

## CONTROL OF PROPERTIES OF NANOSIZED ZEOLITES

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This presentation will review the nanosized zeolites with the overall aim of reviewing the possibility to control their properties. The rational control of the template-free synthesis of nanosized zeolites produces materials with exceptional properties: (1) extremely small crystallites (10-15 nm) with narrow particle size distribution, (2) very high crystalline yields (above 80 %), (3) micropore volume (0.30 cm<sup>3</sup>g<sup>-1</sup>) comparable to their conventional counterparts (micron-sized crystals), (4) adjustable Si/Al ratios, and (5) excellent thermal stability. Another important feature is the excellent colloidal stability of nanosized zeolites that facilitates a uniform dispersion on supports for applications in catalysis, sorption, and thin-to-thick coatings. In addition, the preparation of zeolite nanocrystals free of silanol defects will be presented.<sup>2</sup> The point defect-free nanosized MFI-type zeolite was prepared by introducing atomically dispersed tungsten as a defect-preventing element. Tungsten (WVI) blocks the formation of defects in nanosized W-MFI zeolite crystals, i.e. external and internal silanol groups, by forming flexible W-O-Si bridges with W(=O) or W(=O)<sub>2</sub> species. The incorporation of tungsten and other metals in nanosized zeolites also modifies other important properties such as (i) structural features, (ii) hydrophobicity (absence of silanols), and (iii) Lewis acidity. The defect-free nanosized zeolites open new perspectives for applications in fields such as catalysis, emerging field of biomass upgrading to fuels and chemicals, separation and adsorption. Finally examples on the advanced applications of nanosized zeolites with tunable size- and shape-dependent chemical and physical properties will be presented.

**Keywords:** Nanozeolites, Synthesis, Properties, Advanced characterizations

## FINDING VALUE IN FOOD WASTE – BIOMASS RESOURCE UTILISATION OPPORTUNITIES

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Globally, one third of food produced for human consumption is categorised as lost or waste, which equates to 1.3 billion tonnes per annum. Food is considered lost when it is not transferred from the producer to market due to pest infestations or issues during harvesting and food is considered waste when it is removed from the supply chain due to not meeting quality or size specifications based on appearance. Fruits and vegetables have the highest loss and wastage rates at 40-50% of that grown, followed by cereals and fish at approximately 30-35% each and then dairy and meat at 20%. With the loss or waste of food there is also the inefficient use of inputs including water, energy and labour and the production of greenhouse gas emissions. In Australia, high quality produce is grown and sold as a result of low pollution levels and high food safety standards. It is estimated that 2.2 million tonnes is wasted by the commercial and industrial sector and 3.1 million tonnes is disposed of by consumers. Up to 25% of all vegetables produced don't leave the farm due to insect damage, inefficient harvesting or not meeting produce specifications based on size or quality standards. The Australian government has committed to a National Food Waste Strategy, the aim of which is to halve food waste by 2030. At Monash University, to support local growers, processors and manufacturing industries to reuse or find value in their food waste, we have investigated a number of fruit and vegetable crops, the whey waste resulting from cheese processing, waste coffee grounds, anaerobic digestate residues from food feedstocks and fats and oils collected from restaurants and fast food outlets. In collaboration with the Indian Institute of Technology Bombay, India and numerous local industries projects have been undertaken on extracting valuable compounds from discarded peels, cores and seeds from various discarded streams from fruit production and processing. Our approach in finding value in the food waste is to conduct an elemental analysis, quantify the protein component and extract and quantify the lipids, hemicellulose, cellulose, lignin and dietary fibre using standard extraction techniques. Depending on the substrate, investigation continues into the more highly valued compounds such as pectin, polyphenols, oils and other extractives from the peels and seeds. In all cases, Green Chemistry principles are applied to develop innovative extraction methods. For some waste streams that are rich in plant essential nutrients, application of the waste back into the agricultural systems have been investigated. This component of the research included greenhouse pot trials and field trials. The effect of application on crop growth and yield, soil health including the soil microbial community and greenhouse gas emissions can all be assessed. There are challenges to food waste valorisation including the costs involved in the centralisation of processing waste from remote regions, translation of extraction processes from the laboratory to a larger scale and the need for capital investment in infrastructure. Aside from making better use of our food resources, there are many positive aspects to food waste valorisation including increased employment, more efficient use of crop inputs including water and fertiliser and decreased greenhouse gas emissions.

**Keywords:** Elemental analysis, Green Chemistry, Food waste valorisation

## POTENTIAL VALORIZATION OF TRACE ELEMENT-CONTAMINATED BIOMASS FOR BIOFUELS PRODUCTION

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Trace elements (TEs), also known as heavy metals and metalloids, are among the most important contaminants of industrial wastelands. Phytoremediation appears as one of the most cost effective, sustainable and easiest way to implement the detoxification of contaminated soils. However potential valorization of this TEs enriched biomass is a real challenge to avoid the TEs re-dissemination in our ecosystem. Short rotation coppices of TEs (Zn, Mn) contaminated willow (*Salix viminalis*) and TEs (Zn, Cd, Mn) contaminated hyperaccumulator herbaceous plant (*Noccaea caerulea*) were used to address the potential valorization of phytomanaged biomass for biofuels production. Steam explosion (SE) pre-treatment of acid pre-soaked samples was first used to improve subsequent enzymatic hydrolysis of cellulosic material. Indeed, SE is a thermo-mechanico-chemical pretreatment which allows the breakdown of lignocellulosic structural components by the action of heating, shearing forces resulting in the expansion of the moisture and formation of organic acids during the process. Concerning *Salix viminalis*, SE performed at 220°C with 8 min. as a residence time allowed an extraction of up to 80% of Mn and Zn in the water effluent. Cellulase from *Trichoderma Resei* was then used to hydrolyze the remaining cellulosic pulp. The best hydrolysis yield of cellulose to glucose conversion reached 90 % and was observed after a SE pre-treatment with a middle set of temperature and residence time (180°C-8min) demonstrating that enzymatic hydrolysis is not affected by TEs presence. Finally, production of ethanol was achieved by the fermentation of glucose hydrolyzates using yeast from *Saccharomyces cerevisiae*; high-fermentation yields (80%) were obtained despite the TEs presence. Concerning *Noccaea caerulea*, SE performed at 220°C with 8 min. as a residence time allowed an extraction of up to 50% of Cd, 80% of Zn and 99% of Mn in the water effluent. Cellulase (*Trichoderma Resei*) was also used to hydrolyze the remaining cellulosic pulp and the highest hydrolysis yield of cellulose to glucose conversion was 60 %. Finally, production of acetone, butanol and ethanol was tested, in a preliminary approach, with the fermentation of hydrolyzates using *Clostridium acetobutylicum* bacteria; lower fermentation yields (compared to *Salix viminalis*) were observed, but fermentation in the presence of TEs is nevertheless possible. Both studies demonstrate the potential valorization of woody and herbaceous phytomanaged biomass for biofuel production.

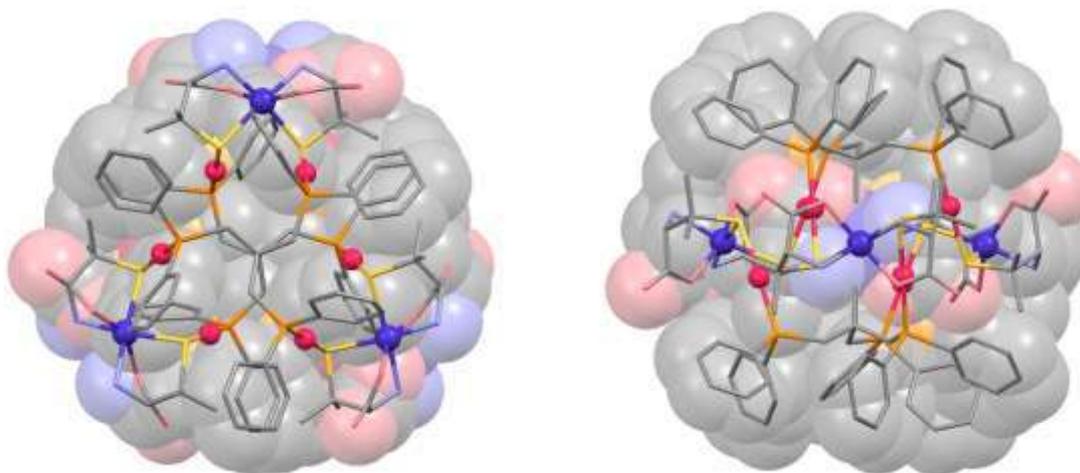
**Keywords:** Trace elements, Potential valorization.

**CREATION OF METALLOSUPRAMOLECULAR COMPOUNDS  
FROM GOLD(I) METALLOLIGANDS WITH AMINO ACIDS****T. Konno**

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Considerable attention has been focused on the design and creation of supramolecular coordination compounds in recent years. In this research field, self-assembly processes, which spontaneously give metallosupramolecular architectures from relatively simple molecular blocks *via* noncovalent interactions, are generally used to construct highly organized structures. Our interest has been directed to the metal-mediated molecular self-assembly processes based on chiral metal complexes with sulfur-containing aminocarboxylate, which serve as a metalloligand toward various metal ions. Recently, we have reported that the aurate(I) complex with two D-penicillaminates (D-pen),  $[\text{Au}(\text{D-pen-S})_2]^{3-}$ , functions as a chiral multidentate metalloligand that can bind to metal ions through coordinated thiolato and non-coordinated amine and carboxylate groups, affording a variety of chiral supramolecular ionic crystals. To expand the range of this chemistry, we thought it worthwhile to introduce a digold(I) unit having a phenylphosphine linker, in place of the  $\text{Au}^{\text{I}}$  ion in  $[\text{Au}(\text{D-pen-S})_2]^{3-}$ . Here we present the unique ionic metallosupramolecular structures developed by the novel coordination functionality of these metalloligands.



