium Kimia Analisis Malaysia" – SKAK1 till SKAM30

Year	Institution
1987	Universiti Kebangsaan Malaysia
1988	Universiti Sains Malaysia
1989	Universiti Teknologi Malaysia
1990	Universiti Pertanian Malaysia
1991	Universiti Malaya
1992	Institut Teknologi MARA
1994	ANALIS and UKM
1995	Universiti Pertanian Malaysia
1996	Universiti Sains Malaysia
1997	Universiti Kebangsaan Malaysia
1998	Universiti Teknologi Malaysia
1999	Universiti Putra Malaysia Terengganu
2000	Universiti Putra Malaysia
2001	Agensi Nuklear Malaysia
2002	Universiti Sains Malaysia
2003	Universiti Malaysia Sarawak
2004	Universiti Teknologi MARA
2005	Universiti Teknologi Malaysia
2006	Universiti Putra Malaysia
2007	ANALIS and IKM
2008	Universiti Malaysia Sabah
2009	Co-Host with ASIANALYSIS X
2010	Universiti Malaysia Terengganu
2011	Universiti Teknologi MARA
2012	Universiti Kebangsaan Malaysia
2013	Universiti Malaysia Sarawak
2014	Universiti Teknologi Malaysia
2015	Universiti Putra Malaysia
2016	Universiti Sains Malaysia
2017	ANALIS

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Probing the Interfacial Solvation and Valence Electronicstructures of Biogenic and Organic Aqueous Aerosols: Implications in the Atmospheric and Environmental Sciences

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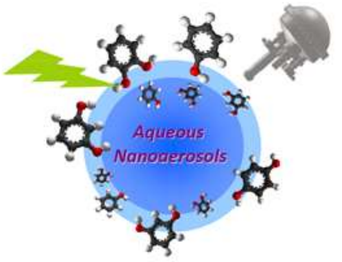
Abstract

Due to the ubiquitous presence of aqueous interfaces in nature, the interfacial properties at/near the aqueous interface are highly crucial in the aerosol science, atmospheric chemistry and environmental science. These surface-specific properties become even more crucial for nanoscaled aqueous aerosols as a result of increased surface-to-volume ratio with decreasing sizes. Therefore, an in-depth understanding of the fundamental energetic and structural properties at/near the interface of nanoscaled aqueous aerosols is essential to fully understand the impacts of organic species, either of biogenic or anthropogenic origins in influencing the cloud formation microphysics and the intrinsic nature of clouds. To address these crucial issues, we recently developed the aerosol VUV photoelectron spectroscopy and utilized this novel aerosol spectroscopy technique to investigate the valence electronic structures and interfacial solvation characteristics of several types of aqueous aerosols that are of particular significance in the atmospheric and marine sciences, including free amino acids [1], small peptides [2] and some amphiphilic organic species such as phenol and dihydroxybenzenes [3] in the form of aqueous nanoaerosols. Because the valence electronic structures and the ionization energies are the most relevant factors in determining how readily the outermost electrons can be removed, the pH-dependent valence electronic structures provide microscopic insights to understand the pH-dependent reactivity of aqueous nanoaerosols and the possible interplay between the amphiphilic organic solutes and water, especially at/near the liquid/vapor interface of aqueous nanoaerosols. The newly developed aerosol VUV photoelectron spectroscopy apparatus sheds new light for one to assess the valence electronic structures of nanoscaled aerosols, complex molecular assemblies and other novel nanomaterials in a size-selective and composition-controlled way. From the technical perspective, we demonstrate the aerosol VUV photoelectron spectroscopy apparatus as a powerful investigation tool to characterize valence electronic structures and interfacial solvation characteristics of aqueous aerosols under conditions highly relevant to the atmosphere, in terms of both aerosol size and temperature. Owing to the surface-sensitivity, the present aerosol VUV photoelectron spectroscopy technique also provides a novel way to characterize the surface pH, specifically for nanoscaled aqueous aerosols.



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[3] P.-C. Lin, Z.-H. Wu, M.-S. Chen, Y.-L. Li, W.-R. Chen, T.-P. Huang, Y.-Y. Lee and C. C. Wang, *J. Phys. Chem. B* 121, 1054-1067 (2017).

Challenges on Drinking Water Quality: Where Are We?

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Abstract

The demand for clean and potable water has increased in line with the rapid development and a growing population of Malaysia. This has created unique challenges to water resources availability and quality. The discovery of organic contaminants of distinctly human origin in the water cycle including drinking water has created a great deal of attention mainly due to their persistency and potential risk to human health. In addition to those chemicals generated by the treatment process itself (generally known as disinfection byproducts), the components of concern include those originate from pharmaceuticals and personal care products, which have known biological impacts to the environment including human. New developments in analytical instrumentations make detection of these substances in waste water, river and drinking water possible although at very low concentration levels of up to part per trillion (ng/L). Research performed by in Malaysia indicated the presence of various pharmaceuticals in surface water and in sewage treatment plant effluent. These include prescribed and also the over the counter drugs. Current treatment technologies used by nearly all local water and wastewater treatment plants were unable to degrade most of these chemicals. This means that the chemicals may have great possibility to remain in the treated water or water supplies to consumers. Although no regulatory requirement yet for the monitoring of pharmaceuticals in drinking water, a wide range of proactive actions could be implemented to reduce and eventually prevent the entry of these contaminants into the environment. Current lifestyle particularly waste disposal remains as one of the key factors to be handled in ensuring the security and sustainability of our scarce water supply. The newly drafted act on the drinking water quality (RUUKAM) by Ministry of Health Malaysia will be something that everyone is looking forward to. Imposing legislation concerning drinking water quality may contribute to better management of water supply and contaminants elimination.

Green Approach for Determination of Organo-Chlorine Pesticides in Aqueous Matrices using Magnetite-Polypyrrole-Reinforced Activated Carbon Solid Phase Extraction Sorbent

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Abstract

Agricultural waste is an emerging adsorbent for the removal of various pollutants from water as it is cheaper and more sustainable alternatives. In this work, modification of magnetite (Fe_3O_4) and polypyrrole (Ppy)-reinforced activated carbon from *oil palm empty fruit bunch (OPAC)* was studied to produce an alternative adsorbent composite, OPAC-Fe_3O_4-Ppy for the extraction of two organochlorine pesticides (OCPs) using magnetic solid phase extraction (MSPE) coupled with gas chromatography-micro electron capture detector (GC-μECD). The optimum conditions for OPAC-Fe_3O_4-Ppy composite synthesis were 1 g $FeCl_3$, 0.1 mL pyrrole monomer and 2:1 (w/w) magnetization precursor: OPAC ratio. Adsorption studies of the two OCPs (dieldrin, DIE and endosulfan, ESO) using OPAC-Fe_3O_4-PPy followed pseudo-second order reaction (chemisorptions process). The isotherm of OPAC-Fe_3O_4-PPy follows Freundlich model, suggesting multilayer adsorption. Extraction of DIE and ESO from different aqueous matrices (tap water, river water and palm oil mill effluent-POME) using OPAC-Fe_3O_4-Ppy MSPE was optimized. The OPAC-Fe_3O_4-PPy MSPE method gave LOD (3 *S/N*) as low as 10 ng/L for DIE and ESO respectively. Good linearity (20 – 1000 ng L^{-1}), correlation ($r^2 > 0.991$) and repeatability (< 6.79%) were obtained. Excellent DIE and ESO recoveries (98.56 – 102.97%) with acceptable RSDs (< 14%) showed the high capability of the OPAC-Fe_3O_4-Ppy sorbent for the extraction of the OCPs from the different aqueous medium. The developed OPAC-Fe_3O_4-PPy MSPE-GC-μECD method for OCPs analysis from aqueous matrices is an excellent green analysis method based on the Analytical Eco Scale (AES_{MSPE}: 79).

Green Synthesis of Nanoparticles

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Abstract

Over the past few years considerable interest has been focused on metal nanoparticles (NPs) due to the potential applications in different fields, such as catalysis, biomedical and sensor. Nanoparticles are of great interest due to their extremely small size and large surface to volume ratio, which lead to both chemical and physical differences in their properties compared to bulk of the same chemical composition. Therefore, design and production of materials with novel applications can be achieved by controlling shape and size at nanometer scale. The numerous preparation routes of nanoparticles can be grouped into two main different strategies: Top-Down (Physical methods); from large to small dimensions and Bottom-Up (wet chemical methods); from molecular scale to nanoscale. The Top-Down method is relatively easy, but the cost of fabrication is high. This approach is suitable for scale up to large scale fabrications, but the crystalline morphology and size distribution of particles obtained is highly broad. The development of efficient green chemistry methods for synthesis of metal nanoparticles has become a main attention of researchers. The green synthesis of NPs involves three main steps based on green chemistry perspectives; the selection of a biocompatible and nontoxic solvent medium, environmentally benign reducing agents, and non-toxic substances for stabilization of the nanoparticles. Nanomaterials have been synthesized using various routes and one of the most considered methods is production of NPs using organisms. Plants seem to be the best candidates and they are suitable for large-scale biosynthesis of nanoparticles. The advantages of using plant and plant-derived materials for biosynthesis of metal nanoparticles have interested researchers to investigate mechanisms of metal ions uptake and bioreduction by plants, and to understand the possible mechanism of metal nanoparticle formation in plants. Several green methods have been developed by our research group to synthesis silver and copper nanoparticles.

SKAM30 Program

	26 August 2017 (Saturday)
Time	**Activity**
4:00 – 6:30 pm	Early Registration

	27 August 2017 (Sunday)
08:00	Registration Arrival of Participants and Guests
09:00 – 9:30	Opening Remarks • Welcoming Remark by Prof. Dr. Amran Ab Majid (Chairperson, Organizing Committee) • Opening Remark by Prof. Dr. Norhayati Mohd Tahir (Acting President, ANALIS) *Venue: Ballroom*
9:30 – 10:15	**Keynote 1**: “*Probing the Interfacial Solvation and Valence Electronic Structures of Biogenic and Organic Aqueous Aerosols: Implications in the Atmospheric and Environmental Sciences*” Speaker: Prof. Wang Chia Chen (National Sun Yat-sen University, Taiwan) Chairman: Prof. Dr. Mohd Basyaruddin Abdul Rahman (UPM) *Venue: Ballroom*
10:15 – 10:45	Tea Break
10:45 – 12:45	Parallel Session 1 (A, B, C, D) Poster Session Exhibition
13:00 – 14.30	Lunch Break / Poster Session / Exhibition
14.30 – 16.30	Posters Evaluation Session Exhibition
16:30 – 17:00	Tea Break & Adjourn
20:00 – 22:00	**Opening Ceremony** by **YAB Datuk Seri Utama Ir. Hj. Idris bin Hj. Haron** **Ketua Menteri Melaka** Conference Dinner ANALIS Awards *Venue: Ballroom*

28 August 2017 (Monday)	
Time	**Activity**
8:30 – 9:15	**Keynote 2:** *"Challenges On Drinking Water Quality: Where Are We?"* Speaker: Prof. Dr. Md Pauzi Abdullah (UKM/ANALIS) Chairman: Prof. Dr. Amran Ab Majid (UKM/ANALIS) *Venue: Ballroom*
9:15 – 10.00	Talk Session with ANALIS's founders Chairman: Prof. Dr. Amran Ab Majid (UKM/ANALIS) *Venue: Ballroom*
10:00 – 10:30	Tea Break
10:30 – 13:10	Parallel Session 2 (A, B, C, D) Poster Session Exhibition
13:00 – 14:30	Lunch Break / Poster Session / Exhibition
14:30 – 15:30	Parallel Session 3 (A, B, C, D) Poster Session Exhibition
15:30 – 16:30	**ANALIS ANNUAL GENERAL MEETING** *Venue: Ballroom*
16:30 – 17:00	Tea Break & Adjourn

29 August 2017 (Tuesday)	
Time	**Activity**
8:30 – 10:50	Parallel Session 4 (A, B, C, D) Poster Session Exhibition
11:00 - 11:30	Tea Break / Poster Session / Exhibition
12:00 - 13:00	Award Presentations Closing Ceremony *Venue: Ballroom*
13.00 - 14:30	Lunch Break
14:30	End of Conference

Oral Presentation Schedule

	Day 1 (27 August 2017)			
Time (am)	**Room A**	**Room B**	**Room C**	**Room D**
10.45 – 11.05	**Plenary 1**	**OGC01**	**Plenary 2**	**ORN01**
11.05 – 11.25	**OES01**	**OGC02**	**ONA01**	**ORN02**
11.25 – 11.45	**OES02**	**OGC03**	**ONA02**	**ORN04**
11.45 – 12.05	**OES03**	**OGC04**	**ONA05**	**ORN03**
12.05 – 12.25	**OES04**	**OGC05**		**ORN05**
12.25 – 12.45	**OES05**	**ONP01**		**ORN06**

OES – Oral Extraction and Separation Sciences
OGC – Oral Green Chemistry
ONP – Oral Natural Product Analysis
ONA – Oral Nano and Advanced Materials
ORN – Oral Radiochemistry and Nuclear Sciences

	Day 2 (28 August 2017)			
Time (am)	**Room A**	**Room B**	**Room C**	**Room D**
10.30 – 10.50	**OCI01**	**OFT01**	**OEA01**	**OOI01**
10.50 – 11.10	**OCI02**	**OFT02**	**OEA02**	**OOI02**
11.10 – 11.30	**OCI03**	**OFT03**	**OEA03**	**OOI03**
11.30 – 11.50	**OCI04**	**OFT04**	**OEA04**	**OOI04**
11.50 – 12.10	**OCS01**	**OOP01**	**OEA05**	**OOI05**
12.10 – 12.30	**OCS02**	**OOP02**	**OEA06**	**OOI06**
12.30 – 12.50	**OCS03**	**OOP03**	**OEA07**	**OOI07**
12.50 – 13.10	**OCS04**	**OOP04**	**OEA08**	**OOI08**

OCI – Oral Catalysis & Industrial Application
OCS – Oral Chemical Sensor and Biosensor
OFT – Oral Food & Trace Analysis
OOP – Oral Oleochemistry & Polymer
OEA – Oral Environmental Analysis
OOI – Oral Organic & Inorganic Synthesis

	Day 2 (28 August 2017)			
Time (pm)	Room A	Room B	Room C	Room D
14.30 – 14.50	OEA9	OES06	OOI9	
14.50 – 15.10	OEA10	OAB01	OOI10	
15.10 – 15.30	OEA11	OES07	OOI11	
15.30 – 15.50	OEA12	OAB02	OOI12	
16.10 – 16.30	ORN07	OES08		
16.10 – 16.30	ORN08			

OEA – Oral Environmental Analysis
ORN – Oral Radiochemistry and Nuclear Sciences
OES – Oral Extraction and Separation Sciences
OAB – Oral Analytical Biotechnology
OOI – Oral Organic & Inorganic Synthesis

	Day 3 (29 August 2017)			
Time (am)	Room A	Room B	Room C	Room D
08.30 – 08.50	OEA13	OEA20	ONA07	OOI13
08.50 – 09.10	OEA14	OEA21	ONA08	OOI14
09.10 – 09.30	OEA15	OEA22	ONA09	OOI15
09.30 – 09.50	OEA16	OEA23	ONA10	OOI16
09.50 – 10.10	OEA17	OEA27	ONA11	
10.10 – 10.30	OEA18	OEA25	ONA12	
10.30 – 10.50	OEA19	OEA26	ONA13	

OEA – Oral Environmental Analysis
ONA – Oral Nano and Advanced Materials
OOI – Oral Organic & Inorganic Synthesis

Poster Presentation

ORAL ABSTRACTS

REMOVAL OF COD AND BOD IN DOMESTIC WASTEWATER USING MOVING BED BIOFILM REACTOR

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Abstract

This research used Moving Bed Biofilm Reactor that has the main advantage: shorter achievable retention time; and the capacity of domestic wastewater treated will be higher. The mechanism of the wastewater treatment process with biofilm process is done in the biological reactor (volume is 42 L) which is filled with medias (spesification is 124,31 m^2/m^3 and made of Polyamide) to cultures of microorganisms in aerobic conditions. This research was conducted through two stages, these are preliminary research (including preparation, seeding, acclimatization) and main research (operating reactor continuously with the variation of retention time 45 minutes, 1hour, 1.5 hours, and 3 hours). The retention time of 3 hours is the optimum retention time due to the concentration of all parameters resulted has quality standards based on the Ministry of Environment and Forestry's Standard for wastewater Nr. P.68/2016. The research results the highest removal efficiency of COD is 83.33%, 87.33% of BOD, 81.5% of Ammonia 79.2% of Nitrate, 93% of Nitrite, 85.82% of Oils and Greases. Parameters kinetic on the substrate by using approach of Complete Mix type, COD removal efficiency was resulted Kd=0.0006/h, Y=0.19 mg VSS/mg COD, qmax=0.02l/h, K=0.0006l/mg.h, Ks=119.14mg/l. Meanwhile, for BOD was resulted Kd=0.0002 h, Y=0.64 mg VSS/mg BOD, qmax=0.04l/h, K=0.0005l/mg.h, Ks=89.61mg/l.

Keywords: biofilms, domestic wastewater, fluidized, *Moving Bed Biofilm Reactor,* media, biofilms, organic materials.

TOFU INDUSTRY WASTEWATER TREATMENT USING BIOREACTOR ANAEROB-AEROB BIOBALL MEDIA WITH VARIATION OF RETENTION TIME

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Abstract

Food industry such as Tofu, in their production generates wastewater. Before discharge wastewater into the waterbody, the wastewater should be treated to reduce the possibility of negative impact and the contamination of the waterbody. For these small industries, the best alternative of wastewater treatment is one which has the following criteria: easy in operation, low cost operation, low volumes of sludge produced, and can be used in high concentration wastewater. In this research, bioreactor anaerobe-aerobe with media bioballs is used. The highest removal efficiency of COD took place on anaerobe zones. Reactors were operated with variations of retention time, 24 hours, 18 hours, and 12 hours. The results of this research are the removal efficiency of COD for 24 hours, 18 hours, 12 hours retention time are about 90,3% (the highest removal organic loading is 15,1 kg COD/m^3.day); 84,4%; 76,3%. The experiments showed that the longer of the retention time, the higher of removal efficiency could be reached. These occurred because the longer of retention time will extend the contact time between wastewater and microorganisms attached. Therefore microorganisms have longer time to degrade organic matter in wastewater. Although in the three-detention time, removal efficiency was high, the effluent of reactor was still above of effluent standard (Kep-51/MENLH/10/1995). Kinetics using Eckenfelder Equation results R^2=0.9991 n = 0.293 dan K = 7,3577 mg/L.

Keywords: tofu wastewater, anaerobe, aerobe, bioball, wastewater, treatment, attached growth

BIOSTABILIZED PALLADIUM SUPPORTED ON MAGNETIC NANOPARTICLES: CATALYST FOR THE COPPER-FREE SONOGASHIRA COUPLING REACTION

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Abstract

Palladium catalysis has achieved an impressive place in numerous commercial chemical processes. In particular, palladium nanoparticles (PdNPs) due to its high surface area to volume ratio showed high reactivity and made them a powerful catalyst for organic transformations. Green synthesis of PdNPs employing plant extract has been suggested as possible eco-friendly alternatives to chemical and physical methods. Ligand assisted method employing functionalized ligands will covalently anchor the PdNPs thus controlling the PdNPs particle size and preventing agglomeration. In this research, 3-(2-aminoethylamino)propyl trimethoxysilane(AEAPTS) functionalized silica-coated magnetite supported biosynthesized PdNPs was synthesized. The biostabilized PdNPs magnetic catalyst were characterized using HRTEM-EDX, XRD, AAS and Zeta Potential analyses. The catalyst was then tested in the copper-free sonogashira reaction between phenylacetylene and iodobenzene to yield diphenylacetylene. Effect of temperature, base and solvent on the reaction conversion were investigated and monitored using gas chromatography-flame ionization detector (GC-FID).

Keywords: palladium nanoparticles, biosynthesis, magnetite, copper-free sonogashira reaction

THE EFFECT OF TEMPERATURE ON THE CATALYTIC ACTIVITY OF SYMMETRICAL TETRADENTATE ALIPHATIC PALLADIUM(II) SCHIFF BASE COMPLEXES

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Abstract

Palladium complexes have been extensively studied due to their outstanding catalytic performance in many chemical reactions, especially in coupling reactions. Their excellent catalysis has been linked partly to the properties of the ligands coordinated to them. However, most of the ligands that are commonly employed are phosphines which are known as environmentally harmful, cost-ineffective and unstable in air and moisture. The application of Schiff bases, specifically *ONNO* tetradentate ligands coordinating to palladium(II) has offset the aforementioned problems. Thus, the present work aims at the synthesis of four aliphatic Schiff bases namely, 2,2'-(((2,2-dimethylpropane-1,3-diyl) bis(azanylylidene))bis(methanylylidene))bis(4-chlorophenol) LC, 2,2'-(((2,2-dimethyl propane-1,3-diyl)bis(azanylylidene))bis(methanylylidene))bis(4-methoxyphenol)LOMe. The Schiff bases were reacted with palladium(II) acetate yielding four palladium(II) complexes namely PdLC, PdLN, PdLMe and PdLOMe. The compounds were characterized through elemental analysis, IR, NMR, UV-Visible, magnetic susceptibility, molar conductivity and melting point. The complexation was indicated to have occurred due to shifting of characteristic v (C=N), v (C-O) and v (HC=N). This was supported by appearance of new v (M-N), v (M-O) and disappearance of v (O-H). The diamagnetic and non-eletrolytic nature of the complexes suggest square planar d^8 palladium(II) geometry and absence of counter ions, respectively. The catalytic activity of the palladium(II) complexes was tested in Sonogashira reaction between iodobenzene and phenylacetylene in DMSO at 80 ^{o}C, 100 ^{o}C and 140 ^{o}C. The percent conversion of iodobenzene was monitored by GC-FID where the samples were taken at 12 hour. At 100 ^{o}C, the best performing catalysts were PdLN and PdLC, indicating that electron withdrawing substituents, $-NO_2$ and –Cl, enhanced catalytic properties of the complexes. At 140 ^{o}C, all palladium(II) complexes successfully converted 100% of iodobenzene after 12 hours of reaction.

Keywords: Schiff bases, palladium(II) complexes, temperature, catalysis, Sonogashira

ALUMINA SUPPORTED Ru/Mg/Ce CATALYST FOR CO_2 METHANATION REACTION: OPTIMIZATION AND PHYSICOCHEMICAL STUDIES

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Abstract

The century of the urbanization and industrialization have gave a great impact on the environment due to more flue gases sectors present which contributed to the emission of CO_2 gas. It was well known that CO_2 gas was one of the greenhouse gases and lead to the earths' climate change. A green and recycling technology should be used in order to convert the CO_2 gas into CH_4 gas, which can also be used as alternative fuel in generating the electricity. Thus, the resources like coal and natural gas used can be reduced as well. Ce was acknowledged with its highly basic and unique redox properties. Therefore, Ce was used as based catalyst and impregnated with Ru/Mg to boost the activity. The catalysts were supported on alumina with different ratios and calcination temperatures of 500°C-700°C for 5 hours. In this study, the potential catalyst was $Ru/Mg/Ce/Al_2O_3$ catalyst with the best working conditions for catalytic methanation reaction was 60 wt% of Ce loading at calcination temperature of 600°C. This catalyst was able to convert about 79.04% of CO_2 with 58.08% of CH_4 formation. The characterization results obtained suggest that CeO_2, $Mg(Al_2O_4)$ and RuO_2 species were the active species for the potential catalyst.

Keywords: methanation, power plant, flue gases, carbon dioxide, catalyst, ceria

SYNTHESIS AND PHYSICOCHEMICAL PROPERTIES OF NANO-MAGNETITE (Fe_3O_4) AS A SUPPORT FOR RECYCLABLE CATALYST

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Abstract

The utilization of recyclable catalyst is an inevitable trend with the development of green chemistry. Recently, magnetic nanoparticles (MNPs) have been used widely by modification of the MNPs surface due to easily separated from the reaction media by applying an external magnetic field. The interest in this research is to synthesis the magnetically-recoverable nanoparticles as a solid support for palladium(II) Schiff base complexes. In this study, magnetically separable iron oxide nanoparticles was successfully synthesized via co-precipitation method from ferrous and ferric solutions. The physicochemical of this support was characterized by various spectroscopic techniques such as Fourier Transform Infrared (FTIR) spectroscopy, Thermogravimetric analysis (TGA), X-ray diffraction analysis (XRD), Scanning Electron Microscopy (SEM) connected to an energy dispersive spectrophotometer (SEM-EDX), and Brunauer Emmett Teller (BET) to determine surface area and porosity.

