PREPARATION AND CHARACTERISATION OF SOL-GEL HYBRID SORBENT METHYLTRIMETHOXYSILANE-CHLOROPROPYLTRIETHOXYSILANE FOR SOLID PHASE EXTRACTION

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Abstract

A new sol-gel hybrid methyltrimethoxysilane-chloropropyltriethoxysilane (MTMOS-CPTES) was produced and applied as sorbent for solid phase extraction (SPE). Three selected organophosphorus pesticides (OPPs) namely chlorpyrifos, profenofos and malathion were employed as test analytes to assess the extraction performance of the synthesized sol-gel hybrid MTMOS-CPTES. Analysis was performed using gas chromatography-mass spectrometry. Several vital parameters were optimised to identify the best extraction conditions. Under the optimum extraction conditions, the MTMOS-CPTES SPE method showed good linearity in the range of 50-1000 µg L\textsuperscript{-1} with coefficient of determination, \( r^2 > 0.995 \). The limits of detection (LOD) at S/N = 3 obtained were in the range of 0.01-0.07 µg mL\textsuperscript{-1} and limits of quantification (LOQ) ranging from 0.03-0.21 µg mL\textsuperscript{-1}. The method showed good repeatability (RSD 2.4-4.7\%, \( n=3 \)). The performance of the MTMOS-CPTES SPE was compared to commercial C\textsubscript{18} SPE sorbent. The LOD obtained for MTMOS-CPTES SPE was 2.3-6.5\% lower than the LOD of commercial C\textsubscript{18}. The developed MTMOS-CPTES SPE method was successfully applied to real sample analysis of the selected OPPs from two fruits samples. The proposed method provided acceptable recoveries (88.33-120.7\%) with good RSDs ranging from 1.6\% to 3.3\% (\( n=3 \)). Recoveries and RSDs of OPPs from fruits samples using commercial C\textsubscript{18} SPE sorbent were 70.3-100.2\%, RSDs 6.3-8.8\%, \( n=3 \). The proposed MTMOS-CPTES SPE method demonstrates the potential as an alternative extraction sorbent for OPPs.

Keywords: sol-gel; methyltrimethoxysilane-chloropropyltriethoxysilane; solid phase extraction; organophosphorus pesticides, gas-chromatography-mass spectrometry
ONE STEP ACTIVATION AND RECYCLABILITY OF KOH AND CaO MODIFIED CARBON IN TRANSESTERIFICATION OF RICE BRAN OIL

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Abstract

Carbon materials is a promising future in catalysis of chemical reactions. In this work palm kernel shell modified carbon was prepared by using one step activation with potassium hydroxide (KOH) and calcium oxide (CaO) as dopant. The modified carbon was prepared by mixing various concentration of calcium oxide while keeping the concentration of potassium hydroxide with palm kernel shell constant and subsequently activating at 500°C for 5 hours. The prepared catalyst were characterized by thermal gravimetry analysis (TGA), back titration analysis, nitrogen adsorption analysis, field emission electron microscope (FESEM), energy dispersive x-ray (EDX) and x-ray diffraction (XRD). The catalytic performance was evaluated by transesterification of rice bran oil with methanol. Gas chromatography–flame ionization detector (GC–FID) was used to analyze the product while X-ray fluorescence (XRF) was used to check for the possibility of leaching. A low BET surface area of around 3.62 m\textsuperscript{2}/g was obtained indicating that the KOH and CaO loading covers the pores of the carbon. This study shows, as the percentage of calcium increases, the basic strength also increases followed by the increase in biodiesel production. The percentage conversion of biodiesel for 0% CaO/KOH/C, 10% CaO/KOH/C, 15% CaO/KOH/C, 20% CaO/KOH/C, 25% CaO/KOH/C and 30% CaO/KOH/C calculated about 80.9%, 86.2%, 90.4%, 92.8%, 93.6% and 94.3%, respectively. Recyclability test of the prepared catalyst was still good for 3 consecutive runs however, for the fourth run the percentage conversion of biodiesel drops.

Keywords: One step Activation; Biodiesel; Recyclability; Rice Bran Oil
DEGRADATION OF CHLORAMPHENICOL USING GRAPHENE OXIDE FROM BAGASSE-Fe$_3$O$_4$

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Abstract

A new method for chloramphenicol degradation using graphene oxide (GO)-Fe$_3$O$_4$ has been developed. The GO has been made from bagasse with pyrolysis process and Hummers method then. GO-Fe$_3$O$_4$ has been produced by co precipitation process of iron (III) chloride hexahydrate and iron (II) sulfate heptahydrate. It characterized using X-Ray Diffraction, Surface Area and Pore Analyzer (SAA), Raman spectroscopy and Fourier Transform Infrared Spectrometer (FT-IR) to ensure the structure of the composite. HPLC instrument was used for chloramphenicol content analysis for each optimization. The conditions used of HPLC instrument was used mobile phase of water/methanol 35/65 (v/v) at wavelength of 278 nm. The optimum conditions of chloramphenicol degradation which include the dosage of the catalyst, the concentration of H$_2$O$_2$, pH, and temperature were 0.15 g/L; 25 mM, 5, and 25$^\circ$ C, respectively. Application of chloramphenicol degradation under optimum conditions of various concentration of 1, 3, 10, 15 and 20 ppm shows the higher chloramphenicol concentration, the lower the degradation rate. However, in the degradation process there was no significant difference when applied to a standard concentration of 20 ppm chloramphenicol.

Keywords: Degradation; chloramphenicol; graphene oxide; bagasse; Fe$_3$O$_4$
Abstract

Magnetic nanoparticles modified with activated charcoal and nonionic silicone surfactant was synthesized in order to extract the phenolic compounds (2, 4–dichlorophenol and 2, 4-dinitrophenol) from waste water samples. The nature of the synthesized material was examined by XRD, SEM, TEM, VSEM and FTIR techniques. First the MNP was synthesized via co-precipitation method and then was coated with non-ionic silicone surfactant. The material was further optimized for future use. The parameters that were optimized in this study were pH, contact time, amount of adsorbent, concentration of analyte and temperature. All the parameters were analysed using UV-VIS spectrometer. The chosen experimental parameters and their ranges were: pH 6 and pH 4 for 2, 4-dichlorophenol and 2, 4-dinitrophenol respectively, extraction time, 40 - 60 minutes for both the analytes; amount of adsorbent was used 40 mg for both the analytes. After the optimization process the adsorption equilibrium results were further used in adsorption kinetic, isotherm and thermodynamic study to identify the suitable model. Then the optimized material was further subjected in the use of real sample analysis.

Keywords: magnetic nanoparticles; activated charcoal; nonionic surfactant; phenolic removal; UV-Vis spectroscopy
INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS OF INTEREST ELEMENTS IN FARMLANDS AT THE CENTRAL AREA OF KATSINA STATE, NIGERIA

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**Abstract**

In this study, macro and micro essential elements for plants growth in the Guinea Savannah region of Katsina State in Northern Nigeria have been identified in the farmlands using instrumental neutron activation analysis. Two fundamental soil physical parameters for healthy growth of some common crops revealed the soil pH in H\textsubscript{2}O in the range of 6.0 pH – 7.0 pH, soil pH in 0.01M CaCl\textsubscript{2} and the percentage organic carbon (OC) ranges from 0.0375 - 1.3315, which are optimal for plant nutrients availability in the soils within the study area. The Percentage Deviation from the Certified (PDC) Reference Material NIST Coal Fly Ash 1633b are within tolerable range and the results obtained showed a good agreement between the values obtained in our study with the certified values. The mean concentration of various elements in the samples investigated are (in mg/kg) Mg (253.8 ± 57.53), Al (23047.73), Ca (987.67 ± 229.50), Ti (2825.033), V (26.1187 ± 2.75), Mn (184.71 ± 2.567), Na (1596.4 ± 6.527), K (10651.53 ± 271.8), As (1.2358 ± 0.112), Br (1.08467 ± 0.144), Cr (23.107 ± 1.98), Fe (10475.57 ± 1984), Co (3.8897 ± 0.235), Zn (18.533 ± 3.367), Rb (55.7233 ± 4.35), Sb ( 0.1167 ± 0.031), Cs (1.716 ± 0.2503) and Ba (290.062±26.441). The results of the pH and organic matter content shows near acidic and very low content respectively. The result of the analyzed samples indicate that Mg and Ca are less available to support plant growth while Al has reached toxic level, with other elements of interest being moderately available in this area of study.

**Keywords:** Elements of Interest; INAA; HpGe Detector; WISPAN 2004; pH; Organic Carbon
The objective of this study is to synthesize and characterize the performance of the protic ionic liquids (PILs) to assess its use as a potential antioxidant in drug design. The PILs based on salicylate anion with the 3-dimethylamino-1-propanol (3DMAP) and 3-diethylamino-1-propanol (3DEAP) were synthesized. Proton nuclear magnetic resonance (\(^1\)H NMR), Fourier transformation infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA) were used to characterize the synthesized PILs. Furthermore, the antioxidant activity of the synthesized PILs was determined using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical assay. 3DMAPS and 3DEAPS show higher DPPH free radical scavenging than parent acid (salicylic acid) which leads to good antioxidant activity.

**Keywords:** protic ionic liquids, salicylic acid, radical scavenging, free radical, DPPH assays
SYNTHESIS, CHARACTERIZATION, REACTION MECHANISM AND THEORETICAL STUDY OF AN ANTIMICROBIAL INHIBITOR FROM HETEROAROMATICS BASED THIOSEMICARBAZONE

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Abstract

Invasive fungal and bacteria diseases are the major cause of morbidity and mortality in the critically ill and immunocompromised patients. The impact of current situation was encouraged researcher to develop a better drug against microbial activity. Previous study has reported single prologue of heteroaromatic and thiosemicarbazide have the unique ability as biomimetics as well as active pharmacophore. This study is successfully synthesised through combination of both 2-acetylthiophene and thiosemicarbazide in one molecule structure to form 2-acetylthiophenethiosemicarbazone. Both experimental and theoretical approaches have been applied to comprehend its structure synthesised compound as well as computational drug design. Structure of synthesised compound was characterized using spectroscopy methods. Computational drug design was conducted to calculate the binding interaction between protein and inhibitor. Density Functional Theory (DFT) was used to calculate the chemical properties of title compound such as highest occupied molecular orbital (HOMO), lowest unoccupied molecular orbital (LUMO), chemical hardness, softness, energy gap, chemical potential, and Fukui functions. Solvent determination and predictive interaction element were confirmed using COSMO-RS method calculation. The synthesised compound was tested in vitro against two Gram-positive and one Gram-negative bacterial strains which are Staphylococcus aureus, Staphylococcus epidermidis and Klebsiella pneumonia respectively and a fungus Candida Albicans. Synthesis compound was found that susceptible with all Gram-positive bacterial strain and fungus while not to Gram-negative bacterial strain. The synthesised compound was evaluated the inhibition zone and showed that Staphylococcus epidermidis was active at concentration 100µg/mL with 16±1.5mm while Staphylococcus Aureus at 50µg/mL with 15±2.0mm of inhibition zone. Candida Albicans showed the highest active activity at concentration 50µg/mL with 19±3.2mm.

Keywords: Heterocyclic compounds, Thiosemicarbazide, Density Functional Theory, COSMO-R
COMPARISON OF LABELLING REACTIONS FOR MONOSACCHARIDE COMPOSITION ANALYSIS USING HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

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Abstract
Monosaccharides are the basic units of carbohydrates and are hydrophilic molecules multiple hydroxyl groups. Compared to other molecules, monosaccharides do not possess intrinsic fluorescent or chromophore moiety which makes detection difficult and challenging. Thus, pre-column labelling techniques via derivatisation reactions have been carried out for monosaccharide analysis using high performance liquid chromatography (HPLC). In this study, a series of monosaccharide standards namely mannose (Man), glucose (Glc), and N-acetylglucosamine (GlcNAc) were analysed. These monosaccharides are the building blocks in asparagine-linked oligosaccharides (N-glycans) in all eukaryotes. Derivatisation was performed using 2-aminobenzoic acid (2-AA; fluorescence), 2-aminobenzamide acid (2-AB; fluorescence), 2-aminobenzoic acid ethyl ester (ABEE; UV and fluorescence), and 1-phenyl-3-methyl-5-pyrazolone (PMP; UV) labels possessing different levels of sensitivity. Labelled monosaccharides were then analysed using a HPLC instrument connected to both ultraviolet-visible (HPLC-PDA) and fluorescence (HPLC-FD) detectors using a C18 reversed-phase column. In this study, the sensitivity and resolution of the derivatised monosaccharide peaks were compared. The most suitable label was found to be ABEE which was then used to determine the limits of detection (LOD) and quantification (LOQ) values. Subsequently, the monosaccharide composition of the total hydrolysate of intracellular free oligosaccharides (fOS) prepared from Saccharomyces cerevisiae and Pichia pastoris was determined using fluorescence detection of the ABEE label.