**Keywords:** Metallosupramolecular structure, Metalloligand, S-bridged structure, Ionic crystal.

## MITOCHONDRIAL TARGETING CANCER PHOTOTHERAPY BY NEAR-INFRARED LIGHT AND PI-EXTENDED PORPHYRIN/DRUG DELIVERY CARRIER CONJUGATE

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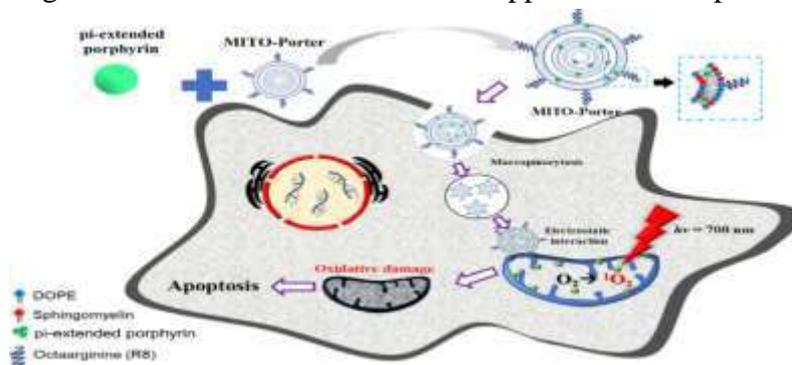
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Cancer treatment by light is a non-invasive treatment that does not put a scalpel on the body, so it can be expected to improve postoperative quality of life (QOL) compared with conventional cancer surgery. In the past decades, photodynamic therapy (PDT) has been studied for a noninvasive strategy that effectively kills cancer cells by minimizing toxicity to healthy cells. Although various photosensitizers have been developed and studied, many of them have low ability to absorb near-infrared light, which possesses high bio-permeability, and require relatively high amount of dose of the drug to kill cancer cells ( $EC_{50} > 3 \mu\text{M}$ ).<sup>1)</sup> These induce adverse reactions as a cancer therapeutic drug, resulting in a decrease in quality of life. In this study, we synthesized and developed a molecule possessing an absorption maximum about 700 nm by extending the pi-conjugation of the porphyrin skeleton. This molecule showed high singlet oxygen generating ability with light irradiation around 700 nm. This molecule was combined with a drug delivery carrier having a mitochondrial transferring ability.<sup>2)</sup> This conjugate was incorporated into HeLa cells and SAS cells, and the effect of killing cancer cells upon light irradiation was examined. As a result, it showed a killing cancer cell effect higher by one order of magnitude than conventional medical approved cancer phototherapy drug.



**Keywords:** Photodynamic therapy, mitochondria, singlet oxygen, porphyrins, fullerenes.

KN-4

**AT THE INTERSECTION OF SPORT, SCIENCE AND  
SUSTAINABILITY**

**A. Behr**

Sport has a great power to unite people and help advance a common goal. The role of sport has been recently recognized by the UN, as part of their Sustainable Development Goals, covering all 3 pillars of Sustainability: social, economic and environmental. Sports marketing has become increasingly sustainability-conscious in the recent years, with various organizations and initiatives popping up with a common goal: advance adoption of more sustainable practices, solutions and technologies in and through sports. In this talk, we'll explore a non-traditional career path of a researcher into sports marketing, and connect the dots between chemistry, sustainability and sport to help demonstrate how sports marketing, when done on the foundation of science, can help accelerate sustainable development.

**SENSITIVITY ENHANCEMENT OF ELECTROCHEMICAL  
BIOSENSORS BY USING NANOMATERIALS FOR DETECTION OF  
SOME CHEMICALS AND BIOMARKERS**

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Carbon-based nanomaterials such as carbon nanotubes, graphene, graphene oxide, carbon nanodots and those that decorated with metal or metal oxide nanoparticles have been used to modify on screen-printed carbon electrodes (SPCEs) for the development of electrochemical sensors and biosensors. Sensitivity and catalytic activity of electrochemical reaction of different analytes or redox probes were studied by cyclic voltammetry. The materials were characterized by SEM, TEM, FTIR, EDS, and EIS. The amperometric sensors was developed for determination of paraquat, and methyl dopa. The suitable electrodes were selected to further develop electrochemical biosensors for determination of some chemicals/biomarkers, i.e., glucose, immunoglobulin G, prostate specific antigen, and Hepatitis B virus surface antigen. The implementation of flow-based analysis to assist in solution manipulation was proposed. The principle of detection, analytical characteristics and performance of the developed methods will be presented.

**Keywords:** Biosensors, Nanomaterials, Catalyst, Biomarkers, Hepatitis B, Cancers

**MESOPOROUS CATALYSTS ENHANCED BIOFUEL PRODUCTION  
VIA DEOXYGENATION**

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There are raising environmental concern owing to greenhouse gas emission through combustion of fossil fuel and depletion of fossil fuel urged the need for sustainable biofuel production. The high oxygen content of fatty acid methyl ester (FAME) used as biodiesel hinders the direct application as a fuel substitute. Therefore, deoxygenation is used for production of hydrocarbon from non-edible oil. In general, the catalytic deoxygenation of plant oils involves two major routes: i) hydrodeoxygenation (HDO) and ii) decarboxylation and decarbonylation (DCO). The DCO pathway is more preferable in principle due to the cost and environmental impact. Generally, the plant oil which mainly comprised of triglycerides is a large molecules and thus encounter diffusional limitations inside the pores of the catalyst. Therefore, the pore size plays a critical role on the catalytic behaviour. To increase the performances, we have synthesized many types of mesoporous material such as mesoporous titania, mesoporous silica (HMS and MCM-41), mesoporous zeolite, etc. These mesoporous catalysts have been evaluated for deoxygenation of triolein as a model compound. Besides, the DCO was conducted under the absence of hydrogen and solvent. These mesoporous catalysts demonstrate a remarkable catalytic acidity which contributed to more than 85-100% of hydrocarbon yield. This study suggested that the effect of surface area, porosity, and acidity have play an important role in determining DCO activities. This finding shows that mesoporous type of catalysts are a potential candidate in producing sustainable hydrocarbon like biofuel from non-edible oil.

**Keywords:** Mesoporous, Catalysis, Deoxygenation, Hydrodeoxygenation, Biofuel

**DESIGN, SYNTHESIS AND BIOLOGICAL EVALUATION OF SOME  
PYRROLIDINE-TYPE COMPOUNDS**

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Heterocycles are an important class of compounds, making up more than half of all known organic compounds. Heterocycles are present in a wide variety of drugs, most vitamins, many natural products, biomolecules, and biologically active compounds. Nitrogen heterocycles such as pyrrolidine compounds represent an important class of the most studied pharmacophore groups in medicinal chemistry. The pyrrolidine ring is present in many pharmacological active synthetic or natural products. Compounds containing a pyrrolidine ring were reported to have antimycobacterial and various other pharmacological activities such as antiviral, anticonvulsant and anticancer activity. In continuation of our work on these pyrrolidines we have discovered new synthetic approaches to synthesize some unique pyrrolidine-based molecules such as pyrrolidine imino sugars, fused pyrano pyrazole, fused pyrrolidine lactam or lactone ring systems etc. The key step in these synthetic investigations involves the multi component reaction (MCR) strategy which gives a quick access to the key intermediate pyrrolidine ring system. Subsequent chemical transformations to this highly functionalised intermediate gave rise to the targeted desired molecules. This lecture will give an insight of the synthesis and biological activity of some pyrrolidine based compounds investigated in our laboratory.

**Keywords:** Heterocycles, Pyrrolidine, Synthesis, Biological activity

**SOME ADVANCES IN THE DETERMINATION OF MYCOTOXINS IN  
FOOD AND ANIMAL FEEDS**

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Mycotoxins are a group of secondary metabolites produced by certain fungi species. The threat from mycotoxins contamination is especially serious in the tropics and sub-tropics where high temperature and humidity are ideal for the production of molds. The complexity of samples and the low levels of mycotoxins poses considerable challenge in the analytical determinations. In this presentation, two major strategies to meet these challenges will be discussed, i.e., (i) Enhancements of analytical signals using post-column photochemical derivatization (for aflatoxin M1), and (ii) new sample preparation based on micro-solid phase extraction (for ochratoxin). Green features in these approaches will be highlighted. The analysis of mycotoxins in different food matrices such as peanuts, rice, milk and animal feeds will be presented.

**Keywords:** Mycotoxins, Analytical, Food, Contaminants

## IV-1

### **EMERGING CONTAMINANTS IN THE ENVIRONMENT : CHALLENGES AND RESEARCH DIRECTION**

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Water pollution is a global water quality challenge that has increased in both developed and developing countries. It impacts negatively on the sustainability of water resources, human health, economic growth and the ecosystem. It reduces total water availability, as the treatment cost is high and in some instances, polluted waters are not treatable for consumption. In recent years, there has been a growing concern over the environmental risks of emerging pollutants, due to their increasing discharge into water bodies and the environment. Even at trace levels, some are capable of potentially causing endocrine disruption in humans and aquatic wildlife and the development of bacterial pathogen resistance. A number of research and technologies have been developed to treat the hazardous pollutants from various pollution sources. However, effective tools and policies to monitor, regulate and control these pollutants in water resources have not been well established. Hence, there is an urgent need to strengthen scientific knowledge and adopt appropriate technological approaches to detect, monitor and treat these pollutants, prevent and control their disposal to water resources and the environment. Water treatment technology is facing challenges to overcome this problem to ensure water quality at a clean level. Research and development initiatives are needed to get the best technology that is cost-effective and environmentally friendly. National Hydraulic Research Institute of Malaysia (NAHRIM) as a government research body on water resources and its environment has play a prominent role in conducting research and developing various water technology innovations to ensure sustainable water resources with collaboration of relevant stakeholders. Towards achieving Sustainable Development Agenda 2030, Malaysia will conserve and implement a holistic integrated approach for water resource management to ensure adequate and safe water for all (including the environment).