Keywords: magnetite support, recyclable catalyst, supported Pd(II) complex

FUNCTIONALIZED ORGANIC SEMICONDUCTOR MATERIALS FEATURING ACETYLIDE-THIOUREA DERIVATIVES AS CHEMIRESISTIVE CO_2 SENSING

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Abstract

Hybrid molecular system of acetylide and thiourea generally provides a wide range of electronic properties as they exhibit electronic delocalization in extended like-rod rigid π-orbital molecular system in their designated molecular framework. In this occasion, new members of 4-methylbenzoyl-3-(4-ethynylbenzonitrile-phenyl)-thiourea (**T1**) and 4-methoxybenzoyl-3-(4-ethynylbenzonitrile-phenyl)-thiourea (**T2**) were successfully designed, synthesised, and characterised for their spectroscopic, thermal analysis, electrochemical, and semiconductor properties. In turn, both **T1** and **T2** sensor candidates were fabricated onto interdigitated electrodes (IDEs) by drop-casting method followed by drying process in an ambient environment to remove any remaining solvent. The surface morphology of **T1** and **T2** on IDEs were investigated via Scanning Electron Microscopy (SEM) to show uniform distribution of **T1** and **T2** on IDEs. The performance of **T1** and **T2** on ionic conductivity, sensor response and reproducibility characteristic towards carbon dioxide (CO_2) at different concentration (10 ppm – 500 ppm) for 30 minutes of time exposure have eventually been measured and investigated at room temperature, via changes in resistivity measurement before and upon CO_2 exposure. Additionally, theoretical studies on **T1** and **T2** towards CO_2 gas were evaluated via density functional theory (DFT) at B3LYP/6-31G (d,p) TD-SCF level of theory. The unique properties of both **T1** and **T2** are due to the presence of two potential active site of –NH-C=O and acetylide C≡C for H-bonding and Van der Waals interaction to take place between the molecule and CO_2 analyte. The sensing behavior of both active sites were evaluated in term of interaction and stabilisation energy that give an effective evidence for sensing ability between **T1** and **T2** towards CO_2. In fact, the response of sensing properties was also obtained from the difference in Mulliken charge distribution of **T1** and **T1**-CO_2; **T2** and **T2**-CO_2 to provide mechanism response of **T1**, **T2** with CO_2. Theoretical results revealed that –NH-C=O site of **T1** and **T2** is a stable interaction site for sensing process to occur.

Keywords: acetylide, thiourea, chemosensor, Gaussian, chemiresistive

MODIFICATION OF 2,2'-BIPYRIDINE SILVER ELECTRODE FOR METAL IONS DETECTION

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Abstract

Polymerized 2,2'-bipyridine-PVC membrane was constructed as an ionophore reagent material for chemically modified silver (Ag) electrode in the determination of selected metals ions. The characterization of modified electrode was done using cyclic voltammetric technique where the electrochemical behaviour of 2,2'-bipyridine modified Ag electrode surface was investigated in the range of -1.0 to +1.0 V with a scan rate of 100 mVs^{-1} and in the presence of 0.1 M KCl as supporting electrolyte. Comparative studies towards metal ions of Cu^{2+}, Fe^{2+} and Pb^{2+} exhibited good redox reaction responses with linear relationship obtained between peak currents and concentrations. The correlation coefficients of the electrode-metal ions responses were 0.9694, 0.9965 and 0.9936 for Cu^{2+}, Fe^{2+} and Pb^{2+} respectively. Meanwhile, the evaluated limit of detection (LOD) for Cu^{2+}, Fe^{2+} and Pb^{2+} measurement were 5.41×10^{-5} M, 5.58×10^{-6} M and 4.18×10^{-6} M respectively.

Keywords: PVC membrane, 2,2'-bipyridine, cyclic voltammetry, redox reactions PVC membrane, 2,2'-bipyridine, cyclic voltammetry, redox reactions

A BIOSENSOR FOR HYDROGEN PEROXIDE BASED ON IMMOBILIZATION OF HORSERADISH PEROXIDASE ON ACRYLIC MICROSPHERE

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Abstract

This paper presents a development of hydrogen peroxide biosensor based on acrylic microspheres. Hydrophobic poly(*n*-butyl acrylate-*N*-acryloxysuccinimide) [poly(nBA-NAS)] microspheres were synthesized using photopolymerization in an emulsion form and it work as an enzyme immobilization matrix. The acrylic microspheres and hydrogen peroxidase enzyme were immobilized together *via* covalent bonding. Field emission scanning electron microscope (FESEM) has been utilized to characterize acrylic microspheres and modified HRP/nBA-NAS/AuNPs/SPE electrode. Differential pulse voltammetry has been used to determine the biosensor performance. The linear response range of hydrogen peroxide biosensor has been obtained from 1.0×10^{-2}M to 1.0×10^{-12}M. The limit of detection (LOD) hass been calcualted to be 5.96×10^{-20}M ($R^2 = 0.99$) . The good response of the biosensor can be utilized for further development of double or multiple enzymatic biosensor and other applications.

Keywords: horseradish peroxidase, acrylic microsphere, hydrogen peroxide biosensor

FORMATION OF CARBOXYL-CALIXARENE MONOLAYER ON GOLD SURFACES: AN ELECTROCHEMICAL SENSING PLATFORM TOWARDS DETECTION OF LEAD

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Abstract

Calixarenes which is one type of cyclic oligomer have attracted a great deal attention as receptors in cation sensing due to their rigid structure that provide a scaffold to be incorporated into functional groups and a central cavity that allowing the macrocycle to bind target analytes with high specificity. In this study, functionalized carboxyl-calixarene namely 5,17,dinitro-11,23-ditert-butyl-25,27-dicarboxymethoxy-26,28-dihydroxycalix[4]arene was modified on gold screen printed electrode through self-assembly technique. Differential Pulse Voltammetry (DPV) measurement has demonstrated that the modified gold SPE was highly selective towards lead ion compared to other studied ion which are zinc and nickel. The confirmation of modified electrode with calixarene has been investigated by Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Photoelectron Spectroscopy (XPS) methods. The parameters of deposition time, deposition potential and supporting electrolytes were varied in order to optimize the electrode performance. Under optimized condition, the limit of detection lead ion was found to be 29.17 μg/L in concentration range of 1-10 μM with RSD of 8.33% for its reproducibility. The developed sensor also able to retain its performance in presence of several metal ions as interferent. For simultaneous study, the fabricated sensor acquired LOD of 46.56μg/L specifically in presence of zinc and nickel ion. The sensitivity of the modified calixarene electrode towards lead was found to be satisfactory as it had passed the permissible level permitted by WHO, thus seems promising for real sample application.

Keywords: calixarene, electrochemical sensor, heavy metal

ASSESSMENT OF RARE EARTH ELEMENTS IN ROCKY SHORE ECOSYSTEM USING BIOINDICATOR

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Abstract

This study stresses on the assessment of REEs in rocky shore, describing their partitioning in selected body parts of chosen bioindicator and considering inter-tissue and interspatial variability. Concentrations of 14 naturally occurring REEs were measured using ICP-MS, along with standard reference material validation as well. The REEs fractionation patterns normalized to chondrite were remarkably similar, indicating a common source of the REEs for the whole region. Consistent enrichment of LREEs over HREEs were discovered in all samples, implies that REEs are transported as a coherent group through aquatic ecosystems. There are variations in the REEs abundance for each site, but they demonstrate similarities in their distribution patterns, which propose that they are of parallel origins. Variation in concentration was observed in all samples with significant differences associated with sites, suggesting interspatial variability. Comparison with permissible limits showed that the values are considerably below the safety limit, except for Ce and Nd.

Keywords: rare earth elements, rocky shore, bioindicator, soft tissue

STUDY OF POLLUTANTS ELEMENTS LINKAGES BETWEEN SEAWATER-MARINE SEDIMENT-MARINE BIOTA IN MANJUNG COASTAL WATER AREA, PERAK, MALAYSIA

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Abstract

Rapid socioeconomic development in Manjung area poses the risk to the pollution of the Manjung coastal water area. One of the main causes of marine pollution is the uncontrolled release of pollutants such as heavy metal elements through anthropogenic activities. The pollutants transported to the atmosphere and deposited back over the land or run off to the river and flow into coastal water area, accumulate in reservoir and finally enter the food chain. The concentration of pollutants contained in the marine ecosystem can be adversely affect human health and the environment. Linkages between seawater-sediment-marine biota potential acts as indicator of pollution level and health risk to the human population of the study area. Therefore, this study focused on determination of elements (Cd, Cr, Cu, Pb, As, Se, Fe and Zn) in seawater, marine sediment and marine biota from Manjung coastal water area in order to assess pollution level, health risk and linkages. Thirty six (36) locations of seawater and marine sediment samples were taken along the coastal water area of Manjung. Meanwhile, five types of marine biota were collected near Manjung coastal included *Pseudorhombus malayanus* (Ikan Sebelah), *Arius maculates* (Ikan Duri), *Johnius dussumieri* (Ikan Gelama), *Portunus pelagicus* (Ketam Renjong) and *Charybdis natator* (Ketam Bunga). The determination of elemental concentrations was carried out using Energy Dispersive X-Ray Fluorescence (EDXRF) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The elements of Fe and Cu are in the category of low pollution level, while elements Cr, Zn and Pb in the category of medium pollution level, whereas elements Cd, As and Se in the category of high pollution level. The significant high risk was found for consumption of marine biota due to high concentration of elements Cr, As and Pb but the risk depends on intake rate per day and the species of biota consumed. Based on transfer factor value, the accumulation of pollutant elements in marine biota is mainly through seawater. Hence, it is important to carry out regular monitoring of pollution level in Manjung coastal water area in order to provide information health risk assessment for human population in the study area.

Keywords: metal, pollution, Manjung coastal

SEASONAL VARIATIONS OF SELECTED METALLIC ELEMENTS CONCENTRATION IN SURFICIAL SEDIMENT OF SUNGAI TERENGGANU, TERENGGANU, MALAYSIA

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Abstract

Sungai Terengganu is rural with a relatively pristine environment in the upstream catchment area becoming urbanized and industrialized downstream with the major settlement of Kuala Terengganu city at the river estuary. Recently, huge amount of pollutants including metallic elements had been discharged to the estuary system due to these urbanization and industrial activities. Therefore, in order to assess the pollution status in term of metallic elements, a study is carried out to determine the concentration of selected metallic elements in the recent sediment of Sungai Terengganu estuary. A total of 61 samples were collected in October 2016 and April 2017 respectively to represent pre-monsoon and post-monsoon seasons. Inductively Coupled Plasma Mass Spectrometer (ICP-MS) was used to analyze the samples after were digested with mixed acid in a closed Teflon Bomb digestion. Generally, the mean concentration of metallic elements studied namely, Pb, Zn. Cu. Cr and As in post-monsoon season was much higher when compared to the pre-monsoon season. The surficial sediments at the river mouth contained high concentration of metallic elements during both pre and post-monsoons. This also proved by assessment with the Pollution Load Index (PLI) where the calculated value also suggested that the mouth of estuary is the most contaminated area. These pollutants maybe derived from the increasing of anthropogenic activities at the area. Further monitoring should be implemented for a better future development of the area.

Keywords: Sungai Terengganu, metallic elements, sediment, closed acid digestion, pollution load index

DETERMINATION OF COPPER (II) COMPLEXATION IN SEAWATER AT PULAU PERHENTIAN, TERENGGANU

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Abstract

The profile of seawater samples was analyzed from a few selected stations at Pulau Perhentian, Terengganu for Cu(II) speciation determination in order to determine the concentration level of Cu(II) in dissolved seawater. The concentration of natural organic Cu(II)-ligand binding (CuL) and its conditional stability constant (Log K_{CuL}) in each sample were determined by using competitive ligand exchange–adsorptive cathodic stripping voltammetry (CLE-AdCSV) method with salicylaldoxime (SA) as competitive ligand. Our present data indicated the presence of natural organic Cu(II) complexing ligands in all of our seawater samples. Its distribution was similar to the dissolved Cu (dCu) throughout the water column, where the concentrations increased from the surface (3m) to the middle layer (15m), before decreasing at the bottom layer. The CuL and dCu concentrations ranged between 3.65 – 18.95 nM and 1.28 – 16.58 nM, respectively. The Log K_{CuL} ranged between 12.11 – 12.92 throughout the water column and this indicated the presence of strong natural organic ligands in our study area. This initial study on Cu(II) speciation analysis in this area has suggested that more than 99% of dissolved Cu was complexed to the natural organic ligands which prevents the toxicity of Cu(II) to the marine organisms.

Keywords: speciation, trace metal, CLE-AdCSV, monsoon, Cu toxicity, organic ligands

MACRO AND MICRO ELEMENT IN THE DIFFERENT PARTS OF *Rhizophora apiculata* AT KUALA SEPETANG MANGROVE FOREST

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Abstract

A total 50 samples of different parts (root, stem disk, bark, trunk, twig and leaves) from five individuals of 20-year-old *Rhizophora apiculata* were sampled at Matang Mangrove Forest, Kuala Sepetang, Perak. The aims of this study are to determine the concentration of selected macroelements (K, Mg, Na and Ca), microelements (Al, Fe, Zn, Mn, Cu, Co and Ni) and nonessential elements (Pb and Cd) in different parts of *Rhizophora apiculata*. The elemental analysis was conducted by using inductively conductive plasma-optical emission spectrometer (ICP-OES). The mean value of studied elements were recorded in the decreasing order of Ca > Na > K > Mg > Mn > Al > Fe > Zn > Cu > Pb >Ni >Cd > Co. The highest value of Ca than other elements in the major parts of *Rhizophora apiculata* is closely related to the role of this element in the cell wall formation. The variable concentration of elements in the different parts of *Rhizophora apiculata* is attributed to the absorption mechanism of the trees. High values of some macroelement than microelements suggest that macroelement is more significant for the growth of *Rhizophora apiculata*. The lower mean value of Pb and Cd than other elements suggest that non-essential elements are not significantly involved in the major process of this mangrove species. This study is crucial in providing a preliminary data of the different type of element in the different part of the *Rhizophora apiculata* species, and it is important for the future development of charcoal industry in Kuala Sepetang, Perak.

Keywords: mangrove forest, *Rhizophora apiculata*, macroelement, microelement, non-essential elements

SEASONAL INFLUENCES ON THE LEVELS OF DISSOLVED AND PARTICULATE Cd, Cr AND Pb IN KUANTAN RIVER, PAHANG

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Abstract

Temporal and spatial variations of selected toxic elements distributions were studied in the Kuantan River waters. Water samples from 9 sampling stations were taken from downstream of the estuary towards the upstream of Kuantan River. Cd, Cr and Pb were analyzed using ICP-MS after being passed through the Chelex-100 and Teflon Bomb digestion processes for dissolved and particulate fractions, respectively. Dissolved elements concentrations measured were varied from not detectable (ND) to 0.037 µg L-1 for Cd, 0.309 to 4.981 µg L-1 for Cr and 2.011 to 19.983 µg L-1 for Pb, respectively. The concentration of particulate Cd, Cr and Pb were in the range of ND to 14.480 mg L-1, 6.047 to 271.497 mg L-1 and 0.646 to 174.859 mg L-1, respectively. The distribution of the elements in dissolved and particulate fractions were found influenced by the rainy season and low-rainy season. Higher concentration of the dissolved elements was found mainly in September 2102 and particulate elements concentration mostly higher in June 2012. The toxic elements studied in Kuantan River waters were still below the Interim Marine Water Quality Standard (IMWQS) permissible limits. The order of Log Kd found in this river, Pb > Cr > Cd confirmed that Pb and Cr have the strongest affinity to become suspended particulate matter and suggested that dissolved phase was Cd as major carrier in the water columns.

Keywords: dissolved metals, suspended particulate matter, toxic metals, river water, cadmium, chromium, lead

LOWER TEMPERATURE DURING MIS-3 IN SOUTHWESTERN TAIWAN WATERS AS REVEALED BY ALKENONE-DERIVED SST RECORDS

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Abstract

We established the estimated sea surface temperature (SST) record based on the alkenone-paleothemometry in samples retrieved from core MD103264 offshore southwestern Taiwan covering the last 45 kyr. $U^{K'}_{37}$-derived SST of coretop sample (27.8°C) is comparable to the modern late spring SST (27.8°C). The published report from nearby location revealed that the alkenone-producing organism of *Emiliania huxleyi* and *Gephyrocapsa oceanica* were predominant in winter and spring. $U^{K'}_{37}$-derived SST varied between 23.3 and 27.8°C with large estimated temperature different (~3°C) was clearly documented during MIS-3. Diunsaturated alkenone ($C_{37:2}$) was relatively higher and more fluctuated compared to the triunsaturated alkenone ($C_{37:3}$). However, the concentration of both alkenones tended to be lower in the interval between 8 and 0 ka than the intervals of early and late MIS-3. A cold temperature in MIS-3 was relatively lower than the value of MIS-2 and LGM. We suppose that $U^{K'}_{37}$-temperature may reflect the changing interaction between Asian Monsoon and Kurashio Current (KC). The fluctuated temperature during MIS-3 suggests that the environmental change during MIS-3 is slightly different than the recent interglacial interval, and lower estimated temperature during MIS-3 might be due to the weaker KC and stronger East Asian winter monsoon event.

Keywords: $U^{K}_{37}{}'$-thermometry, South China Sea, Asian Monsoon, deglaciation

STUDY OF DISSOLVED NUTRIENT CONDITION AT PULAU PERHENTIAN, TERENGGANU

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Abstract

During this study, we have compared the distribution of dissolved nutrients (NO_3^- and PO_4^{3-}) between two seasons (pre-monsoon and post-monsoon) in order to identify the nutrient condition at Pulau Perhentian, Terengganu. The concentration of dissolved nutrients was higher during post-monsoon (April 2015) compared to pre-monsoon (Oct. 2014) due to the huge input of nutrients (especially dissolved PO_4^{3-}) from bottom water. For dissolved PO_4^{3-}, its concentration during post-monsoon was 16 to 83 times higher than its concentration during pre-monsoon. On the other hand, the increasing of dissolved NO_3^- during post-monsoon was about 2 to 3 times higher than its concentration during pre-monsoon. These nutrients input were converted P limitation condition during pre-monsoon to N limitation condition during post-monsoon at our study area. Our present study has suggested that the seasonal Northeast monsoon has influence the distribution of dissolved nutrients and shifted the nutrient condition between seasons at our study area.

Keywords: monsoon, dissolved nutrients, seawater, P-limitation, N-limitation

ADSORPTION STUDY ON THE THE REMOVAL OF Cu IONS FROM AQUEOUS SOLUTION USING NaOH-MODIFIED *Carica papaya* PEELS

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Abstract

Recently, environment contamination by heavy metals has gained much attention due to the significant impact on public health. These heavy metals such as copper (Cu) are non-biodegradable pollutants and they are very difficult to eliminate naturally from the environment. Therefore, this study was conducted to deal with the removal copper (Cu) from an aqueous solution using a highly efficient, environmentally friendly adsorbent from *Carica papaya Peels (CPP)* prepared by NaOH-treatment modification. The effects of adsorbent mass, pH, initial metal ion solution concentrations and contact time were studied in batch experiments. The optimum adsorbent mass, pH, and contact time for Cu removal are 0.9 g, pH = 3, and 45 min, respectively. SEM and FT-IR were employed to validate the efficiencies of the adsorbents towards lead and copper. As a conclusion, the chemical-treated CPP can be utilized as a potential biosorbent for Cu removal from aqueous solutions.

Keywords: *Carica papaya* peels, adsorbent, copper, scanning electron microscopy, Fourier Transform Infrared Spectroscopy

ASSESSMENT OF HEAVY METALS IN PADDY SOIL AND RICE GRAINS (MR269 & MR253): A CASE STUDY IN MARDI PARIT, PERAK

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Abstract

Heavy metals are still a major threat for those who consume rice as their main dishes due to several health risks was reported associated with it. Even though there are heavy metals classified as essential for human growth, but the accumulated properties of heavy metals remain harmful especially if present in larger amount in food chain and eco-environmental system. Due to an awareness of its effects, an assessment of heavy metals (As, Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn) in rice grains and paddy soil were carried out in Malaysian Agricultural Research and Development Institute (MARDI), Parit, Perak using a combination of EDXRF and ICP based techniques (ICP-OES and MS). Soil samples was detected using EDXRF with minimum sample preparation. While rice grain was determined using ICP-based techniques and requires sample to be digested using HNO_3 and H_2O_2. Two varieties of paddy rice consist of MR253 and MR269 were collected together with paddy soil as well to evaluate the impact of long-term application of chemical fertilizer and pesticides that may contaminates paddy soil and its effect on the safety level of heavy metals in these two varieties of paddy rice grain. The average concentration of heavy metals in rice grain of MR253 and MR269 were as followed, As (357 ± 79, 185 ± 35), Cd (50.1 ± 14.3, 250.5 ± 135.4), Pb (169.8 ± 221.7, 75.1 ± 53.1), Cr (157.7 ± 177.2, 196.9 ± 282.6), Ni (167.6 ± 90.9) µg/kg, Cu (2.49 ± 0.76, 3.2 ± 0.2), Mn (19.6 ± 8.1, 32.7 ± 6.5), Zn (19.2 ± 2.0, 25.0 ± 1.6) mg/kg and Fe (25.7 ± 37.5, 15.9 ± 3.3) mg/kg respectively. The results indicated that the average concentration of As in MR269 and Cd in MR253 were higher than as stipulated in CODEX which recommended should be $\leq$ 0.35 mg/kg and $\leq$0.2 mg/kg respectively. But both varieties contain less than 0.3 mg/kg (CODEX) of Pb. The concentration of As, Cd, Cr, Cu, Pb, Ni and Zn in paddy soil meet the CCME (Canada) requirements for agricultural purposes. However, these guidelines may not suitable for rice plant due to the ability and efficiency of rice in accumulating of As and Cd from soil. The present study also evaluates the status of pollution in paddy soil based on Pollution Index (Igeo) and also Transfer Factor (TF) in soil-rice grain system.

Keywords: rice grain, paddy soil, heavy metals, pollution index, transfer factor

IMMOBILIZATION OF CROSS-LINKED CHITOSAN ONTO GLASS PLATE FOR REMOVAL REACTIVE RED 120 FROM AQUEOUS SOLUTION

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Abstract

Fabrication of an immobilized cross-linked chitosan-epichlorohydrine thin film (CS-ECHF) onto glass plate for adsorption of Reactive Red 120 (RR120) dye was successfully studied using the direct casting technique. The characterizations of CS-ECHF were performed by Scanning Electron Microscopy (SEM), The Fourier Transform Infrared Spectroscopy (FTIR), X-ray Power Diffraction (XRD), Point of Zero Charge (phpzc), and potentiometric titration. Adsorption experiments were performed as a function of contact time, initial dye concentration (10 mg/L to 200 mg/L), and pH (4 to 10). The dye adsorptions follow the Langmuir model with maximum monolayer adsorption capacity 344.83 mg/g at 27 ± 2 ◦C. The adsorptions kinetic follow the second-order model with good correlation. Results supported the potential use of an immobilized CLECTF as effective adsorbent for the treatment of reactive dye without using filtration process.

Keywords: chitosan, Reactive Red 120, cross-linked, epichlorohydrine, adsorption, immobilization

VERTICAL PROFILE OF METALS IN CORE SEDIMENT OF THE SUNDA SHELF SOUTHERN SOUTH CHINA SEA DURING HOLOCENE

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Abstract

Marine core sediment TRC3 (~158 cm) was collected from the inshore area of Terengganu, Malaysia to understand the environmental changes based on the records of sediment grain size and selected metals (Ca, Mg, Al, Fe, Cu, Pb, Zn, Co). Those proxies were analyzed by using particle size analyzer (PSA) and inductively coupled plasma mass spectrometer (ICP-MS). The age-sediment and sedimentation rates were established from the ^{14}C records of the intact shell fragments. The established age-sediment is consistent with the early Holocene sea level rise (EHSLR) in the Sunda Shelf which occured between 7000 and 11650 cal yr BP. The sediment mean size between 5.48 (medium silt) and 8.18 (clay) phi may reflect the variability of current intensity in the study area. Interestingly, all metals showed a similar fluctuated trend between 4,000 to 8,000 cal yr BP which indicates a significant changes in the sedimentation process of sediment deposits. The Fe/Ca ratio suggest the sediment deposits are mainly dominated by terrigenous than marine inputs. The applied environmental assessment of enrichment factor (EF), index of geoaccumulation (Igeo) and pollution load index (PLI) are in agreement that the study area is considered unpolluted with the studied metals. Thus, this work is important to reconstruct the environmental changes in the inshore area of Sunda Shelf based on the geochemical proxies.

Keywords: marine, tropical, South China Sea, elements, grain size, holocene

HEAVY METALS CONCENTRATION IN TROPICAL ANGUILLID spp. *Anguilla bicolor bicolor* AND SURFACE SEDIMENTS FROM SELECTED RIVERS IN PENANG, MALAYSIA

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Abstract

Tropical freshwater eel (*A. bicolor bicolor*) and river surface sediment from Sg. Titi Teras, Sg. Bayan Lepas, Sg. Pinang and the upstream of Sg. Pinang (Titi Kerawang waterfall) in Penang, Malaysia were studied. The specimens and samples were examined and analyzed for Fe, Cu, Zn, Cd, Pb, Ni, As and Se concentrations using ICP-MS while Hg was measured using mercury analyzer, MA-3000. The range for the total concentration (µg/g dry wt.) in *A. bicolor bicolor* were 0.15-339.89 (Fe), 0.35-51.03 (Ni), 1.00-83.35 (Cu), 4.52-286.68 (Zn), BDL-290.70 (As), BDL-111.88 (Se), 0.05-1.99 (Cd), 0.05-16.84 (Pb) and BDL-2.47 (Hg). Mean metal concentrations for all metal understudied were highest in the liver followed by gills and muscle tissues and concentration of metals in different tissues varied significantly ($P < 0.05$) depending upon the locations. The concentrations of heavy metals in the muscle of *A. bicolor bicolor* collected from the sites were slightly higher compared to a previous study. While the concentration range for surface sediments were found to contain 0.86-3.80 (Fe), 8.81-135.04 (Cu), 216.11-547.25 (Zn), 0.17-1.19 (Cd), 71.11-129.73 (Pb), 8.11-46.95 (Ni), BDL-0.17 (As), 51.84-129.90 (Se) and BDL-0.04 (Hg) respectively. The calculated enrichment factors (EF) and the Index-Geo assessment obtained indicate only Pb, Zn and As were influence by anthropogenic sources.