Keywords: High performance liquid chromatography (HPLC), 2-aminobenzoic acid (2-AA), 2-aminobenzamide acid (2-AB), 2-aminobenzoic acid ethyl ester (ABEE), 1-phenyl-3-methyl-5-pyrazolone (PMP), free oligosaccharides (fOS), Saccharomyces cerevisiae, Pichia pastoris.
PHYSICOCHEMICAL INVESTIGATION OF TRIAZOLYL BENZOATE ANIONIC SURFACANT AND ITS MIXTURE WITH GLYCOLIPIDS

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Abstract

Recently, conjugated fatty acids such as linoleic acid mostly from nuts, aromatic fatty acids from butter fats, and furan fatty acids from algae were reported and potentially have remarkable health benefits. These inspire us to synthesize and investigate a synthetic fatty acid contains phenyl and triazolyl that connecting the carboxylic acids head group and the hydrophobic tail. The synthetic fatty acid has been previously reported having extremely lower cmc value compared to natural fatty acid with similar length using conductivity and absorbance measurements. However, investigations of the surfactant behavior using conventional and appropriate methods like surface tension and pH is not fully described. Besides, correlations between the unexpected lower cmc of the substance with the presence of both aromatic and heteroaromatics moieties are not well understood. In these present works, the surfactant was analyzed under 1H-NMR in basic condition with selected surfactant concentrations. At 0.05 mM surfactant concentration, which below cmc value, proton signals on phenyl were indicated as doublets at 8.1 and 7.9 ppm, while proton on triazolyl as a singlet at 8.6 ppm. At above the cmc with 5 mM surfactant concentration, the signals were shifted to higher field at 8.3 ppm for proton triazolyl, and proton aromatic shifted to 7.75 and 7.0 ppm. pH profile indicates micelle-to-vesicle phase transition of the surfactant when pH decreased from 10 to 9. The surface tension of the surfactant was only reduced to 65.4 mN m⁻¹ while showing the trend of micelization with cmc value at 0.13 mM. Although the substance displays an example of fatty acid with additional interactions occurred from pi-pi interaction of phenyl groups, plausibly hydrogen bond between the triazolyls and well-ordered aggregation structures, it is not excellent to further reduce the surface tension of water. Therefore, glycolipids with different chain lengths to the fatty acid solution introduced to the surfactant solution in order to improve its surfactant behavior. A series of surfactant-glycolipids mixtures have been prepared with molar ratio of 9:1. The samples were analyzed with surface tension, conductivity, pH and fluorescence spectroscopy. The results suggest the presence of glycolipid with C12 is significantly reduces the surface tension from 65.4 mN m⁻¹ to 32.1 mN m⁻¹. Aggregation of the surfactant-C12 glycolipid is much stable than in C8 and C10, suggesting by larger hydrophobic interaction of glycolipid C12 with the surfactant. The results imply the possible additional interactions that could only exist between non-ionic glycolipids and fatty acids with aromatic and heterocyclic moieties.

Keywords: Surfactant, Heterocyclic, Self-assembly, Critical micellar concentration
SYNTHESIS AND CHARACTERIZATION OF N-SUBSTITUTED THIOSEMICARBAZONE DERIVATIVES AS CORROSION INHIBITORS FOR MILD STEEL IN 1 M HCl

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Abstract

Two N-substituted thiosemicarbazones derivatives namely as, 2-(4-hydroxybenzylidene)-N-phenylhydrazinecarbothioamide (L2OH) and 2-(4-methylbenzylidene)-N-phenylhydrazinecarbothioamide (L4CH₃) were synthesized using condensation method. The synthesized compounds were successfully characterized by melting point, elemental analysis (C, H, N, and S), fourier-transform infrared spectroscopy (FT-IR) and NMR (¹H and ¹³C) spectroscopy. The ligands were tested as corrosion inhibitors on the corrosion of mild steel in 1 M HCl using Tafel polarization and electrochemical impedance spectroscopy (EIS) techniques. The Tafel results showed that the inhibition efficiency (IE%) of L2OH increased with increasing inhibitor concentrations as compared to L4CH₃. The highest (IE%) obtained for L2OH was 88.96%, while for L4CH₃ was 78.59%. The mild steel surface morphology was studied using scanning electron microscopy (SEM) and atomic force microscopy (AFM) to proves formation of protective film protects the surface of mild steel from the attack of acidic solutions.

Keywords: Thiosemicarbazone, corrosion, mild steel, 1 M HCl
SUBSTITUENTS EFFECT OF SCHIFF BASE DERIVED FROM ANILINE AS CORROSION INHIBITOR ON MILD STEEL IN 1M HCl

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Abstract

Two series of azomethine compounds both derived from 2-hydroxy-3-methoxybenzaldehyde (o-vanillin) with 3-nitroaniline and p-toluidine were synthesized and characterized through condensation method. The structure of synthesized products were elucidated via elemental analysis (CHNS), nuclear magnetic resonance (NMR), infrared spectroscopy (IR) and single crystal X-ray diffraction. The corrosion inhibitory effects of two azomethine compound namely 2-methoxy-6-((p-tolylimino)methyl)phenol (D2) and 2-methoxy-6-((3-nitrophenylimino)methyl)phenol (D3) were studied on mild steel in 1.0 M HCl by electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization measurements. Based on the electrochemical measurement, the azomethine compound being studied can possibly achieve inhibition efficiency up to 90% on mild steel in acidic environment. It was found that the presence of electron donating group substituent on the compound increases the corrosion inhibition efficiency on the mild steel. The surface morphology of mild steel specimen was further investigated by atomic force microscopy (AFM) and scanning electron microscopy (SEM).

Keywords: Azomethine, O-vanillin, Aniline, Mild Steel, Hydrochloric Acid.
NON-SYMMETRICALLY SUBSTITUTED BIS-BENZIMIDAZOLIUM SALTS 
AND THEIR RESPECTIVE DINUCLEAR SILVER(I)-NHC COMPLEXES:
SYNTHESIS AND ANTIBACTERIAL ACTIVITIES

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Abstract
This work described the synthesis of the non-symmetry bis-benzimidazolium salts as precursors for the dinuclear Ag(I)-NHC complexes (NHC = N-heterocyclic carbene). Through the reaction of 3-(2-bromoethyl)-1-butylbenzimidazole bromide (I) with n-alkylbenzimidazole (alkyl = methyl, ethyl, propyl, pentyl) with 1:1 ratio in acetonitrile, non-symmetry bis-benzimidazolium bromide salt (1a, 2a, 3a and 5a) were obtained. On the other hand, the symmetry bis-benzimidazolium bromide salt (4a) was synthesized through the easier reaction of n-butylbenzimidazole with 1,2-dibromoethane with 1:2 ratio in 1,4-dioxane. A part of these salts were converted to their hexafluorophosphate salts (1b-5b), respectively for characterization purpose while the other remaining were then undergo \textit{in-situ} deprotonation with Ag\textsubscript{2}O to produce dinuclear Ag(I)-NHC complexes, 6-10. The successful complexations were proved by the disappearance of the most downfield H2' peak in \textit{\textit{1}H NMR (ca 9.8-10)} and confirmed by the presence of doublet peaks C2'-Ag at 187-180 and 188-190 ppm in \textit{\textit{13}C NMR. The bis-benzimidazolium salts 1-5b and Ag(I)-NHC complexes 6-10 were screened for their antibacterial potential against \textit{E. coli} (ATCC 25922) and \textit{S. aureus} (ATCC 12600). All the bis-benzimidazolium salts exhibited no activity against both bacteria while the Ag(I)-NHC complexes showed similar or medium activities compared to standard antibiotic, amoxicillin.

\textbf{Keywords:} N-Heterocyclic carbene; non-symmetry; bis-benzimidazolium; Ag(I)-NHC; \textit{in-situ} deprotonation; antibacterial activity
INVESTIGATION OF RUTHENIUM ALKYNYL COMPLEXES FOR NONLINEAR OPTIC APPLICATION USING COMPUTATIONAL METHOD

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Abstract
Hartree Fock (HF) and density functional theory (DFT) methods based on a 3-21G set level were used to computationally assess the nonlinear optic (NLO) response of six ruthenium (Ru) arylalkynyl complexes. Substitution of Ru-phenyl with six simplified models of Ru-H and Ru-methyl complexes revealed that DFT-based calculations were more accurate than HF in estimating the NLO response. The calculated bond lengths and angles of Ru-methyl was in good agreement with Ru-phenyl. Given that the calculated C≡C stretching vibration and UV-vis maximum absorption for Ru-methyl was comparable to Ru-phenyl, with values corresponding to 2154.56 cm$^{-1}$ and 460.93 nm, respectively, it was evident that Ru-H, Ru-methyl and Ru-phenyl complexes undergo intraligands $\pi-\pi^*$ and Laporte forbidden metal d-d transition. Henceforth, it is affirmed that calculations using simplified Ru-H complexes were as much as reliable as the full structure of Ru to assess the NLO response. Assessment of electron inductive effect on Ru-carbonyl (Ru-Co), Ru-cyclopentadienyl (Ru-Cp) and Ru-bipyridine (Ru-bpy) complexes revealed two absorption maxima that appeared in regions 320–375 nm and 382–460 nm, which represent an intraligand $\pi-\pi^*$ orbital and Laporte forbidden d-d-transition, respectively. Migration of electrons from Ru center to the bipyridine ligand suggests a greater electron acceptor effect than Ru center to the arylalkynyl group. However, Ru conjugated to an electron withdrawing group i.e. carbonyl tend to render lower NLO response while elevating HOMO-LUMO energy gap and Ru to C$\alpha$ bond lengths.

Keywords: ruthenium; nonlinear optic; computational studies; Gaussian; ADF
ONE POT GREEN SYNTHESIS AND ANTIMICROBIAL STUDIES OF SALICYLALAZINE DERIVATIVES SCHIFF BASE

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Abstract
Two azine Schiff base ligands, Hla and HLb derived from thiocarbohydrrazide and salicylaldehyde derivatives were synthesized using microwave assisted synthesis approach. The confirmation of both ligands were elucidated through physiochemical and spectroscopy technique as well as single x-ray crystallography diffraction. The analyses shown that the ligands synthesized were formed as azine instead of thiocarbohydrrazone based on the missing thione, C=S moiety throughout all spectral data. The molecule stucture was further conclude by x-ray crystal analysis. The biological properties of these ligands were screened using Disc diffusion method. The result shows that HLb give significant inhibition toward all of the bacteria tested.

Keyword: antimicrobial, azine, disc diffusion, microwave, Schiff base
SYNTHESIS, CHARACTERISATION & CYTOTOXICITY STUDY OF BENZYL 2-((1E,4E)-1,5-BIS(4-BROMOPHENYL)PENTA-1,4-DIEN-3-YLIDENE)HYDRAZINECARBODITHIOATE & BENZYL 2-((1E,4E)-1,5-BIS(4-CHLOROPHENYL)PENTA-1,4-DIEN-3-YLIDENE)HYDRAZINECARBODITHIOATE AND THEIR Ni(II), Cu(II), Fe(II), Zn(II), & Cd(II) COMPLEXES

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Abstract
Dithiocarbazate Schiff bases and their derivatives have drawn considerable attention due to their unique properties and applications. Many dithiocarbazate metal complexes have been synthesised and applied in many applications such as antibacterial, antifungal, antioxidant agents and in catalysis. Dithiocarbazate metal complexes have also shown significant cytotoxicity against many types of cancer cell lines. This study aimed to synthesise non-toxic compounds by synthesising halogenated chalcones and studying the effect of halogen electronegativity on the cytotoxicity of the metal complexes. Two chalcones, p-chlorodibenzalacetone and p-bromodibenzalacetone were synthesised using base-catalysed Aldol condensation. These chalcones were then reacted with S-benzyldithiocarbazate to form two novel Schiff bases. Ten novel metal complexes were synthesised by reacting these two Schiff bases with five divalent transition metal acetates which were Ni²⁺, Fe²⁺, Cu²⁺, Zn²⁺, and Cd²⁺. These Schiff bases and their metal complexes were fully characterised using various characterisation techniques including FTIR, UV-Vis, ¹H & ¹³C NMR spectroscopy, mass spectral and elemental analysis, and single crystal X-ray diffraction. The cytotoxic properties of these compounds were also tested against two types of bladder cancer cell lines which were RT112 and EJ28. Copper(II) complexes showed better activity than the rest of the metal complexes and better selectivity toward EJ28 than RT112 cell lines. The bromo-substituted complexes showed better effect on the cytotoxicity than chloro-substituted complexes. The copper(II) complex containing di-p-bromobenzalacetone-S-benzyldithiocarbazate Schiff base was observed to have the strongest cytotoxicity with an IC₅₀ value of 2.62 μM against the EJ28 bladder cancer cell line.

Keywords: Schiff base; dithiocarbazate; S-benzyldithiocarbazate; SBDTC; metal complexes; bladder cancer; cytotoxicity; dibenzalacetone
CRYSTAL GROWTH AND PHYSICAL CHARACTERIZATION OF NICOTINAMIDE CRYSTALLIZED WITH CINNAMIC ACID

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Abstract

Pharmaceutical cocrystals can be defined as compounds that contain two or more different molecular components at certain stoichiometry, usually present in different characteristics from the precursors. In this work, cinnamic acid (CIN) was employed to form the cocrystal with the active pharmaceutical ingredients nicotinamide (NIC), which characterized to compare with the precursors. The cocrystallization of NIC with CIN was studied in different molar ratios. It was anticipated that acid-amide heterosynthon as the driving force for cocrystallization. Prior to cocrystallization of NIC with CIN, the estimation of miscibility using Group Contribution Method (GCM) and pKₐ difference rule showed possible miscibility and the formation of cocrystal respectively. NIC-CIN cocrystal was prepared by the slow evaporation method using ethanol solvent. The synthesized cocrystal was characterized using DSC, FTIR, PXRD and ¹H-NMR. The PXRD analysis revealed significant peaks shift which indicates the feasible formation of cocrystal. The DSC thermogram of NIC-CIN mixture showed a lower melting point at 98.95 °C compared to NIC at 129.94 °C and CIN at 134.33 °C precursors. The difference in the melting point of the mixture from the precursors supports the PXRD data which confirmed the feasible formation of cocrystal. The crystal structure of NIC-CIN cocrystal was determined by single crystal X-ray diffraction (SCD). The crystal system of NIC-CIN cocrystal was monoclinic with space group P2₁/c and Z=4. Amide-carboxylic acid heterosynthon was observed in the cocrystal. The intermolecular hydrogen bond, O-H-N, N-H-O and π-π interactions were detected in packing of NIC-CIN cocrystal.