**Keywords:** Polluted waters, Emerging pollutants, Endocrine disruptors, Water technology,  
Safe water

## IV-2

### CONVERSION OF PBAPP WATER TREATMENT PLANT RESIDUE INTO CLAY BRICKS

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The Environmental Quality (Scheduled Wastes) Regulations 2005 classifies water treatment plant residue (WTR) as scheduled wastes (SW204) which have to be disposed at approved designated landfill sites. It is estimated that 300 mTons per day of WTR of 30% dry solid content need to be disposed from Sg Dua WTP. The cost of compliance is very prohibitive and it is estimated to cost about RM10 million per year by transporting to the nearest municipal landfill at Pulau Burong. This gives additional burden to PBAPP as water operator which operates at lowest domestic water tariff in Malaysia. If this incurred into water tariff, the tariff hike will give a big impact to the public. Alternatives needed to handle such situation. Though this, there is a provision for wastes generator to apply for special management from DOE where the WTR can be converted into value added products such as bricks. PBAPP embarked into a project involves R & D since 2009 on the possibility and methodology of converting the WTP residue to a value-added product such as clay bricks. Drying beds method was chosen to dewater the WTR into around 50% dryness before mix with laterite earth and subsequently put into mould to form clay bricks. The clay bricks will then undergo high temperature burning process as a final process. The bricks were tested accordance with a) BS EN 771-1:2003, b) MS 76: 1972, c) Fish Acute Toxicity Test, d) Toxicity Characteristic Leaching Procedure (TCLP) and e) Total Threshold Limit Concentration (TCLP) etc. From the results, Clay bricks made from WTP residue at Sg Dua WTP do not exhibit any effect of toxicity, corrosivity, ignitability or reactivity. The strength of the bricks is able to comply to MS 76:1976 ( $\geq 5.2$  MN/m<sup>2</sup>) and EN 771-1:2003 for clay bricks, which can be used as building materials. There will be immediate saving of 30% - 40% in raw material cost. PBAPP would also benefited by eliminating the cost of disposal, which with estimated saving of RM 10 million yearly. And finally, there is high potential that the same concept can be duplicated to other WTPs in Malaysia. This would help the whole water industry to operate in more sustainable manner, comply to legal requirements and creating more avenue for revenue using the by-products generated as Green Products.

**Keywords:** Water treatment plant, Clay bricks

**CARBON NANOTUBES/GOLD NANOPARTICLES BASED  
ELECTROCHEMICAL SENSOR FOR TRACE DETERMINATION OF  
AMOXICILLIN IN BOVINE MILK**

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In this work, a novel electrochemical sensor was fabricated for determination of amoxicillin in bovine milk samples by decoration of carboxylated multi-walled carbon nanotubes (MWCNTs) with gold nanoparticles (AuNPs) using ethylenediamine (en) as a cross linker (AuNPs/en-MWCNTs). The constructed nanocomposite was homogenized in dimethylformamide and drop casted on screen printed electrode. Field emission scanning electron microscopy (FESEM), energy dispersive X-Ray (EDX), X-Ray diffraction (XRD) and cyclic voltammetry were used to characterize the synthesized nanocomposite. The results show that the synthesized nanocomposite induced a remarkable synergetic effect for the oxidation of amoxicillin. Effect of some parameters, including pH, buffer, scan rate, accumulation potential, accumulation time and amount of casted nanocomposite, on the sensitivity of fabricated sensor were optimized. Under the optimum conditions, there was two linear calibration ranges from 0.2–10  $\mu\text{M}$  and 10–30  $\mu\text{M}$  with equations of  $I_{pa} (\mu\text{A}) = 2.88C (\mu\text{M}) + 1.2017$ ;  $r = 0.9939$  and  $I_{pa} (\mu\text{A}) = 0.88C (\mu\text{M}) + 22.97$ ;  $r = 0.9973$ , respectively. The limit of detection (LOD) and limit of quantification (LOQ) were calculated as 0.015  $\mu\text{M}$  and 0.149  $\mu\text{M}$ , respectively. The fabricated electrochemical sensor was successfully applied for determination of Amoxicillin in bovine milk samples.

**Keywords:** Electrochemical sensor, MWCNTs, Ethylenediamine, AuNPs, Amoxicillin, Bovine milk

**CHEMICAL ANALYSES OF *BORRERIA STACHYDEA* [(DC.) HUTCH. & DALZIEL], *CASSIA ABSUS* LINN AND *ASPILLIA KOTSCHYI* (SCH.BIPEX, HOCHST) OLIVE PLANTS**

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In this present work, three locally used medicinal plants namely: *Borreria stachydea*, *Cassia absus* and *Aspillia kotschyi* belonging to *Rubiacea*, *Leguminosae* and *Compositae* families respectively were extracted using methanolic and petroleum ether 60-80 °C. The extracts were subjected to microwave plasma Atomic Emission Spectroscopy (MPES) to determine the following metals Se, Ag, Fe, Cu, Ni, As, Co, Mn and Al. From the result Ag, Cu, Ni, and Co is of very negligible concentrations in all the three plants. However, selenium is found to be 0.780 (mg/kg) in *Cassia absus* methanolic extract, 0.556 (mg/kg) in *Borreria stachydea* methanolic extract, and 0.530 (mg/kg) in *Aspillia kotschyi* methanolic extract. Iron on the other hand was found to be 3.712 (mg/kg), in *Aspillia kotschyi* methanolic extract, while in *Borreria stachydea* petroleum pirit extract was 3.674 (mg/kg). Arsenic was found to be 0.506 and 1.301 (mg/kg) in *Aspillia kotschyi* extracts, 2.373 (mg/kg) and 0.789 (mg/kg) in *Borreria stachydea* extracts, and 3.315 (mg/kg) in *Cassia absus* extract. The concentration of aluminium was found to be of the range of 3.050mg/kg in *Aspillia* extract and 2.99 mg/kg in *Borreria stachydea* extract. Functional group analysis of the three plants extracts were also carried out using Fourier Transform Infrared (FTIR) spectroscopy. The functional groups determined in all the three extracts showed that the plant materials contained the following functional groups; O-H alcohols, phenols, C-H for alkanes, C=O unsaturated aldehydes and ketones and C=O aliphatic amines. The results of this study suggest some merit in the popular use of the plants in herbal medicine.

**Keywords:** *Aspillia kotschyi*, *Borreria stachydea*, *Cassia absus*, MPES

**ASSESSMENT OF SOME HEAVY METALS IN SOIL SAMPLES OF METAL-POTS PRODUCERS DUMPING SITES (‘YAN-TUKWANE, GASHUA TOWN OF BADE LGA, YOBE STATE, NIGERIA)**

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The local industrialization of urban areas over the last few decades is causing high accumulation of various pollutants in the environment, mostly soil and water. This caused the build-up of heavy metals (Cu, Fe, Pb, Cr, Cd, Zn, Ni, Co) that are toxic to both plants and animals. Soil samples were taken from the dumping sites of metal pots manufacturers of Sabon Gari Area of Gashua town, Bade LGA of Yobe State. The samples were analyzed using AAS spectrophotometer. The results showed mean concentrations of heavy metals (Cu up to 4.65 mg/kg, Pb up to 7.68 mg/kg, Cr up to 3.53 mg/kg, Cd up to 0.95 mg/kg, Zn up to 16.83 mg/kg, Ni up to 0.28 mg/kg and Co up to 0.097 mg/kg). The results indicated potential environmental pollution and health threat to the communities surrounding the area.

**Keywords:** Heavy metals, Pollution, Gashua.

**COMPARISON STUDY BETWEEN HYDROCHLORIC ACID AND  
SULFURIC ACID BY POTENTIOMETRIC TITRATION FOR  
DETERMINATION OF ALKALINITY IN NATURAL RUBBER LATEX  
CONCENTRATES**

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In this paper, a comparison study between hydrochloric acid (HCl) and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) for determination of alkalinity in natural rubber latex concentrate was conducted. Malaysian Rubber Board (MRB) is currently revising the standard method of ISO 125. The objective of this project is to revise ISO standard test method and to establish new precision statement. The current version of this test method did not specify for the procedure for standardization of H<sub>2</sub>SO<sub>4</sub>. In the first stage of the study, method validation work on ISO 125 test method was performed and procedures for standardization of H<sub>2</sub>SO<sub>4</sub> and HCl were established. Highly ammoniated latex concentrate was titrated to pH 6.0 (potentiometric titration) in the presence of a non-ionic stabilizer and its alkalinity was calculated from the quantity of acid required. The data obtained from both acids were analyzed and evaluated using statistical analysis of F-test and T-test. In summary, the average differences between coefficients of variation obtained by using both acids for titration were 0.1% in which the trend showed HCl was slightly higher compared to H<sub>2</sub>SO<sub>4</sub>. Nevertheless, statistical analysis showed no significant differences between H<sub>2</sub>SO<sub>4</sub> and HCl, hence both acids can be used to achieve comparable results. An inter-laboratory testing programme (ITP) was conducted with reliable precision data obtained in terms of repeatability (r) as well as reproducibility (R). The findings from this study are essential in order to support the revision work of the ISO 125. In addition, a new ISO standard method for determination of alkalinity in natural rubber latex concentrate will be established soon; MRB has successfully added procedures for the standardization of sulfuric acid and addition of new precision data in this revision work.

**Keywords:** Alkalinity, Sulfuric acid, Hydrochloric acid, ISO standard

**DISPERSIVE MICRO-SOLID PHASE EXTRACTION (D- $\mu$ -SPE) USING C18 AS SORBENT COUPLED WITH HIGH PERFORMANCE LIQUID CHROMATOGRAPHY FOR THE DETERMINATION OF OFLOXACIN AND SPARFLOXACIN IN HUMAN PLASMA**

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A specific and sensitive analytical method based on Dispersive Micro-solid phase extraction (D- $\mu$ -SPE) combined with HPLV-UV method was developed and validated for the determination of ofloxacin and sparfloxacin in human plasma is described. C18 was used as sorbent in D- $\mu$ -SPE of the antibiotics compounds from biological fluids. The Optimum separation conditions were obtained carried out on a Hypersil GOLD ODS C18 (250 x 4.6 mm x 5 $\mu$ m particle size), using a mobile phase of mobile phase consisted of a mixture of (5 mM, pH 3) (50:50, v/v) of phosphate buffer and methanol to which phosphoric acid had been added and to sit wavelength at 245 nm with flow-rate on 1 ml/min and the retention times were 5.32 min. Important D- $\mu$ -SPE parameters, namely Type and amount of sorbent mass, type desorption solvent, extraction time, sample pH, salt addition, desorption time, amount of sorbent and sample volume were optimized. Under the optimized conditions, the calibration curves showed good linearity in the range of 0.5 – 1000  $\mu$  g/L with satisfactory limit of detection ( 0.73 and 1.81  $\mu$  g/L) and limit of quantitation ( 2.44 and 6.03 $\mu$  g/L). The proposed method also showed acceptable intra-day and inter-day precisions for ofloxacin and sparfloxacin from human plasma with RSD  $\leq$  12.5% and good relative recoveries in the range 88.5 – 90.3%. The D - $\mu$ - SPE method proved to be rapid and simple and requires a small volume of organic solvent (300  $\mu$  L); thus it is advantageous for routine drug analysis.