Keywords: tropical freshwater eel, heavy metal accumulation, rivers in Penang, surface sediment, ICP-MS, Mercury analyzer, Enrichment factor (EF), I-Geo index (*I-geo*)

ELECTRONIC WASTE MANAGEMENT USING RECYCLING TECHNOLOGY: A LIFE CYCLE ASSESSMENT STUDY

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Abstract

The number of electrical appliances will continue increase on the global scale which at the end will consume as an electronic waste. Electronic waste is dissimilar compared with other municipal waste due to electronic waste comprises thousands of toxic substances. The study aims to evaluate the environmental impact produced from managing electronic waste using recycling technology. The research methodology are field study, data collection, model development of recycling system, computer calculation development and impact analysis study. The recycling waste component facility considered in this study area are waste collection facilities, waste recycling facilities and landfill disposal site facilities as well as effluent treatment plant facilities. The input of the recycling system are management of 700 tonnes/day of electronic waste which were generated from residential, commercial and industrial premises. The results of this study shows that the recovery materials from the recycling system are 472.2 tonnes per day. The emissions produced from the transportation and processing activity in the recycling system are 47.32 kg water pollutant and 940.75 kg air pollutant. The findings of the study shows that, recycling technology able to recover world raw materials such as plastics, metals, glass and others. However, the emission such as water, air and residual waste generated from the system. The presence of pollution control such as air pollution control and water pollution control is important to eliminate the pollutant that is generated from the recycling system before it is released to the atmosphere and water bodies.

Keywords: emission, life cycle inventory, transportation, processing activities

SOURCES AND DISTRIBUTION OF ALIPHATIC AND POLYCYCLIC AROMATIC HYDROCARBONS IN COASTAL SEDIMENTS OF BRUNEI BAY, EAST MALAYSIA

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Abstract

Brunei bay is an area with a vast array of biological diversity and productive ecosystems providing suitable habitats for wildlife. Several rivers discharges into the bay. The bay also serves as an important maritime route serving the Federal Territory of Labuan, Brunei Darussalam, southwestern Sabah, and northeast Sarawak, with possible anthropogenic input of hydrocarbons contaminants to the bay. This study represents a first detailed study to determine the concentrations and distributions of aliphatic and polycyclic aromatic hydrocarbons (PAHs) in coastal surface sediments of Brunei Bay. Possible source of these compounds is assessed using selected hydrocarbon molecular markers. Thirteen surface sediments were collected, freeze-dried and then ultrasonic extracted using dicholoromethane-methanol solvent system. Identification and quantification of these hydrocarbon compounds were carried out using gas chromatography mass spectrometry after sample derivatisation. Total *n*-alkanes, unresolved complex mixtures (UCMs) and total PAHs and its alkylated homologues ranged between 0.61-9.88 μgg^{-1}, 2.92-16.15 μgg^{-1} and 0.02-2.19 μgg^{-1}, respectively. The low organic carbons in sediments showed weak correlation with total n-alkanes, total PAHs, and UCM. The carbon preference index (CPI) ranged 1.42-2.77 and other biomarkers ratios such as terrestrially derived alkanes, terrigenous over aquatic (TAR), isoprenoid hydrocarbons and UCM suggested a mixed contribution of terrigenous and anthropogenic fossil fuel inputs. The PAH profiles showed a predominance of petrogenic sources contamination in the area, with lesser contributions from pyrolytic PAHs and natural sources. Perylene, widely accepted as the naturally derived PAHs, comprises only between 2.61-39.39% of the total PAHs. The elevated petrogenic PAHs is probably due to surface runoffs from surrounding town or direct discharges of fossil fuel from maritime related activities such as boating and fishing. The values of n-alkane, PAHs and related biomarkers suggest that there is a moderate to high pollution in the area of study.

Keywords: biomarkers, marine sediment, terrigenous alkanes, petrogenic hydrocarbons, pyrolytic PAHs

DETERMINATION OF NATURAL RADIOACTIVITY AND HEAVY METALS AT FORMER MINING SITE OF KOLEJ UNIVERSITI ISLAM ANTARABANGSA SELANGOR (KUIS) LAKE

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Abstract

Ex-mining site has created an undesirable impact to the environment such as destruction of plants and contaminations. Thus, this study was carried out in order to determine the natural radioactivity and heavy metals presence in ex-mining site at Kolej Universiti Islam Antarabangsa Selangor's (KUIS) lake. Both parameters were measured using Gamma Spectrometry and ICP-MS. The results showed that the concentrations of ^{238}U (0.44 ± 0.31 Bq/L), ^{232}Th (0.04 ± 0.02 Bq/L), ^{226} Ra (0.19 ± 0.11 Bq/L) and ^{40}K (0.62 ± 0.37 Bq/L) in water samples were below the recommended values by WHO for drinking water source, which implied the safety comsumption of these water. However, the presence of ^{238}U, ^{232}Th, ^{226} Ra and ^{40}K in sediment samples were exceeded the limit proposed by UNSCEAR, with the radioactivity of 49.1 ± 18.8 Bq/kg, 102.3 ± 3.3 Bq/kg, 133.2 ± 18.8 Bq/kg and 297.1 ± 25.7 Bq/kg, respectively. For heavy metals determination, Zink was found in sediment with the highest concentration (17.34 ± 15.79 mg/kg) compared to other heavy metals, while concentration of Arsenic was highest in water with 0.58 ± 0.26 µg/L. All hazard indices were found to be below the safety limit except for the gamma index of 1.6 ± 0.7 Bq/kg and 104.1 ± 48.4 nGy/h for the gamma dose rate.

Keywords: radioactivity, heavy metals, water, sediment, gamma spectrometry

REMOVAL OF MERCURY IONS FROM AQUEOUS SOLUTION BY MODIFIED TURMERIC POWDER IMMOBILIZED ON SOL-GEL

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Abstract

The adsorption ability of sol-gel derived from turmeric powder functionalized with xanthate was investigated in an attempt to produce more economical and effective adsorbent for the removal of Hg(II) ion from aqueous solution. The result from the adsorption study showed that with the presence of xanthate functional group, sol-gel - modified turmeric (SG-MT) exhibited the highest selectivity toward the adsorption of Hg^{2+} with lesser response to Cr^{3+}, As^{5+}, Cd^{2+}, and Pb^{2+}.The effects of pH, dosage of adsorbent, adsorption time and initial metal ion concentration had been done to optimize the adsorption of Hg(II) by SG-MT adsorbent. Experimental data were exploited for kinetic evaluation related to the adsorption processes. The correlation coefficient values indicated a better fit of pseudo-second order equation compared to pseudo-first order model. The characteristics of the adsorption process were evaluated by using the Langmuir and Freundlich adsorption isotherm. It was determined that Hg(II) adsorption follows Langmuir adsorption isotherm model. The monolayer sorption capacity of this optimum adsorbent was found to be 37.59 mg/g.

Keywords: turmeric, xanthate, silica gel, adsorption, mercury(II) ion

HISTRORICAL CHANGES OF HEAVY METALS CONTENT IN A SEDIMENT CORE FROM SUNGAI KILIM LAGOON, LANGKAWI, MALAYSIA

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Abstract

Core sediment (LKC2) was collected from Sungai Kilim Lagoon to understand the history of heavy metal accumulation and pollution status during the recent past. The age-sediment was established by using AMS ^{14}C dating of the identified shell fragments which was ranged between 430 and 1240 cal yrs BP. The selected heavy metals (Cu, Pb, Co, Zn, Ni, Cd, Mn, Fe and Al) were measured using inductively couple plasma-mass spectrometer (ICP-MS). Generally, the concentration of Fe, Mn, Pb, Ni, Cu, Co and Cd showed a similar decreasing trend towards the surface. Meanwhile, the increase value of Al and Zn towards the surface is possibly due to the input from the surrounding area. The concentration of all metals was more fluctuated between 600 and 650 cal yr BP. The enrichment factor (EF), pollution load index (PLI) and geoaccumulation index (Igeo) were applied to assess the history of metals pollution in the study area. The EF indicates that Fe, Cu, Co, Zn, Ni, Cd and Mn fell in the category of deficiency to minimal enriched, and Pb is the only metal which was considered in the category of significant enrichment. The Igeo also showed similar result where Pb is the only metal in class 1 (uncontaminated to moderately contaminated) compared to the other metals. This finding may suggest that the enrichment of lead in this environment is originated from anthropogenic and/or natural sources. However, the PLI value which represent the entirety status of studied metals illustrate no serious metals pollution in the Sungai Kilim Lagoon within the past 1240 cal yrs BP. Hence, a continuous monitoring of metals content in this lagoon is very important to ensure Sungai Kilim remains pristine and unpolluted.

Keywords: tropical, sediment core, lagoon, AMS ^{14}C, heavy metals

ESTIMATION OF ELEMENTAL POLLUTION LEVEL USING *Leucobryum aduncum* IN MANJUNG DISTRICT, PERAK, MALAYSIA

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Abstract

Mosses have been used in many studies to monitor air pollution levels by measuring their elemental deposition. This recent study aimed to determine the deposition of Fe, Cr, Cu, Zn, Se, Pb, As and Cd in moss species of *Leucobryum aduncum* using EDXRF Spectrometer. The samples were collected from 24 locations in Manjung, Perak. The sampling locations were divided towards three directions (North, North-East and South-East) centered in Teluk Rubiah. From the calculated enrichment factors (EF), the moss samples were highly contaminated with Cd and slightly contaminated with Cr. In addition, PCA analysis shows the elements mostly were contributed from anthropogenic activities such as metal industries, chemical industries, smelting, soil dust, coal fired power plant and the used of fertilizers.

Keywords: Moss, EDXRF, PCA, bio-monitor

LIFE CYCLE ASSESSMENT STUDY FOR MANAGING ELECTRONIC WASTE USING LANDFILL TECHNOLOGY

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Abstract

Electronic waste has become a serious environmental issue due to its rapid growth in volume and its hazardous content. As a new addition to the waste stream, the emergence and rapid increase of electronic waste demands an inclusive management system. The management of electronic waste using landfill technology will pose significant amount of problem for environment and human health. Toxic substances from electronic waste dumping at the landfill site may be found in various type of emission such as leachate and landfill gas from landfill site. This study aims to evaluate the environmental impact produced by management of the electronic waste using landfill technology. In this study, a life cycle assessment technique will be used to evaluate the impact of the management of electronic waste using landfill technology. The methodologies of this study are field study, data collection, model development of landfill system, computer calculation development and impact analysis study. The inputs of the system are management of 700 tonnes/day of electronic waste, which were generate from residential, commercial and industrial premises. The fuel usage for transportation activities are 1,069.44 liters per day. The energy usage for processing activities at secured Landfill site are 2,800 liters per day for diesel usage and 2.33 MW/day for electricity usage The results of this study shows that there is no recovery material produced from the landfill. The emissions produced from the processing activity in the landfill system are 583.1 tonnes/day of leachate as well as 2,249,100 m3/day landfill gas. The landfill system produces 59.19 kg water pollutant and 866.9 kg air pollutant. The finding of the study shows that, landfill technology unable to recover world raw materials such as plastics, metals, glass and others. In addition, the emissions such as water and air emission also generated from the system. To minimize the impact of Landfill Technology for managing electronic waste, pollution control facilities such as air pollution control and water pollution control is essential to eliminate the pollutant from released to the atmosphere and water bodies.

Keywords: emission, Life Cycle Inventory, transportation, landfill, e-waste

ACCUMULATION OF TOXIC AND ESSENTIAL METALLIC ELEMENTS IN EDIBLE BIVALVES COLLECTED FROM MUAR RIVER ESTUARY, JOHOR, MALAYSIA

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Abstract

In recent years, the pollution of various river and coastal area by metallic elements has been the subject of intense public concern in Malaysia. People can be exposed to these toxic chemicals that accumulate in shellfish taken from the contaminated waters. In Muar region, oyster, green-lipped mussel, cockles and clams collected in the Muar river are the most popular seafood besides fishes landed by fisherman. Therefore, to estimate the potential human risk toward these metallic element, concentration of toxic (Cd, Pb, Hg, As) and essential metallic elements (Cu, Zn) in 300 bivalves including oyster, green-lipped mussel, cockles and clams collected were analyzed in the edible tissue using a fast and sensitive Inductively Coupled Plasma Mass Spectrometer (ICP-MS). Results showed that the concentration of metallic elements in bivalves ranged at the following sequence: Zn>Cu>As>Pb>Cd>Hg. Generally the average concentrations of all metallic elements studied were below and comparable with the limit set by Malaysian Food Regulation, European Commission, Ministry of Public Health Thailand and China National Standard and Malaysia Food Regulation. Metals concentrations in the in-toto tissue were assessed for human safety consumption according to Provisional Tolerable Weekly Intake (PTWI) and Provisional Tolerable Daily Intake (PTDI). The weekly and daily intake of metallic elements through the consumption of these shellfish was estimated based on both of the metal concentration in bivalve and the consuming amounts of bivalve. The calculated daily intakes of these metals through consuming the bivalve were 0.122 (Cd), 0.137 (Pb), 0.102 (Cu), 1.20 (Zn), 0.005 (Hg) and 0.905 (As), which below the limit set by Joint FAO/WHO Expert Committee on Food Additives. In addition, the target hazard quotients (THQ) were used to evaluate the potential risk of these elements in the organism on human body. The highest THQ values of Pb and Cd were observed in the species of oyster and clam. However, the results indicate that the intake of these selected elements by consuming shellfish collected from Muar estuary do not present an appreciable hazard risk on human surrounding area health, but attention should be paid to consuming those with relatively high THQ values such as oyster and clam. Due to their accumulation capacity of heavy metals, these bivalves had the potential of being used as biomonitor to control the aquatic contamination by these elements.

Keywords: toxic, essential metallic elements, bivalve, edible tissue, Muar river estuary, potential risk, consumption

A PROPOSED CLOSED-TUBE REFLUX COLORIMETRIC METHOD FOR THE DETERMINATION OF TOTAL ORGANIC CARBON IN BOTTLED DRINKING WATER

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Abstract

The American Public Health Association (APHA) analysis of chemical oxygen demand (COD) by the closed-tube reflux colorimetric method requires the use of Kalium dichromate ($K_2Cr_2O_7$), silver and mercury sulfates. It involves using large volumes of both reagents and samples. This placed a restriction on analysis reagent, increase cost of COD determination and risk of environmental pollution. The present method used Kalium chromate (K_2CrO_4) and reduced the amounts of reagent and sample needed for the analysis. The use of kalium chromate was possible because it exists as the dichromate in strong acid mixture, a medium suitable for the digestion. The UV-Vis spectra of the $K_2Cr_2O_7$ and K_2CrO_4 in H_2SO_4 acid medium and a comparison of the proposed method with APHA method proved it. The proposed method eliminates the dependent on kalium dichromate and reduces the amount of the sample by 60%, and digestion mixture (chromate and mercury sulfate) and catalyst (silver sulfate) by 33.33% and 60% respectively. The method was applied for the UV-Vis spectrometry determination of COD in bottled drinking water.

Keywords: Total organic carbon, potassium chromate, APHA

SEASONAL VARIABILITY AND FRACTIONATION OF P-BASED NUTRIENTS IN A TROPICAL RIVER SYSTEM IN MALAYSIA

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Abstract

A seasonal study of phosphorus (P)-based nutrients was carried out in the Setiu River basin which is located in the southern region of the South China Sea (Malaysia) from July 2010 until June 2011. Parameters measured were dissolved inorganic P (DIP), dissolved organic P (DOP) and total particulate P (TPP). In addition, DOP concentration was size fractionated into high molecular weight (HMW, 0.7 µm-100 kDa), middle MW (MMW, 100-30 kDa) and low MW (LMW, <30 kDa). The results show that the mean concentration of P-based nutrients ranged between 3.2-7.0 µg/L P for DIP, 5.6-12.1 µg/L P for DOP and 9.2-119.4 µg/L P for TPP. DOP therefore represented a substantial component of dissolved P. The measured concentrations of the P-based nutrients were low compared to many other river systems. In general, higher mean concentrations of P were recorded at urban and agricultural areas suggesting sources in the discharge from these activities. In addition, the distribution of P was affected by season as higher mean concentrations of P were observed during the north-east monsoon due to the surface runoff of nutrients during this heavy raining season. The fractionation study of DOP further revealed that HMW fraction was abundant in Setiu River, making up approximately 60% of the total DOP. This high percentage of HMW DOP was recorded at stations in the vicinity of agriculture area and towns suggesting an important contribution from anthropogenic activities. The LMW DOP fraction was not significantly associated with chlorophyll-a, suggesting that the phytoplankton might not be the predominant contributor for LMW fraction in this river. DOP and dissolved organic nitrogen (DON) concentrations within the river system are correlated suggesting that organic forms of N and P are responding to somewhat similar forcing. Therefore, we suggest that the transport of P-based nutrients in Setiu River basin is dominated by organic and particulate forms. Comparison with data from other rivers in the region suggests this pattern is widespread. We suggest that these organic nutrient inputs and should be monitored as part of the management of the aquatic system especially the coastal area of the South China Sea.

Keywords: Seasonal variability, fractionation, P-based nutrients, monsoon and non-monsoon seasons, tropical area

SINGLE AND BINARY TOXICITY OF TEMPERATURE, SALINITY, CADMIUM AND COPPER ON THE HEARTBEAT OF *Daphnia* sp.

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Abstract

We evaluated single and binary toxicity of temperature, salinity, cadmium and copper on the heartbeat of *Daphnia* sp. in 24 hours. *Daphnia* sp. is a good test organism looking into their crucial function as secondary producer in the aquatic food chain. The rising of water temperature, salinity and bioavailability of contaminant (cadmium and copper) to the living things also require continuous investigation as the climate change impacts keep progressing. This study was conducted in laboratory in order to examine the impacts of each parameters in control condition following OECD guidelines 218. The 24 hours median lethal value of temperature is 38.2^{0}C, while the 24 hours median lethal concentration (24hr LC50) of salinity, cadmium and copper are 0.84 ppt, 3.25 µg/L and 3.41 µg/L, respectively. The binary toxicity were examined by the combination of salinity and temperature; temperature and cadmium; cadmium and copper. The number of heartbeat was recorded to be higher in binary toxicity of salinity and temperature with values ranged from 138 to 195 beat/minute compared to 90 to 163 beat/minute in single effect of salinity. No significant difference of heartbeat/minute values found in single and binary toxicity of temperature and 0.5 µg/L of cadmium ($p>0.05$). However, the survival period of *Daphnia* sp. was shortened to 16 hr compared to 24 hr in single effects of temperature (25 to 32°C). The toxicity of respective cadmium and copper on heartbeat/minute of *Daphnia* sp. was not significant difference ($p>0.05$) to the binary mixture of cadmium and copper. However, the survival period has shortened to 8 hr in binary toxicity experiment. Differ from parameters such as temperature and salinity (90 to 195 beat/min), the heartbeat/minute value rapidly increase after *Daphnia* sp. were exposed to either single or binary toxicity of contaminant (130 to 326 beat/min). Thus, we can conclude that heartbeat and survival period could be a good endpoint of toxicity test using *Daphnia* sp. In addition, binary toxicity showed significant impacts to the *Daphnia* sp., hence further investigation of mixture effects is required as stressors (physical parameter or contaminants) in natural environment exerted impacts in combination forms.

Keywords: single, binary, toxicity, temperature, salinity, cadmium, copper, Daphnia

PHYTOREMEDIATION OF HEAVY METALS BY A*locasia puber* IN LABORATORY-SCALE CONSTRUCTED WETLANDS

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Abstract

The contamination of water by toxic heavy metals is a worldwide environmental problem. This problem has led to the development of alternative technologies for removal of heavy metals from contaminated sites. One such novel approach is phytoremediation, which is considered as a green alternative treatment. This study focus on the potential of *Alocasia puber* (Araceae) as a new plant for remediation of heavy metals in wastewater. The objective of this study is to determine the efficiency of *Alocasia puber (A. puber)* to treat heavy metals contamination in synthetic waste water using laboratory-scale constructed subsurface wetlands. The most hazardous heavy metals i.e. Cd, Cr, Cu, Hg, Ni, and Zn were selected as target heavy metals in this study. The heavy metals concentrations of water inflows to the systems were set to be 5 mg/L and 10 mg/L. Six different HRTs were applied to the treatment system with corresponding HRTs of 2, 4, 6, 8, 10 and 12 days. Average removal efficiencies of heavy metals removal ranged from 91.86 to 99.03%. The removal of Ni from 10 mg/L solutions (initial concentrations) reached highest removal efficiency of 99.91% after 12th day, followed by Hg, Cd, Cu, Cr and Zn. The results in this present study shows the great potential of *A. puber* in constructed wetlands for phytoremediation of water contaminated with heavy metals. This study will contribute to a new knowledge of heavy metals remediation using phytoremediation by *A. puber*.

Keywords: phytoremediation, constructed wetlands, *Alocasia puber*, heavy metals

PATTERN RECOGNITION ON DISTRIBUTION OF MERCURY SPECIES IN WATER COLUMN: A CASE STUDY OF JOHOR STRAITS

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Abstract

This research work is aim to evaluate the pattern recognition of trace metal distribution towards three main species, namely CH_3Hg^+, $C_2H_5Hg^+$ dan Hg^{2+}. The distribution pattern was evaluated on surface and bottom water-sediment interface. Mercury species were extracted using the developed method (2^4 Central Composite Design) namely solid phase micro extraction – assisted dispersive derivative agent. Supervised and unsupervised pattern recognition namely cluster analysis, principle component analysis, factor analysis, discriminant analysis and time series analysis were applied during this study. The mercury content from Johor Straits marine water samples were recorded with maximum value at 1.38 μgL^{-1} which frequency of detection based on species as follows: 100% for CH_3Hg^+ and Hg^{2+} but only 54.5% (surface) and 50.2% (bottom) for $C_2H_5Hg^+$ species. Total mercury content were calculated exceed the permissible level for Malaysian Marine Water Quality Criteria under class E. Hierarchical agglomerative cluster analysis was able to explain the spatial variation of field study and was separated into two main clusters at linkage distance $D_{link}/D_{max} \times 100 < 70$ and < 30. Principal component analysis and factor analysis (PCA/FA) were able to distinguish six hydrochemistry parameters namely water pH, dissolved oxygen, organic carbon, suspended solids, sulfate and ferrous ions which were known to have influences toward the availablity of mercury species. All the six parameters represent cummulative variance of 96.70% and 97.70% for surface and bottom levels, respectively. A stepwise mode of discriminant analysis was able to discrimate the spatial and temporal variation during period of study toward six parameters which represent 91.7% and 99% of total variance. The ARIMA models were obtained as follows: CH_3Hg^+ (1,1,2) or (1,1,1), respectively and $C_2H_5Hg^+$ (1,1,1) and Hg^{2+} (1,0,1) similar for both surface and bottom of straits.

KAJIAN KELIKATAN PELARUT EUTEKTIK DALAM RELINA TERHADAP PENGEKSTRAKAN α-TOKOFEROL DARIPADA MINYAK SAWIT MENTAH

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Abstrak

DES Relina diklasifikasikan sebagai pelarut hijau dan serasi untuk digunakan bagi pengekstrakan α-tokoferol (α-T) daripada minyak sawit mentah (CPO) kerana kemampuannya untuk membentuk ikatan hidrogen yang kuat dengan molekul α-T. Kepekatan dan hasil α-T dikuantifikasi menggunakan RP-HPLC. Dua parameter dikaji melalui penggunaan lima siri nisbah CPO: DES relina (1: 1, 1: 2, 1: 3, 1: 4 dan 1: 5) pada 2 dan 3 jam masa pengekstrakan untuk menentukan kondisi optimum bagi pengekstrakan α-T. Kepekatan α-T untuk semua sampel yang diekstrak dibandingkan dengan kawalan (tanpa DES relina) memberikan nisbah 1: 2 (CPO: DES relina) dengan kelikatan pelarut 5.7 ± 0:58 cP pada 3 jam masa pengekstrakan sebagai kondisi pengekstrakan terbaik bagi mendapatkan kepekatan α-T tertinggi iaitu 825.321 ± 74.27 mg / L dengan keberhasilan 0.1554 ± 0.0156 (w/w %). Perkara ini menunjukkan bahawa kondisi ini merupakan keadaan yang paling kondusif bagi penyerapan molekul α-T ke dalam ruang kosong dalam struktur pelarut di mana molekul α-T dan DES relina berinteraksi melalui ikatan hidrogen. Ikatan hidrogen yang kuat terbentuk antara kumpulan –OH α-T dengan kumpulan amina $-NH_2$ DES relina merupakan faktor peningkatan keupayaan DES relina untuk mengekstrak sebatian α-T yang tinggi daripada CPO.