Keywords: cocrystal; physical characterization; molecular interaction; hydrogen bonding; single crystal XRD; crystal structure
SYNTHESIS AND CHARACTERISATION OF FERROCENE-INDOLE DERIVATIVES VIA SIMPLE ESTERIFICATION AS HEla INHIBITOR

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Abstract

The rise of organometallics in various applications is noticeable in the past few decades. Conventionally, organometallics are usually perceived as difficult to synthesis, isolate and involves high maintenance in terms of storage due to high susceptibility to oxidation especially on the metal centres. Hence, ferrocene is chosen as the organometallic core of interest due to its classical representation of an organometallic compound. A conjugate of ferrocenephényl diamide-indole hybrids were synthesised via a one-step reaction that resembles Steglich Esterification. As the use of ferrocene-indole for biology application is still new to be ventured. A precursor molecule ferrocenephénylendiamine is also synthesised, with the same method proposed and dicyclohexylcarbodiimide (DCC) as coupling agent. However, 1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide (EDC) was used for the synthesis of the final hybrids, as the use of DCC yield excessive insoluble by-products (dicyclohexylurea) that are very difficult to separate. Both reactions mentioned are successful, the novel precursor and a series of 6 compounds with all 6 possible substitution site of indole substituted by ferrocenephénylendiamine were obtained.

All 7 mentioned compounds are obtained in reasonable yields. Surprisingly, among the 6 ferrocene-indole hybrids, only substitution of position -7 has a significant lower yield of 32% while the others are pretty much similar at around 60%. Besides, precipitates of the final products are stable under room condition, they does not undergo drastic change of colour over time. Structure of the compounds are confirmed by the help of Nuclear Magnetic Resonance (NMR) and Fourier-Transform Infrared Spectroscopy (FT-IR). The obtained ferrocene-indole hybrids were treated to HeLa cell lines in MTS assay for 24h, 48h and 72h, the IC\textsubscript{50} values obtained show that all 6 of the compounds (with substitution site of indole at position -2, -3, -4, -5, -6 and -7 replaced by precursor) are cytotoxic towards HeLa, and the most potent compound has its indole substituted at position 3 (-3), showing a IC\textsubscript{50} value as low as 1.9\textmu M. This project is believed to be able to serve as a pioneering approach to any future ferrocene-indole based anticancer drugs design.

Keywords: Organometallics, ferrocene, indole, Steglich esterification, HeLa
WASTES FROM KITCHEN: A PROMISING MATERIALS FOR ANALYTICAL SAMPLE PREPARATION

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Abstract

In this study, we are discovering the hidden potential of the kitchen wastes as useful resources for analytical sample preparation in environmental studies. Kitchen wastes that are available have a potential to be used as inexpensive materials for analytical sample preparation. Transformation of kitchen wastes into valuable material is of double interest: on the one hand, a waste is converted into value added product, and, on the other, alternative way in waste management. In this presentation, we focus mainly on various strategies in the preparation of new materials from kitchen wastes and their corresponding applications in separation science.

Keywords: Kitchen wastes, sample preparation, separation science.
CHEMOSENSOR DEVELOPMENT USING 2-ACETYLPYRROLE THIOSEMICARBAZONE FOR Cu²⁺ ION RECOGNITION IN AQUEOUS MEDIUM: EXPERIMENTAL AND THEORETICAL STUDIES

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Abstract

A sensitive and selective colorimetric of 2-acetylpyrrole thiosemicarbazone for the efficient detection of Cu²⁺ has been developed. The colorimetric and optical properties of a new chemosensor of 2-acetylpyrrole thiosemicarbazone were studied by the naked-eye detection and UV-VIS spectroscopy. The ligand was synthesised from thiosemicarbazone and 2-acetylpyrrole through condensation reaction. The ligand was further characterized by melting point, elemental analysis CHNS, IR, UV-visible spectroscopy, and 1H-NMR spectroscopy. The sensitivity of 2-acetylpyrrole thiosemicarbazone was done by optimizing the solvent, ratio of solvent:co-solvent and the pH buffer that was used. The selectivity test of the chemosensor was also conducted. DMSO was chosen as the best solvent with a 5:5 ratio at pH 7. All the optimization that was used has given a significant result in the detection of Cu²⁺ ion. The 2-acetylpyrrole thiosemicarbazone chemosensor did not encounter any interference from other metal ions. The detection limit of the probe towards Cu²⁺ was 1.88 x 10⁻⁵ M. The interaction by the formation of the 2-acetylpyrrole-Cu complex is 1:1 stoichiometry that was calculated using Job’s plot method. The sensing behavior of the chemosensor was further emphasized by computational studies. The sigma profile was calculated using COSMO-RS. Density Functional Theory (DFT) calculations, such as MEP, Fukui function and HOMO-LUMO energy gap were successfully calculated to visualize and clarify the interaction between 2-acetylpyrrole thiosemicarbazone and Cu²⁺.

Keywords: Thiosemicarbazone, Chemosensor, COSMO-RS, DFT
ELECTROKINETIC SUPERCHARGING IN CAPILLARY ELECTROPHORESIS FOR ONLINE PRECONCENTRATION OF 5-FLUOROURACIL AND ITS METABOLITES IN HUMAN PLASMA

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Abstract

In this work, an online preconcentration method termed electrokinetic supercharging (EKS) combined with capillary electrophoresis with diode array detector (CE-DAD) system, was developed and evaluated for the determination of 5-fluorouracil (5-FU) and its metabolites in human plasma. Several important parameters such as buffer composition and concentration, terminating electrolyte, organic modifier, and injection voltage and injection time of both terminating electrolyte and sample were comprehensively optimized. The optimum EKS conditions used were as follows: type of background electrolyte, 45 mM sodium hydrogen phosphate at pH 8; temperature, 20 ºC; voltage, 15 kV; injection time, 200 s; total capillary length, 65 cm; leading electrolyte, 50 mM NaCl and terminating electrolyte, 50 mM TRIS. Under the optimized EKS and CE-DAD conditions, the proposed method will be validated in terms of its linear dynamic range, precision (repeatability) and accuracy (relative recovery). To test the applicability of the method, the developed EKS-CE-DAD will be applied to the analysis of 5-FU and its metabolites in human plasma samples from cancer patients after pretreatment procedure.

Keywords: electrokinetic supercharging, capillary electrophoresis, 5-fluorouracil and its metabolites, human plasma
CHEMOSENSOR DEVELOPMENT OF Cu$^{2+}$ RECOGNITION USING 1,5-DIPHENYL CARBAZONE: OPTIMIZATION, COSMO-RS AND DFT STUDIES

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Abstract

The sensitive and selective chemosensor for copper (II) ions (Cu$^{2+}$) was successfully optimized using the 1,5-diphenylthiocarbazone (DPT) compound. Results showed that Dimethyl Sulfoxide (DMSO) in a 9:1 ratio with a co-solvent at a pH 3 was the optimum condition for DPT to act as chemosensor of Cu$^{2+}$ recognition. The DPT chemosensor did not encounter any interference from other metal ions, including Fe$^{3+}$, Ag$^{+}$, Cr$^{3+}$, Pb$^{2+}$, Cu$^{2+}$, Mg$^{2+}$, Cd$^{2+}$, Zn$^{2+}$, K$^{+}$, Ni$^{2+}$ and Co$^{2+}$. The presence of Cu$^{2+}$ led to an absorption peak at 658 nm, where the colour changed from cantaloupe to grey-green color indicating the interaction by the formation of the DPT-Cu complex in 2:1 stoichiometry. The theoretical σ-profile calculation using conductor-like screening model for real solvents (COSMO-RS) showed the compatibility of DPT with the DMSO solvent through hydrogen bonding. In the density functional theory (DFT) calculations, the formation energy of DPT and DPT-Cu were -1113.79645660 a.u. and -2435.71832681 a.u., respectively. Under optimal conditions, a detection limit of 1.47 ppm for the DPT chemosensor for Cu$^{2+}$ recognition can compete with the atomic absorption spectroscopy (AAS) value of 1.50 ppm. The preliminary results show that DPT was able to provide less expensive, more portable and convenient chemosensor for Cu$^{2+}$ recognition in aqueous medium, as compared to AAS.

Keywords: Chemosensor, 1,5-diphenylthiocarbazone (DPT), COSMO-RS, DFT
SIMULTANEOUS ENANTIOMERIC RESOLUTION OF IMIDAZOLE ANTIFUNGAL AGENTS USING HYDROXYPROPYL-β-CYCLODEXTRIN AS CHIRAL SELECTOR IN CAPILLARY ELECTROPHORESIS

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Abstract
Capillary electrophoresis (CE) method employing native β-cyclodextrin (β-CD) and modified hydroxypropyl-β-cyclodextrin (HP-β-CD) was studied and they were being compared as a chiral selector for simultaneous enantioseparation of two imidazole antifungals. The CE method was developed and applied to discriminate two stereoisomers of cis-ketoconazole and two stereoisomers of miconazole as the subjects of study. The background electrolyte (BGE) type and concentration, and chiral selector types and concentrations, pHs, running voltage, and capillary temperature were studied and optimized. In this study, the modified cyclodextrin was proved to enhance the enantioseparation of ketoconazole and miconazole compared to native β-CD as it exhibited a higher resolving power than the native one. Under optimum conditions such as 35 mM tris-phosphate buffer at pH 2.5 containing 1.5 mM HP-β-CD with applied voltage of 15 kV at 15°C, the ketoconazole and miconazole enantiomers are successfully resolved within 30 minutes. In order to understand the possible chiral recognition mechanism of both imidazole compounds with native β-CD and HP-β-CD, the host-guest inclusion complex and binding constant studies were done using UV-vis spectroscopy, FTIR and NMR analyses.

Keywords: hydroxypropyl-β-cyclodextrin, imidazole, capillary electrophoresis
DEVELOPMENT AND VALIDATION OF HPLC METHOD FOR SIMULTANEOUS DETERMINATION OF CARBAMAZEPINE AND GABAPENTIN IN FIXED-DOSE COMBINATION

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Abstract
This paper describes a simple, sensitive and selective high-performance liquid chromatographic (HPLC) method for the separation and determination of carbamazepine (CBZ) and gabapentin (GBP) simultaneously in fixed-dose combination. The chromatographic separation was performed using COSMOSIL (250 mm x 4.6 mm i.d., 5 μm particle size) at 30 ± 0.5°C, with a mobile phase composed of phosphate buffer (pH 4.0 ± 0.02) and an organic phase (methanol:acetonitrile; 62.5:37.5) in the ratio of 60:40. The flow rate was maintained at 1.0 ml/min, injected volume was 5 μl and detection wavelength at 210nm. The method was validated according to ICH Q2 (R1) guidelines and found to be linear over a range of 375-2250 μg/ml (R² = 0.9999) and 125-750μg/ml (R² = 0.9985) for GBP and CBZ, respectively. The drugs in combination were subjected to various stress degradation studies as per the International Conference Harmonization (ICH) guidelines. Results obtained from the stress degradation studies revealed that the developed method is applicable for stability studies.

Keywords: Carbamazepine; Gabapentin; HPLC; method development; validation
ELECTROCHEMICAL LIQUID-LIQUID EXTRACTION OF PHARMACEUTICAL COMPOUNDS

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Abstract

Quantification of drugs in biological sample such as urine and plasma remain challenging for the study of their pharmacokinetics. Unfortunately, drug traces in biological sample have a very low concentration. Therefore, sample preparation plays significant role in drugs determination as it can improve the limit of detection. The classical methods such as liquid-liquid extraction do not operate with hydrophilic medical drugs. Electrochemistry at the liquid-liquid interface allows the control of drugs distribution between the two immiscible phases [1]. We propose here a method for drug extraction based on electrochemistry at the interface between two immiscible electrolyte solutions (ITIES). The principle is based on the application of an electrical driving force to transfer desired ions across the ITIES. Cyclic voltammetry studies at ITIES showed that metformin, phenyl biguanide, phenformin can be extracted from urine to dichloroethane despite their hydrophilicity. The application of different Galvani potential differences enables selective extraction of drugs. Metformin which is the most hydrophilic drug needs a higher potential to transfer across interface compared to phenformin (more hydrophobic). Chemical potential modulation method was developed as an instrument-free extraction method based on electrochemical principles. The potential was applied by introducing concentration gradients of tetramethylammonium chloride (TMA+ Cl-) between two phases. Important parameters such as volume of aqueous and organic phase, pH and concentration of TMA+ Cl- were optimized to improve the enrichment factor. Values of 40.8, 44.2, 49.3 for the enrichment factor were obtained for extracted metformin, phenyl biguanide, phenformin respectively. The results showed that by using this method, the targeted compounds can be extracted from aqueous phase and analysed by classical chromatographic methods.