**Keywords:** Dispersive Micro-solid phase extraction, HPLC-UV, Ofloxacin, Sparfloxacin, Human plasma.

**CATION SELECTIVE ELECTROKINETIC INJECTION AND  
MICELLAR ELECTROKINETIC CHROMATOGRAPHY FOR  
ANALYSIS OF SULFONAMIDE ANTIBIOTICS IN MARINE  
ENVIRONMENTAL PRODUCTS**

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A rise in the issues surrounding antibiotic residues in the marine environment has triggered worldwide concern recently because of high detection rate in the marine environmental samples. In order to protect public health, the European Commission has established a maximum residue limit of 100 µg/kg for sulfonamides in fish muscle. The main goals of this work are to combine cation selective electrokinetic injection-micellar electrokinetic chromatography (CS-EKI-MEKC) and capillary electrophoresis-diode array detector (CE-DAD) to develop stacking strategy that is simple, sensitive, rapid and improvement over the existing methods and to test its ability to separate, preconcentrate and detect sulfonamide antibiotics (SAs) from marine environmental samples. Several important variables affecting the stacking methodology of SAs were optimized including type of buffer, buffer composition and concentration, buffer pH, surfactant concentration, applied voltage, temperature, injection voltage and injection time. Through the optimization study, 45 mM sodium dihydrogen phosphate with 45 mM 1-hexyl-3-imidazolium chloride and 40% acetonitrile was selected as optimum background electrolyte (BGE) at pH 4.6. The separation voltage was applied at 15 kV with temperature at 20°C, using fused silica capillary of 65 cm x 50 µm i.d. (56 cm from inlet end to the detection window). Injection mode was performed through hydrodynamic injection (HDI) at 50 mbar for 5 s. Detection wavelength was monitored at 270 nm. The developed preconcentration strategy will be validated in terms of its linearity, repeatability and recovery, and finally, will be applied to the determination of SAs in real samples.

**Keywords:** Cation selective electrokinetic injection-micellar electrokinetic chromatography, Capillary electrophoresis, Sulfonamide antibiotics, Marine, Environmental, Fish.

**DEVELOPMENT OF WATER QUALITY INDEX OF EX-MINING PONDS IN MALAYSIA**

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There is a need to develop a suitable index to assess the water quality of the abundant ex-mining ponds in Malaysia for proper utilization. The existing Malaysian river water quality index did not accommodate the toxic metal pollutants dominantly in ex-mining water, the acceptable index should therefore incorporate the metal pollutants. Heavy metals were analyzed using inductively coupled plasma mass spectrophotometer (ICPMS), and physico-chemical parameters were analyzed in-situ using the appropriate equipments. Multivariate statistical analysis was successfully applied for the parameter selection process. Two water quality indexes were developed with reference to Malaysia's water quality standard (INWQS). The heavy metal index comprises of Pb, As, and Cd as the selected metals, while the physico-chemical parameter indexes are BOD, pH, DO, and AN. Multivariate statistical analysis precisely reveals the dominance of the selected heavy metals ex-mining water, and physico-chemical parameters are of importance in monitoring the water quality. Heavy metal water quality index reveals that most ex-mining ponds in Klang Valley are classified very poor, hence cannot be used for human consumption. Due to domestic input in some of the lakes in Klang Valley, the physico-chemical water quality index reveals very poor status. In Melaka and Negeri Sembilan, ex-mining ponds and lakes are classified excellent in both indexes.

**Key words:** Water Quality Index, Chemometric, Heavy metals, Ex-mining pond, Pollution.

## A KINETIC STUDY OF THE REDUCTION OF METHYLENE BLUE BY TETRAMETHYLTHIOUREA IN AQUEOUS ACIDIC MEDIUM

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Tetramethylthiourea, (TMTU), a thiocarbamide, is known to be a chemical teratogen, carcinogen mutagen, which possesses strong antithyroid activity in rats. TMTU differs from other substituted thioureas in that it does not possess a detachable proton on any of the nitrogen groups, and thus is unable to form stable oxoacids such as sulphinic and sulphonic acids without resorting to the formation of zwitterions. The varied metabolic effects induced base on very small changes in substituent of thiourea is important in order to rationally design thiourea-containing drugs. Thus it is necessary to develop structure-toxicity relationships through studying oxidation mechanism of thioureas. As a continuation of our work on reduction of methylene blue by thiourea derivatives, we now report kinetic findings on MB<sup>+</sup>/TMTU reaction. This was carried out by investigating the stoichiometry and order of reaction, effects of [H<sup>+</sup>], ionic strength and change in dielectric constant, tests for intermediate and radicals, product analysis and Michaelis-Menten's plots. The rate, first order in both [oxidant] and [reductant], was found to be in linear relation with tetramethylthiourea [TMTU], methylene blue [MB<sup>+</sup>], and [H<sup>+</sup>]. A 1:1 mole ratio of [MB<sup>+</sup>]: [TMTU] respectively was observed for the stoichiometry of the reaction. Ionic strength dependence was positive and reaction rate was catalysed by cations. The results of spectroscopic investigation and Michaelis-Mentens analysis did not support the formation of an intermediate complex formation in the course of the reaction. The mechanistic consequences of these results gave a 10-step mechanism of reaction which was rationalized on the basis of outer-sphere mechanism, and conforms to the rate law:

$$-\frac{d[MB^+]}{dt} = (a + b [H^+])[MB^+][TMTU]$$

This finding supports earlier assertion that changing the substituent on the nitrogen of thiourea may affect the rate of reaction but not the mechanism since the major reaction is at the sulphur center. This change in rate of reaction could be attributed to the inductive effect of the methyl group in TMTU. However, increases in [H<sup>+</sup>] and  $\mu$  shows the same effect in the earlier studied substituted thiourea reactions i.e. a linear dependence within the concentrations range studied.

**Keywords:** Kinetics, Mechanism, Oxidation, Stoichiometry, Tetramethylthiourea.

**MAGNETIC POLY (CYCLODEXTRIN-IONIC LIQUID) BASED  
FERROFLUID FOR DISPERSIVE LIQUID PHASE  
MICROEXTRACTION OF POLYCYCLIC AROMATIC  
HYDROCARBONS**

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A new class of ferrofluid-liquid phase microextraction (FF-LPME) based on magnetic poly(cyclodextrin-ionic liquid) nanocomposite and 1-octanol as carrier liquid has been developed. Seven representative polycyclic aromatic hydrocarbons (PAHs) were used as model compounds in the development and evaluation of the extraction procedures in non-fatty solid and liquid food matrices coupled with gas chromatography- flame ionization detector. One variable at-a-time (OVAT) analysis and response surface methodology (RSM) based on Box-Behnken designs (BBD) were used for efficient optimization of the main variables in the extraction procedure. Under the optimum experimental conditions, the calibration curves found to be linear in the range of 0.1-150 ng mL<sup>-1</sup> with correlation determinations (R<sup>2</sup>) ranging from 0.9944 to 0.9986. The limits of detection (LODs) of the proposed method for all the studied PAHs were 0.02 to 0.07 ng mL<sup>-1</sup>. The intra and inter-day precision (RSD %) were in the range of 1.80% -7.56% and 2.97%-8.23%. The ferrofluid showed satisfactory reproducibility in 1.72% to 5.90% range and acceptable recovery values at 84%-110% were obtained for the real sample analysis. The optimized method was successfully applied to access the content safety of studied PAHs in selected food matrices.

**Keywords:** Ferrofluid, 1-octanol, Polycyclic aromatic hydrocarbons, Gas chromatography-Flame ionization detector

**NEPHROTOXIC METALS IN DIET AS INDICATORS OF CKD IN  
GASHUA, NORTHEASTERN NIGERIA**

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Chronic Kidney Disease (CKD) is a major health issue of serious concern in the Northern part of Yobe State, Nigeria. Therefore, in an attempt to investigate the possible causes of the disease, we investigated the relationship between dietary exposure to nephrotoxic metals (As, Cd and Pb) and the prevalence of CKD in the area of study. The levels of As, Cd and Pb in vegetables, the blood and urine samples from CKD patients were determined using analytical techniques. The results showed that the levels of the metals were significantly higher than the control samples as well as the maximum permissible limits based on WHO/FAO standards. The chronic daily intake (CDI), the hazard index (HI) and the incremental lifetime cancer risk (ILCR) indicate that high risk to the health of consumers. It was found that the levels of the Pb in the blood and Cd in the CKD patients were higher than in the non-CKD patients, which demonstrated a relationship between the concentration of the metals and declines in renal function. Our findings suggest that the high levels of the nephrotoxic metals in the tested samples are strong indicators for CKD in this study.

**Keywords:** Chronic kidney disease, Nephrotoxic heavy metals, Renal function

**REVERSED PHASE MONOLITHIC COLUMN FOR THE  
DEVELOPMENT OF MINIATURIZED HPLC SYSTEM**

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High-Performance Liquid Chromatography (HPLC) is an analytical technique that is widely used as a qualitative and quantitative method in the pharmaceutical industries and laboratories. This separation method allows a broader range of analyte separation, mainly based on the molecular weight of the analyte of interest. However, the selection of right column dimension and stationary phase is often important for the desired column efficiency. Particle-packed columns are commonly selected as separation media as it guarantees good separation. With a variety of pumps available in the market, the demand for smaller internal diameters and particle sizes, which yields better separation, is rapidly increasing. However, high back-pressure always the biggest drawback. To improve the permeability of the column, the monolithic column has been introduced since the 1990s as an alternative to particle packed column. The monolithic column is a porous solid that is hermetically sealed against the inner wall of the tubing. Anchoring of the monolith onto the inner wall of the tubing is a major challenge in the fabrication of monolithic column, especially for those with a larger internal diameter. We successfully fabricated a monolithic column in fused silica tubing with an internal diameter of 500 $\mu$ m, without shrinkage and cracking. This 10 cm long monolithic column was then derivatized with TMSI to create a reversed phase surface for non-polar analyte separation. It gave a plate height of 10.3 mm and resolution of 0.832. The backpressure of the monolithic column was greatly reduced by 45%. The current work provides a starting point for the fabrication of monolithic column for future specialized HPLC applications.