Kata kunci: penderma ikatan hidrogen, pelarut eutektik dalam relina, tokoferol, kelikatan, kolin klorida

RP-HPLC FOR SEPARATION OF L-ARGININE AND L-CITRULLINE IN RED AND YELLOW CRIMSON WATERMELON (*Citrullus lanatus*) JUICES EXTRACT USING GRADIENT MODE

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Abstract

Watermelons (*Citrullus lanatus*) is one of medicinal fruit that has attracted scientific interest due to its health benefits phytonutrients such as carotenoids and amino acids. Watermelon juices are the richest known source of amino acids including L-arginine and L-citrulline. For complete separation of amino acids in watermelon juices extracts, a gradient mode of reverse phase high performance liquid chromatography (RP-HPLC) with UV detection is conducted due to its supremacy over isocratic mode. The present study aims to achieve good separation of chromatography profiles and baseline between L-arginine and L-citrulline in two types of watermelon juices extracts, red and yellow crimson watermelon. The edible parts of watermelons were juiced, stored at – 80 ^oC and freeze dried to obtain dried juice powder. The samples were analyzed by gradient mode of RP-HPLC using 0.1% orthophosphoric acid in water (v/v) and acetonitrile as mobile phase A and B. The chromatograph separation was performed using Gemini C_{18} column at flow rate 1.0 mL/min, maintained temperature of 25 ^oC and detection was made at different wavelengths: 195 nm, 200 nm, 207 nm and 210 nm with total analysis time, 35 min. Excellent chromatographic separation was achieved at condition of gradient elution (0%B - 95%B, 0-18 min and 95%B, 18-23 min, 95%B-0%B, 23-25 min, 0%B, 25-35 min) with baseline between L-arginine and L-citrulline in both watermelon juices extracts. The gradient mode of this RP-HPLC achieved efficient separation of chromatographic profile in watermelon juices extracts and also potentially be used for complete analysis of amino acids profile in commercial fruit juices samples.

Keywords: RP-HPLC, Gradient mode, Separation, Watermelon, Juice extract, L-arginine, L-citrulline

EXTRACTION OF NSAIDs BY USING MAGNETIC SOLID PHASE EXTRACTION OF SPOROPOLLENIN BASED BETA CYCLODEXTRIN

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Abstract

Current study reports the preparation of supramolecular host beta cyclodextrin functionalization/magnetization onto surface bio-polymeric spores of sporopollenin (MSp-TDI-βCD) for magnetic solid phase extraction (MSPE) technique. The synthesized adsorbent was characterized using Fourier transform infrared spectroscopy, scanning electron microscopy, and vibrating sample magnetometer analysis Four types nonsteroidal anti-inflammatory drugs (NSAIDs) were selected; indoprofen (INP), ketoprofen (KTP), ibuprofen (IBP) and fenoprofen (FNP) were separated, preconcentrated and being optimized for several operating variables. Optimum extraction conditions showed MSp-TDI-βCD as MSPE sorbent gives linear range between 0.5-500 ng/mL, low limit of detection at S/N=3 (0.1-0.4 ng/mL) and limit of quantification at S/N=10 (0.5-1.0 ng/mL). The precision for proposed technique gives interday (n=3) and intraday (n=5) relative standard deviation (RSD%) in the range of 2.5-3.9 and 2.1-5.5 respectively. For reusability performance, it can be proposed that MSp-TDI-βCD could be act as MSPE sorbent until five cycles. MSPE method also was implemented for analysis in real samples from various water samples, whereby percentage recovery in range 92-123% with RSD% lower than 12.4 was achieved.

Keywords: magnetic nanoparticles, beta-cyclodextrin, sporopollenin, NSAIDs, magnetic solid phase extraction

REVIEW ON EXTRACTION SOLVENTS IN THE DISPERSIVE LIQUID-LIQUID MICROEXTRACTION (DLLME)

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Abstract

Dispersive liquid-liquid microextraction (DLLME) is a novel sample preparation technique that offering high extraction efficiency using microvolumes of solvents. It has been received noticeable attention because of several advantages, including simplicity, short extraction time, low cost and high enrichment factor. In conventional DLLME, chlorinated solvents had been widely used as extraction solvents. However, these solvents are toxic and environmental-unfriendly. In recent years, many researches focused on using of nontoxic or low toxic extraction solvents and better practical procedures which improve extraction efficiency. This review discusses the development of low toxic extraction solvent recently used in the DLLME. The improvement of extraction solvent from the green analytical chemistry perspectives also discussed in this review.

Keywords: extraction solvents, dispersive liquid-liquid microextraction

LEACHING OF YTTRIUM FROM LCD WASTE USING SULFURIC, NITRIC AND HYDROCLORIC ACID

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Abstract

Thermodynamic modeling, experiment measurement and characterization technique were used to evaluate the leaching process of yttrium (Y) elements from liquid crystal display (LCD) waste. Thermodynamic modelling using HSC 6.0 software revealed that the reaction of leaching out Y are exothermic thus require less energy and at the same time decrease free energy at the minimum temperature of 283.15 K. The reaction with sulfuric and nitric acids identified to be reversible from temperature 283.15 K and above due to the negative entropy change, while the reaction with hydrochloric acid was found irreversible due to the positive entropy change at the similar minimum temperature setting. The significance of reversibility versus irreversibility is their relationship to the efficiency. Pourbaix diagram modeling showed that Y dissolved in HCL at pH below 7 therefore strong reducing agent such sulfuric acid (sulfide) can improve the dissolution of Y. Inductively coupled plasma mass spectroscopy (ICP-MS) results showed that Y is viable to be efficiently leached from the studied LCD waste either in a single leaching or in two-stages leaching mode. Sulfuric and nitric acids are found to be the most practical solutions in leaching out them whereby around 0.00515 ppm and 0.00507 ppm of Y were dissolved in both solutions respectively based on the two-stages leaching approach

Keywords: yttrium, Liquid Crystal Display, leaching, modeling, spectroscopy

EFFICIENT EXTRACTION METHOD FOR PHARMACEUTICAL COMPOUNDS OF CARBAMAZEPINE AND CAFFEINE

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Abstract

Carbamazepine and caffeine are pharmaceutical compounds abundantly contaminated the wastewater. To allow optimum detection of these compounds by gas chromatography-mass spectrometry, extraction methods for the compounds from wastewater samples are addressed. This study focuses on determining the efficient extraction methods between Liquid-liquid Extraction and Solid Phase Extraction for carbamazepine and caffeine. Optimization on methanol volume was investigated. The efficiency of extraction method was evaluated through mean recovery percentage. Optimum methanol volume for carbamazepine and caffeine extraction was determined at 11 mL. Overall, analytical mean recovery of carbamazepine and caffeine was greater than 80% for liquid-liquid extraction meanwhile, mean recovery for solid phase extraction method was very low (5% to 35%). As a conclusion, Liquid-liquid Extraction method using 11 mL methanol was selected as optimum methanol volume and efficient method for carbamazepine and caffeine extraction. This study gains an insight into optimum extraction method for carbamazepine and caffeine hence, further study results will be more reliable.

Keywords: caffeine, carbamazepine, liquid-liquid extraction, pharmaceutical compounds, solid phase extraction

THE EXTRACTION OF THORIUM HYDROXIDE IN HYDROCHLORIC ACID BY USING DI (2-ETHYLHEXYL) PHOSPHORIC ACID IN KEROSENE

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Abstract

Di-(2-ethylhexyl) phosphoric acid (DEHPA) dissolved in kerosene has been chosen as a solvent extraction method to extract Thorium from Thorium hydroxide with hydrochloric acid. Different parameters were carried out to test the following variables: separation time and reaction time, optimum ratio of DEHPA in kerosane, Concentration of Thorium in thorium hydroxide with different acidity with pH 2.2 and the analysis has been carried out by using inductively coupled plasma mass spectrometry (ICPMS). The result has been calculated for thorium extracted. The data obtained showed that the best result for thorium extraction based on the variable tested, the optimum ratio of DEHPA in kerosene is 30%, the optimum mixing and contact time is 10 minute. As the final result showed that the extraction of thorium with pH 2.2 reach achieve 98% respectively.

Keywords: thorium, extraction, DEHPA, kerosene

THE EFFECT OF ELECTROLYTE pH ON THE ELECTRODEPOSITION OF NICKEL COATING ON ZINCATED ALUMINIUM

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Abstract

Nickel coating was successfully deposited on zincated aluminium using electrodeposition technique. A detailed study was made about the effect of electrolyte pH (i.e: 2, 6,10) containing 0.1 M nickel sulphate ($NiSO_4$) by cyclic voltammetry (CV). A constant potential of -0.8 V (pH 2), -1.1 V (pH 6) and -1.5V (pH 10) was choosen from mixed region of each CV for deposition of Ni using chronoamperometry (CA). The surface morphology, chemical composition and crystallographic structure of nickel coating were investigated by field emission scanning electron microscopy (FESEM), energy dispersive spectroscopy (EDAX) and X-ray diffraction (XRD), respectively. The adhesion strength of nickel coating on the zincated aluminium substrate was analysed using Scotch® tape test. It was found that the nucleation potential for nickel deposition on zincated aluminium increased with increasing electrolyte pH. Notably, pH 2 produced the highest current during deposition of nickel as compared to pH 6 and pH 10 due to the presence of simultaneous hydrogen evolution reaction (HER). Due to HER, a lot of pits were present on the zincated aluminium surface and only few nickel particles were formed as imaged by FESEM. Meanwhile, deposition using pH 6 solution (without HER) has produced almost fully covered nickel coating with uniform nodular morphology. However, the morphology has changed to cauliflower-like agglomerates when using pH 10 solution with less surface coverage. EDAX analysis showed that the coating using pH 6 has the highest nickel content with 73.6 wt%. XRD analysis indicated that all nickel deposits consisted only cubic crystalline structure. Nickel deposited using pH 6 solution was the most well-adhered coating. Thus, pH electrolytes has greatly influenced the deposition of nickel coating on zincated aluminium surface.

Keywords: zincated aluminium, nickel underlayer, electrodeposition

MICROEMULSION ELECTROKINETIC CHROMATOGRAPHY COUPLED WITH DISPERSIVE MICRO SOLID PHASE EXTRACTION FOR DETERMINATION OF ISOFLAVONES IN SOYBEAN PRODUCTS

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Abstract

Nutritional supplements based on soy extract are a rapidly growing segment in the food and health care market. However, isoflavones in soy products have been reported to reduce the risk of cardiovascular disease and improve bone health by alleviating symptoms of osteoporosis occurring after menopause. Therefore, it is imperative to establish methods for accurate determination of isoflavones levels in the soy products. The aim of this work is to develop a microemulsion electrokinetic chromatography (MEEKC) coupled with dispersive micro solid phase extraction (D-µ-SPE) method for the determination of isoflavones in soy products. MEEKC uses an aqueous buffer containing droplet of oil, surfactant and co-surfactant to form a microemulsion background electrolyte solution (BGE). The microemulsion containing ionic surfactant allows chromatographic separations to be obtained as solutes can partition between the charged oil droplets and the aqueous buffer phase. D-µ-SPEs of real samples were carried out using multi-walled carbon nanotubes (MWCNTs) as adsorbent prior to MEEKC. Separations of selected isoflavones namely daidzein, genistein and formononetin by MEEKC were carried out using fresh daily-prepared microemulsion BGE. It was found that the optimized MEEKC conditions for the separation of isoflavones were 4 mM borate buffer pH 8.5, 6.6% (w/v) 1-butanol, 0.9% (w/v) sodium dodecyl sulphate, 0.75% (w/v) ethyl acetate, 3% (w/v) acetonitrile at 3 s injection time, 27 kV and 35°C. The optimum extraction conditions were 5 mg of MWCNTs and 300 µL of methanol as desorption solvent. Under the optimized conditions, the calibration curves for the isoflavones showed good linearity in the concentration range of 1 - 10 mg/L with coefficients of determination (r^2) > 0.99 and limit of detections of 0.27-0.95 mg/L. The D-µ-SPE-MEEKC method was successfully applied to the analysis of five real samples namely soy supplement, tofu, tempeh, egg tofu and fujook for the determination of isoflavones. The analysis of the selected isoflavones in the samples showed good results with recoveries in the range of 74.5-112.5% and RSDs of < 3%. The newly developed D-µ-SPE-MEEKC has proved to be simple, offers low consumption of organic solvent and relatively short extraction time, thus a potentially viable alternative method for extraction and determination of isoflavones in soybean products.

Keywords: microemulsion electrokinetic chromatography, background electrolyte, dispersive micro solid phase extraction, isoflavones, soybean products

COMPARATIVE STUDY OF RAW AND COOKED FARMED SEA BASS (*Lates calcarifer*) IN RELATION TO TRACE METALS AND ITS ESTIMATED HUMAN HEALTH RISK

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Abstract

Metallic elements contamination in seafood, especially in fish has been of increasing concern to the human health. And with increasing dependency towards farmed fish for fish resources, the metallic elements contamination in them are still questionable. Therefore, this study was carried out to investigate the effects of cooking (steaming) on metallic elements concentration in farmed fish and to estimate its potential human health risk. Farmed seabass (*Lates calcarifer*) from Sg. Setiu were used to study the difference of metal uptake through human consumption of raw and cooked (steamed) fish samples. Metals namely, Zn, As, Cd, Hg and Pb were analysed using ICP-MS after closed digestion methods using HNO_3. In our finding, cooked fish showed to have significant effects on these metal concentrations in the fish muscle, where the general trend of metal accumulation in the fish muscles were found to be significantly higher in cooked fish with the exception of As and Pb. However, the metals concentration in the cooked fish muscles were mostly all well below the standard limits set by FAO/WHO. Despite such, long-term intake of these fish may pose a risk to human health due to the high Hg and As concentration found in these fishes.

Keywords: metallic elements, farmed sea bass, cooked, raw, human health risk

CHEMICAL CONSTITUENTS IN E-CIGARETTE REFILL SOLUTIONS AND AEROSOLS FOUND IN KLANG VALLEY

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Abstract

Electronic cigarettes (e-cigarettes) are rapidly increasing in popularity. E-cigarettes are battery powered devices that vaporize a glycerin or propylene glycol solution. This solution is flavored, sometimes with the addition of nicotine as well in order to give it a similar satisfaction such as cigarette smoking. The act of smoking e-cigarettes is also known colloquially as 'vaping'. Main purpose of the study is to evaluate the chemical composition of e-cigarette refill solutions and their resultant aerosol for possible differences. 75 samples of e-cigarette refill solutions were obtained from multiple locations throughout Klang Valley. Chemical composition of e-cigarette refill solutions and their resultant aerosol were analyzed using Gas Chromatography Mass Spectrometry (GC-MS). 116 compounds were identified from e-cigarette refill solutions. On the other hand, 275 compounds were identified from their resultant aerosol samples. At least 60% of the e-cigarette refill solutions and the resultant aerosol contain nicotine. It was also found that at least 9 out of 35 nicotine free labelled refill solutions contains nicotine. Propylene glycol and glycerin were found in 96% of the refill solution and aerosol samples. Various compounds such as N-ethyl-4-hydroxypiperidine, butanoic acid, ethyl ester, ethyl maltol, vanillin, 1-butanol 3-methyl-acetate, benzyl alcohol, ethyl acetate were found in more than 30% of the refill solutions samples. Results demonstrated distinctly different chemical composition between refill solution and aerosol samples. Compounds such as acrolein, 2-propanone 1 hydroxy-, acetic acid, nicotyrine and 2-propen-1-ol were found in aerosol but not in refill solution samples. The compounds were found in more than 55% of the aerosol samples. Formation of the compounds could be linked to the aerosolization process during the usage of e-cigarettes. Further research on the quantification of chemical compound present in e-cigarette refill solution and aerosol samples need to be done in order to accurately evaluate its potential health implication for humans.

Keywords: e-cigarettes, aerosol, refill

GROWTH AND BIOREMEDIATION OF FUNGI IN PHENANTHRENE AND WASTE ENGINE OIL

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Abstract

The ability of *Aspergillus* sp. and *Pleurotus ostreatus* to grow and biodegrade polycyclic aromatic hydrocarbons phenanthrene in MSS solution at different conditions were investigated. The application of fungi in 1 % waste engine oil degradation was also evaluated. The fungi growth was observed through optical density and the bioremediation was evaluated through biodegradation percentage. *Aspergillus* sp. and *Pleurotus ostreatus* demonstrated an increase growth and then achieved saturation. The growth of both fungi decreased when the phenanthrene concentration increased. For bioremediation study, *Aspergillus* sp. was inhibited when increased of phenanthrene concentration. However, *Pleurotus ostreatus* showed contradicted result where higher phenanthrene concentration, increased production of enzymes and hence increased phenanthrene biodegradation. *Aspergillus* sp. worked well at low pH but the optimum pH for *Pleurotus ostreatus* was pH 9. Hence, *Pleurotus ostreatus* is a potential degrader for phenanthrene biodegradation. In application of waste engine oil study, *Aspergillus* sp. showed a higher biodegradation activity than *Pleurotus ostreatus* as 1 % waste engine oil is considered under low pollutant concentration environment. Optimum pH of waste engine oil biodegradation for *Aspergillus* sp. and *Pleurotus ostreatus* were acidic and alkaline, respectively. The obtained results play important role to select potential fungi in further application study.

Keywords: aspergillus, biodegradation, concentration, pH, phenanthrene, pleurotus, waste engine oil

FABRICATION OF CHITOSAN- ACTIVATED CHARCOAL COMPOSITE BEADS FOR ADSORPTION OF THIONINE DYE

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Abstract

Chitosan-Activated charcoal composite (CS-AC) beads were prepared and studied in a batch mode operation for the adsorption of cationic dye Thionine from aqueous solution. Characterization on the surface of CS-AC was performed by using Scanning Electron Microscopy (SEM), Fourier Transform Infrared (FTIR) and point of zero charge (pHpzc) method. Batch adsorption studies were carried out to optimize the adsorption equilibrium experiments by observing the effect of experimental parameters, which are adsorbent dosage (0.04-0.40 g), initial pH (4-11), initial concentration (10-100 mg/L) and temperature (30-50 °C). The results show that the equilibrium uptake increased with increasing initial concentration. The adsorption equilibrium data show that the adsorption process obeys the Langmuir model with maximum adsorption capacity 59.52 mg/g. The kinetics of the Thionine uptake was well-fitted to the pseudo second order kinetics. Results supported the potential use of CS-AC as effective adsorbent for the treatment of cationic dyes such as Thionine.

Keywords: chitosan, activated charcoal, thionin, adsorption, beads

SYNTHESIS OF MAGNETIC IRON OXIDE NANOPARTICLES VIA BIOCOMPATIBLE MICROEMULSION AND MODIFIED WITH GLYCINE FOR HEAVY METAL REMOVAL

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Abstract

Substantial heavy metals are applied as a group of metals and metalloid with moderately high density and harmful at low fixation. They are found naturally in Earth's crust. They are not easily degraded or pulverized. Some of substantial heavy metals, for example copper and zinc are esssential in our body to maintain metabolism but prolong exposure to heavy metals can cause serious health impacts including reduced growth and development, cancer and death for an extreme cases. Heavy metals are released in environment through fast industrialization. Thus, heavy metals accumulated in water are serious problem globally as it can prompt to lifetime undermining sickness. Application of iron oxide for heavy metal removal is more appealing due to its small size, high surface area and magnetic properties. Magnetic property of iron oxide nanoparticle empowers simple isolation of adsorbents from the system and could be reused for further application. Various techniques had been produced to viably treat heavy metal-containing effluent, including electrocoagulation, reverse osmosis, solvent extraction, chemical precipitation, and ion exchange. These methods have some impediments and inconveniences. For example, there are huge amount of sludge being produced in the chemical precipitation process. Therefore, an extra effort is needed to further treat the sludge produced which can be a long term environmental issue in term of disposal. Among these methods, microemulsion method is an adaptable method that can deliver nanoparticles of small size and low polydispersity. In various types of microemulsion, W/O microemulsion is more alluring because of the capacity of solubilizing polar soluble molecule (by reverse micelle) in low extremity condition. Besides that, results showed that both effect of pH and time, percentage removal of copper ion with magnetic iron oxide nanoparticle modified with glycine (MNPs-Gly) is higher than using magnetic iron oxide nanoparticle (MNPs).

Keywords: microemulsion, iron oxide nanoparticle , biocompatible, heavy metal

PREPARATION AND PROPERTIES OF PMMA SPHERES AS 3 DIMENSIONAL PHOTONIC CRYSTALS AND ITS THIN FILM VIA SURFACTANT-FREE EMULSION POLYMERIZATION

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Abstract

3-dimensional (3D) photonic crystals have been extended use in wide research and application from material to sensor. Nanoparticles of poly (methyl methacrylate) (PMMA) latex beads have been successfully prepared by green-chemistry approach where no surfactant, linking agent and solvent were involved. Regardless of the effect of initiator in polymerization reaction, this study presents the effect of reaction condition where temperature, monomer concentration, stirring speed and reaction period as parameters in order to tune the particle size of spheres. Its morphology of uniformity sized-tuned was confirming by using particle size analyzer (PSA) and scanning electron microscopy (SEM). The fabrication of 3D photonic crystals film by using self-assembly method to pattern the desired PMMA layers which is the most feasible, low cost method are also presented. The detailed properties of PMMA nanoparticles from this experimental study will be discussed and its potential used in photonic application will be explained.

Keywords: photonic crystals, surfactant -free, poly(methyl methacrylate), nanostructured polymers, colloids, films, self -assembly

GREEN SYNTHESIS AND CHARACTERIZATION OF SILICA NANOPARTICLES USING COCONUT SHELL

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Abstract

This study was conducted to synthesis silica nanoparticles (SiNPs) from coconut shell. Coconut shell is an agricultural waste, which can be used as a renewable source to produce silica as coconut shell contains approximately 88% of silica oxides. As most of the coconut shell is thrown away after harvest, reusing coconut shell as an alternative source in silica extraction may reduce waste generated from the plantation industry. At the earlier stage of this study, coconut shell was treated with NaOH for the formation of sodium silicate solution with the pH range of 12 to 13. Neutralization of sodium silicate solution was carried out using three types of acids, which are nitric acid, sulphuric acid and orthophosphoric acids at consistent acid concentration. Silica gel was formed at pH <10, and the effect of different solvents used during aging process also was determined. SiNPs produced were then characterized using Fourier Transform Infrared (FTIR) Spectroscopy. The presence of Si-H, Si-O-Si anti-symmetric group, and O-H stretching vibration indicates the silica formation, while the crystallinity nature of SiNPs formed was confirmed using X-Ray Diffraction (XRD) analysis. A broad peak centered at $2\theta = 25^\circ$ shows that SiNPs have an amorphous nature and highly disorder structure. The surface morphology of obtained SiNPs was observed using Scanning Electron Microscopy (SEM). The different acids and solvents used during the synthesis give a significant result to the shape and size of the nanoparticles obtained.

Keywords: coconut shell, silica nanoparticle, sodium silicate solution, green synthesis

SYNTHESIS AND CHARACTERIZATION OF Zn-Al-LAYERED DOUBLE HYDROXIDE – CAPTOPRIL NANOCOMPOSITE

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Abstract

A nonsteroidal anti-hypertensive drugs, captopril (CPL) was successfully intercalated into zinc-aluminium-layered-double hydroxide, LDH for the formation of zinc-aluminium-captopril nanocomposite, LDHCPL by self-assembly method. As the result of the successful intercalation of nanocomposite, the basal spacing has increased from 8.91Å in LDH to 9.69 Å in LDHCPL hybrid nanocomposite. The FTIR spectra of the LDHCPL hybrid nanocomposite shows the resemblance functional groups of LDH and CPL, suggesting the inclusion of the organic compound into the LDH interlamellae. The percentage loading of CPL was found to be 30.63% (w/w) calculated from the percentage of carbon in the resulting material of LDHCPL. It was also found that the BET surface area increased from 1.15 m^2/g to 10.96 m^2/g for LDH and LDHCPL, respectively. The pore texture of the resulting materials was also changed as the result of the intercalation and the expansion of the basal spacing together with the pore formation during the intercalation to form LDHCPL nanocomposite.