Keywords: Metformin; phenformin; phenyl biguanide; electrochemically modulated liquid-liquid extraction; ITIES; concentration gradient
THE ETHICAL CONCERNS OF BIOANALYTICAL CHEMISTRY: THE CASE OF FORENSIC SCIENCE

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Abstract

Due to its pragmatic nature, bioanalytical science, a sub-discipline of analytical chemistry, is increasingly becoming a central point to address the practical needs of modern community life. It seems to be very promising and potential to play a major role in many sectors of industrial, environmental and medical applications. It nevertheless, encompasses various forms of analytical chemistry, such as: (i) bioanalysis for medical and clinical purposes, especially in the pharmaceutical industry; (ii) quality assurance purposes in food industry and products; (iii) environmental chemistry and ecosystem; and (iv) bioanalysis for forensic purposes. The practical part of all these sciences depends on bioanalytical chemistry, which is based on techniques and technologies that are characterized by accuracy, preciseness and honesty. To harness potentials of the newly growing science of bioanalysis, the analytical chemists need to address, not only matters of accuracy and preciseness of measurement methods and techniques, but also the ethical implications of bioanalysis. In fact, the ethical concerns are arising alongside with the various techniques adopted, application methods, fraudulent cases, negligence, as well as other problems that may jeopardize the integrity of bioanalytical science. This paper focuses, especially, on the ethical aspects of forensic science which depends solely on bioanalytical chemistry. Forensic science is mainly concerned with using results of bioanalysis for legal purposes, to draw evidences for conviction. Although there is a considerable literature on forensic science, however, the ethical dimension of this science needs further investigation, especially based on technical study of bioanalytical chemistry, as aimed by this paper. The paper, firstly, outlines briefly the basic forms of bioanalytical chemistry, as summarized above, and their technical methods of analysis; secondly, the paper investigates the ethical implications of forensic science. The method adopted will be theoretical, analytic and critical in nature.

Keywords: Bioanalysis; analytical techniques; forensic evidences; biological fluids; ethical implications; legal investigation
FABRICATION OF MAGNETIC POLY(β-CYCLODEXTRIN FUNCTIONALIZED IONIC LIQUID) NANOCOMPOSITES AND ITS APPLICATION IN THE MAGNETIC SOLID PHASE EXTRACTION OF POLYCYLIC AROMATIC HYDROCARBONS FROM RICE SAMPLES

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Abstract

Ionic liquid and chemical cross-linker were employed as a structural modifier for magnetized β-cyclodextrin to increase its merit, also to overcome some limitation of β-CD alone in the solid phase extraction process. In-situ chemical functionalization and polymerization were used to prepare Fe₃O₄@βCD-Vinyl-TDI nanosorbents. Application of this designed material in the magnetic SPE (MSPE) of selected polycyclic aromatic hydrocarbons (PAHs), as model analytes in the rice samples coupled with GC-FID was evaluated. The characterization of the nanocomposite was performed using transmission electron microscopy (TEM), scanning electron microscopy (SEM), X-ray diffraction analysis (XRD), Fourier Transform Infrared (FT-IR) spectroscopy, 1H and 13C NMR spectrometry, zeta potential analysis, thermogravimetric analysis (TGA), wetting analysis, Brunauer-Emmett-Teller (BET) Surface Area and Barrett-Joyner-Halenda (BJH) Pore Size and Volume Analysis, elementary analysis (EA) and vibrating sample magnetometry (VSM). Nine important parameters, affecting the extraction efficiency of PAHs, including: sorbent dosage, sample solution, effect of pH, salt addition, organic modifier, extraction time and desorption conditions were investigated. The optimum extraction conditions were obtained as: 20 mg of sorbents in 25 mL sample solution without pH adjustment, 3% (w/v) of salt addition, extraction time of 30 minutes, 200 μL of acetonitrile without organic modifier as desorption solvent and a desorption time of 15 minutes under shaking. Good performance data were obtained at the optimized conditions. Detection limits were in the range of 0.01-0.18 μg/kg in real matrix. The calibration curves were linear over the concentration ranges from 0.1 to 500 μg/kg with correlation determinations (R²) from 0.9970 to 0.9982 for all the studied analytes. The RSDs values were found to be between 2.95%-5.34% for intra-day and between 4.37%-7.05% for inter-day precision in six different days. The sorbents exhibited a satisfactory reproducibility in the range of 2.9% to 9.9% in extracting the five selected analytes. Acceptable recoveries values 80.4% -112.4%, were also obtained for the real sample analysis using the proposed method. The fabricated adsorbent combines the advantages of the superior adsorption capability of modified cyclodextrin cross-link polymer and separation ability of magnetic nanoparticles to provide high adsorption capacity, and easy isolation from sample solutions.

Keywords: Ionic liquid; β-cyclodextrin; Fe₃O₄@βCD-Vinyl-TDI nanosorbents; Magnetic solid phase extraction; GC-FID
SYNTHESIS, CHARACTERIZATION AND CATALYTIC APPLICATION OF SYMMETRICAL PALLADIUM(II) N₂O₂- SCHIFF BASE TOWARD MIZOROKI-HECK REACTION

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Abstract
A symmetrical square planar palladium(II) complex with N₂O₂-tetradentate ligand was synthesized by condensation reaction of 3,5-di-tert-butyl-2-hydroxybenzaldehyde with 2,2-dimethyl-1,3-propanediamine followed by complexation with palladium(II) acetate using equimolar amount to give C₃₅H₅₂N₂O₂Pd. The catalyst was then characterized by several techniques such as elemental chemical analysis CHNS, FTIR, NMR spectroscopy and Single X-ray crystallography. This air/moisture stable symmetrical catalyst was investigated for Mizoroki-Heck reaction of aryl bromide with methyl acrylate using several parameters such as different of bases, catalyst loading and temperatures. The catalytic reactions were monitored by GC-FID. The isolated product from the catalytic testing obtained as 3-(4-acetylphenyl) acrylic acid methyl ester.

Keywords: N₂O₂-tetradentate ligand; palladium(II) complex; Mizoroki-Heck reaction
SIZE-CONTROLLED SYNTHESIS OF PALLADIUM NANOPARTICLES SUPPORTED ON TITANIA FOR HYDROGENATION REACTION

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Abstract

Sol-immobilisation method was employed in the preparation of 1wt% palladium nanoparticles (Pd NPs) supported on TiO\textsubscript{2} (different phases: P25, rutile, anatase). The size of Pd NPs were controlled by controlling the reduction temperature of Pd nucleation, in which the kinetic growth was altered by variation of the temperature from 1 °C up to 75 °C. The method lead to the formation of Pd NPs with diameter range between 2.6 nm up to 5 nm. The process of reduction was monitored by UV/Vis spectrometer. Whereas, TEM, IR-CO adsorption and EXAFS were used to characterized catalysts properties. Various attempts have been made by other researchers to develop a suitable catalytic system for cinnamaldehyde hydrogenation, however the selectivity is still an important subject. The immobilized Pd NPs catalysts from our work demonstrated an outstanding activity and selectivity of reaction at C=C which can be tune with controlled particles size and metal support interaction.

Keywords: Palladium nanoparticles; EXAFS; TEM
A COMPARATIVE STUDY ON THE STRUCTURE-ACTIVITY RELATIONSHIP OF Ru/M*/Ce/Al₂O₃ PROMOTED WITH Mg AND Mn FOR CO₂/H₂ METHANATION REACTION

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Abstract
In this paper, the influence of Mg and Mn incorporation method on the ceria based catalyst for CO₂ methanation was systematically investigated. A series of trimetallic Ru/M*/Ce/Al₂O₃ (M*= Mg or Mn) catalysts with different ratios and calcination temperatures were prepared by wetness impregnation method. Among the catalyst screened, the Ru/Mn/Ce (5:30:65)/Al₂O₃ catalyst with calcination temperature of 1000°C exhibited the highest CO₂ conversion of 97.73% and nearly 100% selectivity to methane at a reaction temperature as low as 200°C. The TPR-TPD analysis of Ru/Mn/Ce (5:30:65)/Al₂O₃ catalyst disclosed the addition of Mn improving the reducibility of the catalyst due to its higher surface basicity as compared with the Mg modification. The presence of Mn₂O₃ species could help to form a moderated interaction with the support which inhibited the particles agglomeration in high temperature, improved the dispersion thus enhanced in the CO₂/H₂ adsorption capacity.

Keywords: methanation; carbon dioxide; catalyst; ceria, magnesium; manganese
GLYCEROL DEGRADATION WITH ABSENCE OF EXTERNAL HYDROGEN GAS BY USING WASTE EGGSHELL AS HETEROGENEOUS CATALYST

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Abstract
The demand of biodiesel production nowadays is getting high every day. The function of biodiesel itself as alternative fuel to replace the usage of fossil fuels is one of the best technique to reduce the carbon dioxide (CO2) gas release in the atmosphere. However, the booming of biodiesel’s demand resulted in increasing of glycerol as major by-products. Although glycerol could serve in various industries, the demand and usage are still limited. Therefore, an alternative has been taken in this research in order to convert glycerol into more value-added chemical of methanol (MeOH), propanol (PrOH) and 1,2-propanediol (1,2-PDO) through low-cost and simple reflux technique. The calcium oxide catalyst derived from waste eggshells was utilized as heterogeneous catalyst. The optimum condition was obtained at temperature of 170 °C, 7 hours, 1 g of catalyst and 20 wt% of glycerol concentration. The optimum glycerol conversion (82 %) and products selectivity to MeOH (7.95 %), PrOH (70.8 %) and 1,2-PDO (21.2 %) were observed.

Keywords: glycerol; degradation; external hydrogen; waste eggshell; heterogeneous catalyst
POSTER SESSIONS ABSTRACTS
ELECTROCHEMICAL TREATMENT OF AQUEOUS C. I. REACTIVE BLUE 21 AND SYNTHETIC TEXTILE EFFLUENT USING METAL/GRAFTHITE-POLYVINYL CHLORIDE COMPOSITE ELECTRODE

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Abstract

In this study, the electrochemical oxidation of C. I. Reactive Blue 21 (RB21) on a cobalt/graphite-polyvinyl chloride (Co₄₇.₅/C₄₇.₅-PVC₅) composite electrode was investigated using electrochemical technique. Co₄₇.₅/C₄₇.₅-PVC₅ and graphite rod were used as an anode and cathode, respectively, in the presence of NaCl as a supporting electrolyte in the electrolysis of RB21 solution. Optimum electrolysis conditions for the decolorization of RB21 solution using Co₄₇.₅/C₄₇.₅-PVC₅ electrode is by using 20 V of applied voltage for 45 min of electrolysis time in the presence of 0.5 mol L⁻¹ NaCl solution as a supporting electrolyte. Under the optimum electrolysis conditions, 99.95% of RB21 decolorization percentage has been achieved. The Co₄₇.₅/C₄₇.₅-PVC₅ electrode also shows high efficiency in the decolorization of synthetic textile effluent containing azo and anthraquinone dyes using similar optimum electrolysis conditions as mentioned above. This is further confirmed by the wastewater parameter analyses in which the high removal percentage (>75%) of COD and BOD₅ was achieved for the treated solution. This shows that the prepared electrode provides high efficiency in the removal of dye compounds. Characterization of the electrode surface using FESEM-EDX shows no significant changes in the composition of C, Co and Cl elements in the prepared electrode before and after electrolysis of RB21 solution. This shows that the prepared electrode has a good binding ability between C and Co by using PVC as a binder agent.

Keywords: Electrochemical technique; C. I. Reactive Blue 21; synthetic textile effluent; composite electrode
REMOVAL OF Cu AND Pb FROM AQUEOUS SOLUTION USING CORN LEAVES (Zea Mays) AS ADSORBENT

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Abstract
Corn leaves are considered as an agricultural waste which causes environmental problems due to the way they have been disposed. The potential to remove Pb and Cu ion from wastewater treatment systems using untreated corn leaves (CLUT) and treated corn leaves (CLT) through adsorption was investigated in batch experiments. CLT were treated by 0.1 M NaOH. Batch mode experiments were conducted in various parameters such as adsorbent mass (0.1-0.9 g), pH (1-11), concentration of toxic metals (5-25 mg/L) and contact time (0-120 min). Highest percentage removal of Cu (41 % and 78 %) and Pb (72 % and 92 %) were achieved using 0.9 g of CLUT and CLT. Maximum adsorption was obtained at pH condition of 3-4 for both adsorbents. At contact time of 120 min, for concentration of 5 mg/L, Pb was removed up to 80 % using CLUT and 90 % using CLT. Meanwhile, as for Cu ion, at contact time of 120 min with concentration of toxic metal of 5 mg/L, 46 % was removed using CLUT while the removal percentage achieved up to 93 % using CLT. Low initial concentration of toxic metals would give high percentage removal of toxic metals. The adsorption data fitted well to the Langmuir isotherm model which assumes that the adsorption process occurred is a monolayer adsorption which occurs on the same sites on the adsorbent surface. Therefore, this study demonstrated that both adsorbents which are CLUT and CLT could be used to remove Cu and Pb from industrial wastewater.