**Keywords:** Monolithic column, HPLC, Monolith, Backpressure.

**RECENT ADVANCES OF MOLECULARLY IMPRINTED POLYMERS  
BASED STRATEGIES FOR EXTRACTION OF MYCOTOXINS FROM  
FOOD MATRICES**

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Mycotoxins, are secondary metabolites produced by fungi, are potentially hazardous to humans, animals and crops. Ochratoxin A (OTA) and citrinin (CIT) are mycotoxins which are known to have teratogenic, nephrotoxic, hepatotoxic, genotoxic, immunotoxic and carcinogenic effects in humans. Determination of mycotoxin concentrations is challenging because the analytical methods need to have low limits of detection, generally in the mg/kg (ppm) or µg/kg (ppb) range. Conventional extraction methods, such as liquid-liquid extraction (LLE) and solid phase extraction (SPE) require large volumes of environmentally hazardous waste and the common sorbents used in SPE often suffered from low selectivity, especially for trace level of analytes. Micro-solid phase extraction (µ-SPE), based on the packing of sorbent material in a sealed porous polypropylene membrane envelope to overcome these disadvantages has emerged as potential alternative. During the extraction, the µ-SPE device is placed into a stirred sample. Tumbling of the µ-SPE device facilitated mass transfer and due to the porosity of the membrane sheet, the analyte was able to diffuse through and be extracted by the sorbent. After the extraction, desorption was carried out *via* ultrasonification with the extraction device immersed in a suitable organic solvent. To increase selectivity, molecularly imprinted polymer (MIP) has been introduced. MIPs are synthetic materials that contain artificially generated recognition sites that are able to specifically rebind a target molecule. In this presentation, I will share how MIP was used in µ-SPE for the determination of OTA. Besides that, hybrid MIP membrane which prepared by embedding the MIP particles into the polyethersulfone scaffold using phase inversion technique for the extraction of CIT will also be presented. The analytical characteristics of the methods, their merits over the conventional methods will also be highlighted.

**Keywords:** Molecularly imprinted polymer, Microextraction, Mycotoxin

## GEOGRAPHICAL VARIATION IN QUALITY ATTRIBUTES AND ANTIOXIDANT POTENTIAL OF PAKISTANI CITRUS FRUIT JUICES

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Geographical conditions affect the yield and characteristics of crops due to various factors including temperature, sun light, rain fall and soil type etc. The present study is also focused on variation in quality attributes of citrus fruit juices from geographically different districts of Punjab, Pakistan and correlation with bioactivities. Quality attributes (yield, brix, total titratable acidity, sugars) of juices of *Citrus reticulata* (Kinnow), *Citrus sinensis* (Musammi) and *Citrus paradisi* (Grape fruit) from district Faisalabad, Sargodha and Layyah were assessed by standard methods. Yields of juices from Sargodha were recorded to be: 45.7, 33.11 & 36.19% followed by Faisalabad; 41, 30.12 & 35.22% and Layyah: 35.4, 28.43 & 32.46% for *C. reticulata*, *C. sinensis* & *C. paradisi* respectively. Brix in juices from Faisalabad were recorded to be: 11.9, 9.8 & 12%, followed by Faisalabad: 11.08, 10.7 & 13.0% and Layyah: 11.6, 10.2 & 13.5% for *C. reticulata*, *C. sinensis* & *C. paradisi* respectively. Total titratable acidities in juice from Faisalabad were found to be 2.24, 1.88 & 2.59, followed by Sargodha: 2.20, 1.85 & 2.62% and Layyah: 2.14, 1.92 & 2.68% respectively. Antioxidant activities of the juices were assessed by different methods like total phenolic content (TPC), total flavonoids content (TFC) and DPPH assay. TPC in juices were: 34.63, 24.73 & 28.41mg/g; 32.74, 30.41 & 29.17mg/g and 27.58, 15.19 & 23.66mg/g for *C. reticulata*, *C. sinensis* and *C. paradisi* from Faisalabad, Sargodha and Layyah, respectively. TFC in juices were: 4.21, 1.91 & 4.91mg/g; 5.10, 3.58 & 2.94mg/g and 4.11, 6.42 & 3.62mg/g for *C. reticulata*, *C. sinensis* and *C. paradisi* from Faisalabad, Sargodha and Layyah respectively. DPPH radical scavenging activities were found to be: 41.98, 43.98 & 46.16µg/ml; 40.89, 41.32 & 43.79µg/ml and 44.12, 46.43 & 49.46µg/ml for *C. reticulata*, *C. sinensis* and *C. paradisi* from Faisalabad, Sargodha and Layyah respectively. The study suggests that geographical conditions affect the quality attributes and antioxidant activities of the citrus fruit juices

**Keywords:** *C. reticulata*, *C. sinensis*, *C. paradisi*, Yield, Sugars, Antioxidant, DPPH

**AGAROSE IMMOBILIZED HOLLOW POROUS MOLECULARLY  
IMPRINTED POLYMER FOR SEPARATION AND  
PRECONCENTRATION OF TETRACYCLINE ANTIBIOTICS IN  
FOOD PRODUCTS OF ANIMAL ORIGIN**

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In this work, a novel hollow porous molecularly imprinted polymer (HPMIP), utilizing mesoporous silica MCM-48 as sacrificial support with molecular shape, size and functionality of template complementary to tetracycline was developed and incorporated in agarose polymer matrix as high capacity adsorbent for extraction of four tetracycline antibiotics (ie; tetracycline (TC), oxytetracycline (OTC), doxycycline (DTC) and chlortetracycline (CTC) from animal based food samples. The polymers (imprinted and non-imprinted) were characterised comprehensively using various tools including Fourier transform infrared spectrophotometer (FTIR), field emission scanning electron microscope (FESEM), transmission electron microscope (TEM), x-ray diffraction (XRD), nitrogen adsorption, and thermogravimetric analysis (TGA). As compared to traditional MIP, the HPMIP is expected to have higher selectivity, sensitivity and capacity contributing to higher recognition sites spreading in the inner, outer and within hollow surfaces. The HPMIP incorporated with agarose polymer matrix will be applied as mixed matrix membrane microextraction for separation and preconcentration of tetracycline antibiotics. The microextraction procedure will be optimized and validated, prior to the application to food products of animal origin.

**Keywords:** Hollow porous molecularly imprinted polymer; Tetracycline antibiotics; MCM 48, Mixed matrix membrane microextraction; Food products of animal origin.

**GRAPHENE AND ZEOLITE AS SORBENTS IN BAR- $\mu$ -SOLID PHASE  
EXTRACTION FOR THE DETERMINATION OF SELECTED  
PHARMACEUTICAL COMPOUNDS**

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A bar-micro-solid phase extraction filled with graphene and zeolite coupled with high performance liquid chromatography was developed for determination of metformin, buformin, phenformin and propranolol. The targeted compounds quantitatively adsorbed onto the graphene due to the  $\pi$ - $\pi$  interaction and their hydrophobicity properties. The adsorption of these polar molecules by zeolite is caused by their strong electrostatic and host-guest interactions with the micropore channels of zeolite. Several parameters influencing the analytical performance for each sorbent were investigated such as conditioning solvents, pH of sample, sample volume, amount of sorbent, stirring rate, time of extraction, type and volume of desorption solvent and time of desorption. Sodium ion heptane sulfonate as ion pair was used during desorption step to attract hydrophilic drugs. Under optimum conditions, detection limit for graphene and zeolite are  $0.18 - 0.27 \mu\text{g L}^{-1}$  and  $0.67 - 93.3 \mu\text{g L}^{-1}$ , respectively. The performance of extraction analytes using mixture of these sorbent will be investigated.

**Keywords:** Graphene, Zeolite, Metformin, Buformin, Phenformin, Propranolol, Bar- $\mu$ -Solid Phase Extraction

**MAGNETIC MOLECULARLY IMPRINTED POLYMER AS ADSORBENT FOR EFFECTIVE REMOVAL OF PHENOLIC COMPOUNDS FROM ENVIRONMENTAL WATER SAMPLES**

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A novel magnetic molecularly imprinted polymer based on methacrylic acid-beta cyclodextrin (MMIP MAA- $\beta$ -CD) was synthesized via the bulk polymerization process for the selective removal of phenolic compounds from environmental water samples. MAA- $\beta$ -CD was used as the monomer, bisphenol A as the template, trimethylolpropane trimethacrylate, and benzoyl peroxide as the crosslinker and initiator respectively. The adsorbent showed excellent removal percentage (96%) when compared to its corresponding non-imprinted polymer (77%). The kinetic studies showed that the removal process followed a pseudo-second order adsorption rate and the isotherm data is explained well by the Freundlich model. The obtained results indicate that MMIP MAA- $\beta$ -CD can be used as a smart and efficient adsorbent material for adsorption of bisphenol A from aqueous media.

**Keywords:** Magnetic molecularly imprinted polymer, Phenolic compounds, Cyclodextrin, Adsorption.

**DEVELOPMENT OF ULTRASONIC-ASSISTED LIQUID-LIQUID  
MICROEXTRACTION USING MAGNETIC HYDROPHOBIC DEEP  
EUTECTIC SOLVENT (mDES) FOR DETERMINATION OF  
HERBICIDES RESIDUE IN WATER SAMPLES.**

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A simple, and cost-effective ultrasonic assisted liquid-liquid microextraction (ULLME) based on the magnetic hydrophobic deep eutectic solvent (mDES) has been developed for the determination of five different herbicide residues (bromacil, ametryn, 2,4-D, MCPA, chlorothalonil) in water samples. A series of magnetic hydrophobic DES was prepared using fatty acids (octanoic acid, nonanoic acid, decanoic acid and dodecanoic acid) and ferrum (III) chloride (FeCl<sub>3</sub>) as an alternative extraction solvent. These mDESs were characterized using Fourier Transform Infrared (FTIR). The ULLME technique is able to pre-concentrate the target analytes with a minimum amount of DES and thus, enhanced the sensitivity of the procedure. The key parameters affecting the extraction efficiency such as the initial concentration of the sample, type of mDES, the volume of mDES, sample pH, and time of sonication were studied in order to obtain the most optimal conditions of effective parameters. The remained concentration of herbicides in the aqueous phase was analyzed using high-performance liquid chromatography combined with UV-Vis detector (HPLC-UV) at wavelength 235 nm within 32 min.