Keywords: captopril, anti-hypertensive drugs, layered double hydroxide, intercalation, nanocomposite

THE EFFECT OF pH ON COPPER (II) LOADED ON TITANIANANOTUBES AS ELECTRODE FOR PHOTOELECTROCHEMICAL CELL

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Abstract

In this study, copper metal loaded onto titania nanotubes (CuTNT) were successfully prepared via facile anodization followed by Successive Ionic Layer Adsorption Reaction (SILAR) method using sodium borohydride as the reducing agent. The effect of precursor solution pH on the formation of CuTNT was investigated using Field Emission Scanning Microscopy (FESEM), Energy Dispersive X-Ray (EDX), and X-Ray Diffraction (XRD). Copper metal was successfully deposited onto the TNT as proven by FESEM, EDX and XRD analysis. From the photoelectrochemical test, CuTNT synthesized at neutral pH labeled as CuTNT/pH7 exhibits the highest PEC performance (η=7.05%) compared to the other CuTNTs and even two times better than blank TNT (η=3.32%). However, reducing the pH of copper (II) solution to pH 3 and 5 has decreased the PEC performance of CuTNTs probably due to the undefined shaped copper which forms covering layer on TNT surface. Increasing the pH further to 9 also results in similar trend but probably due to precipitation of copper metal cations in the alkaline medium and large clumps resulted that blocked the TNT surface from light entrance. The improvement of PEC performance of CuTNT/pH7 was due to the narrowing band gap effect and the broadened utilization of light spectrum resulted from the introduction of copper into TNT, proven by UV-DRS analysis, as well as the uniform distribution of copper which maximizes the effect.

Keywords: titania nanotubes, copper loaded titania nanotubes, Successive Ionic Layer Adsorption Reaction, photoelectrochemical cell

EFFECT OF CONCENTRATION OF GOLD SOLUTION ON THE PHOTOELECTROCHEMICAL PROPERTIES OF GOLD DECORATED TITANIUM DIOXIDE NANOTUBES

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Abstract

Solar photoelectrochemical (PEC) water splitting for hydrogen production is a promising technologies that provides a clean, eco-friendly and cost-effective technology by taking advantage of the solar light as energy source. Metal oxides such as TiO_2 is more preferable as photoanode in PEC water splitting as it has relatively high reactivity, stable in aqueous solution and lower cost than other non-oxide semiconductors. However, the large band gap of TiO_2 (3.2 eV) only allowed it to function under irradiation of UV light. Thus, gold nanoparticles were decorated onto the TiO_2 nanotubes (TNT) to extend the spectral response of TNT to visible region. Gold decorated titanium dioxide nanotubes (AuTNT) were synthesized by pulse electrodeposition of gold nanoparticles onto the titanium dioxide nanotubes. The electrodeposition was carried out in 0.5M H_2SO_4 containing 50µM , 100 µM, 500 µM and 1000µM of Au solution with 75% duty cycle and an applied potential of -0.5V for 20 minutes with in a three-electrode electrochemical cell. The titanium dioxide nanotubes were annealed at 500^{o}C for 2 hours in order to induce the crystallinity of TNT prior to deposition. The physicochemical properties of the AuTNT were characterized by using FESEM, EDX and XRD. Photoelectrochemical properties of AuTNT electrode was evaluated in 0.5M Na_2SO_4 and 2M C_2H_5OH under illumination from a halogen lamp. The AuTNT electrode prepared with concentration of 50µM Au solution gives the highest photocurrent compared to other concentrations of Au.

Keywords: gold nanoparticles, titanium dioxide nanotube, pulse electrodeposition, photoelectrochemical properties

EFFECT OF AL-NANOSTRUCTURED COATING ON THE CORROSION BEHAVIOR OF MILD STEEL FOR CURRENT DEPENDENT TIME BY ELECTRON BEAM THERMAL EVAPORATOR

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Abstract

One way to protect mild steel from corrosion is by coating it with Nanostructured film. In this study Aluminium nanostructured coatings were successfully coated on the mild steel substrate by electron beam thermal evaporator technique. The deposition of Al-nanostructured on mild steel substrate was carried out by applying a constant deposition time of 5 min with various current deposition such as 55 A, 60 A, and 70 A. The morphology of Al-nanostructured coatings was characterized by FESEM. The deposition current of Al-coated on mild steel is optimized at 60 A, changed the morphology of coatings with more smooth compact coatings structure. The Al-nanoparticles will fill up the uneven surface mild steel substrate at the size of nanometer compared to the normal coating only cover the substrate surface. The void will be created between the substrate and coating material. Corrosion rate measurements show that Al-nanostructures coatings can enhance the corrosion resistance of the mild steel in 5.0 M sodium chloride solution. The presence of Al-nanostructured has greater corrosion protection for mild steel. The order of the corrosion resistance of the sample coatings in protection mild steel in 5.0 M NaCl is 55A>60 A>70 A.

Keywords: Al-nanostructured, mild steel, corrosion resistance

PHASE FORMATION AND PARTICLE SIZE OF Ti DOPED $LiMn_2O_4$

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Abstract

Lithium cobalt oxide ($LiCoO_2$) is currently being used commercially as a cathode material for lithium-ion batteries. Unfortunately, due to its high cost and not abundant in source, spinel lithium manganese oxide ($LiMn_2O_4$) have gained attention as an alternative. In this research, Mn has been partially substituted with Ti in order to overcome the problem of severe capacity fading by $LiMn_2O_4$. $LiMn_{2-x}Ti_xO_4$ (x = 0.1 and 0.2) was prepared by a self-propagating combustion method (SPC). The materials were annealed at 700 °C for 24 h. Phase analysis was done by performing X-Ray Diffraction (XRD). The morphologhy and the particle size of the materials were done *via* Field emission Scanning electron Microscopy (FESEM)(JEOL JSM-7600F).

Keywords: lihium-ion battery, $LiMn_2O_4$, phase, particle size

THE EFFECT OF PEG ON THE CRYSTALLITE SIZE OF SnO_2 NANOPARTICLES

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Abstract

Tin Oxide (SnO_2) is well known metal oxide which has wide range of applications in the industry. In this study, SnO_2 powders were synthesized by a simple and low cost method which is the sol-gel method. This method has been modified using polyethylene glycol (PEG) as a surfactant. 5 ml and 10 ml of PEG is added during synthesis. The products were annealed at 350 °C for 24 h. The annealed samples were characterized using X-Ray Diffraction (XRD) for phase studies. Subsequently, the crystallite size and morphology of SnO_2 were determined using High Resolution Transmisson Electron Microscopy (HRTEM). It is found that the 10 ml PEG SnO_2 has smaller crystallite size compared to the 5 ml PEG material.

Keywords: crystallite size, phase, tin oxide

CHARACTERISTICS OF $LiMn_2O_4$ DOPED WITH Ti AND Sn CATHODE MATERIALS FOR LITHIUM-ION BATTERIES

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Abstract

Lithium manganese oxide $LiMn_2O_4$ with the spinel structure is a well-known cathode material used in high energy density batteries as it has several advantages such as lower cost and less toxic. But, $LiMn_2O_4$ has a drawback of large capacity fading when cycling at high temperature. In this research work, Mn has been partially substituted with Ti and Sn in order to overcome this problem. $LiMn_2O_4$ a nd $LiMn_{1.9}$ $Ti_{(1.9-x)}Sn_xO_4$ (x = 0.001 and 0.005) powder were synthesized by a self-propagating combustion method (SPC). The materials were annealed at 800 °C for 24 h. Phase analysis was done by performing X-Ray Diffraction (XRD). XRD results showed that the materials are pure and single phase with FCC structure. The morphologhy and the particle size of the materials were done *via* Field emission Scanning electron Microscopy (FESEM)(JEOL JSM-7600F).

Keywords: lihium-ion battery, $LiMn_2O_4$, phase, particle size

ROLE OF SILANOL GROUPS IN IONIC MOBILITY OF SILICON DIOXIDE FILLED POLYMER ELECTROLYTES

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Abstract

In this study, silanol (Si-OH) groups on the surface of silicon dioxide (SiO_2) were removed by chemical modification with hydrochloric acid (HCl) at various acid concentrations. The HCl modified SiO_2 (HCl-SiO_2) then was used as filler in preparation of polymethyl methacrylate/50% epoxidized natural rubber (PMMA/ENR 50) electrolytes with lithium tetrafluoroborate ($LiBF_4$) as dopant salt. Fourier transform infrared (FTIR) analysis revealed that HCl-SiO_2 and SiO_2 show similar bands characteristic of O-H, Si-O-Si asymmetric straching, Si-O-Si bending, Si-O-Si rocking and Si-OH. However, the intensity of these peaks were varied when the concentration of HCl used was varied. Meanwhile, HCl-SiO_2 filled PMMA/ENR 50 (PELM15) electrolytes show similar FTIR spectrum as SiO_2 filled PMMA/ENR 50 (PEL15) electrolyte with C=O, O-CH_3, C-O-C and BF_4^- ion peak recorded at ~1700, ~1400, ~1200 and ~700 cm^{-1}, respectively. Interestingly, the position and intensity of these peaks were non identical and were HCl concentration dependence. Surprisingly, room temperature ionic conductivity of PELM15 electrolytes are one magnitude lower than PEL15 electrolyte.

Keywords: silicon dioxide, modified silicon dioxide, silanol group, polymer electrolyte, 50% epoxidized natural rubber, polymethyl methacrylate, lithium tetra-fluoroborate

GREEN SYNTHESIS OF ZINC OXIDE NANOPARTICLES WITH ENHANCED DEGRADATION OF PHENOL

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Abstract

In this study, ZnO nanoparticles were synthesized by a green approach via electrochemical method, and were characterized by XRD, TEM, BET surface area, and particle analyzer. The physicochemical analyses revealed high purity and wurtzite hexagonal structure of ZnO with a large surface area and lessen particle size (< 10 nm). The formation of ZnO nanoparticles affected its photoactivity, which clearly observed when it was tested on a degradation of phenol compound under visible light. Nearly complete degradation of phenol was achieved compared to commercial ZnO after 2 h exposure to light irradiation.

Keywords: ZnO nanoparticles, green synthesis, electrochemical, phenol, photodegradation

DETERMINATION OF POLYPHENOL CONTENTS IN *Hevea brasiliensis* AND RUBBER-PROCESSING EFFLUENT VIA FTIR AND REVERSE PHASE-HPLC ANALYSIS

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Abstract

The information on polyphenol composition in *Hevea brasiliensis* is limited albeit the importance on understanding the value of this crucial phytochemical, especially in terms of plant protection. *H. brasiliensis* produces valuable latex and has become one of the important commodities in the world. The profiling of polyphenols is therefore vital to understand its abundance in the system of the crop and also towards sustaining rubber production. There is also no report on the content of polyphenols in rubber processing effluents. Knowing the composition of valuable compounds in the waste can further generate profit from the industry and creating a sustainable wealth from source of waste. The objective of this study is to determine the presence of polyphenol compounds in C-serum and effluent using Fourier transform infrared spectroscopy (FTIR) analysis. This study also aimed to develop and profile specific phenolics via High Performance Liquid Chromatography (HPLC). Results from the FTIR analysis show the presence of the polyphenol in both latex and effluent of rubber processing. An optimal method for determining polyphenol from the selected samples on HPLC has been determined. HPLC analysis showed detection of several polyphenol peaks in both latex and effluent when compared to aunthentic polyphenol standards. The current achievement in this study marks the potential of understanding polyphenol composition in latex of *H. brasiliensis* and effluent from rubber processing which has not been explored before.

Keywords: polyphenols, *Hevea brasiliensis*, latex, HPLC, FTIR

SYNTHESIS AND ANTIOXIDANT ACTIVITIES OF 6-HYDROXYL-4-METHYLCOUMARIN AND ITS DERIVATIVES

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Abstract

Four coumarin derivatives compounds namely 6-hydroxyl-4-methylcoumarin (**I**), 6-hydroxyl-4-methyl-5-(p-nitrophenyl azocoumarin) (**II**), 6-hydroxyl-4-methyl-5,7-(bis-*p*-nitrophenyl azocoumarin) (**III**) and 6-hydroxyl-4-methyl-5,7-(bis-*p*-chlorophenyl azocoumarin) (**IV**) were successfully synthesized by reacting hydroquinone with ethylacetoacetate and selected amines. The compounds were characterized by CHN Micro-Elemental Analysis, Nuclear Magnetic Resonance (NMR) and Fourier Transform Infrared (FTIR) spectroscopic methods. The infrared spectra of these compounds exhibit five important stretching vibrations: ʋ(-OH), ʋ(C=O), ʋ(C=C), ʋ(C-O) and ʋ(C-N) at the range of 3441-3359 cm^{-1}, 1604-1632 cm^{-1}, 1581-1496 cm^{-1}, 1331-1225 cm^{-1} and 1251-1109 cm^{-1}, respectively. ^{1}H NMR spectra of these compounds show the presence of proton aromatic at the chemical shift of δH 7.00-8.70 ppm, proton methyl (δH 2.20-2.50 ppm) and proton pyrone ring (δH 6.10-6.90 ppm). CHN analysis results of all compounds are in good agreement with the calculated values. All the synthesized compounds were evaluated for their antioxidant activity using DPPH method, where ascorbic acid was used as a standard. UV-Vis spectroscopic technique was used to investigate the absorbance of these compounds. Compound (**II**) exhibits high antioxidant activitiy compared to compounds (I), (III) and (IV) which possess moderate to low activities. The colour changes from yellow to purple of the compounds were easily observed by naked eyes.

Keywords: coumarin, antioxidant activity, DPPH, ascorbic acid

COMPUTATIONAL APPROACH AND SYNTHETHIC STUDIES OF RUTHENIUM ALKYNYL COMPLEXES FOR NONLINEAR OPTIC APPLICATION

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Abstract

Metal alkynyl complexes are of interest for a variety of possible applications, one being advanced nonlinear optical materials. Octupolar symmetry organometallic complexes can give high nonlinear optical responses. In this paper, linear ruthenium alkynyl complexes, Ru(C≡C-4-C6H4C≡CPh)(dppe)(Cp) have been prepared and fully characterized. Studies of octupolar complexes were carried out using Amsterdam Density Functional (ADF) software. The linear and nonlinear optical properties of the complexes have been examined and the experimental results have been rationalized by computational studies employing time-dependent density functional theory. The synthesized complexes are shown to have high potential in nonlinear optic application, as proven by computational works.

Keywords: ruthenium, nonlinear optic, computational studies, ADF

SYNTHESIS AND CHARACTERIZATION OF BENZOHYDROXAMIC ACID AND METHYLBENZOHYDROXAMIC ACID METAL COMPLEXES AND THEIR CYTOTOXICTY STUDY

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Abstract

Hydroxamic acids (RCONHOH) are flexible compounds for organic and inorganic analyses due to their frailer structures compared to carboxylic acid. These acids are easy to deprotonate and produce hydroxamate ions. The syntheses, physico-chemical and characterization of benzohydroxamic acid (BHA) and methylbenzohydroxamic acid (CH_3-BHA) and their metal complexes (VO(IV), Cr(III) and Ni(II)) are reported herein. The metal complexes were synthesized by condensation reaction of BHA and CH_3BHA with metal salts in 2:1 molar ratio. The compounds were characterized by elemental analysis, infrared spectroscopy, ^{1}H and ^{13}C NMR, UV-Vis, TGA, magnetic susceptibility and molar conductance. From IR and magnetic susceptibility, each complexes coordinated to the metal via oxygen atoms (*O,O*) in bidentate manner to form octahedral geometries. The molar conductance values suggested that all complexes were non-electrolytes. A cytotoxicity study against HCT116 displayed that all of the complexes have better anticancer than their parent ligands.

Keywords: hydroxamic acid, vanadium(IV), chromium(III), nickel(II), cytotoxicity, HCT116

GREEN SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL APPLICATION OF Ni(II) AND Zn(II) BIS-THIOCARBOHYDRAZONE COMPLEXES

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Abstract

Two series of Ni(II) and Zn(II) complexes derived from bis-salicylaldehyde derivative thiocarbohydrazones were synthesized. These series were synthesized in alcoholic solution by microware irradiation. All compounds were characterized by elemental analysis, magnetic susceptibility, molar conductance, UV, IR, 1H NMR spectroscopies. The chelation between the thiocarbohydrazone and metal occurred at the azomethine nitrogen atom, thio-sulfur group and phenolic oxygen atom. Both Ni(II) and Zn(II) complexes exhibit tetrahedral structures. Preliminary antimicrobial screening using Disc diffusion method against Escherichia coli, Bacillus subtilis, Enterobacter cloacae, and Klebsiella pneumoniae showed that the ligands and their respective complexes possess antimicrobial activity towards bacteria.

Keywords: Ni(II) complexes, Zn(II) complexes

SYNTHESIS, CHARACTERIZATION AND CYTOTOXICITY OF S-BENZYLDITHIOCARBAZATE SCHIFF BASES WITH DIBENZALACETONE, DICINNAMALCAETONE AND DI-*P*-METHOXYBENZALACETONE AND THEIR TRANSITION METAL COMPLEXES

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Abstract

Three Schiff bases have been synthesized by acid catalyzed reaction between S-benzyldithiocarbazate (SBDTC) and three different ketones which are dibenzalacetone, dicinnamalacetone & di-*p*-methoxybenzalacetone. These ketones have been synthesized by using base catalyzed Aldol condensation method from acetone with benzaldehyde, cinnamladehyde or *p*-methoxybenzaldehyde. Fifteen metal complexes have been synthesized from these three Schiff bases with Ni(II), Cu(II), Fe(II), Cd(II) & Zn(II) acetates. All Schiff bases and their metal complexes have been fully characterized by physical and spectroscopic techniques such as melting point, molar conductivity, magnetic susceptibility, IR spectroscopy, UV-Vis spectroscopy, GC-MS spectra, ^{1}H & ^{13}C NMR, ICP-OES & CHNS analysis. Some metal complexes have been characterized by single crystal-XRD after growing crystals from metal complexes by using tetrahydrofuran and cyclohexane solvents in a slow diffusion crystal growth method. The yielded metal complexes have been tested to evaluate their cytotoxicity toward two types of bladder cancer cell lines which are EJ-28 & RT-112. This study aimed to determine the main affecting factor on the anticancer activity of this type of Schiff bases. All of these compounds have high conjugation single bond–double bond system. Dicinnamalacetone has longer aliphatic conjugation system while di-*p*-methoxybenzalacetone used to test the effect of methoxy group on the cytotoxicity. As a result from this study, the cytotoxicity of transition metal complexes toward bladder cancer cell line EJ-28 depends on the type of functional group substituted on benzene ring rather than the elongation of aliphatic conjugation system. Beside this, the cytotoxicity activity of these metal complexes depends on the type of transition metal used. Fe(II) & Cu(II) metal complexes with di-*p*-methoxybenzalacetone-SBDTC have been showed better cytotoxicity than the others. All Schiff Bases and their metal complexes were inactive toward bladder cancer cell line type RT-112.

Keywords: S-benzyldithiocarbazate, Schiff base, aldol condensation, dibenzalacetone, dicinnamalacetone, di-p-methoxybenzalacetone, SBDTC, metal complexes

SYNTHESIS, CHARACTERISATION AND EFFECT OF TEMPERATURE ON CORROSION INHIBITION BY THIOSEMICARBAZONE DERIVATIVES AND ITS TIN(IV) COMPLEXES

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Abstract

The thiosemicarbazones derivatives ligands and its tin complexes used in this study were as follows: 2-acetylpyridine 4-ethyl-3-thiosemicarbazone (A), 2-acetylpyridine 4-methyl-3-thiosemicarbazone (B), phenyltin trichloride 2-acetylpyridine 4-ethyl-3-thiosemicarbazone (C) and phenyltin trichloride 2-acetylpyridine 4-methyl-3-thiosemicarbazone (D). All the title compounds were characterised using elemental analyser, FTIR, UV-Vis and NMR. The efficiency of the synthesised compounds were investigated as a corrosion inhibitor of mild steel in 1.0 M HCl by using weight loss technique at different concentrations, 1mM, 2mM and 3mM and also at temperature range 30-60°C. The thiosemicarbazone ligand and its tin complexes proved the hypothesis where the inhibitor efficiency tends to increase as an inhibitor concentration increase, indicating their potential use as a corrosion inhibitor for mild steel. Moreover, the inhibitors were more effective when the temperature increases.

Keywords: thiosemicarbazones, corrosion inhibitor, temperature, mild steel

SYNTHESIS AND CORROSION INHIBITION STUDIES OF *N*-((*o, p* -AMINOPHENYL) CARBAMOTHIOYL) BENZAMIDE WITH Cu(II) COMPLEXES IN 1M HCl

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Abstract

N-((2-aminophenyl) carbamothioyl) benzamide(A1) and N-((4-aminophenyl) carbamothioyl) benzamide(A2) with its copper(II) complexes were successfully synthesized using microwave method. The synthesized compounds were characterized by elemental analysis (CHNS), IR spectroscopy, NMR (^{1}H and ^{13}C) spectroscopy, melting point and magnetic susceptibility determination method. The inhibition effect of A1, A2 and its copper complexes on the mild steel in 1M HCl medium has been investigated using potentiostatic polarization technique. From the obtained results, the highest inhibition efficiency of A1 is 98% while for A2 is 99%. The percentage inhibition efficiency of A2 is higher than A1 due to the difference position of substituent at ortho and para.

Keywords: thiourea, corrosion inhibition, potentiostatic polarization, mild steel

INVESTIGATION ON CONJUGATED ALKOXY SUBSTITUTED STILBENE IMINE DERIVATIVES AS SEMICONDUCTIVE MATERIALS

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Abstract

Recently, the development of organic semiconducting materials have drawn intensive attention for molecular electronic applications due to their unique characteristics such as low cost of production, lightness and compatibility with large-scale flexible substrates. Therefore, remarkable progress of organic semiconductor classes have been extensively studied based on conjugated molecules to enhance carrier mobility, electrical performance and can perform function at an optimum level. In this present study, a new type of hybrid moieties of alkoxy stilbene imine derivatives inspired from two well-known individual conjugated π-systems; vinylene (C=C) and azomethine (CH=N) have been successfully integrated into an addition of organic semiconducting materials. The assessment of 4-[(ethoxyphenyl)methylene] amino)-4’-stilbene (**3a**), 4-[(hexyloxyphenyl) methylene]amino)-4’-stilbene (**3b**) and 4-[(decyloxyphenyl)methylene]amino)-4’-stilbene (**3c**) based on Donor (D)-π-Acceptor (A) system have been successfully synthesized as active semiconductor material candidates. These compounds were evaluated via several spectroscopic and analytical techniques namely infrared (IR), UV-visible (UV-vis), ^{1}H and ^{13}C Nuclear Magnetic Resonance (NMR). The relationships between electronic and optical properties, chemical modeling at molecular interactions and thermal stability of the designated system were evaluated. In addition, the quantum mechanical calculation proved that the value of energy separation of **3a-3c** between HOMO and LUMO exhibits around 3.50-3.55 eV which was in good agreement with the experimental result of optical band gap. From the preliminary result, a good relation between the experimental and theoretical data provides strong support of being a potential candidate in organic semiconductor application, particularly in the respect of their low and good stability of HOMO-LUMO energies. The findings from the thermal analysis revealed that the targeted compounds exhibited thermal stability at high temperature and it gave good indications for the potential of thin film fabrication be applied as semiconductive film for it displays remarkable performance under prolonged thermal stress. Therefore, this type of molecular framework has given an ideal indication to act ideally as organic semiconductor materials potentially use in any designated application particularly in optoelectronic devices.

Keywords: stilbene imine, optical band gap, semiconductive materials

MICROWAVE-ASSISTED SYNTHESIS, CHARACTERIZATION AND ANTICANCER SCREENING OF TETRANUCLEAR SCHIFF BASE COMPLEXES

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Abstract

Hexadentate Schiff base (OVanMPD) ligand was synthesized via condensation of *o*-vanillin (OVan) and *m*-phenylenediamine (MPD). Their Cu(II), Co(II) and Zn(II) complexes were obtained through microwave-assisted complexation with corresponding acetate salts. The compounds were characterized by elemental analyser, molar conductivity, magnetic susceptibility, TGA, IR, UV–Visible and ^{1}H NMR spectroscopy. The IR spectra showed the shifting of $\upsilon(C=N)$, $\upsilon(C\text{-}O)_{phenolic}$ and $\upsilon(C\text{-}O)_{methoxy}$ peaks to lower frequencies with appearance of new peaks assignable to $\upsilon(M\text{-}N)$ and $\upsilon(M\text{-}O)$ in the ranges 529-565 and 424-490 cm^{-1}, respectively, indicating the coordination of the metal centres to the azomethine N, phenolic O and methoxy O. The Cu_4(OVanMPD) and Co_4(OVanMPD) complexes were paramagnetic with μ_{eff} of 1.85 and 3.84 B.M., respectively. The anticancer activities of all compounds were evaluated against *colon cancer cell* (HCT116) cell lines. The parent ligand revealed a lower anticancer activity than its metal complexes with Cu_4(OVanMPD) showing the highest activity having IC_{50} of 6.56±1.26 µM.