Keywords: agriculture waste, corn leaves, adsorbent, toxic metals
SEDIMENT QUALITY ASSESSMENTS IN RELATION TO SOCIO-ECONOMIC DEVELOPMENT IN KAMPUNG TEKEK, TIOMAN ISLAND, PAHANG DURING SURVEY IN AUGUST 2015

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Abstract

The study to identify the sediment quality changes affected by the increasing human activities along the coastal environment in Kampung Tekek, Tioman Island has been conducted in August 2015. In total, 15 surface sediment samples were collected from three transects in the bay of Kampung Tekek to be analyzed with particle size analyzer (PSA) and loss on ignition (LOI) methods. The results demonstrates good relationship between grain size classification, sediment sorting, skewness and kurtosis which shows differences between the coarser grain and poorly sorted zone up to the finest grain and best sorted zone. However, the highest percentages of sediment composition were recorded to be sand content (23 to 64%) with a mean of 48%, followed with silt content (29 to 68%) with a mean of 43%. The total organic matter (TOM) were ranged from 4.88 to 14.15% with a mean value of 7.61%. This sediment texture contains mostly fine and medium silts could expose poten threat to the coral reefs in the bay of Kampung Tekek due to sedimentation and higher partitioning of contaminants in sediment.
SEASONAL INFLUENCES ON THE LEVELS OF PARTICULATE METALS IN KUANTAN RIVER, EAST COAST MALAYSIA USING PRINCIPAL COMPONENT ANALYSIS

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Abstract
Principal component analysis (PCA) were performed to evaluate temporal variations of trace metals of Kuantan River waters. Water samples from 12 sampling stations were taken from downstream of the estuary towards the upstream of Kuantan River during the Northeast Monsoon (NEM) and Southwest Monsoon (SWM). Particulate metals were filtered, dried, weighed, analyzed using Teflon Bomb digestion processes and detected using ICP-MS. The metals distribution in suspended particulate matter was found influenced by monsoon seasons particularly during NEM. The PCA/FA identified six varifactors, which were responsible for 83.30% of total variance in the dataset. The PCA results showed that the main source of river water pollution is mostly due to the point sources such as domestic wastewater, wastewater treatment plants and industries as well as non-point sources namely agriculture and oil palm plantations. This study illustrates the usefulness of PCA for identification of pollution sources and understanding temporal variations in river water for effective river water management.

Keywords: Trace Metals, River water, Principal component analysis, Suspended particulate matter
DEGRADATION OF METHYL ORANGE BY USING SILVER PHOSPHATE/TITANIUM DIOXIDE PHOTOCATALYST

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Abstract

Azo dyes have been widely used in the industries and they contributed to the largest group of pollutants found in water. They are not only highly toxic and hazardous, but also causing various diseases and disorders to aquatic life and human being. Hence, photodegradation is an important method for wastewater treatment. In this research, six samples of Ag₃PO₄/TiO₂ photocatalysts were prepared via simple precipitation method by varying the mass ratio of Ag₃PO₄ to TiO₂ and these were used in the photodegradation of methyl orange (MO) dye solution. Three samples were uncalcined while the rest were calcined for 4 hours at 400°C. These photocatalysts were characterized by its phase and band gap energy using X-ray diffraction analysis and UV-Visible spectrometer, respectively. The photocatalytic activities of these photocatalysts were tested by degrading MO dye in aqueous medium under visible light irradiation. For uncalcined photocatalyst, 4A/T1 exhibits highest photodegradation efficiency (100%) compared to 2A/T1 and 1A/T1. The effect of MO concentration and mass of photocatalyst used on the photocatalytic performance of 4A/T1 was evaluated for optimization of MO degradation. 4A/T1 photocatalyst exhibit 100% degradation for 0.3g used and also for 10ppm MO. The synthesized photocatalysts are analyzed using X-Ray Diffraction (XRD) to study the crystallinity where Ag₃PO₄ is cubic and TiO₂ is tetragonal both for rutile and anatase. The band gap energy for 1A/T1, 2A/T1 and 4A/T2 are 2.17eV, 2.25eV and 2.29eV, respectively. Meanwhile, for calcined photocatalysts, the activities of Ag₃PO₄/TiO₂ towards MO degradation were found to be greatly dependent on the mass ratio of Ag₃PO₄/TiO₂ where 4A/T-1 showed the highest photocatalytic degradation which showed an enhancement of approximately 22.68% in catalytic activity when using 4A/T-1 compared to 1A/T-1 and 2A/T-1. This improvement in photocatalytic activity may be because of induced changes of the Ag₃PO₄/TiO₂ physical properties during calcination process and the highest Ag₃PO₄ content in the catalyst. Moreover, the photocatalytic performance of 4A/T-1 was studied particularly by carried out the effect of MO concentration and photocatalyst loading on the photocatalytic performance of 4A/T-1 was studied in order to optimize the photocatalytic degradation of MO. 4A/T-1 exhibited the highest photodegradation efficiency at optimum conditions of 0.5g photocatalyst and 10ppm of MO. The calculated band gap for 2A/T-1, 1A/T-1 and 4A/T-1 are 1.70eV, 1.98eV and 2.15eV respectively while the XRD results shown that Ag₃PO₄ was cubic and TiO₂ was tetragonal both for rutile and anatase.

Keywords: Methyl orange, Ag₃PO₄/TiO₂, visible light, photodegradation, photocatalyst.
Penentuan kepekatan logam berat dalam sedimen di muara Sungai Kemaman, Terengganu

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Abstrak
Penentuan kepekatan dan taburan beberapa logam berat seperti Al, Fe, Cr, Cu, Mn, Ni, Pb dan Zn di dalam enapan dasar telah dijalankan di muara Sungai Kemaman (Laut China Selatan) menggunakan kaedah pencernaan basah. Selepas pencernaan, logam berat di dalam larutan ekstrak dianalisa dengan spektrofotometer serapan atom (AAS) dan spektrofotometer serapan atom relau grafit (GFAAS). Umumnya, keputusan menunjukkan bahawa kepekatan kebanyakan logam berat di kawasan kajian adalah lebih rendah atau setara jika dibandingkan dengan kawasan lain. Bagi penyampelan pertama, purata kepekatan logam adalah 4.44 ± 2.55% bagi Al, 2.36 ± 1.01% bagi Fe, 44.29 ± 23.82 μg.g⁻¹ bagi Cr, 12.13 ± 6.03 μg.g⁻¹ bagi Cu, 135 ± 83.08 μg.g⁻¹ bagi Mn, 27.08 ± 13.47 μg.g⁻¹ bagi Ni, 25.11 ± 10.34 μg.g⁻¹ bagi Pb dan 69.85 ± 29.13 μg.g⁻¹ bagi Zn. Bagi penyampelan kedua pula purata kepekatan adalah 10.18 ± 6.64% bagi Al, 1.78 ± 0.75% bagi Fe, 27.00 ± 18.27 μg.g⁻¹ bagi Cr, 10.15 ± 6.75 μg.g⁻¹ bagi Cu, 120.71± 72.69 μg.g⁻¹ bagi Mn, 11.88 ± 5.18 μg.g⁻¹ bagi Ni, 20.49 ± 9.20 μg.g⁻¹ bagi Pb dan 34.17 ± 29.97 μg.g⁻¹ bagi Zn. Analisis statistik anova menunjukkan terdapatnya perbezaan bererti (p<0.05) di antara penyampelan pertama dan kedua. Penentuan faktor pengkayaan menunjukkan kepekatan Pb di kawasan kajian dipengaruhi oleh sumber-sumber antropogenik. Beberapa aktiviti di sekitar Sungai Kemaman telah dikenalpasti sebagai penyumbang sumber antropogenik iaitu aktiviti-aktiviti perikanan, perindustrian, aktiviti-aktiviti di dermaga dan daripada kawasan perbandaran.

Katakunci: Logam berat, faktor pengkayaan, sedimen, Sungai Kemaman (Laut China Selatan).
REMOVAL OF CRUDE OIL FROM AQUEOUS SOLUTION BY BIVALVE SHELL AS LOW-COST ADSORBENT

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Abstract

The removal of crude oil from aqueous solution by bivalve shell (BS) was studied via batch adsorption under varying parameter such as pH (3-11), adsorbent dosage (10-50 g/L), initial oil concentration (10000-100000 ppm) and temperature (303-323 K). The crude oil was completely removed at pH 9 using 40 g/L of BS dosage under 10000 ppm of crude oil concentration at 303K within 30 min of time contact. The significant uptake of crude oil from aqueous solution onto the BS was proven by FT-IR and FE-SEM analyses. The isotherm studies revealed that the experimental data agree with Langmuir Isotherm type 1 model with $R^2 = 0.9999$. The pseudo-first-order kinetics model fitted well with the experimental results. The negative value of enthalpy ($\Delta H^\circ$) indicate that the nature of the adsorption process is exothermic, the negative value of entropy ($\Delta S^\circ$) show that the decrease in the randomness at sorbate-solution interface during the adsorption process. A negative value of Gibbs free energy ($\Delta G^\circ$) means that the reaction is favourable; increased in the value of $\Delta G^\circ$ with rise in temperature show that the adsorption is more favourable at lower temperature.

Keywords: Bivalve shell, crude oil, batch adsorption, low-cost
WATER QUALITY AND ANTROPHOGENIC POLLUTANTS DETERMINATION IN SUNGAI BERTAM, CAMERON HIGHLANDS, PAHANG.

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Abstract

Water quality in Sungai Bertam declining because of clearing land for settlement, logging, agriculture and development. Therefore, a study to determine the current status of water quality and both nutrient and heavy metal content in the water and sediment was conducted on November 2017 in Cameron Highlands. Six sampling stations along the river were selected by considering the anthropogenic activities nearby to represent the entire river. In-situ parameters determined in this study were pH and dissolve oxygen (DO). Laboratory analysis have determined the parameters of biochemical oxygen demand (BOD), chemical oxygen demand (COD), total suspended solid (TSS) and ammonia nitrogen (NH3-N) according to standard methods. The results showed that Bertam River is classified as class II based on WQI. Those determined nutrients content comprised flouride (F-), chloride (Cl-), nitrate (NO3-), sulphate (SO42-), phosphate (PO3-), and bromide (Br-). The concentration of (F-) in water was in the range of 0.035 to 0.058 mg/L, 1.488 to 4.157 mg/L for (Cl-), 0.056 to 1.273 mg/L for (Br-), 3.425 to 9.688 mg/L for (NO3-), 2.902 to 7.444 mg/L for (SO42-), and 0.112 to 0.242 mg/L for (PO3-). Meanwhile, the concentration of (F-) in sediment was in the range of 0.34 to 0.66 μg/g, 11.29 to 16.37 μg/g for (Cl-), 0.12 to 0.25 μg/g for (Br-), 10.04 to 17.07 μg/g for (NO3-), 26.33 to 51.87 μg/g for (SO42-), and 1.03 to 2.85 μg/g for (PO3-). The composition of heavy metals in the organic oxidation fraction (OO) and resistant fraction (RR) were determined with inductively coupled plasma mass spectrometry (ICP-MS). Those determined heavy metals content comprised of Fe, Mn, Zn, Cu, Cd, Cr and Pb. The results showed that the total amount of heavy metals extracted from sediment sequentially was in the order Fe>Mn>Zn>Cr>Pb>Cu>Cd. Amount of heavy metals extracted sequentially in OO was in the order of Mn>Fe>Zn>Pb>Cr>Cu>Cd and in the order of Fe>Mn>Cr>Pb>Cu>Mn>Cd for RR. Meanwhile, the composition of heavy metals in water samples were in the order Fe>Zn>Cu>Cr>Pb>Mn>Cd. Sandy and sandy loam were the dominant texture of sediment in the sampling areas. This study overall contributes to identify the sources and level of pollution on Bertam River in Cameron Highlands.

Keywords: nutrients, heavy metals, water quality index, sandy, sandy loam
ULTRASOUND ASSISTED DISPERSIVE LIQUID–LIQUID MICRO EXTRACTION (USADLLME) FOR THE DETERMINATION OF BIOGENIC AMINES IN FOODS

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Abstract
Ultrasound assisted dispersive liquid-liquid microextraction (USADLLME) was developed for the extraction of seven biogenic amines (BAs) in foods. BAs were derivatized with dansyl chloride and 1-butyl-3-methylimidazolium hexafluorophosphate (extraction solvent) was dispersed into the aqueous sample solution as fine droplets by ultrasonication. Acetonitrile was used as disperser solvent respectively. The factors affecting the extraction efficiency, such as the dansylation condition, volume of ionic liquid, ultrasonication time and extraction temperature have been optimized. Analyte was analyzed using C18 monolithic column of high performance liquid chromatography coupled with diode array detector after extraction and centrifugation. The proposed method was linear over 0.1-100 mg L\(^{-1}\) with a correlation coefficient of 0.991-0.999. The limits of detection and quantification based on signal to noise ratio ranges from 0.06-0.3 mg L\(^{-1}\) and 0.19-0.99 mg L\(^{-1}\) respectively. The relative standard deviations (RSD) for intra-day and inter-day assay were found to be less than 3.6%. Relative recoveries ranging from 78.3 to 114% were established using mango juice, tempe and sardine samples with %RSD less than 5.5%. The developed method was found to be sensitive, rapid, green, and cost-effective for the determination of biogenic amines in wide range of sample matrices.

Keywords: Biogenic amines, foods, dansylation, USADLLME, HPLC
CANDIDA RUGOSA LIPASE IMMobilized ON DIETHYlaminoethyl-CELLULOSE (DEAE) FOR ESTERIFICATION OF OLEIC ACID AND BIOALCOHOL

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Abstract
Lipase from Candida rugosa was immobilized onto diethylaminoethyl-cellulose (DEAE) through physical adsorption method with high percentage of protein absorption obtained at 83.4%. The enzymatic synthesis of butyl oleate, was tested by reacting oleic acid and butanol using immobilized lipases. The reaction was carried out in hexane as reaction medium. The effect of reaction temperature, thermostability of the immobilization lipase, stability in organic solvent for 10 days, leaching with hexane and storage studies under various conditions of immobilization lipase were investigated. The optimum esterification was found to be more than 90%. Only a slight of lipase leached out after being washed by 20 ml of hexane. This showed that lipases were strongly attached to the support via physical adsorption method, and it could be used as industrial biocatalyst.