**Keywords:** Ultrasonic assisted liquid-liquid microextraction, Magnetic hydrophobic DES, HPLC-UV, Herbicides residue, Water samples

**APPLICATION OF S-QUINOLIN-2-YL-METHYLDITHIOCARBAZATE FUNCTIONALIZED MAGNETIC NANOPARTICLES AS MAGNETIC SOLID PHASE EXTRACTION ADSORBENT FOR THE DETERMINATION OF CADMIUM AND COPPER IN WATER SAMPLE**

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A new and simple method for Cd<sup>2+</sup> and Cu<sup>2+</sup> separation from water sample by magnetic solid phase extraction (MSPE) using Fe<sub>3</sub>O<sub>4</sub> magnetic nanoparticles functionalized with S-quinolin-2-yl-methyl-dithiocarbazate (MNP-SQ2MDTC) adsorbent has been developed. MNP-SQ2MDTC based MSPE was carried out by adding adsorbent in the sample solution which is to be sonicated followed by separation of MNP-SQ2MDTC adsorbent using an external magnet. Subsequently, Cd<sup>2+</sup> and Cu<sup>2+</sup> were desorbed by sonicating MNP-SQ2MDTC in acid solution prior to decantation with the external magnet. The parameters affecting the MSPE method such as pH, the weight of adsorbent, sonication time, sample volume, eluent volume, and type of eluent have been investigated using atomic adsorption spectrometer (AAS). Under the optimized conditions, the limit of detection (LOD) of the developed method for Cd<sup>2+</sup> and Cu<sup>2+</sup> were found to be 0.175 and 0.016 µg mL<sup>-1</sup>, and their limit of quantifications (LOQs) were found to be 0.582 and 0.053 µg mL<sup>-1</sup>, respectively. The proposed method has been validated by applying in seven different water samples, collected from various local rivers and the results showed that our developed method is able to recover the target metal ions up to 98.7%. Overall, the proposed method has the advantages of simplicity, rapidity, selectivity, and high efficiency for the removal of heavy metal ions in water.

**Keywords:** Magnetic solid phase extraction, Heavy metals removal, Magnetic nanoparticles, S-quinolin-2-yl-methyl-dithiocarbazate.

**MAGNETIC SOLID PHASE EXTRACTION BASED ON MAGNETITE GRAPHENE OXIDE NNOPARTICLES FOR DETERMINATION OF ANTIBIOTICS IN ENVIRONMENTAL WATER SMAPLES**

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Graphene oxide-based magnetic nanoparticles (GO- Fe<sub>3</sub>O<sub>4</sub>) was developed as magnetic solid-phase extraction (MSPE) adsorbent for the determination of antibiotics (ofloxacin, enrofloxacin, levofloxacin, sparfloxacin, gemifloxacin and norfloxacin) in environmental water samples. Graphene oxide (GO) was successfully synthesised using a modified Hummers method, the reaction has been performed by oxidizing of graphite flakes using KMnO<sub>4</sub> in presence of H<sub>2</sub>SO<sub>4</sub>/H<sub>3</sub>PO<sub>4</sub>. Then, GO-Fe<sub>3</sub>O<sub>4</sub> was synthesised by one step coprecipitation method using ammonium ferrous sulfate ((NH<sub>4</sub>)<sub>2</sub>FeSO<sub>4</sub>) and ferricchloride hexahydrate (FeCl<sub>3</sub>·6H<sub>2</sub>O) as iron sources and ammonium solution as precipitant. The structure and morphology of GO-Fe<sub>3</sub>O<sub>4</sub> were characterised by using Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and Energy Dispersive X-ray spectroscopy (EDX). The MSPE was performed prior to HPLC-DAD for the determination of antibiotics. Based on the result obtained, GO-Fe<sub>3</sub>O<sub>4</sub> has been successfully utilized as MSPE adsorbent for selective and efficient extraction of antibiotics.

**Keywords:** Graphene oxide-based magnetic nanoparticles, Magnetic solid phase extraction, Antibiotic, High-performance liquid chromatography

**EMULSIFICATION LIQUID-LIQUID MICROEXTRACTION BASED ON DEEP EUTECTIC SOLVENT: AN EXTRACTION METHOD FOR THE DETERMINATION OF DICAMBA AND MCPA IN ENVIRONMENTAL WATER SAMPLES**

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A new and rapid emulsified liquid-liquid microextraction (ELLME) method was successfully developed by using phenolic based deep eutectic solvent (DES) as the extraction solvent coupled with high performance liquid chromatography and ultra-violet detector (HPLC-UV) in the determination of phenoxy acid herbicides, DICAMBA (3,6-dichloro-2-methoxybenzoic acid) and MCPA (2-methyl-4-chlorophenoxyacetic acid) in environmental water samples. Five different phenolic based DES were successfully synthesized by using phenol, 2-chlorophenol, 3-chlorophenol, 4-chlorophenol and 3,4-dichlorophenol as the hydrogen-bond donor (HBD) and choline chloride as the hydrogen-bond acceptor (HBA). These DESs were characterised by using Fourier Transform-infrared (FTIR) and Nuclear Magnetic Resonance (NMR). The ELLME method was further developed by studying the effect of different parameters; type and volume of extraction solvent, type and volume of emulsifier solvent, sample pH and vortex time. Under the optimum conditions, enrichment factor (EF) were in the range of 43.1 – 59.7. Limit of detection were 0.96 µg/L and 1.01 µg/L for DICAMBA and MCPA, respectively. This method was applied on the real environmental water sample, with extraction recoveries in the range of 23-90% for dicamba and 82-99% for MCPA.

**Keywords:** Phenolic based deep eutectic solvent, Emulsified liquid-liquid microextraction, Phenoxy acid herbicides, HPLC

**ELECTROCHEMICAL DEGRADATION OF REACTIVE RED 4  
USING GRAPHITE/CHITOSAN-PVC COMPOSITE ELECTRODES**

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Reactive Red 4 (RR4) is known as one of the azo dyes which are the major contaminants that enter both aquatic and atmospheric systems via textile industries. In this research, RR4 degradation was studied by using graphite/chitosan-PVC (C/Chi-PVC) as anode and graphite rod as cathode in the presence of sodium chloride (NaCl) as supporting electrolyte. Five electrodes with different C: Chi composition ratios have been tested using cyclic voltammetric technique. The result showed that the electrode with 70:30 composition ratio (known as C<sub>49</sub>/Chi<sub>21</sub>-PVC<sub>30</sub>) shows better electrochemical behaviour compared to the other electrodes. Therefore, C<sub>49</sub>/Chi<sub>21</sub>-PVC<sub>30</sub> was chosen as the electrode used for electrolysis at different concentration of NaCl (0.01-1.0M), voltage (1-20V), and times (15-75min) for the electrolysis parameters optimization. The UV-Vis spectra of RR4 showed complete decolorization of solution in a short of time. The mineralization studies (COD and TOC analyses) using treated and untreated RR4 solution proved that the prepared electrode was able to degrade the solution. This concluded that C<sub>49</sub>/Chi<sub>21</sub>-PVC<sub>30</sub> was the most efficient for the treatment of RR4.

**Keywords:** Electrochemical treatment, Composite electrodes, Reactive Red 4, Graphite, Chitosan

**PLATINUM GROUP ELEMENTS ANALYSIS IN BUKIT BUNUH  
METEORITE IMPACT STRUCTURE, LENGGONG, PERAK**

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Bukit Bunuh archaeological sites were found in 2001 during palaeoenvironmental survey by Centre for Global Archaeological Research USM team. Bukit Bunuh revealed important evidence of meteorite impact and Palaeolithic stone tools. Based on archaeological evidence, the impact happened at 1.83 Ma and bombarded a Palaeolithic stone tools workshop at Bukit Bunuh. Geophysical studies suggested a complex type of crater with the size of 8km in diameter. Boulders of impact melt and seuvite rocks which found at the site strengthen the evidence of impact event from the geological perspective. 19 impact related rocks from the potential impact structure site were analysed for platinum group elements (Os, Ir, Ru, Rh, Pt, Pd, Re) content using INAA and ICPMS technique after Nickel-sulphide (NiS) fire assay procedure. The results showed some significant concentration of PGEs content when compared to the continental crust concentration. For example, concentration of Iridium (Ir) in the Bukit Bunuh samples are 0.2-0.3 ppb, which are 10x higher concentration than the bulk continental crust value (0.037 ppb). However, most of the results from Bukit Bunuh samples showed consistently lower PGEs content compare to other impact site in the world such as Morokweng (South Africa), New Quebec (Canada), Roter Kamm (Namibia), and Clear Creek (USA). The findings suggested the type of meteorite which fell on the Bukit Bunuh archaeological sites was achondrite type that has a lower concentration of PGEs than the iron or chondritic types.

**Keywords:** PGE, Bukit Bunuh, meteorite impact, archaeological site, INAA, ICPMS

## CATIONIC PERYLENE PROBE AND SILVER NANOPARTICLE-BASED APTAMER SENSOR FOR LOW COST DETECTION OF AFLATOXIN B1 IN FOOD AND FEED SAMPLES

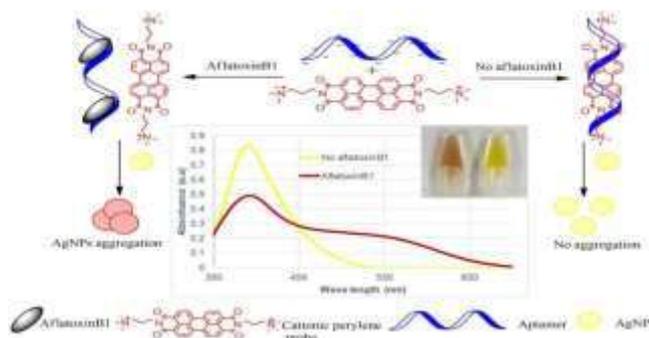
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Herein, the aggregation of silver nanoparticles-based aptamer induced by cationic perylene probe for aflatoxin B1 detection in food and feed samples was studied. Initially, the aptamers, single strand DNA, containing negative charges interacted with cationic perylene probe through electrostatics. In the presence of aflatoxin B1, the aptamers were competed between aflatoxin B1 and cationic perylene probe to form complex structures. Since the aptamers were more specific to aflatoxin B1 than cationic perylene probe. Consequently, cationic perylene probe was released into the solution. Afterward, silver nanoparticles were added in order to be aggregated by cationic perylene probe resulting in the color changing from yellow to orange. These color changes could be quantitatively measured by colorimetry. The absorbance ratio at 390 nm and 520 nm varied with the corresponding change in different concentrations of aflatoxin B1. Moreover, the developed method was applied to detect aflatoxin in food and feeds at limit of quantitation lower than the maximum residue level of aflatoxin in food. In conclusion, this method was simple, sensitive and cost effective. It could be used for rapid screening test of aflatoxin B1 in food and feed.