Keywords: microwave-assisted, tetranuclear complexes, Schiff base, anticancer screening

SYNTHESIS AND CHARACTERIZATION OF CARBONYL THIOUREA DERIVATIVES AS ORGANOCATALYST

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Abstract

A present work deals with the synthesis of a series carbonylthiourea derivatives namely*N*-((4-aminobutyl)carbamothioyl)-2-methylbenzamide **(2ABMB)**, *N*-((4-aminobutyl)carbamo thioyl)-3-methylbenzamide **(3ABMB)** and *N*-((4-aminobutyl)carbamothioyl)-4-methyl benzamide **(4ABMB)** using condensation reaction between *o ,m,p*-methylbenzoyl chloride and 1,4-diaminobutane. The synthesized compounds were characterized by FTIR and^{1}H and ^{13}C NMR Spectroscopy. The infrared spectra of these compounds showed four significant stretching vibrations, (N-H), (C=O), (C-N) and (C=S) at 3288–3312 cm^{-1}, 1629-1637 cm^{-1}, 1384-1430 cm^{-1} and 741-753 cm^{-1} respectively. There are two significant amine protons showed in the ^{1}H NMR spectra which are-C=S-NH (δ_H 8.4 ppm) and -C=O-NH (δ_H10.9 ppm).In ^{13}C NMR spectra, the signal of carbon carbonyl (C=O) can be observed at δ_C 167-168 ppm. Chemical shift of the carbon thione groups (C=S) appeared at δ_C179-180 ppm. The synthesized compounds were tested as organocatalyst in Michael addition reaction of *N*-phenylmaleimide and isobutyraldehyde. The performances of the catalytic study were monitored by Gas Chromatography- Flame Ionization Detector (GC-FID) at 6 hour reaction period with 20% mol of organocatalyst. The percentage conversion of N-phenylmalemide to the product was up to 70%.

Keywords: carbonylthiourea, Michael addition reaction, *N*-phenylmaleimide, isobutyraldehyde, organocatalyst

SEMICONDUCTIVE ETHYNYLATED SCHIFF-BASE DERIVATIVES IN THE INTEREST OF MOLECULAR ELECTRONICS

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Abstract

The flexibility and rigid rod-like nature of the conjugated system in ethynylated Schiff-base derivatives enhance the development of donor-π-acceptor (D-π-A) and donor-π-donor (D-π-D) system which can act as organic molecular wires namely organic thin-film transistor (OTFT) and organic light emitting diode (OLED). Inspired by these well-known π-systems, the combination of acetylide (C≡C) and imine (C=N) moieties have been integrated into a new class of molecular system. The exploration into this frontier have been made in this work through the synthesis of three new ethynylated Schiff-base derivatives, namely N-(4-nitrobenzylidene)-4- phenylethynylaniline (2a), N-(4-hexyloxybenzylidene)-4-phenylethynylaniline (2b), and N-(4-hexyloxybenzylidene)-4-tolylethynylaniline (2c). 2a-2c were successfully synthesised, and characterised for their spectroscopic, thermal analysis, electrochemical behaviour, surface morphology analysis, and semiconductor properties via electrical and optical investigation prior to form active layer significantly for organic molecular wires application. Thermal analysis of 2a-2c revealed that all compounds were stable up to 326°C and gave great indication to be further fabricated as molecular wires devices. Moreover, the surface morphology of *2a-2c* were observed in stacked and layered form. The energy band gaps (E_g) measured exhibited that 2a-2c are indeed organic semiconductor materials. Ultimately, electroluminescence analysis have shown that 2a and 2b possessed excellent electroluminescence behaviours with maximum light emission at 20V. Meanwhile, the study of electrical conductivity of 2c thin film by using four point probe and two point probe respectively revealed 2c is a conductive material due to the presence of alkoxy chain acts as electron donating group that can decrease the resistivity value. Therefore, this type of molecular framework featuring 2a-2c derivatives are an ideal candidates as OTFT and OLED potentially to be applied in various microelectronic devices.

Keywords: ethynyl, Schiff-base, conductivity, molecular wires, spectroscopic, OTFT, OLED

THE EFFECT OF SUBSTITUENTS GROUP OF SCHIFF BASES ON CORROSION INHIBITION OF MILD STEEL

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Abstract

Schiff base ligands namely salicylideneaniline(L1) and ovan-aniline(L2) were syntheses via condensation reaction giving yield of 80.74% and 81.7%, respectively. The ligands were then characterized by physicochemical and spectroscopic techniques namely melting point, micro elemental analysis (C, H and N), ^{1}H Nuclear Magnetic Resonance (NMR) and Infrared (IR) spectroscopy. The characteristic υ(C=N) peak for salicylideneaniline and ovan-aniline were observed at 1615 and 1613 cm^{-1} and OH) at 13.27 and 13.37 ppm, respectively. Cyclic voltammetry (CV) and chronoamperometry (CA) techniques were employed to electrodeposit both compounds on mild steel at 0.05 M concentration in 0.3 M NaOH. The formation of yellow and brownish imine layers were observed on all coated mild steel. The corrosion behavior of coated and uncoated mild steel was studied using Tafel Extrapolation Method in 0.5 M NaCl. The coated mild steel showed better corrosion resistance compared to the uncoated. The mild steel coated with L2 through CA technique revealed the highest inhibition efficiency, hence indicating a better surface coverage, at potential +0.90 V with 97.20 % of inhibition efficiency while L1 gives highest inhibition efficiency with 93.83 % at potential +1.35 V. SEM observations confirmed the presence of protective film on mild steel surface.

Keywords: Schiff base, electrodeposition, cyclic voltammetry, chronoamperometry, LPR

SYNTHESIS AND CHARACTERIZATION OF NEW *N*-((5-CHLORO-2-PHENOXYPHENYL)CARBAMOTHIOYL) THIOUREA DERIVATIVES

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Abstract

A series of acyl thiourea derivatives namely N-((5-chloro-2-phenoxyphenyl)carbamothioyl)hexanamide **(I)**, N-((5-chloro-2-phenoxyphenyl)carbamothioyl)heptanamide **(II)**, and N-((5-chloro-2-phenoxyphenyl)carbamothioyl)octanamide **(III)** were synthesized and characterized by typical spectroscopic method. The FTIR spectra of the synthesized compounds revealed the existence of three important peaks in the range of 3191-3196 cm^{-1}, 1688-1691 cm^{-1} and 744-747 cm^{-1}, which corresponded to ν(N-H), ν(C=O) and ν(C=S), respectively. The ^{1}H NMR spectra of all compounds showed the appearance of two significant amine proton resonances which were -C=O-N*H* and -C=S-N*H* at δ_H 11.54-11.57 ppm and δ_H 12.97-13.00 ppm, respectively. Whilst, the C=O and C=S carbon signal were detected within the range of δ_C175 - δ_C178 ppm, which has proven that thiourea compounds have been successfully synthesized.

Keywords: acyl chloride, aniline, acyl thiourea derivatives, amides

SCHIFF BASE METAL COMPLEX: POTENTIAL OPTICAL PROBE FOR PORCINE DNA

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Abstract

The salphen ligand, N,N'-phenylenebis(salicylideneimine) is a Schiff base which can be prepared from a condensation reaction using one equivalent of a diamine and two equivalents salicylaldehyde. The Schiff base's imine is formed via nucleophilic addition by amine to the electrophilic carbon atoms of the carbonyl group of salicylaldehyde. This reaction yields an hemiaminal intermediate followed by the elimination of water generating a diimine. Their respective metal complexes can be easily obtained by treatment with metal salts (normally acetate) or in some cases via activation of the oxygen of the phenolic group with base (e.g. triethylamine). Square planar metal salphen complexes was reported to interact with different types of DNAs. Studies on identification of pork content in food have grown rapidly to meet the Halal food standard. Targeting porcine mitochondria DNA is thought to be the most accurate marker for porcine identification. In this project N,N'-Bis-5-(hydroxysalicylidene)-phenylenediamine-Ptl(II) complexes were synthesized and characterized. The binding mode and interactions of this complex with porcine DNA were studied by UV-Vis and emission DNA titration. The phosphorescence emission of Pt(II) complex when interacted with the DNA has been studied. Also, the application of the chosen complex as an optical porcine DNA sensor was investigated. These findings will be valuable for exploring the potential use of the Schiff base metal salphen complex as new DNA biosensor optical sensing material, for the detection of porcine DNA in food products analyses to provide a visual, fast and simpler technique compared to other conventional methods.

Keywords: metal salphen, Schiff base, porcine DNA, optical sensor, DNA biosensor

INTERACTION OF BIS SALPHEN COMPLEXES WITH HUMAN TELOMERIC G-QUADRUPLEX DNA

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Abstract

Apart from the normal double helix DNA conformation, certain guanine rich sequences of DNA can adopt unusual four stranded structure called G-quadruplex DNA. Stabilization of human telomeric (Htelo) G-quadruplex DNA was reported to inhibit telomerase, an enzyme which is overexpressed in 85-90% of cancerous cells. This research aimed to investigate the potential of bis salphen complexes as G-quadruplex DNA stabilizers. Stabilization of G-quadruplex DNA by metal complexes had gained more attention recently because the presence of metal centre can give optimal structural geometry for G-quadruplex DNA binding. In this research nickel(II) and zink(II) salphen complexeshad been synthesised and characterized. The wide planar bis salphen aromatic structure was expected to increase the ability of the complexes to bind strongly with G-quadruplex DNA via stacking interaction. All of the complexes were analyzed by Nuclear Magnetic Resonance spectroscopy (NMR), Infrared Spectroscopy (IR), elemental analysis (CHNS), mass spectrometry and Ultraviolet-Vis spectroscopy (UV-vis). The ability of complexes to stabilize human telomeric G-quadruplex DNA was carried out using UV-Vis and fluorescence titration studies. The binding affinity (K_b) of complexes to DNA was calculated using UV-Vis spectroscopic data. Bis salphens complexes showed selectivity towards DNA G-quadruplex over duplex CT-DNA with zinc(II) bis salphen complex bearing ester substituent showed highest binding affinity ($K_b = 6.61 \pm 0.66 \times 10^6$ M^{-1}). The complex showed good selectivity with 11- folds preference to G-quadruplex DNA over duplex DNA (CT DNA). Hypochromicity of 23% and red shift of 4 nm were observed from UV-Vis spectrum suggested that the complex interacts with G-quadruplex DNA via □□□ stacking interaction. From FID analysis and emission titration study suggested that the complex bind moderately to G-quadruplex DNA and showed enhancement of intensity upon addition of G-quadruplex DNA compared to CT-DNA by two folds. Finally, the anticancer activity of zinc(II) bis salphen complexes bearing ester substituent have been tested against human lung cancer line (A549) and normal cell line (MRC5). The complex showed higher cytotoxicity towards cancer cell line than normal cell. Zinc(II) bis salphen complexes bearing ester substituent showed the potential to be a good candidate for anti-cancer drug development.

Keywords: metal complexes, DNA binding, G-quadruplex DNA, Htelo

EFFECTS OF FATTY ACID COMPOSITIONS ON THE PRODUCTION OF FURFURAL COMPOUNDS DURING THE OXIDATION OF USED AND HEATED PLANT OILS

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Abstract

The presence of volatile compounds furfural and 5-hydroxymethylfurfural (5-HMF) were determined in selected fresh plant oils including canola oil, olive oil, palm oil, rice bran oil and virgin coconut oil. Additionally, the presence of these compounds were also determined in used plant oils and heated plant oils at deep-fat frying temperature of 175 ºC with different time duration (10 min, 20 min and 30 min). A modified steam distillation method was used to extract the volatile compounds from the selected plant oils before being analyzed using high performance liquid chromatography (HPLC) with ultraviolet (UV) detector at 284 nm.. The chromatograms obtained were compared to the standard furfural and 5-HMF. There is no presence of furfural and 5-HMF compound in fresh plant oils but they present in used plant oils and heated plant oils. The presence of this volatile compound was due to the lipid oxidation of the plant oils when exposed to heat. Results show that more unsaturated fatty acid components in plant oils yielded more furan compound compared to plant oils with higher saturated fatty acid components. Furthermore, furfural oxidise better than 5-HMF due to its low polarity.

Keywords: fatty acids, furfural, 5-hydroxymethylfurfural, oxidation, palm oil, canola oil, rice bran oil, virgin coconut oil

CHARACTERIZATION OF PALM FATTY ACID DISTILLATE OF SIME DARBY PLANTATION OIL REFINERY, MALAYSIA

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Abstract

Palm fatty acid distillate (PFAD) is cheap and valuable by product of edible oil processing industries. Palm fatty acid distillates are generally used in the soap industry, animal feed industry, and as raw materials for oleochemical industries, in the manufacture of candles, cosmetics and toiletries. Other applications include their use as food emulsifiers, an aid in rubber processing, in flavours and fragrance industries well as in pharmaceutical products. Vitamin E has been extracted commercially from PFAD for encapsulation. This study was carried out to determine the physicochemical properties of Malaysian palm fatty acid distilled (PFAD). The physicochemical properties showed that the free fatty acid (FFA %), acid value, iodine value, saponification value, unsaponifiable matter, hydroxyl value, specific gravity at 28°C, moisture content, viscosity at 40°C and colour at 28°C values were 87.04± 0.1 %, 190.6± 1 mg/g, 53.3±0.2 mg/g, 210.37±0.8 mg/g, 1.5±0.1%, 47±0.2 mg/g, 0.87 g/ml, 0.63 %, 30 cSt and yellowish respectively. Gas chromatography (GC) was used to determine the fatty acid (FA) composition in PFAD. The fatty acids were found to be comprised mostly with 48.9 % palmitic acid (C16:0), 37.4 % oleic acid (C18:1), 9.7 % linoleic acid (C18:2), 2.7 % stearic acid (C18:0) and 1.1 % myristic acid (C14:0). The analysis of high performance liquid chromatography (HPLC) has resulted with 99.2 % of FFA, while diacylglycerol and monoacylglycerol were 0.69 and 0.062 % respectively.

Keywords: palm oil, fatty acid, characterization, gas chromatography

OPTIMIZATION AND SWELLING STUDY IN DIFFERENT MEDIA OF CARBOXYMETHYL SAGO STARCH (CMSS) – ACID HYDROGEL

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Abstract

The present investigation deals with the optimization of carboxymethyl sago starch (CMSS) in order to produce a valuable smart hydrogel. The fabrication of the hydrogel was done by the addition of acetic acid to the CMSS. The degree of substitution (DS) of the CMSS obtained was 0.6410. The optimization was based on the gel content and degree of swelling of the CMSS - acid hydrogel. In this study, four parameters were studied in order to optimize the formation of CMSS – acid hydrogel. The parameters were ; percentage of CMSS, concentration of acetic acid, reaction time and reaction temperature. The optimum conditions to produce CMSS – acid hydrogel were at 80% (w/v) of CMSS, 2.0 M of acetic acid, 24 hours and 60°C reaction time and temperature, respectively. From the data analyzed, the percentage of optimum gel content of the CMSS – acid hydrogel was 76.69 % with degree of swelling of 33.77 g/g. In addition, the swelling properties of CMSS – acid hydrogel in water, different pH and media were also investigated. The results show that the CMSS – acid hydrogel swells in both alkaline and salt solutions. While in acidic solution, the hydrogel tends to shrink and deswell. The production of CMSS – acid hydrogel as a smart material can offer a lot of auspicious benefits in the future especially due to the swelling behavior in different media.

Keywords: carboxymethyl sago starch, hydrogel, optimization, gel content, swelling study

PREPARATION AND CHARACTERIZATION OF CMC HYDROGEL AS POTENTIAL VACCINE CARRIER

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Abstract

Carboxymethylcellulose (CMC) is a water-soluble polymer, which is widely used in various fields such as food additives, textiles, pharmaceuticals and cosmetics. CMC is the most important cellulose derivatives and carboxymethylation of cellulose is widely studied since there are numerous potential applications for this materials. Nowadays, chickens are susceptible to many infectious diseases. One of the viral disease known as Newcastle disease. Newcastle disease can be controlled by the use of Newcastle disease vaccine (NDV). In this study, hydrogel was prepared from CMC by using calcium chloride as a crosslinking agent and NDV was encapsulated in CMC hydrogel. Optimization of the CMC hydrogel was based on the gel content and swelling properties. 7% w/v amount of CMC in 2 % of calcium chloride solution for 24 hours at room temperature were the optimized conditions for the preparation of CMC hydrogel. The CMC and CMC hydrogel were characterized by using Fourier Transform Infrared (FT-IR), thermogravimetric analysis (TGA) and scanning electron microscopy (SEM). Optimization of CMC hydrogel load with NDV vaccine (strain Lasota) had been carried out at several different parameters which were loading time and amount of CMC hydrogel. The highest loading was 48.11% at 45 minutes loading time with 0.075g of CMC hydrogel. Antibody response to the haemagglutinin protein in the Newcastle disease virus envelope had been quantified using Haemagglutination Assay (HA) test which showed 32 HA titer value. In conclusion, CMC hydrogel shows that it can be used to encapsulate NDV vaccine.

Keywords: carboxymethylcellulose, hydrogel, polymer, calcium chloride, NDV vaccine

MODIFIED LEACHING OF ^{238}U AND ^{232}TH FROM WATER LEACH PURIFICATION (WLP) RESIDUE

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Abstract

In recent years, the importance of actinide elements mobile fractions is increasing in risk assessment evaluation of contaminated sites. In order to understand the mobility of ^{238}U and ^{232}Th, it is important to know about its sorption kinetics and the thermodynamics behind the sorption process on radioactive waste. In present study the leaching process of ^{238}U and ^{232}Th from Water Leach Purification (WLP) residue of *Lynas Advanced Materials Plant* (LAMP) was investigated to obtain the optimum leaching conditions using combination of Synthetic Precipitation Leaching Procedure (SPLP) and batch method, as simulation of acid rain and heavy flooding, respectively. In this study, initial ^{238}U and ^{232}Th concentration, pH and contact time of ^{238}U and ^{232}Th in WLP residue were investigated. The results show that the initial concentration of ^{238}U and ^{232}Th in WLP residue are 6.6 mg/kg and 206.1 mg/kg, respectively. In general, the highest values of the concentration after leaching process of ^{238}U and ^{232}Th observed are 0.363 mg/kg and 8.288 mg/kg, respectively. Both results shows that the maximum potential remobilization of ^{238}U and ^{232}Th at pH = 4 with same contact time which is at 14 days. From the finding, the maximum percentage of leached of 5.50 % and 3.99 % for ^{238}U and ^{232}Th, respectively. In the other hand, at pH = 7, the minimum leaching efficiencies of ^{238}U and ^{232}Th were 4.7% and 3.61 %, respectively. Thus, the effect of different pH prior to the remobilization of ^{238}U and ^{232}Th was found that the rate of leaching was influenced by the pH used. The study shows that the maximum concentration of ^{238}U and ^{232}Th were obtained at lower pH such as pH = 4. Meanwhile, pH values 7 and 8, the leached amount of ^{238}U and ^{232}Th is minimum. It could be concluded that the SPLP leaching method are practicle for estimating the leaching and remobilization of ^{232}Th and ^{238}U radionuclides from WLP residue,which could be useful for monitoring and risk assessment purposes apply to environmental and contaminated samples.

Keywords: leaching, ^{238}U, ^{232}Th, Water Leach Purification (WLP) residue

THE DEPTH DISTRIBUTION OF BERYLLIUM 7 IN THE SOIL STUDY

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Abstract

The main objective of this research paper is to examine the depth distribution of 7Be in the soil .The study was conducted in an open land area in Bangi, Selangor. All soil core samples were taken after heavy rainfall using metal corer and brought to Radiochemistry and Environment Group Laboratory (RAS), Malaysian Nuclear Agency for further treatment. All samples have been sectioned into 2 mm increments to a depth of 4 cm and subsequently, oven dried at 45- 60 ºC and gently disaggregated. The sample is passed through a < 2 mm sieve and packed into geometry plastic container for 7Be analysis using gamma spectrometry with a 24- hour count time. From the findings show that the concentration of 7Be is deposited into decreases exponentially with depth, is confined within the top few centimeters at most and similar with other works been reported. However, a detailed study that is by increasing the number of sampling stations throughout Peninsular Malaysia should be done in ensuring a complete data for a future reference.

Keywords: ^{7}Be, depth distribution, Gamma spectrometry, exponentially, centimeters

THE SPATIAL VARIABILITY OF BERYLLIUM 7 FALLOUT STUDY

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Abstract

The main objective of this study was to identify the concentration of ^{7}Be in rainwater in different places in Bangi and Senawang stations, Peninsular Malaysia. All rainwater samples were collected using a plastic container containing 50 ml of concentrated nitric acid for a period of November 2015 until Jan 2016. Twenty-five (25) samples of rainwater that has been collected during the sampling were taken to the laboratory for analysis using the method of chemical precipitation. Counting rainwater samples were carried out using a numerator gamma spectrometry for 24 hours counting with 20% of the detector efficiency. The uncertainty of for each sample, as calculated as a γ – detector-counting error at the 95% confidence level, is in the order of ± 10%. Based on the analysis results obtained, the concentration of ^{7}Be in rainwater in Bangi showing higher average concentration compared to Senawang stations. However, the results of the analysis of the concentration of ^{7}Be from both the station did not show different results been reported by other researchers. Nevertheless, a further study could be carried out, namely by increasing the number of sampling stations in order for the data can be used as a comparison reference data in the future.

Keywords: spatial variability, Beryllium 7, Rainfall samples, Chemical precipitation, Gamma Spectrometry

ASSESSMENT OF NATURALLY OCCURING RADIOACTIVE MATERIAL IN BUILDING MATERIALS USED IN PENINSULAR MALAYSIA

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Abstract

Naturally Occurring Radioactive Materials (NORM) present naturally in the environment, but the increase of the concentration of radionuclide may come from the anthropogenic activities. NORM at certain limit becomes hazardous to environment and human. The amount of activity concentration of NORM such as ^{238}U, ^{232}Th and ^{40}K is the important factor in assessing whether it is harmful to human.The assessment of radionuclides concentrations (^{238}U, ^{232}Th and ^{40}K) in building materials from peninsular Malaysia were carriedout. Concentrations of uranium, thorium and potassium were determined using Energy Dispersive X-ray Fluorescence (EDXRF) method.The hazard risk of NORM was determined by calculating the radiological assessment.The study shows some building material such as gravel contain higher concentration of radionuclide. This indicates higher radiological risk in term of external hazard index. It is suggested that the radiological risk of raw material is predetermined before being used as building material.

Keywords: NORM, building material, EDXRF, radiological risk assessment

ASSESSMENT OF RADIONUCLIDES, LEAD AND CADMIUM IN SELECTED MARINE FISHES FROM KUALA LANGAT COASTAL AREA

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Abstract

The environment of the Kuala Langat coastal area receives pollutants such as radionuclides and heavy metals from various terrestrial and marine sources. Beside the entire pollution load from the river, this locality also affected by a great variety of pollutants from the shipping and port activities as well as the intensive agriculture, industrialization, and urbanization activities along the Langat River. Radionuclides and heavy metals that presence in the marine environment can be adversely affect human health when it enters the food chain. The present study, focus on the presence and transfer of these pollutants from various species of fishes into human food chain. This would enable to estimate the health risk due to the consumption of marine fishes. This study reports the levels of radionuclides ^{238}U, ^{232}Th, ^{40}K and heavy metals (Cd and Pb) in fish tissues of nine difference marine fish collected from Kuala Langat coastal area. These species of fish are regularly consumed by the population of the area. The concentrations of Cd and Pb in all fish sampled were found to be below the allowable limit set by Malaysian Food Act (1983) and Regulation (1985). The radiological risk based on daily intake radioactivity, internal hazard index and lifetime cancer risk were estimated.

Keywords: coastal, marine fishes, radionuclide, ICPMS, Kuala Langat

SHIELDING EFFECT ON RADON EMANATION FROM COMMON BUILDING MATERIAL

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Abstract

Building materials are said to be one of the main contributor to indoor radon emanation. Radon is one of the main cause of lung cancer, second after the smoking habit. Building materials investigated in this study are sand, cement, and paint. The study employs Continuous Radon Monitor, CRM-1029 to measure the radon emanation by individual samples and being compared with its mixture which made into sand brick. The effect of paint as shielding for radon emanation was also studied to emulate the real situation in building construction. The layer is then being coated with a layer of emulsion paint and few layers of paint. From each stage, radon emanation rate was measured using CRM-1029 for 72 hours with measurement taken every one hour interval. The study was conducted in a tight-chamber environment and sealed to avoid leakage of radon gas to the surrounding. The emanation rate per volume of the container can be calculated. The result shows that, by each painting layer, the amount of radon emanation rate to surrounding has been reduced. As a consequence of these, emanation rate from building materials can be reduced.

Keywords: radon emanation rate, continuous radon monitor, shielding, building materials, indoor radon

RADIATION VULCANIZATION OF NATURAL RUBBER LATEX WITH ORGANIC PEROXIDE AS THE CO-SENSITIZER

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Abstract

At present, there are three type of vulcanization process being used in natural rubber latex industries; which is sulphur, radiation and peroxide vulcanization. Sulphur vulcanization produced products with superior tensile strength compared to radiation and peroxide vulcanization. This paper discussed on th e effect of combining radiation and peroxide vulcanization in improving the tensile strength of radiation vulcanized natural rubber latex (RVNRL). Latex formulations based on 2.5 parts perhundred rubber (phr) of hexanediol diacrylate **(**HDDA) as the sensitizer, 0.1 phr of tert-butyl hydroperoxide (tBHPO) as the co-sensitizer and 2.5 phr of Aquanox LP antioxidant was prepared and irradiated at various radiation dose of 2, 4, 6, 8, 10 and 12 kiloGray (kGy). The rubber film obtained from irradiation at 6 kGy had tensile strength of 27.0 MPa, which is more than 37% increment compared to control; 19. 6 MPa**.** Besides crosslink percentage of the rubber films also showed 4 % increment from 89.4% to 93.4%.