Keywords: lipase, immobilized, esterification, oleyl ester
TERNARY PHASE BEHAVIOUR OF WATER/GLYCOLIPID/OIL SYSTEM

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Abstract

The question on how glycolipids would behave in surfactant-oil-water (SOW) systems was still understudied, especially in regards to its effect with medium-chain triglycerides (MCT) and long-chain triglycerides (LCT), which predominated in coconut oil (CO) and olive oil (OO) respectively. Several Cremophor® EL (CrEL)-stabilized systems were compared in the absence and the presence of dodecyl glucopyranoside (DDG). Preliminary results showed that DDG played consequential role in the size and stability of nanoemulsion and microemulsion produced. The systems with coconut oil were also found to be more stable than the systems with olive oil. In order to elucidate the mechanism behind these observations, the components were individually characterized: fatty acid profile of oils using gas-chromatography-mass-spectrometry (GCMS); thermal analysis of glycolipid using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC); and solubility of ketoprofen using ultra-visible (UV-Vis) spectrometry. Three ternary phase diagrams of CrEL/CO/W, CrEL:DDG/CO/W, and drug-loaded CrEL:DDG/CO/W systems were constructed. Selected formulations were prepared by phase inversion composition, which then were characterized to gain information on size and polydispersity using dynamic light scattering (DLS), morphological features using transmission electron microscopy (TEM); storage stability and its potential towards pharmaceutical applications.

Keywords: Glycolipid, Nonionic surfactant, Coconut oil, Olive oil, Nanoemulsion, Microemulsion, Cremophor EL, Phase inversion composition
CARBOXYMETHYL SAGO STARCH/POLY(ETHYLENE OXIDE) HYDROGEL NANOFIBERS AND ITS CONTROLLED RELEASE BEHAVIOR

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Abstract

Starch is a natural polymer that readily available, low cost and biodegradable [1]. Sago starch is product of sago palm tree or scientifically known as metroxylon sagu. It is come from the spongy center or pith of sago palm tree. The major producers of sago in Southeast Asia are Malaysia and Indonesia. In Malaysia, sago was largely produced in Sarawak, and was exported to many countries such as Japan, Taiwan, Singapore and other countries [2]. In this study, sago starch was chemically functionalized to produce carboxymethyl sago starch to improve processability of sago starch in water. CMSS and poly(ethylene oxide) (PEO) composite hydrogels nanaofibers were prepared by using electrospinning technique and non-toxic and biodegradable cross-linker, citric acid. The characterization of the CMSS/PEO hydrogel was done by using scanning electron microscopy (SEM), Fourier transform infrared (FT-IR), and thermogravimetric analysis (TGA). The optimum condition swelling capacity of CMSS/PEO hydrogel was found to be at 5% concentration of citric acid, 70°C curing temperature and 3 hours curing duration. The CMSS/PEO hydrogel shows the highest swelling percentage in pH 8 followed by pH 7.4, pH 4 and pH 1.2. The loading and release of methylene blue (MB) in CMSS/PEO hydrogel at different pH was investigated by using UV-vis spectroscopy at ʎmax 664 nm. The release profile of MB exhibits pH dependent, where the highest MB release was in pH 1.2 and slowest release in pH 4. The results show the potential of CMSS/PEO composite to be used drug delivery application.

References


Keywords: Sago starch, drug delivery, controlled release, hydrogel, citric acid, nanofibers, electrospinning
BIPHASIC NANOHYBRID OF LAYERED DOUBLE HYDROXIDE INTERCALATED WITH 4-CHLOROPHENOXYACETATE AND 2,4,5-TRICHLOROPHENOXYACETATE HERBICIDES

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Abstract

This template Simultaneous intercalation of 4-chlorophenoxyacetate (4CPA) and 2,4,5-trichlorophenoxyacetate (TCPA) into zinc-aluminium-layered double hydroxide (ZAL) was successfully accomplished by anion-exchange method for the formation of a biphasic organic-inorganic nanohybrid. The 4CPA anion was found to be more preferentially intercalated into the inorganic layered double hydroxide (LDH) interlayer galleries than the TCPA anion with percentage loadings of 35.5 and 21.0 % (w/w), respectively. Further studies using this biphasic nanohybrid showed that controlled release of both the 4CPA and TCPA anions occurred at the same time but the kinetics of their release was different, with higher percentage release of 4CPA compared to TCPA anion. However simultaneous release of both anions are fitted well to pseudo-second order kinetics with optimum amount of 70% and 46% for 4CPA and TCPA, respectively.

Keywords: Simultaneous intercalation, simultaneous controlled release, anion-exchange, chlorophenoxyacetates, organic-inorganic nanohybrid.
ELECTROSPUN POLY (VINYL ALCOHOL) NANOFIBERS DOPED WITH MESOPOROUS SILICA NANOPARTICLES FOR CONTROLLED RELEASE OF METHYLENE BLUE

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Abstract

Nanofiber materials have often been reported as transporters for clinical drugs but face the limitation of burst releasing the drugs. Therefore, mesoporous silica nanoparticles (MSNs) have raised much interest to be used in drug delivery system because of their large pore volume and high surface area. In this study, nanofiber drug delivery system based on poly(vinyl alcohol) (PVA) loaded with novel ionic liquid templated MSNs were successfully prepared by the electrospinning method. The composite fiber mat was designed for the prolonged and sustained release of drug. MSNs were synthesized by co-condensation method with average particles size of ~70 nm and then loaded with hydrophilic model drug methylene blue (MB). The effect incorporation of MB-loaded MSNs into the polymer solution to form fibrous structure was investigated. Uniform PVA/MB nanofiber mat was also produced as controls. The morphologies of nanoparticles and composite nanofiber were characterized by field emission scanning electron microscope (FESEM). After electrospinning, electron microscope revealed that MSNs were randomly distributed in the regions of nanofiber. Drug release profiles of MB from MSNs and electrospun mats were evaluated. The results indicated that adsorption of model drug MB into MSNs and incorporation them into nanofiber are effective way of minimizing burst release of drug. Sustained delivery was achieved with controllable release during the 120h releasing period.

Keywords: poly(vinyl alcohol), mesoporous silica nanoparticles, nanofiber, electrospinning, drug delivery
SYNTHESIS OF MESOPOROUS SILICA NANOPARTICLE FROM BAGASSE ASH FOR METHYLENE BLUE DYE REMOVAL

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Abstract
In this study, mesoporous silica nanoparticles (MSNs) was synthesis from bagasse ash using formaldehyde as growth suppressant. The removal of methylene blue (MB) by bare silica and MSNs were investigated by batch adsorption after varying pH (3-11), adsorbent dosage (0.1-0.5 g/L), initial dye concentration (10-100 ppm) and temperature (303-343 K). The modification of MSNs using formaldehyde (MSNF) significantly increased the surface area of the adsorbent, thus creating MSNF with much better adsorption capacity for MB removal. The adsorption kinetic and equilibrium isotherm of the MSNF were studied using pseudo-first-order and pseudo-second-order kinetic equation as well as Langmuir isotherm (Type I-IV), Freundlich and Temkin models. The experimental data obtained with MSNF fits best to the Langmuir Type I model and exhibits a maximum adsorption capacity ($q_{\text{max}}$) of 52.9 mg/g; data followed the pseudo-second-order model. The thermodynamic study showed that the adsorption is endothermic, random and spontaneous at high temperature. The results indicate that MSNF adsorbs MB efficiently and could be utilized as a low-cost alternative adsorbent for the removal of dye effluent in wastewater treatment.

Keywords: Sugarcane bagasse ash, MSNs, methylene blue dye, adsorbent, adsorption capacity
CRYSTALLISATION VIA MELTING

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Abstract

Self-assembly is a dynamical process of units coming together without external intervention into ordered organisation. A common example of self-assembly is crystallisation. We investigate crystallisation using a well-known colloid-polymer mixture. The addition of nonadsorbing polymer to a colloidal suspension with hard sphere interactions introduce effective attraction between the colloids. By cooling the system close to θ-temperature, the polymer radius of gyration decreases and leads to a reduced effective attraction between the colloids. In turn, the deeply quenched system is “warmed-up” bringing it to a more shallow quenched region. Structural rearrangement is expected when the effective attraction is weaker due to bond reversibility, and an improved self-assembly takes place when the system is brought back to room temperature. The temperature change investigations were carried out by using a temperature stage fitted to a confocal microscope. After carrying out a series of cooling and heating, we have identified a temperature close to θ-temperature where more improved crystallisation was observed.

Keywords: self-assembly, colloid-polymer mixture, depletive attraction, crystallisation
SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL STUDY OF ACRIDINE IMIDAZOLIUM SALT.

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Abstract
A series of acridine- imidazolium salts were synthesized based on 9-chloroacridine and N-substituted imidazole. The compounds were characterized by FTIR, UV-Vis, ¹H and ¹³C NMR, and evaluated for in vitro cytotoxicity against non-tumorigenic cell line MCF-10a and breast cancer cell line MCF-7 by MTT assay. The results indicated the non-toxic behaviour for all compounds. While simple alkyl substituted imidazole only showed low bioactive against MCF-7, the benzyl substituted derivative furnished promising therapeutic potential with an IC₅₀ of 5 µg/mL.

Keywords: 9-chloroacridine, breast cancer (MCF-7), MTT and imidazolium salts
EVALUATING THE INHIBITION OF THE ENZYME α-GLUCOSIDASE BY IMINOSUGAR INTERMEDIATES OF DEOXYNOJIRIMycin

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Abstract

α-Glycosidase enzymes hydrolyse α-glycosidic linkages and are involved in bodily processes such as the catabolism of glycans, intestinal digestion, and the degradation of glycoproteins. Various types of diseases which are caused by the failure of this enzyme to function properly can be treated through enzyme inhibition. Although the iminosugar, deoxynojirimisin (D-DNJ) is an excellent micromolar glycosidase inhibitor, α-glucosidase inhibition activity of D-DNJ synthesis intermediates have yet to be reported. Therefore, the scalable synthesis of the D-DNJ intermediates 1,2-O-isopropylidene-α-D-glucurono-3,6-lactone 2, 1,2-O-isopropylidene-β-L-idurono-3,6-lactone 3 and 5-azido-5-deoxy-1,2-O-isopropylidene-α-D-glucurono-3,6-lactone 4 was carried out using D-glucuronolactone (1) as the starting material based on Best et al. 2010, and subsequently, evaluated for anti-α-glucosidase activity. The synthesis was started with D-glucuronolactone 1 using acetone and concentrated sulfuric acid to obtain the acetonide 2. Purification of 2 by recrystallisation using hot toluene afforded a yield of 55%. Triflic anhydride added to intermediate 2 in dichloromethane and pyridine formed the major product 2a. Crude trflate derivative containing 2a was obtained from the organic fraction after washing with aqueous HCl. Addition of sodium trifluoroacetate to crude trflate dissolved in DMF followed by extraction with EtOAc partitioned with aqueous sodium bicarbonate formed the inverted L-idoo-lactone 3. Purification of 3 by recrystallisation using hot toluene followed by vacuum liquid chromatography (hexane:ethyl acetate = 2:1) afforded a yield of 46%. Triflic anhydride was added to the L-idoo-lactone 3 in dichloromethane and pyridine to obtain the corresponding trflate 3a and was subsequently reacted with sodium azide in DMF to form the D-gluco-azide 4. Purification of 4 was carried out by recrystallisation using hot toluene, vacuum liquid chromatography (hexane:ethyl acetate = 3:1) and preparative TLC (hexane:ethyl acetate = 2:1) to obtain 4 in 1% yield. Purity analysis using HPLC-ESLD gave a single peak at the retention times of 3.72, 3.59 and 3.52 minutes for 2, 3 and 4, respectively. All products were identified by mass spectrometry (DI-ESI-MS) and NMR spectroscopy (via comparison of 1D 1H and 13C with previously reported values). The inhibitory activity of 1, 2, 3 and 4 towards α-glucosidase from Saccharomyces cerevisiae using pNP-glucoside substrate was tested. Compound 3 showed 29.5% inhibition followed by 2 (21.4%), 1 (15.8%) and 4 (15.7%) compared to the positive control, quercetin (72.7%).

Keywords: D-Glucuronolactone 1, 1,2-O-Isopropylidene-α-D-glucurono-3,6-lactone 2, 1,2-O-Isopropylidene-β-L-idurono-3,6-lactone 3, 5-Azido-5-deoxy-1,2-O-isopropylidene-α-D-glucurono-3,6-lactone 4, Iminosugar, Deoxynojirimycin, α-Glucosidase enzyme inhibition
SYNTHESIS OF AMINOANTHRAQUINONE DERIVATIVES

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Abstract

Amino derivatives of anthraquinone have been known to have a wide range of reactivities as anticancer agents. Structure modification of anthraquinone such as via reduction, alkylation or acylation also play important roles to increase their bioactivities. Twelve aminoanthraquinones including eight new aminoanthraquinones were synthesized via two different sequence of synthetic pathways that consisted of two step-step reactions. In the first synthetic pathway, quinizarin was subjected to reduction, alkylation and acylation separately before treated with amination in the presence of iodobenzene diacetate to produce seven aminoanthraquinones. In the second synthetic pathway, quinizarin was subjected to amination and then further reduction, alkylation and acylation separately to produce another five aminoanthraquinone derivatives. All aminoanthraquinone derivatives were characterized using melting point measurement, common spectroscopic techniques and comparison with the literature data. 2-(Butylamino)-9,10-dioxo-9,10-dihydroanthracene-1-yl acetate exhibited strong antimicrobial toward Methicillin-resistant Staphylococcus aureus (MRSA), Psedomonas aeruginosa, Candida albicans and Escherichia coli with MIC values of 0.1-0.5 mg/mL. This compound together with other five aminoanthraquinones named 2-(butylamino)anthracene-1,4-dione, 2-(butylamino)-4-methoxyanthracene-9,10-dione, 1-(butylamino)-4-methoxyanthracene-9,10-dione, 2-(butylamino)-1-hydroxy-4-methoxyanthracene-9,10-dione and 2-(butylamino)-1,4-dimethoxyanthracene-9,10-dione showed strong cytotoxic activities against human estrogen receptor positive breast cancer (MCF-7) cell line with IC50 1.1-11.0 µg/mL and human liver hepatocellular carcinoma (Hep-G2) cell line with IC50 1.1-14.0 µg/mL.