**Fig 1.** Schematic representation of the aflatoxin B1 detection by colorimetric technique of silver nanoparticles induced by cationic perylene probe.

**Keywords:** Aptamer, Cationic perylene probe, Silver nanoparticles, Aflatoxin

**PAPER-BASED SENSOR COUPLED WITH SMARTPHONE FOR  
FLUORESCENCE DETECTION OF PHENOLIC POLLUTANTS  
BASED ON AMPHIPHILIC QUANTUM DOTS**

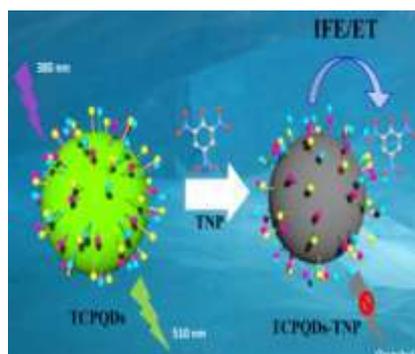
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Herein, we present paper-based sensors combined with amphiphilic quantum dots to detect phenolic pollutants. The proposed paper-based sensor could implement visual detection of 2,4,6-trinitrophenol (TNP). The fluorescence of the amphiphilic quantum dots has been quenched by TNP through inner filter effect (IFE), electrostatic,  $\pi$ - $\pi$ , and hydrogen bonding interactions. Under optimal conditions, the detection limits down to 0.56 nM within 60 s. More importantly, portable, cost-effective, and easy to use paper sensors have been successfully applied to visually detect as low as 2.29 picogram of TNP. The possibility of using a smartphone with a color-scanning app in the detection of TNP has also been established. Moreover, the practical application of the developed sensors for TNP detection in wastewater samples has been successfully described with good recoveries of 98.02–107.50%. This novel paper-based device shows great potential for portable and rapid detection of TNP pollutant in environmental real samples.



**Keywords:** Amphiphilic quantum dots, fluorescence, TNP, wastewater, smartphone, paper sensor

**DEVELOPMENT OF AN ELECTROCHEMICAL SENSOR FOR THE  
DETECTION OF MALTOSE IN STARCH HYDROLYSIS**

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A hybrid sol-gel nano-biosensor has been developed to determine maltose produced by starch hydrolysis by  $\alpha$ -amylase. Glucosidase (GOx) and glucose oxidase (GD) enzymes were covalently immobilized on the sol-gel sensor via carbodiimide reaction. The kinetic study confirmed the successful immobilization of the enzyme. Immobilization conditions, such as the amount of enzyme, immobilization time and pH in working buffer, were optimized using the Design of Expert (DOE) software applying Response Surface Methodology (RSM) through Central Composite Design (CCD). The biosensor showed the maximum response at glucose oxidase concentration 20 U/ml and immobilization time 90 minutes. The optimum pH of the working buffer was pH 7 at 45 °C. Lineweaver-Burk plot gave the  $K_m$  value for the immobilized enzyme in sensor 12.73 mM which was much lower than enzyme activity in solution 24.6 mM. The biosensor showed a linear response to maltose hydrolysis in the range of 0.5 mM to 5 mM. The sensitivity and correlation coefficient of the sensor was 43.15  $\mu\text{A}/\text{mM}$  and 0.9975, respectively.

**Keywords:** Sol-gel, Covalent immobilization,  $\alpha$ -glucosidase, Glucose oxidase, Maltose hydrolysis

**OPTIMIZATION OF PROTIC IONIC LIQUID MEDIATED CURCUMIN EXTRACTION FROM *CURCUMA LONGA*****<sup>1</sup>Z. S Othman, <sup>2</sup>N. A Ali, <sup>3</sup>S. M. S Zahari, <sup>1</sup>M. Y Maskat and <sup>1</sup>N. H. Hassan**

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Turmeric rhizomes have an abundance of curcuminoids (curcumin, demethoxycurcumin and bisdemethoxycurcumin). Curcumin helps to reduce amyloid plaque formation, inflammation and neurodegeneration which were the hallmarks of Alzheimer's disease. However, its chemical instability and low yield extracted raised concern. Therefore, this study prompted to introduce a protic low-cost ionic liquid (IL), triethylammonium hydrogen sulphate, [Et<sub>3</sub>NH][HSO<sub>4</sub>] as an additive in extraction medium and analyze the optimum parameter for higher yield of curcumin extraction with the antioxidant properties of the extract. Responses (curcumin yield, TPC, TEAC) were optimized using response surface methodology (RSM) by employing three factors (temperature, extraction time and % THS) based on a three-level face centered central composite design (CCD). The optimal conditions to obtain maximum curcumin yield, TPC and TEAC were extraction temperature of 53°C at 24 hours extraction time and 20% THS with predicted curcumin yield of 57.99 mg/g, TPC of 71.61 GAE g/100g and TEAC of 1231.85 µgTE/g. The experimental values obtained for the curcumin yield, TPC and TEAC under the optimal condition were 56.89 ± 2.95 mg/g, 69.75 ± 1.10 GAE g/100g and 1186.59 ± 3.50 µgTE/g respectively. The regression coefficients R<sup>2</sup> of predicted were in reasonable agreement with adjusted R<sup>2</sup> for all responses model. The proximity between experimental and prediction values verify the fitness of RSM models applied. Thus, this research outcome able to aid in enhancing the extraction of curcumin by introducing [Et<sub>3</sub>NH][HSO<sub>4</sub>] as an additive in turmeric extraction medium and determine the optimum extraction condition.

**Keywords:** Triethylammonium hydrogen sulphate, *Optimization, RSM, Antioxidant, Curcumin*

**PHYTOCHEMICAL ANALYSIS AND CYTOTOXIC EVALUATION OF  
*CITRUS HYSTRIX* LEAF EXTRACTS ON COLON CANCER CELLS  
(HT29)**

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*Citrus hystrix* locally known as *Limau purut* is a culinary flavoring that is common in Malaysia and South East Asian cuisine as well as in herbal preparations. The present study was aimed to evaluate the antiproliferative activity of ethanolic and aqueous leaf-extracts of *C. hystrix* and to screen for bioactive metabolites present in the leaves. The cytotoxic effects of the extracts on colon cancer cells (HT29) were evaluated by 3(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazoliumbromide (MTT) assay and the bioactive metabolites were screened by qualitative analysis using standard procedures. The cytotoxic activities of the extracts showed IC<sub>50</sub> values of 0.25 and 3.25 mg/mL for ethanolic and aqueous extracts, respectively indicating stronger cytotoxic effects on HT29 cells in ethanolic extract. The phytochemical analysis of the extracts revealed the presence of several bioactive compounds; alkaloid, saponin, flavonoid and fixed oil and fats, with ethanolic extract being more potent compared to aqueous extracts. Relatively, the results obtained in this research have shown high potential of *C. hystrix* leaves to be used as a good source of anticancer agent for the elimination of various disease and infections and this may be attributed to the presence of bioactive compounds present in the leaves.

**Keywords:** *Citrus hystrix*, Extract, Cytotoxic activity, Phytochemicals.

**TOTAL PHENOLIC CONTENT AND ANTIOXIDANT PROPERTIES  
OF *CITRUS HYSTRIX* LEAF-EXTRACTS**

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A significant number of plants have been utilized as dietary and phytomedicinal sources in enhancing our health. *Citrus hystrix* is a culinary flavoring that is common in Malaysia and South East Asian cuisine as well as in herbal preparations. The present study was conducted to evaluate antioxidant property and total phenolic content of aqueous and ethanolic leaf-extracts of *C. hystrix*. Antioxidant activity was evaluated by using DPPH Radical Scavenging Activity Assay and Total Phenolic Content (TPC) was determined by Folin-Ciocalteu method. The percentage yield of extract concentrates and TPC were found to be higher in ethanolic compared to aqueous extracts, respectively. For the antioxidant activity, the present results indicates that both ethanolic and aqueous leaf-extracts showed IC<sub>50</sub> values of > 8 mg/mL in comparison of 0.03 mg/mL of the standard ascorbic acid, indicating low antioxidant activity. Therefore, both ethanolic and aqueous leaf-extracts of *C. hystrix* exhibited a weak, negative correlation between total phenol contents and percentage inhibition of DPPH with R<sup>2</sup> value of 0.2278 and 0.434, respectively. Thus, *C. hystrix* leaves have no potential power to be used as good source of natural antioxidants.

**Keywords:** *Citrus hystrix*; Antioxidant property; Extract; Total Phenolic Content.

### PHYTOCHEMICAL AND IN VITRO ANTIMICROBIAL STUDIES OF ROOT BARK EXTRACTS FROM *STRYCHNOS INNOCUA* (DELILE)

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Many medicinal plants have been reported to have potential antimicrobial activities. The decoction of root bark of *Strychnos innocua* (Delile), a member of *Loganiaceae* family, is used to treat candidiasis, skin infections, and other infectious diseases. This present study is to investigate the phytochemicals presence in the root bark of *S. innocua*, as well as the antimicrobial activities. Preliminary phytochemical study of the root bark extracts (*n*-hexane, ethyl acetate and methanol extracts) showed the presence of alkaloids, flavonoids, triterpenes, steroids, tannins, saponins, phenols, quinones, anthraquinones, carbohydrates, phlobatannins, cardiac glycosides and coumarines. The antimicrobial study of the extracts showed activities against three gram-positive bacteria (Methicillin-resistant *Staphylococcus aureus*, *Staphylococcus aureus* and *Bacillus subtilis*), one gram-negative bacterium (*Pseudomonas aeruginosa*) and three fungi (*Candida albicans*, *Candida krusei* and *Aspergillus fumigatus*). The organisms (*Streptococcus pyogenes*, *Escherichia coli*, *Salmonella typhi*, *Klebsiella Pneumoniae* and *Aspergillus niger*) showed resistance. The inhibitory effect was compared with the standard drugs ciprofloxacin, sparfloxacin, fulacin and fluconazole. Minimum inhibitory concentration (MIC) and minimum bactericidal concentration (MBC) for all the extracts were also determined using the broth dilution method. This study concluded that the root bark of *S. innocua*, used traditionally as a medicinal plant, has antimicrobial activities against some pathogenic organisms.