Keywords: RVNRL, vulcanization, latex, crosslink

EFFECT OF GAMMA IRRADIATION ON PHYSICAL STABILITY OF DPPC LIPOSOMES

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Abstract

Unilamellar liposomes composed of dipalmitoylphosphatidylcholine (DPPC) were prepared by reverse-phase evaporation method and extrusion through polycarbonate membrane filter. Liposomes suspension with 0.7mg/mL lipid concentration in deionized water were exposed to gamma irradiation at dose up to 25 kGy. The effect of gamma irradiation towards physical stability of liposomes was studied by DLS and zeta potential analysis. From DLS analysis, no significant changes can be observed in hydrodynamic size of liposomes. Zeta potential analysis revealed that gamma irradiation of liposomes suspension enhanced the overall stability of DPPC liposomes. However, a drastic rise in magnitude of zeta potential occurred. Radiation oxidation of DPPC which produced fatty acid composed of COO moiety increased the negativity of liposomes surface. Hence, gamma irradiation on DPPC liposomes may produce liposomes with high stability.

Keywords: gamma irradiation, dipalmitoylphosphatidylcholine (DPPC), physical stability, zeta potential

POSTER ABSTRACTS

DEVELOPMENT OF SENSING SYSTEM FOR DOMOIC ACID DETECTION

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Abstract

Domoic acid is a neurotoxin produce by red algae of pseudo nitsczhia species involves in red tides phenomena. This neurotoxic poisoning is classified as amnesic shellfish poisoning (ASP) that can cause short term memory losses and other symptoms such as nausea, vomiting and diarrhea. Human exposure to domoic acid occurs via the consumption of contaminated shellfish. Therefore, in this research study the potential of ninhydrin as a sensing reagent for the detection of domoic acid was investigated. Solution and immobilization studies for the reaction responses of ninhydrin with domoic acid were carried out in order to design suitable sensing system for the domoic acid. The entrapment of ninhydrin into the PVC film provides more mobility and allowing the reagent to be active in its participation in the extraction of the domoic acid in samples. All the sensing measurement was done using UV-VIS spectrophotometric technique. The system shows high sensitivity response towards domoic acid with the limit of detection (LoD) obtained was in the range of 10^{-5} M. Other results and findings will be presented in the poster presentation.

Keywords: domoic acid, ninhydrin, PVC film

NANOEMULSION SYSTEM CONTAINING LIPOPHILIC DRUGS FOR PULMONARY DRUG DELIVERY

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Abstract

Lipophilic drugs such as curcumin and quercetin known to be excellent curative medicinal agents as they possess anti-cancer activity. However, the biomedical application of curcumin and quercetin are limited due to lack of good aqueous solubility, less stable, and easily degraded following administration. Hence attention has been focused on the design of alternative delivery system using nanotechnology based approach. In this study, formulation development of an oil in water (o/w) nanoemulsion system containing curcumin or quercetin capable for being nebulized for pulmonary delivery has been focused. Nanoemulsion consists of three main components which are oil, water, and surfactant were prepared using overhead stirrer and high shear homogenizer. The process parameters were optimized by using Response Surface Methodology (RSM). The nanoemulsions were characterized based on the particle size, zeta potential, polydispersity index (PDI) and pH. The characterization of nanoemulsion system containing curcumin or quercetin showed that the particle sizes were in the nano-range (<200 nm), with surface charge/zeta potential and pH of <-30 mV and ~7 respectively. The morphology study exhibited that the nanoemulsion formulations were round shape and uniformly distributed (PDI <0.3). The obtained results showed that the developed nanoemulsions have the potential for use as pulmonary delivery system for lipophilic drugs and could enhance the anti-cancer activity.

Keywords: curcumin, quercetin, nanoemulsion, lipophilic, pulmonary delivery

INTERACTION STUDIES OF PUTATIVE CHEMICAL LIGANDS IN BINDING SITES OF THERMOSTABLE LIPASE FROM *GEOBACILLUS ZALIHAE* STRAIN T1

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Abstract

One of the focus in industrial biotechnology is on the usage of enzyme as the catalyst in many chemical reactions. However enzyme stability remains a major challenge. Enzymes can be modified via genetic or chemical methods by manipulating its structure. A potential biocatalyst based on lipase enzyme was designed by *in silico* approach. The enzyme-ligand interactions between selected chemical ligands and a thermostable lipase from *Geobacillus zalihae* strain T1 were studied by using molecular docking. The T1 lipase structure was predicted to have a 65 pockets, with nine of them showed potential binding sites for ligands based on their surface area, volume and number of residues. The potential interactions of selected binding sites with 10 chemical ligands based on different aromatic rings, amine and hydroxyl groups were explored and analyzed. Ligands PHE and BEN docked in the pockets with lowest final docked energy when compared to other selected ligands. Interactions like hydrogen bonds, electrostatic interactions, van der Waals and hydrophobic contacts played very important roles in the enzyme-ligand interactions. These results are essential in determining novel binding sites, particularly for T1 lipase to act as new biocatalyst for industrial purposes.

Keywords: molecular docking, enzyme, ligangs, interactions, biocatalyst

SOLVENT FREE OXIDATION OF BENZYL ALCOHOL USING Au-Pt SUPPORTED NANO CERIA CATALYST

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Abstract

Gold catalyst has played a significant role in oxidation reaction and further the utilization of gold catalyst has been tremendously expanded annually. The uses of nano ceria supported nanoparticles with gold and platinum have shown encouraging enhancement toward catalytic performance for the selective oxidation of alcohol. In this study, solvent free oxidation of benzyl alcohol in the absence of a solvent using Au-Pt supported on nano ceria catalyst has been investigated at a mild condition (80 ^{0} C) using hydrogen peroxide as an oxidant. Different preparation methods have been employed in order to prepare the catalyst (impregnation and sol immobilisation methods). In addition, the characterization of catalyst was carried out using four methods of analysis involved in the physical and microscopic analysis, which are X-Ray Powder Diffraction (XRD), Brunauer, Emmett, and Teller surface area measurement (BET), Field Emission Scanning Electron Microscopy (FE-SEM), and Transmission Electron Microscopy (TEM). From the analysis, catalyst prepared using sol immobilization method shows 1.6% compared than using impregnation method which is 1.1% but impressively both shown selectivity of 100% to benzaldehyde. Comparison of bimetallic and monometallic catalyst revealed that the bimetallic catalyst gave more conversion than monometallic catalyst and both were selective to 100% of benzaldehyde. In this reaction, using Pt as a catalyst gave significant factor for switched off the formation of product of toluene which is contradicted when Pd was used from previous reported. The effect of prolonging time of reaction until 24 hours for both methods was shown to give only little increment of conversion but the main product was still selective to 100% of benzaldehyde Further investigation with catalyst prepared by sol immobilization method using different oxidant of *tert*-butylhydroperoxide (TBHP) gave higher TOF for almost three times compared using H_2O_2 but increases the production of benzoic acid as time prolonged. In conclusion, the catalyst prepared using sol immobilisation method demonstrated a good catalytic activity for selective oxidation of benzyl alcohol.

Keywords: gold catalysis, heterogeneous catalysis, benzyl alcohol

EFFECT OF METAL LOADING ON OSMIUM SUPPORTED CATALYST IN HYDROGENOLYSIS OF GLYCEROL

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Abstract

In this study, bentonite supported osmium catalysts with different metal loading (1,3,5, and 7 wt%) were prepared using impregnation method and applied to convert glycerol, a renewable feedstock, to value-added 1,2-propanediol. Catalytic performance of these catalysts were evaluated in glycerol hydrogenolysis using stainless steel autoclave reactor equipped with a magnetic stirrer at 150°C, hydrogen pressure 20 - 40 bar for 7 hours reaction. The result shows that metal loading of 5 wt.% give good conversion (75 %) compared to other metal loading. This study showed that bentonite which is cheap and abundant clay is potentially a good catalyst support material. The Os/bentonite catalyst was characterized by XRD, XPS, FESEM-EDX and TEM for obtaining some physicochemical properties of the catalysts such as crystal phases,oxidation number of metal, morphologies and dispersion of Os metal on support. Study on recyclebility of Os/bentonite catalyst shows that this catalyst was consistent active in 4 cycle of glycerol hydrogenolysis.

Keywords: osmium, bentonite, glycerol hydrogenolysis, 1,2-propanediol

TRANSITION METAL COMPLEXES OF DITHIOCARBAZATE SCHIFF BASE: SYNTHESIS AND CATALYTIC REACTIVITY OF THE OXIDATION OF CYCLOHEXANE

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Abstract

There have been little reports on the catalytic activity for complexes with chelating ligands derived from substituted dithiocarbazates, although they are known as versatile ligands that can be easily derived into multifunctional hetero coordinating agents. We reported herein the synthesis and catalytic studies of six heteroatomic transition-Schiff base complexes (abbreviated as Ni^{II}SBdiAC, Cu^{II}SBdiAC, Co^{II}SBdiAC, Fe^{II}SBdiAC, Mn^{II}SBdiAC and Zn^{II}SBdiAC, in which SBdiAC represents Schiff base derived from *S*-benzyldithiocarbazate and diacetyl benzene). The dithiocarbazate Schiff base complexes were used as a mild and efficient catalyst in the selective oxidation of cyclohexane to cyclohexanol and cyclohexanone in the presence of *tert*-butyl hydroperoxide (TBHP). The complexes were characterised using several techniques such as Fourier transform infrared (FT-IR) spectroscopy, nuclear magnetic resonance spectroscopy (^{1}H-NMR, ^{13}C-NMR), magnetic susceptibility measurements, molar conductivity, UV-visible spectroscopy, and inductively coupled plasma spectrometry (ICP). The activity of these complexes as catalysts in cyclohexane oxidation were investigated and effects of various reaction conditions on the catalytic reaction were optimized to obtain as high 99% selectivity with 48% of oxidation of cyclohexane at 70°C for 6 hours using TBHP as an oxidant in acetonitrile under mild conditions. Gas chromatography was used to analyse the products of the oxidation reaction of cyclohexane and it showed that cyclohexanol and cyclohexanone are the main products. The time of reaction, temperature, and the concentration of TBHP and catalyst played an important role in the selectivity and conversion of cyclohexane oxidation.

Keywords: dithiocarbazate Schiff bases; metal complexes; cyclohexane; oxidation, selectivity

PRELIMINARY STUDY ON GRAPHENE QUANTUM DOTS-ENZYME SYSTEM FOR HYDROGEN PEROXIDE DETECTION THROUGH FLUORESENCE QUENCHING

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Abstract

A biosensor based on graphene quantum dots (GQDs) conjugated enzyme (horseradish peroxidase, HRP) for the determination of hydrogen peroxide (H_2O_2) has been explored. The GQDs was used as an indicator reveal the fluorescence property of the system based on fluorescence quenching of GQDs which is induced from the enzymatic reaction. The presence of H_2O_2 quenches the fluorescence intensity of GQDs/HRP system which is proportional to the concentration of H_2O_2. It showed a linear dependence on the H_2O_2 concentration ranging from 12.5 mM to 200 mM with the detection limit of 6.25 mM.

Keyword: fluoresence quenching, GQDs, hydrogen peroxide

ACCUMULATION OF METALLIC ELEMENTS IN ASIAN SEABASS AND TILAPIA FROM TUMPAT, KELANTAN

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Abstract

This study presents the accumulation of metallic elements in Asian seabass and Tilapia, specifically in their respective muscle, gill and liver tissues. Metallic elements have the potential to bioaccumulate in biota as well as environment, transferred through food chain and cause ecological damage. Thus, this study aims to investigate the accumulation of iron, lead, copper and cobalt in cage culture fish, Asian seabass and Tilapia. Asian seabass and Tilapia could represent the study area as they were confined in the cage for certain period. Twenty individual of each fish species were analysed using microwave digestion method and measured using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). Iron as an essential element in biota was found as the highest concentration in the liver tissues with 386±25.3 mg/kg in Asian seabass and 205±40.0 mg/kg in Tilapia. Copper was found as the second highest concentration in the same tissue with 43.3±6.27 mg/kg deviation in Asian seabass and 90.6±29.4 mg/kg in Tilapia. The accumulation of metals in increasing order is Co<Pb<Cu<Fe. Malaysia Food Regulation 1985 was referred to define the acceptance level of metals in fish for market purpose based on the concentration in muscle, the main tissue consumed by human. The value of lead and copper are decided for 2 mg/kg and 30 mg/kg in Malaysia Food Regulation 1985, respectively which is far higher than the measured concentration in this study. Thus, Asian seabass and Tilapia from Tumpat are allowed for market and safe to be consumed by human. However, iron and cobalt values could not be found in Malaysia Food Regulation 1985. In conclusion, iron, lead, copper and cobalt did accumulated in different tissue of Asian seabass and Tilapia, but the values are low enough to pose ecological threat toward biota and the environment.

Keywords: accumulation, metallic element, Asian Seabass, tilapia, Tumpat, Kelantan

CONGO RED DYE REMOVAL FROM AQUEOUS SOLUTIONS USING PALM OIL EMPTY FRUIT BUNCH

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Abstract

Adsorption of Congo Red (CR), an anionic dye was investigated in a comparison batch study using raw (R-EFB) and biochar (B-EFB) palm oil empty fruit bunch. The investigated parameters were initial dye concentration, pH, temperature, adsorbent dose and contact time. At optimum conditions, B-EFB achieved 99% Congo Red dye removal while R-EFB achieved 81%. Adsorption data for R-EFB showed a better fit to Freundlich isotherm while B-EFB fitted both Langmuir and Freundlich isotherms. The Langmuir isotherm used to correlate equilibrium data of Congo Red adsorption showed maximum monolayer adsorption of 31.45 mg/g and 26.73 mg/g for B-EFB and R-EFB respectively. Kinetic studies for both R-EFB and B-EFB had better fit to the pseudo second order model. Thermodynamic data indicate Congo Red adsorption by both R-EFB and B-EFB is endothermic and spontaneous in nature. This study shows that palm oil empty fruit bunch has good potential for anionic dye adsorption especially Congo Red dye.

.

Keywords: palm oil empty fruit bunch, congo red dye, batch studies

METALS AND SUPEROXIDE DISMUTASE LEVEL IN *Lates calcarifer* FROM SETIU WETLAND, TERENGGANU

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Abstract

Metal accumulation in mariculture fish can cause economic loss as well as ecological damage to the farmers. Exceeding the threshold level, metal causes oxidative stress and impairment such as abnormality in fish. Thus, superoxide dismutase (SOD) could provide quick information of oxidative stress level in fish. Both metals and SOD levels will be compared to justify whether accumulation of metal could be a stress indicator in fish. Therefore, this study aims to investigate metals and SOD level in *Lates calcarifer* from Setiu Wetland, Terengganu. Metals concentration (copper and lead) were analysed in muscle, gill and liver tissues of *L. calcarifer* using microwave digestion method and measurement was done using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). SOD activity was measured in gill and liver tissues by SOD assays, and the measurement was done using microplate reader. Copper concentration is one time higher compared to lead in muscle tissues with 1.35±0.32 mg/kg and 0.56±0.20 mg/kg, respectively due to biological role of copper in fish body. The results also showed that lead is higher in gill (4.92±0.20 mg/kg) than liver (3.34±0.53 mg/kg) tissues, and copper was found higher in liver (20.8±3.20 mg/kg) than gill (3.95±0.46 mg/kg) tissues suggesting the specific metal regulation in different tissues of fish. Metals and SOD values were tabulated in graph to define their relationship but the conclusion could not be made due to inconsistencies between the values. The clear relationship could be expected with high number of replication and frequency of samples collection. Copper and lead concentrations in *L. calcarifer* were referred to Malaysia Food Regulation 1985, and the values were found below the permissible limit. Thus, *L. calcarifer* from Setiu Wetland is not contaminated by copper and lead and safe to be consumed. The results suggest that metals accumulation is not in agreement with SOD levels, hence copper and lead are not the stress indicator of *L. calcarifer* in the present study.

Keywords: metal, superoxidase dismutase, lead, copper, *Lates calcarifer*, Setiu Wetland

FABRICATION OF POLYMER-SILICA HYBRID MONOLITH USING CONVENTIONAL SOL-GEL METHOD AS AN ENZYME SUPPORT

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Abstract

This research involves developing a novel solid support for the enzyme attachment which focusing on fabricating a polymer-silica hybrid monolith via sol-gel polymerization method. The developments of very large surface area of monolith were done using cold mixture of poly(ethylene-glycol) (PEG) with tetraethyl-orthosilicate (TEOS) and acetic acid. Experiments were done at very low temperature followed with overnight gelification and aging. The sol then underwent calcination forming a hybrid monolith. Subsequently, successful hybrid monoliths were activated by glutaraldehyde before covalently immobilized with lipase and calculating the enzyme activity. Characterizations of hybrid monoliths were done by Fourier Transformed Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM) and Brunauer-Emmett-Teller (BET) to monitor the developed monoliths. This fabrication of hybrid monolith on fused silica capillary is very promising for future rapid lipid analysis in small scale analytical reaction.

Keywords: hybrid monolith, fused silica capillary, sol-gel polymerization, enzymatic microreactor, analytical applications

THE EFFECT OF RICE STARCH ON RHEOLOGICAL AND STRUCTURAL PROPERTIES OF GLYCOLIPID-BASED EMULSIONS

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Abstract

Oil-in-water emulsions are largely used in many applications especially in food and health care industries in order to improve the product quality. Therefore, the main purpose of this research is to study the surface tension, droplet size and rheology of rice starch/methyl α-D-glucopyranoside/oil/ water system at 50 °C. In order to determine the surface tension, a KRUSS KISOI tensiometer has been used. The droplet size and rheology have been analyzed by using an optical polarizing microscope and a rheometer, respectively. There are four types of samples containing different rice starch concentrations. Based on the results obtained, a sample D with the highest rice starch concentration (0.6g) shows the highest stability as compared to the other three samples (A-C). This can be seen through oil-water mixture (O/W) composition with 0.6g of rice starch produces the most stable emulsion as the surface tension is 20.1mN/m, average droplet size is 4.92 µm, and it has the highest viscosity which is 0.00294 Pa·s. However, the emulsion mixtures of samples A, B, and C at 50 °C show a slightly unstable as compared to sample D due to the lower rice starch concentration in a homogenous phase. An increase in rice starch concentrations from 0.0g to 0.6g of samples A, B, C, and D is directly proportional to the rheology but inversely proportional to the average droplet size and surface tension.

Keywords: Emulsions, rice starch, surface tension, droplet size, rheology

SIMULTANEOUS ANALYSIS OF VITAMIN D AND K VIA ULTRA HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

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Abstract

This study highlights on the simplified extraction technique and method development of Ultra High Performance Liquid Chromatography (UHPLC) for simultaneous determination of total vitamin D and K in selected samples of cereals and flour. Vitamin D (D_2, D_3) and K (K_1, K_2) were eluted within 25 minutes on a UHPLC Dionex Ultimate 3000 Series system using two Dionex Acclaim Polar Advantage II C_{18} columns; where one served as the On-line solid phase extraction (On-line SPE) (3.0 x 75mm, 3 µm) and the other as analytical column (4.6 x 250mm, 5 µm) at 30 °C and acquisition wavelength of 265 nm. Simultaneous analysis of vitamin D and K was achieved via gradient elution of methanol with ultra-pure water (95:5 – 100:0 – 95:5), at a programmed flow rate of (1.0 – 0.2 –1.0 mL/min) for left pump, whereas constant flow rate of 1 mL/min was maintained for right pump. The limits of detection (LODs) and limits of quantification (LOQs) ranged between 0.05 – 0.12 µg/mL and from 0.17 – 0.41 µg/mL, for vitamin D while vitamin K fell in the range of 0.05 – 0.08 and 0.16 – 0.28 µg/mL, respectively. The developed method showed good linearity (R^2: 0.996 - 0.999) and high accuracy in the range of 85.35 – 105.59% for vitamin D and 100.72 – 106.36% for vitamin K, respectively. Recovery study was performed via standard addition technique into sample representative of cereal and flour; whereby recovery above 70% was achieved for vitamin D, whereas slightly lower recovery in the range of 60 – 70% was observed for vitamin K.

Keywords: Vitamin D, Vitamin K, Ultra High Performance Liquid Chromatography, On-line solid phase extraction

SYNTHESIS AND CHARACTERIZATION OF SILICA-SILVER CORE-SHELL NANOPARTICLES

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Abstract

Silica-silver core shell nanoparticles have gained a great interest as it has been used in various fields and applications. The aim of this study is to synthesis the silica – silver core shell particles using green approach. The silica – silver core shell particles were synthesized using Tollen reagent technique and followed by reduction process of silver using glucose solution. UV-Vis Spectroscopy study revealed the absorption of surface plasmon resonance (SPR) of silver nanoparticles in range of 380 – 450 nm. Crystallinity of silica – silver core shells nanoparticles showed the face centred cubic (FCC) structure. Spherical shapes of silica – silver core shell nanoparticles were determined using Scanning Electron Microscope (SEM). The silica – silver core shell particles will be used as a potential of dye treatment application in the future.

Keywords: Tollen reagent, green synthesis, silica coating, deposition, dye removal

PHOTOCATALYTIC DEGRADATION OF METHYLENE BLUE DYE IN WATER BY UV-IRRADIATED ZnO/TiO_2 RODS ARRAY

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Abstract

A photocatalytic degradation of methylene blue dye has been investigated in UV-irradiated ZnO/TiO_2 nanorods array thin film in aqueous suspension. The feasibility of such degradation was attempted for a rapid removal of the colour and simultaneously degraded into less harmful compounds like CO_2 and H_2O. The preparation of the nanostructures was done using spin-coating method (TiO_2) and solution immersion method (ZnO). The degradation was compared to ZnO thin film, TiO_2 thin film and ZnO/TiO_2 thin film. Optical properties and the surface morphology of the nanostructures were characterized using Ultra-violet Visible Spectroscopy (UV-Vis) and Field-Emission Scanning Electron Microscope (FESEM). The compositional of elements existed in the nanostructures were confirmed by using Energy Dispersive Analyzer X-ray (EDAX). The photocatalysis was carried out on 20 ppm of methylene blue under UV light. The FESEM results reveal that the structure of ZnO on TiO_2 was well-dispersed throughout the glass substrate with hexagonal multipods rod shape of diameter approximately 250 nm. After 40 min irradiation by UV light, 30, 34 and 39 % of methylene blue solution were decolorized by ZnO, TiO_2 and ZnO/TiO_2 thin film respectively. The result shows that the photocatalytic efficiency improved by the combination of ZnO and TiO_2 rather than the metal oxides alone.

Keywords: ZnO, TiO_2, rods array, methylene blue, UV-irradiation

ELECTROCHEMICAL SYNTHESIS AND CHARACTERIZATION OF IRON SENSITIZED TITANIA NANOTUBES AS PHOTOANODE IN PHOTOELECTROCHEMICAL CELLS

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Abstract

Titania is considered as one of the most studied photocatalysts due to its superior features such as chemical inertness, low cost and photostability. Recently, sensitization of titania nanotubes with transition metals has been reported to demonstrate promising reactivity upon visible light illumination via band gap enginnering. Thus, in this study, a two-step method for the fabrication of ordered iron sensitized titania nanotube layer (Fe-TNT) by potentiostatic anodization of titanium foil in a fluorinated electrolyte followed by electrodeposition in iron(III) chloride solution is reported. The effects of deposition voltages and durations on the structural properties and photoelectrochemical performance of the resulting Fe-TNT have been investigated. The FESEM results showed that surface morphology of FeTNT is dependent on the deposition voltage applied. Diffuse reflectance spectra of Fe-TNT showed an enhancement in the visible absorption relative to pure TNT. From the photoelectrochemical measurement, sensitization with iron has effectively enhanced the photocurrent performance. This work demonstrates a simple and feasible method to fabricate an effective photoanode for solar cells application.

Keywords: electrodeposition, titania nanotubes, photocurrent, photoelectrochemical cells, iron

SYNTHESIS AND CHARACTERIZATION OF ZINC LAYERED HYDROXIDE INTERLEAVED WITH *P*-AMINOBENZOIC ACID

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Abstract

p-aminobenzoic acid (4-AB) is a type of topical drug plays important role as a sunscreen to absorb UV-B rays. 4-AB interleaved into zinc layered hydroxide (ZLH) forming ZAB nanocomposite has potential to deliver drug with adequate amount to skin. The synthesis of ZAB from zinc oxide (ZnO) was carried out by using direct method. PXRD pattern with basal spacing of 23.3 Å confirmed that 4-AB has been interleaved into ZLH interlayer. This result is supported by COO^- and $C_6H_5NH_2$ functional groups that appeared in FTIR spectra at 1518 cm^{-1} and 1174 cm^{-1}, respectively, attributing to 4-AB intercalated into ZLH inorganic interlayer gallery. Characterisation of the resulted compounds were also studied by TGA, SEM and BET analyses.