Keywords: aminoanthraquinone, amination, cytotoxic, antimicrobial
SYNTHESIS, MOLECULAR DOCKING OF 5-ACETYL-4-METHYLTHIAZOLE DERIVATIVES AS ANTIMICROBIAL AGENTS

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Abstract

The emergence of antibiotic resistance against bacterial strains has attracted great interest in the discovery and development of new antibacterial agents. Thiazole derivatives had been widely used in the field of biological as well as pharmacological and their efficiency as pharmaceutical drugs are well established. In this study, a series of thiazole derivatives were synthesized by incorporating selected primary amines in one-pot synthesis manner. The compounds were structurally characterized via several spectroscopy analyses such as FTIR, UV-vis and $^1$H NMR. Their antibacterial properties were screened against selected Gram-positive such as Bacillus cereus, Staphylococcus epidermidis as well as Gram-negative bacteria Escherichia coli and Pseudomonas aeruginosa using disc diffusion technique. Molecular docking studies also were performed in order to rationalize the obtained antibacterial results. On the other hand, four compounds exhibited positive result against both types of bacterial strains with 4 was proven to be most promising candidate as antibacterial agent.

Keywords: Thiazole, Amines, Antibacterial, Disc diffusion, Molecular docking
PALM OIL AS ALTERNATIVE BIOLUBRICANTS FOR IMPROVING TRIBOLOGICAL HYDRODYNAMIC

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Abstract

Metal-on-metal (MoM) hip replacements are commonly used in hip implants. However, one of the issues under debate is the interference of friction and wear. The purpose of this feasibility study is to elucidate the performance of palm lubrication between the femoral head and the acetabular cup. In the tribology of hip implants, the use of palm oil as natural lubricants for human joints has shown tremendous potential. Palm oil give significant value as alternative biolubricant due to its advantages, large production in country, and also has potential to replace petroleum based lubricants. A modified pin-on-disc as hip screening has been used to evaluate the friction and wear on an acetabular cup. The wear debris was then observed with microscopy image analysis. This study revealed that the physical and unique chemical properties in palm oil can optimize the rate of friction and wear on the metal acetabular cup and thus allow for a stable implant of MoM.

Keywords: Metal-on-metal, tribology, palm oil, friction, wear
SYNTHESIS AND STRUCTURAL CHARACTERISATION OF LANTHANIDE METAL-ORGANIC FRAMEWORKS CONTAINING DICARBOXYLIC ACID LIGANDS

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Abstract

Metal-Organic Frameworks (Ln-MOFs) are compounds that contain lanthanide metal ions or clusters coordinated to organic ligands. Ln-MOFs have been reported to have diverse architectures due to their high coordination numbers and the large ionic radii of the cations, potentially creating the possibilities of various coordination environments around the central metal ions. Ln-MOFs combine the advantages of MOFs and the intrinsic spectroscopic properties of lanthanides, such as a large Stokes shift, a long fluorescence lifetime, and a wide emission range. Their luminescent properties have been extensively studied and applied in many applications including as chemical and luminescent sensors. In this work, four lanthanide-based Metal Organic Frameworks La(III)-MOFs, Ce(III)-MOFs, Pr(III)-MOFs, and Nd(III)-MOFs were synthesised using mixed dicarboxylic acid ligands, terephthalic acid (H$_2$BDC) and 4,4'-oxybis (benzoic acid) (H$_2$OBA) with dimethyl formamide (DMF) as the solvent. These MOFs were successfully synthesised using a solvothermal method for 72 hours at 150 °C by varying the molar ratio of metal to ligand. MOFs assembled from La(III), Ce(III), Pr(III), Nd(III), H$_2$BDC and H$_2$OBA were named Ce-112 (9), La-112 (10), Pr-112 (11) and Nd-112 (12). These MOFs were characterised to obtain information on their structure using Fourier Transform Infrared Spectrophotometer (FT-IR) Analysis, Powder X-ray Diffraction (PXRD) Analysis, Thermogravimetric Analysis (TGA), Inductively Coupled Plasma - Optical Emission Spectrometry (ICP-OES) Analysis, Scanning Electron Microscopy (SEM) Analysis, and Single-crystal X-ray Diffraction (SXRD). Peaks below 10° present in the PXRD analysis indicated the formation of the large lattice unit of the frameworks. TGA analysis indicated that these compounds 9-12 had excellent thermal stability where all of them decomposed at above 700 °C. The SEM images indicated needle-shaped crystals for all four compounds. The SXRD analysis data revealed that compound 9 crystallized in a monoclinic crystal system with a space group space of P2$_1$/n while compounds 10-12 crystallized in an orthorhombic crystal system with a space group of I2/a. The luminescent properties of these MOFs are currently being investigated.

Keywords: Metal-Organic Frameworks, Lanthanide metals, Single-crystal X-ray diffraction analysis
REDUCED TiO$_2$ MODIFIED POLY (ETHER SULFONE) FILM: A NEW STRATEGY FOR PHOTOCATALYST IMMOBILIZATION

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Abstract

Reported here is a method to synthesize reduced TiO$_2$ (rTiO$_2$) by simple thermal reduction with NaBH$_4$ before immobilized into poly (ether sulfone) (PES) polymer film via immersion precipitation. The combination of reduced TiO$_2$ with PES showed better photoactivity in degrading methyl orange dye with long-term stability under visible light irradiation. The absorption of visible light by the interstitial site of reduced TiO$_2$ was enhanced due to the presence of Ti$^{3+}$ species or oxygen vacancies which acted as electron acceptors facilitating the charge carrier transfer. The addition of rTiO$_2$ could endow the film photocatalyst with excellent hydrophilicity, increasing its interfacial contact with the pollutant in water. The kinetic study of the best film photocatalyst (PES-13 wt% rTiO$_2$) was improved with complete removal in acidic condition, and full recovery of the film photocatalyst is sustained over five cycles without itself being subjected to any regeneration process.

Keywords: Reduced TiO$_2$, Poly (ether sulfone), Photocatalyst, Immobilization
GLYCEROL ETHERIFICATION FOR PRODUCTION OF FUEL ADDITIVE USING ACTIVATED BENTONITE CATALYST

(Tindak balas eterifikasi gliserol untuk penghasilan bahan tambah bahan api menggunakan pemangkin bentonit teraktif)

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Abstract
Glycerol is a by-product from transesterification of vegetable oil and alcohol for biodiesel production. In order to utilize the excess glycerol, it was converted to a value-added chemical. In this study, glycerol was reacted with tert-butyl alcohol (TBA) to produce fuel additives, which are mono-tert-butyl glycerol ethers (MTBGs), di-tert-butyl glycerol ethers (DTBGs) and tri-tert-butyl glycerol ether (TTBG) using acid-activated bentonite as the solid acid catalyst. Higher ethers (DTBGs and TTBG) are preferred due to lower affinity to water and good solubility in biofuel. Acid activated bentonite catalysts were prepared using various of mineral acid (H₂SO₄, HNO₃, HCl) and p-toluenesulfonic acid by impregnation method. The prepared catalysts were tested in glycerol etherification. The reaction was conducted at inert atmosphere for 6 hours at 70°C, mol ratio TBA to glycerol of 4:1 and 5wt% catalyst loading. The glycerol tert-butyl ether was analyzed using GC-FID with HP-INNOWAx column (30m,0.32mm,0.5um) under the following temperature program: the initial column temperature was 45°C (for 3 min), the temperature was then increased at 5°C/min to 240°C and at 240°C for 3 min isothermally. The prepared catalysts were also characterized using BET and TPD-NH₃. The result showed hydrochloric acid treated bentonite (B-HCl) is the best catalyst system as it produced relatively high amount of higher ethers (desired products), which is 94% and the best in conversion of glycerol, which is 52%. BET surface area of B-HCl increases from 80.51 m²g⁻¹ to 202.93 m²g⁻¹ due to cation interchange of H⁺ from the hydrochloric acid into the structure of bentonite smectite. The acid amount of B-HCl is 3.10 mmol H⁺/g compared to 0.80 mmol H⁺/g for untreated bentonite. The improvement of acidity and surface area leads to a better performance of B-HCl catalyst in glycerol etherification.

Keywords: solid acid bentonite catalyst, glycerol etherification, glycerol tert-butyl ethers, fuel additive
ELECTROBIOSYNTHESIS OF NiO USING RAMBUTAN LEAVES FOR PHOTODEGRADATION OF REMAZOL BRILLIANT BLUE DYE

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Abstract
A NiO catalyst was prepared through electrochemical method with rambutan (Nephelium lappaceum) leaves as an electrolyte. The effect of the preparation methods on the physical properties of the catalyst were studied via FTIR. The interaction between nickel species and bio-active compounds of rambutan leaves crude during the electrochemical was found to affect the NiO structure. An amount of 3.0 g L\(^{-1}\) of NiO was found to be optimum dosage for 10 mg L\(^{-1}\) of Remazol Brilliant Blue (RBB) dye, which resulted in 83.7% of maximum degradation after 1 h of contact time at pH 3 under fluorescent light. This study showed that the kinetics follow a pseudo-first order Langmuir–Hinshelwood model with calculated value of \(k_r\) and \(K_{LH}\) were 1.38 mg L\(^{-1}\) h\(^{-1}\) and 0.03 L mg\(^{-1}\), respectively. Measurements of the mineralization of RBB by COD and BOD\(_5\) analysis were 66.6% and 73.4%, respectively, before and after reaction. Therefore, the effectiveness of synthesis NiO by electrobiosynthesis method was established and confirmed through this studies. The synthesized NiO has a great potential as a photocatalyst in photocatalytic reaction for wastewater treatment.

Keywords: Electrobiosynthesis, NiO, RBB dye, degradation, light irradiation
PHOTODEGRADATION OF PHENOL AND METHYL ORANGE USING t-EGZrO₂ NANOPARTICLES CATALYST

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Abstract
A facile one–pot electrosynthesis method of t-EGZrO₂ nanoparticles catalyst was studied. The catalyst were characterized using Fourier transform infrared (FTIR), X-ray diffraction (XRD), transmission electron microscopy (TEM), Brunauer-Emmett-Teller (BET), and ultraviolet–visible diffuse reflectance spectroscopy (UV–vis DRS). A 10 mg L⁻¹ phenol was nearly complete degraded (97.1%) when using 0.4 g L⁻¹ of t-EGZrO₂ catalyst at pH 9 under light irradiation; while a 10 mg L⁻¹ methyl orange was completely degraded (~100%) when using 0.4 g L⁻¹ of t-EGZrO₂ catalyst at pH 3 under light irradiation. The degradation followed pseudo first–order kinetic rationalized Langmuir–Hinshelwood model. An excellent activity towards degradation of phenol and methyl orange promises the catalyst to be used in textile industry wastewater treatment and also other applications.

Keywords: Electrosynthesis, t-EGZrO₂, phenol, methyl orange, degradation, light irradiation
ELECTROSYNTHESIS OF SILVER OXIDE DEPOSITED ONTO HOTSPRING MUD WITH ENHANCED DEGRADATION OF CONGO RED

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Abstract
AgO-supported hotspring mud (AgO-HSM) catalyst was prepared by introducing AgO onto HSM support through electrochemical method. The effect of the preparation methods on the physical properties of the catalyst were studied. The interaction between silver species and HSM during the electrochemical was found to affect the AgO-HSM structure. An amount of 0.2 g L\(^{-1}\) of 11 wt% AgO-HSM was found to be the optimum dosage for 10 mg L\(^{-1}\) Congo red (CR), which resulted in 98.2% of maximum degradation after 2 h of contact time at pH 5 under fluorescent light. This study showed that the kinetics follow a pseudo-first order Langmuir–Hinshelwood model with calculated value of \(K_r\) and \(K_{LH}\) were 172.41 mg L\(^{-1}\) h\(^{-1}\) and 0.005 L mg\(^{-1}\), respectively. Measurements of the mineralization of CR by COD and BOD\(_5\) analysis were 38.4% and 61.1%, respectively, before and after reaction. Therefore, AgO-HSM could be a promising catalyst for the degradation of various dyes in wastewater.

Keywords: AgO-HSM, electrochemical, degradation, Congo red, light irradiation
AN OPTICAL SENSOR BASED ON GRAPHENE QUANTUM DOTS FOR HYDROGEN PEROXIDE DETECTION

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Abstract
Graphene quantum dots (GQDs) is a zero-dimensional material of the carbon family and considered as a small cutting fragment from graphene sheet. It has unique electronic and optical properties due to electron confinement in the finite size of graphene cluster that leads to the opening of energy gap and quantization of electronic energy. In this study, biosensing based on GQDs in combination with enzyme (horseradish peroxidase, HRP) for the determination of hydrogen peroxide (H₂O₂) has been presented. 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 H₂O₂ quenches the fluorescence intensity of GQDs system which is proportional to the concentration of H₂O₂. Parameter optimization such as response time, enzyme concentrations, pH of buffer have been investigated. For linear calibration graph, it showed a linear dependence on the H₂O₂ concentration ranging from 0.1 mM to 1.0 μM with the detection limit of 0.1 μM.