Keywords: sunscreen, zinc layered hydroxide, zinc oxide, 4-aminobenzoic acid

EFFECT OF GAMMA RADIATION ON Gd_2O_3 MAGNETIC NANOPARTICLES COATED IN CHITOSAN AS CONTRAST AGENT IN MAGNETIC RESONANCE IMAGING

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Abstract

In this research, gadolinium nanoparticles (GdNPs) were synthesized in chitosan using gamma ray irradiation-reduction under simple condition. Chitosan was used as natural stabilizer whereas Gd_2O_3 was used as the gadolinium precursor. The properties of GdNPs in chitosan were studied in term of their functional group, surface plasmon resonance and particle distribution. All samples were characterized using UV-visible spectroscopy, transmission electron microscopy, scanning electron microscopy – energy dispersive x-ray and Fourier transform infrared spectroscopy. The MR imaging measurements were carried out on a MRI machine with magnetic field intensity of 3.0 T. The prepared samples were placed stagnantly with a Perspex holder positioned in an isocentre of the MR machine. A volume coil was served as a signal relay for both transmission of radio frequency (RF) pulses and signal reception. The parameters that being used for the examination were field of view (FOV) = 250 mm x 250 mm, echo time (TE) = 10 ms (T1) and 72 ms (T2) and relaxation time (TR) = 520 ms (T1) and 3600 ms for (T2). The results revealed two new peak at 260 nm and 290 nm interpreted as the chemical changes of chitosan after gamma radiation. SEM-EDX showed that the chitosan nanoparticles (CsNPs) were nanofiber in shape meanwhile the Gd-CsNPs (control) were in spherical shape. Further analysis using TEM resulted Gd-CsNPs with an average diameter size of 30-45 nm after gamma radiation. The size decreased when irradiated with gamma ray at different doses. The results from the MR imaging showed that the T1 images were much suitable to use as a contrast agent compared to T2 images. The results suggested that gadolinium nanoparticles coated in chitosan solution at gamma ray dose of 10 kGy was sufficient to achieve maturely formed nanoparticles.

Keywords: chitosan, gadolinium, gamma ray irradiation-reduction, MRI

PREPARATION AND CHARACTERIZATION OF ZINC OXIDE NANOPARTICLES IN CLAY MINERALS FOR HEAVY METALS REMOVAL

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Abstract

Rapid industrialization and population growth has led to the excessive release of toxic metals from agricultural and industrial activities into the environment which poses threat to ecosystem and environment in general. Clay minerals are potentially low-cost materials from abundant natural resources which have the ability to accommodate new species in their structure which are of great importance in developing nanocomposites. The aim of this research is to synthesize, characterize and test the adsorption efficiency of the prepared nanocomposites. In this study zinc Oxide nanoparticle (ZnO-NP) was successfully incorporated into clay minerals montmorillonite (MMT) and talc by a simple green heating method using zinc nitrate and sodium alginate as zinc oxide precursor and stabilizer respectively. The prepared nanocomposites were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), Energy Dispersal X-ray (EDAX), Field emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM). The potentials of the prepared ZnO/MMT and ZnO/talc nanocomposites as adsorbent for the removal of copper and lead ions from their aqueous solution were investigated. The XRD and FESEM measurements revealed the presence of ZnO nanoparticles with hexagonal wurtzite structure in the nanocomposites. Energy dispersive X-ray (EDX) also confirmed the presence of ZnO nanoparticles in the nanocomposites. The percentage removal of by ZnO/MMT was found to be 97.2% (Pb) and 89.5% (Cu) which are higher than that of 90.3% (Pb) and 80.6 % (Cu) for ZnO/talc. Kinetics studies revealed that the adsorption of Cu (II) and Pb(II) onto the prepared nanocomposites followed the pseudo-second-order kinetics and the adsorption equilibrium data were well fitted to Langmuir isotherm model. At pH 4, maximum adsorption capacities for the ZnO/MMT and ZnO/Talc were 88.50 and 48.30 mg/g for Pb^{2+} and 54.06 and 83.30 mg/g for Cu^{2+}, respectively. The regeneration revealed that the nanocomposites adsorbent can be utilized for three times because of their stability and can serve as promising adsorbents for the removal of copper and lead ions from aqueous solutions because of their distinctive chemical and physical properties. Electrostatic attraction and ion exchange could be the main adsorption mechanisms for lead and copper ions adsorption onto the nanocomposites. Results showed that the nanocomposites have high level of adsorption towards heavy metal ions.

Keywords: zinc oxide, MMT, talc, nanoparticles, clay materials, heavy metals

SINTESIS ZARAHNANO ZINK SULFIDA MENGGUNAKAN KAEDAH KOLOIDAL DENGAN PENAMBAHAN POLIETELINAIMIN SEBAGAI AGEN PASIF

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Abstrak

Pengimejan tradisional mempunyai banyak kekurangan seperti mempunyai jangka hayat yang pendek serta bersaiz besar kerana sifat pendafluornya tidak tahan lama atau berlakunya 'photobleaching'. Melalui kajian ini, zarahnano zink sulfida boleh berfungsi sebagai pengimejan titik kuantum yang sesuai dengan sifat optiknya yang pelbagai mengikut saiz zarahnya. Zarahnano ZnS disediakan dengan menggunakan kaedah koloidal. Bahan pemula untuk sintesis zarahnano ZnS adalah zink asetat sebagai sumber zink dan natrium sulfida sebagai sumber sulfur dengan air suling sebagai pelarut. Sifat optik dan tenaga jurang jalur zarahnano ZnS dan bersalut dengan agen pasif polietelinaimin (PEI) dengan nisbah yang berbeza dikenalpasti. Saiz zarahnano ZnS tanpa salutan PEI adalah 14.91 nm dengan tenaga jurang jalur 4.28 eV. Manakala bagi zarahnano ZnS bersalut PEI pula mempunyai saiz 13.04 nm iaitu lebih kecil dari saiz zarahnano ZnS tanpa salutan PEI dan menunjukkan peningkatan dalam tenaga jurang jalur iaitu 4.45 eV. Masing-masing mempamerkan sifat pancaran biru dibawah UV-Vis dan menunjukkan bahawa zarahnano ZnS tanpa salutan PEI dan bersalut PEI berada dalam panjang gelombang yang pendek dan bersaiz kecil. Konklusinya didapati nisbah terbaik untuk ZnS kepada PEI yang mempuyai sifat optik yang maksimum adalah 1:4.

Kata kunci: titik kuantum ZnS, polietelinaimin (PEI), kaedah koloidal, sifat optik

FACILE SYNTHESIS OF PURE TETRAGONAL ZIRCONIA NANOPARTICLES FOR DEGRADATION OF PHENOL

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Abstract

Pure tetragonal ZrO_2 (t-ZrO_2) nanoparticles were prepared by a simple electrochemical method and characterized using XRD, TEM, and BET surface area. The presence of tetraethylammonium perchlorate (TEAP) as supporting electrolyte was responsible for the stabilization of t-ZrO_2 hence, resulting in a decrease in ZrO_2 crystal size. The photoactivity of the t-ZrO_2 was then subjected to various parameters, and the optimum values was observed at pH 11 with a shorter contact time using 1.0 g L^{-1} of catalyst dosage under lower calcination temperature (523 K), which resulted in 97% degradation of phenol compound. In addition, the regeneration study shows that the catalyst could be maintained with a slight decrease in degradation (< 9%) after six cycling runs. The t-ZrO_2 shows good photoactivity towards phenol degradation under UV light in a batch reactor compared with commercial t-ZrO_2 (33%).

Keywords: t-ZrO_2, electrochemical, stabilization, phenol, photodegradation

MICROWAVE INDUCED HNO_3 AND H_3PO_4 ACTIVATION OF OIL PALM FROND FOR REMOVAL OF MALACHITE GREEN

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Abstract

The use of abundantly available and eco-friendly adsorbent oil palm frond (OPF) has been reported as an alternative to the current expensive methods of removing of malachite green (MG) dye from an aqueous solution. The effects of pre-treatment, pH, adsorbent dosage, initial concentration of malachite green and temperature were investigated, and the optimal experimental conditions were ascertained. The adsorption of MG onto OPF was confirmed by FTIR, as it showed the changes in peak of FTIR before and after adsorption. It was found that strong interaction between OPF surface and MG was observed**.** In this study, the use of 3 g/L of 300W N-OPF resulted in the nearly complete removal of 50 ppm of MG after 15 min of contact time at pH 6 and 343 K. The results indicated that the OPF can be used to effectively remove MG from aqueous media.

Keywords: oil palm frond, malachite green, eco-friendly, adsorption

STRONG ION-EXCHANGE CENTRIFUGAL PARTITION CHROMATOGRAPHY (SIX CPC) AS AN EFFICIENT METHOD FOR PURIFICATION OF FOUR ANTHOCYANINS IN *Hibiscus sabdariffa* L.

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Abstract

Anthocyanin is a hydrophilic compound with good solubility in water, ethanol and methanol and their mixtures. The classical CPC method using conventional solvent system does not always produces satisfactory results due to the low retention of solutes in the stationary phase. In order to extend their hydrophobicity windows, new solvent systems are developed. In this study, for the separation of anthocyanins from an ethanolics extracts of *Hibiscus sabdariffa* L. (HS), the biphasic solvent system adapted for strong ionic exchange (SIX) was built to achieve an anion-exchanger behaviour. The separation process was carried out on a gram scale using the ternary biphasic system ethyl acetate: *n*-butanol: water [mobile organic phase: 4:4.6:1.4; aqueous stationary phase: 90:0.5:0.5] in the ascending mode. An Aliquat 336 ® was used as the anion extractant in the aqueous stationary phase and sodium iodide (NaI) was used as the displacer in the organic mobile phase. Four (4) of anthocyanins are successfully isolated and have been identified as delphinidin-3-*O*-sambubioside (DS), cyanidin-3-*O*-sambubioside (CS), delphinidin-3-*O*-glucoside (DG) and cyanidin-3-*O*-glucoside (CG). From 500 mg of crude HS extract, 11.3 mg, 8.2 mg, 7.8 mg, and 10.8 mg of DS, CS, DG and CG were obtained respectively after 75 minutes for total process duration of 90 minutes. High performance liquid chromatography analysis revealed that the isolated compound was 88%, 92%, 93% and 70% purity respectively. This new methodology for isolation of anthocyanin mixtures successfully increased purity and efficiency especially to DG and CG which is higher than the classical CPC method (< 40% and < 30% purity respectively). The availability of high purity anthocyanin mixtures will facilitate anthocyanin studies and will promote the application of anthocyanins in the food and pharmaceutical industries.

Keywords: SIXCPC, centrifugal partition chromatography, *Hibiscus sabdariffa*, anthocyanins

IDENTIFICATION AND QUANTIFICATION OF PHYTOCHEMICALS IN *Carica papaya* LEAF EXTRACTS AND *IN VITRO* ANTI-DENGUE AND CYTOTOXICITY ACTIVITIES

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Abstract

Carica papaya L. belongs to the family of *Caricaceae* and it is one of the most widely grown crops in the tropical and subtropical region including Malaysia. The leaf extract is traditionally used to cure dengue fever and its associated symptoms. Extracts from the *Carica papaya* L. leaves are widely reported to contain phytochemicals with antibacterial, antioxidant and anticancer *in-vitro* activity. These include phytochemicals from the chemical groups alkaloids, flavonoids and phenolics. This study aims to identify and quantify the major phytochemicals present in *Carica papaya* L. leaves juice extract, as well as the growth inhibitory activity of the extract and its compounds on DEN-2 dengue virus and cytotoxicity *in vitro*. Compounds were isolated and purified by centrifugal partition chromatography and confirmed by spectroscopic methods (LC-MS and 1D/2D-^{1}H/^{13}C NMR). Quantification of isolated compounds were carried out by UHPLC-Oribitrap-MS equipped with Acquity UPLC Cortecs C_{18} analytical column and Electrospray ionization (ESI). For evaluating the anti-dengue activity, the replication of DEN-2 dengue viruses were measured by plaque reduction assay. Cytotoxic activities of the extracts and compounds were evaluated *in-vitro* on non-cancerous Vero cell line by MTT assay. Clitorin (m/z 739.20807 $[M-H]^-$), manghaslin (m/z 755.20654 $[M-H]^-$), rutin (m/z 609.14673 $[M-H]^-$) and nicotiflorin (m/z 593.14807 $[M-H]^-$) were confirmed based on LCMS data and full analysis of 1D/2D-^{1}H and ^{13}C NMR data. The content of clitorin (3.12 mg/g) was significantly higher than other identified compounds followed by manghaslin (1.52 mg/g), rutin (0.75 mg/g) and nicotiflorin (0.43 mg/g). For anti-dengue activity, leaves juice extract and all major flavonoids showed no activity against DEN2 dengue virus *in vitro* while the IC_{50} value was more than 30.5 µg/ml. The phytochemical analysis demonstrated the presence of clitorin, manghaslin, rutin and nictotiflorin as the major flavanoid compounds in *C. papaya* juice leaves extract. Although no activity against DEN2 dengue virus was observed *in vitro* for this extract and its compounds, investigation into other mechanism of action of denguecan be further investigated.

Keywords: *Carica papaya,* flavonoids, cytotoxic, dengue

CYTOTOXICITY AND NEUROPROTECTIVE EFFECTS OF APPLE AND DATE VINEGAR ADDED *Centella asiatica* AGAINST LIPOPOLYSACCHARIDE (LPS)-INDUCED INFLAMMATION IN NEUROBLASTOMA SH-SY5Y CELLS

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Abstract

Alzheimer's disease (AD) is a neurodegenerative disease of central nervous system. The progression of this chronic disease may lead to cognitive impairments, behavioral and dementia. Increased rate of hydrolysis of neurotransmitter by acetylcholinesterase enzyme (AChE) could worsens the condition of cognitive dysfunction. Several herbs are known to possess the anti-inflammatory, anti-acetylcholinesterase inhibition and antioxidant activity. The present study was performed to investigate the bioactivities of apple and date vinegar added *C. asiatica* (AVCA and DVCA) at various concentration (0, 0.5, 2 and 5%) on neuronal cells. SH-SY5Y cells was used to resemble neuronal-like systems to research especially in neurodegenerative disease. The neuroprotective effects apple and date vinegar added *C. asiatica* were studied in Lipopolysaccharide (LPS) stimulated inflammation and cell death in SH-SY5Y cells. The effect of apple and date vinegar added *C.asiatica* extract on LPS-induced neuroinflammation were evaluated using SH-SY5Y neuroblastoma cells and determined using a 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide (MTT) assay. *In vitro* studies have shown that extracts from AVCA and DVCA act as antioxidant and neuroprotective agents against inflammation. AVCA and DVCA pretreatment conferred significant protection against the LPS-induced decrease of SH-SY5Y cell viability. Current findings suggest that AVCA and DVCA exert neuroprotective effects against LPS-induced inflammation in SH-SY5Y cells. Therefore, AVCA and DVCA pretreatment have potential neuroprotective effects in neurodegenerative diseases.

Keywords: Alzheimer's disease, vinegar, *Centella asiatica,* anti-inflammatory, neuroprotective

SYNTHESIS AND CHARACTERISATION OF COPPER(II) COMPLEXES CONTAINING SCHIFF BASES DERIVED FROM L-PHENYLALANINE

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Abstract

Amino acid Schiff base complexes have received considerable attention due to their biological importance as antibacterial and anticancer agents. A series of new Cu(II) amino acid Schiff base complexes derived from L-phenylalanine (Phe) and various dicarbonyl compounds (glyoxal, acetyl acetone, 2,5-hexanedione and 5,5-dimethyl-1,3-cyclohexanedione) were synthesized using a template reaction. These complexes were characterized by various spectroscopic techniques, elemental analysis, magnetic susceptibility, molar conductivity, and single Crystal X-ray crystallographic analysis, where possible. The FT-IR spectra indicated the presence of the C=N band around the region of 1600 cm^{-1} which proved the successful formation of the Schiff base. The Schiff base amino acid ligands are expected to coordinate to the metal ion via the azomethine nitrogen atom and oxygen atom. For the cytotoxic assay, two copper complexes derived from glyoxal and acetyl acetone were found to be moderately active against RT-112 (minimally invasive human bladder carcinoma cell line). However, none of the complexes were active against EJ-28 (invasive human bladder carcinoma cell line). Scratch assay was conducted for the complexes that were inactive against EJ-28 in order to determine the anti-migration properties of the complexes.

Keywords: Schiff base, L-phenylalanine, bladder cancer cell lines

SYNTHESIS OF PALM STEARIN BASED BIOLUBRICANT BASESTOCKS

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Abstract

Refined, Bleached and Deodorized Palm Stearin (RBDPS) was chemically modified to synthesis biolubricant basestock. Esterification reaction between palm stearin based free fatty acid (FFA) and trimethylolpropane (TMP) was successfully performed at 150 ºC for 2 hours with the molar ratio of FFA:TMP; 3:1 using H_2SO_4 1wt.% as catalyst. The product conversion was 99%. Infrared spectrum of TMP-esters showed the presence of carbonyl group of esters, ν(C=O) at 1746 cm^{-1} wavenumber. The chemical structure of product was confirmed by proton and carbon nuclear magnetic resonance (^{1}H-NMR and ^{13}C-NMR) spectra. Biolubricant basestock was characterized for its lubrication properties such as kinematic viscosity (40 ^{o}C and 100 ^{o}C), viscosity index, pour point, flash and fire points and oxidative stability.

Keywords: palm stearin, TMP-esters, esterification, biolubricant, renewable source

PREPARATION AND OPTIMIZATION OF CARBOXYMETHYL SAGO STARCH (CMSS) HYDROGELS BY USING CITRIC ACID AS CROSSLINKING AGENT

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Abstract

Sago starch with scientific name of 'Metroxylon sagu' is a natural and biodegradable polymer. It was modified by carboxymethylation reaction produce carboxymethyl sago starch (CMSS). Hydrogel is three-dimensional network composed of hydrophilic polymeric chains and exhibits ability to swell. In this study, CMSS was used to produce hydrogel by using citric acid as crosslinking agent. The sample was prepared by raising the temperature in order to activate the crosslinking process since citric acid dehydrate to yield cyclic anyhride that react with hydroxyl group CMSS. The optimization of hydrogel was done based on four parameters; concentration of CMSS, concentration of citric acid, effect of reaction time and effect of temperature. The optimum condition obtained were; 60% w/v of CMSS, 10% w/w of citric acid, 48 hours of reaction time and at 50°C reaction temperature.

Keywords: carboxymethyl sago starch, citric acid, hydrogel

ELECTROSPUN PAN/LIGNIN/TEOS AS PRECURSOR FOR THE PRODUCTION OF CARBON NANOFIBERS

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Abstract

Carbon nanofibers (CNF) is widely applied as heat-management materials, composite reinforcement, filtration membrance, and energy storage. Polyacrylonitrile (PAN) is the main precursor for fabrication of CNF due to its brilliant properties such as high specific surface area, high aspect ratio, high mechanical strength and flexibility. However, the manufacturing prices is high. Low-cost carbon nanofibers can be fabricated from renewable materials such as lignin. It is a second most abundant raw material on earth and can be obtained easily. Tetraethyl orthosilicate (TEOS) can be added into the CNF as pore generator to generate more porous surface and catalyze the stabilization process. Porous surface is very important in providing the CNF with high electrical performance such as improved electric double-layer capacitance in supercapacitor. In this study, lignin/PAN/TEOS carbon nanofibers waslignin/PAN/TEOS carbon nanofibers were prepared by using electrospinning method followed by the heat treatment of the up to 1000°C. Electrospun nanofibers were characterized by TGA, DSC and SEM while the carbon nanofibers were characterized by using FTIR and FESEM. The TGA results show that the major degradation temperature decreased to around 270-280°C after lignin is added due to the lower thermal stability of lignin. DSC results show the addition of TEOS shifted the exothermic peaks to lower temperature due to catalytic ability of TEOS. This indicated that the stabilization process of TEOS-incorporated CNF is kinetically higher than those without TEOS. SEM images show the morphology and diameter of the sample which decreased as the lignin concentration was increased. IR spectra show peaks around 1100 cm^{-1} which is correspond to Si-O-C bond. This indicated the successful formation of Si-O-C/Si-O-Si structures which will influence the properties of CNF such as electrochemical performances. FESEM shows the fiber diameter decreased as TEOS amount increased and pores were successfully produced at the surface of carbon nanofibers.

Keywords: carbon nanofiber, electrospinning, porous, polyacrylonitrile (PAN), lignin, tetraethyl orthosilicate (TEOS)

CONVERSION OF THORIUM OXIDE (ThO_2) TO THORIUM TETRAFLUORIDE (ThF_4) BY AMMONIA HYDROGEN FLUORIDE (NH_4HF_2) AS FLUORINATION AGENT

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Abstract

Liquid Fluoride Thorium Reactor (LFTR) was considered thorium tetrafluoride (ThF_4) as its fuel in liquid form during operation. ThF_4 can be prepared through fluorination process of thorium oxide (ThO_2). In this study, method for synthesis of ThF4 from ThO_2 using ammonia hydrogen fluoride (NH_4HF_2) as fluorination agent was investigated. ThO_2 and NH_4HF_2 were mixed at different molar ratio and allowed to react at room temperature for a few days. Then, sample was heated with argon gas flow at temperature range 450°C – 750°C. Characterization was done by X-Ray Diffraction (XRD) and Scanning Electron Microscopic- Dispersion X-ray Spectroscopy (SEM-EDX). Reaction of ThO_2 and NH_4HF_2 at room temperature was synthesized ammonia thorium fluoride and produced ThF_4 after heated at 450°C.This study was showed NH_4HF_2 is one of the fluorination agents to synthesis ThF_4 at low temperature.

Keywords: thorium tetrafluoride, Thorium oxide, Ammonium hydrogen fluoride, XRD

ASSESSING ALPHA SPECTROMETRY SYSTEM USING MULTIRADIONUCLIDE STANDARD SOURCE: ENERGY AND EFFICIENCY CALIBRATION

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Abstract

A study of energy and efficiency calibration was carried out to assess the performance of a high resolution multi-chamber alpha spectrometry system. The calibration was performed to assess the capability of the system to produce the best quality data using multi-radionuclide standard source. The multi-radionuclide standard source used was electroplated with four radionuclides namely ^{241}Am, ^{234}U, ^{238}U and ^{238}Pu. As a result, the energy calibration curve was plotted and a linear relationship between peak energy and channel number was obtained. The linear coefficient obtained was 1. In addition, the system considerably reliable and rugged as none of the detector efficiency was in the action limit. To test the reliability of the system, several sets of certified reference materials used to validate the system. The results obtained were in the range of the reference values. In conclusion, the alpha spectrometry system is rugged and able to produce the best quality of analytical results.

Keywords: alpha spectrometry, energy and efficiency calibration, multi-radionuclide standard source

TOWARD GREENER PROCESSING OF MALAYSIAN MONAZITE FOR THORIUM AND RARE EARTH ELEMENTS PRODUCTION

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Abstract

This paper describes a process for the extraction and recovery of thorium and rare earth elements (REEs) from Malaysian monazite using acid digestion, selective precipitation and solvent extraction. The process prevents environmental impacts caused by the removing of thorium as hazardous industrial residue. Results of the study shows that Th and REEs can be recovered from the chemical treatment of monazite and produce in monazite sulphate leach solutions. Selective precipitations at different pH are able to separate more than 97% of Th and 98% of REEs from the studied solutions. Purity up to 91% for Th was obtained using solvent extraction. The study also revealed that various steps of chemical processing of Malaysian monazite followed by selective precipitation and solvent extraction manage to enrich Th effectively and separated it from U and REEs. The enrichment percentage of thorium by selective precipitation was in the range of 5.93 % - 64.48% while by extracting Th using TBP, the enrichment of Th was in the range of 5.17 % - 29.96%. Therefore this study shows that a combination of solvent extraction and precipitation at selected conditions has produce a green Th from Malaysian monazite.

Keywords: thorium, REEs, selective precipitation, solvent extraction, Malaysian Monazite

THORIUM RECOVERY BY ACID DIGESTION USING AMMONIUM HYDROXIDE AS PRECIPITATING AGENT

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Abstract

Thorium content in monazite is the highest compared to the other local minerals like xenotime, ilmenite and zircon. The recovery of thorium (Th) has its importance in the security of prospective nuclear fuel supply along with producing more value-added products from the local sources. In this study, method to recover thorium using acid digestion and selective precipitation by ammonium hydroxide (NH_4OH) as precipitating agent was investigated. Monazite and 98% concentrated Sulphuric Acid (H_2SO_4) were mixed together and heated at different temperature for 4 hours. Then, sample was cooled down to room temperature and leached out using deionized water before thorium is precipitated out using NH_4OH at pH value ranged from 1.67 to 1.82. Inductively coupled plasma mass spectrometry (ICP-MS) was used to analyze the Th content. Reaction of monazite with H_2SO_4 produced thorium sulphate ($Th(SO_4)_2$) which is soluble in aqueous which can be selectively precipitate to produce thorium hydroxide ($Th(OH)_4$). This study shown that the highest Th purity gained was 50.264 ± 0.285 %. Solvent extraction or counter-current extraction can be used to further gain the purity of Th.

Keywords: monazite, thorium, recovery, acid digestion

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