Keywords: Quantum dots, fluorescence, hydrogen peroxide, quenching, sensor

Possible mechanism of the system in the presence of H₂O₂, where H₂O₂ quenched the GQDs and leading to fluorescence intensity of the system.
Preparation and Characterization of Inclusion Complexes Between Pioglitazone and Native β-Cyclodextrin and β-Cyclodextrin Functionalized Ionic Liquid

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Abstract

The complexes of pioglitazone with β-cyclodextrin (β-CD) and β-CD functionalized ionic liquid (IL) were prepared by kneading method. Characterization of products by Fourier Transform Infrared (FTIR) spectrometer and Thermogravimetric Analysis (TGA) had proven the formation of the complexes of β-CD/pioglitazone and β-CD-IL/pioglitazone. The interactions of β-CD and β-CD-IL with pioglitazone was analyzed by ¹H Nuclear Magnetic Resonance (¹H NMR) and Ultraviolet-visible spectroscopy (UV-VIS). The interaction of pioglitazone was occurred in the hydrophobic cavity of native β-CD. However, the interaction of pioglitazone was appeared at external cavity of β-CD-IL. The formation constants of complexes at different pH were calculated using a modified Benesi-Hildebrand equation. The stoichiometry ratio was also determined to be 1:1 for the both complexes of β-CD and β-CD-IL with pioglitazone.

Keywords: β-cyclodextrin, pioglitazone, kneading, ionic liquid, hydrophobic cavity
EXCIPIENTS SELECTION AS AEROSOLIZED NANOCOLLOIDAL CARRIER SYSTEM LOADED QUERCETIN FOR PULMONARY DELIVERY OF LUNG CANCER

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Abstract

Globally, lung cancer has become the most common type of cancer cases. Quercetin (QT) has been extensively investigated for its pharmacological effects on lung cancer. However, clinical applications of QT are limited due to poor solubility and low stability in aqueous medium. Hence, this study focused on the development of nanocolloidal carrier system to enhance the solubility of QT by pulmonary administration. The screening of oils and surfactants as the excipients in the formulation were investigated as it is important to obtain stable formulation. The solubility of drug in the oil phase and emulsification test was taken as criteria for selection of oils and surfactants. Palm oil esters with ricinoleic acid (ratio 1:1 wt/wt) and Tween 80 gave the highest solubilizing effect of QT and smallest droplet size. Oil in water nanoemulsion system loaded with QT was prepared by high energy emulsification method. The formulation was stable against phase separation test and characterization of Zetasizer analysis showed the droplet size, polydispersity index and zeta potential were 131.5 ± 1.70 nm, 0.258 ± 0.004 and -51.9 ± 0.86 mV, respectively. Aerosols performance analysis demonstrated the volume median diameter was 4.644 ± 0.09 µm, Span value of 1.32 ± 0.06 and fine particle fraction (< 5 µm) of 62.87 ± 0.83 %. These results suggest that the nanocolloidal formulation containing QT could be successful carrier system for pulmonary drug delivery application.

Keywords: Palm oil ester, nanoemulsion, quercetin, aerosols pulmonary delivery
MODELLING INHIBITION BY ROSMARINIC ACID ON PORCINE PANCREATIC LIPASE

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Abstract

An enzyme known as pancreatic lipase is responsible for the gastrointestinal absorption of fats. The inhibition of the enzyme is one of the widest studied methods used to determine the potential activity of natural products to inhibit dietary fat absorption. This inhibition by using natural products can be one of an excellent strategy to prevent and treat obesity. In this study, rosmarinic acid (RA), an ester of caffeic acid and 3,4-dihydroxyphenyllactic acid which is mainly found in \textit{Boraginaceae} and \textit{Lamiaceae} plant species was studied as the potential enzyme inhibitor. The purpose of this research is to model the inhibition of RA on a porcine pancreatic lipase (PPL). The effect of inhibition was determined by comparing the structural properties and intermolecular interactions at the active site of PPL with and without the presence of RA. Firstly, molecular dynamics (MD) simulation was performed on PPL in water. Then, the final PPL conformation was docked with RA molecule. The most stable conformation from the molecular docking was chosen based on the binding energy at the active site of PPL. This was followed by aqueous MD simulation on the docked compound. The result from MD simulation of PPL and docked compound of PPL and RA was compared in term of their stability and flexibility. Root mean square deviation and root mean square fluctuation of PPL-RA has lower value compared to the free PPL. The structure of PPL-RA in water showed higher stability and lower flexibility as compared to PPL in water. The results indicated that inhibition of PPL by RA did not induce major conformational changes towards the enzyme.

Keywords: pancreatic lipase; rosmarinic acid; molecular docking; molecular dynamics, enzyme inhibition
ANTIBACTERIAL AND ANTIOXIDANT ACTIVITIES OF EXTRACTS FROM CALOPHYLLUM FERRUGINEUM AND CALOPHYLLUM INCRASSATUM

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Abstract
Calophyllum is a pan-tropical genus belongs to the Guttiferae family and locally known in Malaysia as ‘bintangor’. There has been a continual interest to further investigate the phytochemistry of Calophyllum sp since this genus is a rich source of active secondary metabolites which showed anti-HIV, cytotoxicity and antimicrobial properties. In this study, antibacterial and antioxidant activities of barks and leaves of C. ferrugineum and C. incrassatum were investigated. Cold extraction method employing dichloromethane, ethyl acetate and methanol as solvent was performed. All extracts were tested for their total phenolic content and antioxidant activities by DPPH radical scavenging and Ferric Reducing Antioxidant Power (FRAP) assays. The methanol extract from the leaves of C. ferrugineum showed the highest TPC value at 122.08 mg GAE/g and the lowest DPPH SC50 value at 11.80 µg/mL. The methanol extract from the barks of C. ferrugineum was found to have the highest FRAP value among all extracts. The antibacterial activity of all extracts was tested by minimum inhibition concentration (MIC) test against Bacillus subtilis, Staphylococcus aureus, Escheria coli and Pseudomonas aeruginosa. Only the dichloromethane extract from bark of C. ferrugineum showed moderate MIC value against Gram positive bacteria, B. subtilis and S. aureus at 125 µg/mL.

Keywords: Calophyllum, C. ferrugineum, C. incrassatum, antioxidant, antibacterial, Guttiferae
PHYTOCHEMICAL SCREENING AND ANTIOXIDANT ACTIVITY OF *PSIDIUM GUAJAVA*

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**Abstract**

*Psidium guajava* or commonly known as guava is useful to treat gastroenteritis, dysentery, stomach pain and indigestion. The leaves of *P. guajava* was screened for phytochemical and antioxidant activity. The phytochemicals were extracted by sequential maceration using *n*-hexane, chloroform and methanol, while phytochemical screening was performed using various chemical tests. Antioxidant activity was assessed by 2,2-diphenyl-1-picrylhydrazyl (DPPH) assay. Steroids and terpenoids were present in the *n*-hexane extract, while phenols and terpenoids were detected in the chloroform extract. The methanol extract was found to contain flavonoids, steroids, saponins, phenols and terpenoids. Among the tested extracts, the methanolic extract demonstrated strong DPPH radical scavenging activity with an IC\(_{50}\) value of 51.07 \(\mu\)g/mL.

**Keywords**: *Psidium guajava*, phytochemical screening, antioxidant
COMPARISON OF EXTRACTION TECHNIQUES FOR THREE CALOPHYLLUM SPECIES AND THEIR ANTIOXIDANT ACTIVITY

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Abstract

Calophyllum is a pan-tropical genus belongs to the Guttiferae family and locally known in Malaysia as ‘bintangor’. There has been a continual interest to further investigate the phytochemistry of Calophyllum sp since this genus is a rich source of active secondary metabolites which showed anti-HIV, cytotoxicity and antimicrobial properties. This study was conducted to investigate the effect of extraction techniques on the phytochemicals content and antioxidant activity of the barks, leaves and heartwood extracts of three Calophyllum species, C. incrassatum, C. rubiginosum and C. canum. Soxhlet and maceration extraction techniques by using methanol as solvent were chosen in this study. Maceration extraction technique produced higher percentage yield compared to Soxhlet extraction for leaves and barks of the three Calophyllum species. Highest percentage yield was obtained from bark extract of C. canum (21.76%) followed by bark extract of C. rubiginosum (20.24%) and leaves extract of C. rubiginosum (19.34%). Meanwhile, Soxhlet extraction technique gave higher percentage yield compared to maceration technique for heartwood extracts of all samples. The phytochemical screening test revealed all the extracts contain tannin, phenol, flavonoid, terpenes, cardiac glycoside, coumarin and phytosterol. The antioxidant activity of all extracts was tested by determining the Total Phenolic Content and DPPH radical scavenging activity. The highest Total Phenolic Content was obtained from Soxhlet extraction technique. The bark extract of C. canum displayed the highest phenolic content (461.90 mg GAE/g) followed by bark extract of C. incrassatum (394.52 mg GAE/g) and leave extract of C. incrassatum (227.89 mg GAE/g). Meanwhile, the extracts from Soxhlet extraction technique gave higher antioxidant activity compared to maceration extraction. The bark extract of C. canum showed the lowest IC₅₀ value (3.07 µg/mL) followed by bark extract of C. incrassatum (5.12 µg/mL) and leave extract of C. incrassatum (5.93 µg/mL). Pearson’s correlation test showed positive correlation between Total Phenolic Content and DPPH radical scavenging activity.

Keywords: Calophyllum, C. incrassatum, C. rubiginosum C. canum antioxidant, Guttiferae
FORMULATION AND EVALUATION OF PVA/PEG PEEL-OFF MASK CONTAINING RICH FRACTION OF CENTELLA ASIATICA AND CUCUMIS SATIVUS EXTRACT FOR ANTI-AGEING

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Abstract

Premature ageing can be seen through the formation of wrinkles and altered pigmentation earlier than it should be. Due to premature ageing and high demand for a mask that can dry faster with easy application, peel-off mask can be used to solve the problems due to its ability to remove dead skin cells and able to treat premature ageing with the addition of active ingredients. Generally, peel-off mask is a gel that is thinly applied on face and can be peeled off as thin film from the face. In this research, the benefits of peel-off mask will be enhanced with the addition of rich fraction of Centella asiatica (asiaticoside) and Cucumis sativus extract due to their antioxidant properties and collagen-synthesis inducer. Thus, the objectives of this research are to formulate peel-off mask containing rich fraction of C. asiatica and C. sativus extract and to evaluate the physicochemical properties of peel-off mask formulation. In order to identify the best formulation, screening test was conducted by using different concentration of chemical ingredients to obtain the desired properties of peel-off mask formulation. The modified formulation was made up of 20 % of polyvinyl alcohol (PVA) with some additional ingredients which are polyethylene glycol (PEG), glycerine, propylene glycol, ethanol, parabens, citric acid, rich fraction of C. asiatica and C. sativus extract. The physicochemical properties of this peel-off mask formulation were found to be affected by its storage temperature. Generally, all formulations remained colourless with slight PVA odour. Besides that, they were homogenous with no visible coarse grains. The ranges of spreadability were between 0.47 and 0.77 cm, and they have low drying time which was about five min. The pH values fell within skin pH, which were between 5.74 and 6.19, with viscosity ranges between 0.16 and 0.32 kP. Surface morphology of dried films was affected by the spreadability of the gel, where formulation with ideal spreadability produced smooth surface. Lastly, this formulation was found to be stable against mold and fungi, but not against bacteria. To conclude, peel-off mask made up of 20 % of PVA with rich fraction of C. asiatica and C. sativus extract has good physicochemical properties with good stability at low (8 ± 3 °C) and room temperature (24 ± 3 °C).

Keywords: Centella asiatica, Cucumis sativus, peel-off mask, polyvinyl alcohol, polyethylene glycol, ageing
CUMENE HYDROPEROXIDE AS A CO-SENSITIZER IN THE PREPARATION OF PREVULCANIZED NATURAL RUBBER LATEX VIA COMBINATION OF GAMMA RADIATION AND PEROXIDE VULCANIZATIONS

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Abstract

In order to improve the viscoelastic property of the Radiation Vulcanization of Natural Rubber Latex (RVNRL), an attempt has been made onto the processing method by combining both radiation and peroxide vulcanizations. In this study, hexanediol diacrylate (HDDA) played a major role as a sensitizer during the gamma radiation vulcanization and cumene hydroperoxide (CHPO) with an aromatic molecular structure acted as the co-sensitizer in the peroxide vulcanization. The vulcanized natural rubber latex film via hybrid radiation and peroxide vulcanization has tensile strength of 26.6 MPa, an increment of more than 16% compared to controlled film (22.7 MPa). Besides, the crosslink percentage of the rubber film achieved 6.5% increment from 90.7% to 96.6%. The utilization of HDDA and CHPO during the hybrid vulcanization process was analysed using Fourier transform infrared (FTIR) spectroscopy. The FTIR spectrum confirmed the absence of functional groups such as C=C, C-O and C=O from HDDA and CHPO in the hybrid RVNRL-peroxide samples. It is predicted that all the compounds are fully utilized during the radiation vulcanization.

Keywords: vulcanization, gamma radiation, peroxide, latex, RVNRL
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