**Keywords:** *Strychnos innocua*, Phytochemical, Antimicrobial, Minimum inhibitory concentration, Minimum bactericidal concentration.

## DEVELOPMENT OF CONDUCTIVE NANOWIRES BY TEMPLATE DIRECTED PROCESS ONTO DEOXYRIBONUCLEIC ACID MOLECULES

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American Semiconductor Industry Association (SIA) predicts that current trend in the down scaling of feature sizes on commercial microchip will soon reach a plateau due to fundamental limitation of photolithographic methods currently used in device manufacture. This expected failure at molecular scale presents the challenge for providing alternative strategies which move away from the convectional top-down approaches to bottom-up device construction. In this study, we focus on a scheme which in a simple way enables the generation of polymer nanoparticles onto herring testes deoxyribonucleic acid (HT-DNA) as a platform. Such structures are interesting for applications in different areas such as biosensors and biomedical. The process combines the opportunities afforded by self-assembly and recognition capabilities of HT-DNA which find application in controlled assembly for designed structures. In the first step, the DNA acts a template to achieve pattern deposition followed by ageing to direct the nanoparticle growth. Secondly, the application of scanning probe microscopy elucidates ‘wire-like arrays’ conductivity and the scheme can be extended to microscopy dimensions. The AFM topographies translated the interaction into the directing ability of the template into high aspect ratio nanostructured materials with densely packed nanoparticles linearly arranged as nanowires. The diameter typically lies in the 6.5-33 nm. Analysis of the nanostructures reveals symmetrical distribution of the structure heights around the modal values. Electronic probe of the nanowires by non-contact method via scanned conductance microscopy identifies HT-DNA/Ppy to follow negative parabolic dependence, an indication of conductive characteristic.



Scheme 1: Templating by HT-DNA molecules in directing the growth of Ppy (Zubairu, 2013)

**Keywords:** DNA, AFM, Self-assembly, Nanowires, Nanoparticles.

**CHARACTERIZATION AND NUTRITIONAL QUALITY OF  
BAMBARANUT (*VIGNA SUBTERRANEA (L) VERD C.*) PROTEIN  
GROWN IN MADOBI LGA, KANO STATE-NIGERIA**

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Bambaranut (*Vigna subterranea (L) verd C.*) is one of the neglected leguminous crops in Kano state, Nigeria. The study was aimed at evaluating the amino acids content and nutritional quality of Bambaranut proteins grown in Madobi local Government area of Kano state, Nigeria. The amino acids content of Bambaranut was determined using Technicon sequential Multi-sample Amino acid Analyzer (TSM). The total amino acid content of Bambaranut was 94.76g/100g protein, with total essential and nonessential amino acids of 43.83g/100g protein (46.25%) and 50.93g/100g protein (53.75%), respectively. The total aromatic and sulphur containing amino acids were 11.69g/100g protein (12.34%) and 2.87g/100g protein (3.02%), respectively. All the essential amino acids, aromatic and sulphur containing amino acids are present in good proportion and exceed the WHO/FAO/UNICEF standard values except leucine, which is below the standard value for children. The result indicates that Bambaranut has an excellent amino acid profile and could be used as a food ingredient in baby food as amino acid supplements and in pharmaceutical industries e.t.c.

**Key words:** Bambaranut, Protein characterization, Amino acids, Nutrition

**BIOCHEMICAL AND IN-VITRO ANTIOXIDANT ACTIVITY OF ETHYL ACETATE FRACTIONS OF *BOSWELLIA POPYRIFERA* (DEL) STEM BARK ON CCL<sub>4</sub> INDUCED LIVER DAMAGE IN RATS**

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This study is aimed at evaluating the biochemical effects and antioxidants activity of ethyl acetate fractions on carbon tetrachloride-induced liver damage in rats. Column chromatography of the ethyl acetate stem bark extract of *Boswellia papyrifera* (E2) was carried out using Silica gel [(60-120 mesh) (500g)] in a slurry of n-hexane. The column was eluted using solvents and solvent mixtures. The use of the solvent mixtures was to increase the polarity of the solvent. Then, the column was gradually eluted in a mixture of solvent n-hexane:ethylacetate. The obtained fractions were evaporated to dryness and monitored using TLC on different solvent systems. The similar fractions were pooled into 4 major subfractions (E2A, E2B, E2C, and E2D). These subfractions were administered to CCl<sub>4</sub> induced -rats liver for four weeks prior to liver function indices and antioxidant analysis. A total of 25 white male albino rats were used for each fraction in this experiment and the rats were divided into five groups. Each group consists of five rats. The rats in group I that were not induced with lipid peroxidation and liver damage, they served as a normal control. Groups II to V were injected with a single dose of 120mg/kg carbon tetrachloride prior to oral administration of ethyl acetate fractions of *Boswellia papyrifera* to groups III to V with 20, 30, 50mg/kg of the fractions. The effect of E2B fraction on the activity of Serum Aspartate aminotransferase (AST), Alanine aminotransferase (ALT), Alkaline phosphatase (ALP), and serum concentration of Albumin, total and Direct Bilirubin and Total Protein were significantly ( $p < 0.05$ ) found to be no different from that of the normal control group. However, a significant ( $p < 0.05$ ) increase in glutathione peroxidase (GPX) and catalase (CAT) activities were recorded in all experiments for the treated animal groups as compared to the control group. Malondialdehyde (MDA) concentration was observed to decrease significant ( $p < 0.05$ ) in all animal groups as compared to the control group. Histopathology shows that liver integrity was consistent with the biochemical findings. However, no significant effect observed for E2A, E2C and E2D fractions. This can conclude that the E2B fraction of ethyl acetate of *Boswellia papyrifera* possesses potent curative activity against CCl<sub>4</sub> induced liver damage via a potential free radical mopping activity.

**Keywords:** *Boswellia papyrifera*, Column chromatography, Liver enzymes, Antioxidants, Carbon tetrachloride.

**IDENTIFICATION OF PRO-APOPTOTIC GENE (*Bax*) EXPRESSION  
INDUCED BY ROOT EXTRACT OF PAKU TUNJUK LANGIT  
[*Helminthostachys zeylanica* (L.) Hook.] ON MCF-7 BREAST CANCER  
CELL**

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The high incidence of breast cancer and the emergence of various problems in cancer treatment indicate the need for the development of anticancer drugs to improve the effectiveness of treatment. One of the developments of new drugs can be done by utilizing natural resources, such as the utilization of paku tunjuk langit (*Helminthostachys zeylanica*) plant which is proven to have anticancer activity in several types of cancer cells. This anticancer activity can be supported by apoptotic mechanisms that induced by several protein pathways, including the regulation of the protein family *Bcl-2* through the induction of pro-apoptotic proteins namely *Bax*. The objectives of this study were to examine the cytotoxic activity and apoptotic mechanism through the *Bax* gene expression pathway by the extract of *H. zeylanica* root on MCF-7 cell. The extract of *H. zeylanica* root was obtained by polarity gradient extraction using n-hexane and ethyl acetate as the solvents with ultrasound assisted maceration methods. The cytotoxic assay was carried out by using the MTT assay and *Bax* gene expression was identified by using conventional Polymerase Chain Reaction (PCR). Cytotoxic assay showed that IC<sub>50</sub> values of n-hexane and ethyl acetate extract of *H. zeylanica* root were 771.97 and 104.97 ppm, respectively. Identification of *Bax* gene expression in MCF-7 cells which was treated by the ethyl acetate extract of *H. zeylanica* root in ½IC<sub>50</sub> and IC<sub>50</sub> concentrations, did not show many changes in *Bax* gene expression compared to control cell group. Meanwhile a high increase of *Bax* gene expression occurred in group which treated by 2IC<sub>50</sub> extract concentration. The results of this study indicated the ethyl acetate extract of *H. zeylanica* root at 2IC<sub>50</sub> concentration induced the apoptosis in MCF-7 cells, by increasing the expression of *Bax* gene as a pro-apoptotic protein.

**Keywords:** *Helminthostachys zeylanica*, Breast Cancer, MCF-7, Apoptosis, PCR

**METABOLOMICS APPROACH: ANTIDIABETIC ACTIVITY OF  
MOMORDICA CHARANTIA FRUIT USING STREPTOZOTOCIN  
INDUCED OBESE-DIABETIC RAT MODEL**

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*Momordica charantia* Linn (Cucurbitaceae) has been widely commercialized based on traditional usage as an antidiabetic product. However, the scientific evidence of its antidiabetic activity is not sufficient. Hence, the major aims of this research were to evaluate the antidiabetic activity of *M. charantia* fruit through proton-nuclear magnetic resonance (<sup>1</sup>H-NMR) spectroscopy based metabolomics approach. The 80 % ethanolic extract of *M. charantia* was force fed to obese-diabetic (ob-db) rats. Rats were divided into four groups: healthy, ob-db treated with 300 mg/kg bw metformin, ob-db treated with 300 mg/kg bw of *M. charantia* and ob-db rats. *In vitro* dipeptidyl peptidase-4 (DPP-IV) inhibitory and 3T3-L1-cell glucose uptake activities were also tested for the same extract to explain its mode of action in relation to the metabolomics results. The results showed that the administration of the 80% ethanolic extract of 300 mg/kg bw for 4 weeks significantly ( $P < 0.05$ ) reduced the blood glucose level. However, the data obtained from the metabolomics showed that the metabolite profiles of the serum and urine of rats could not be fully normalized by the 80% ethanolic extract and metformin. The identified biomarkers in serum and urine were 2-hydroxybutyrate, leucine, adipate, alanine, acetate, succinate, 2-oxoglutarate, dimethylamine, creatine, creatinine, betaine, glucose, taurine, phenylacetyl glycine, allantoin and hippurate. The metabolomics approach indicated that the 80% ethanolic extract of *M. charantia* fruit and metformin have altered energy, amino acid, purine, creatine, bile, and gut microflora metabolisms of the serum and urine profiles of the streptozotocin ob-db rats. Furthermore, it was found that the energy metabolism was ameliorated through the improvement of 3T3-L1-cell glucose uptake and inhibition DPP-IV.

**Keywords:** Metabolomics, *Momordica charantia*, Diabetes and obese-diabetic rat.

## BIO-010

### A BIOCHEMICAL STUDY ON THE GASTROPROTECTIVE EFFECT OF *MUNTINGIA CALABURA* LEAVES IN RATS INDUCED WITH GASTRIC ULCER

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*Muntingia calabura* L. (family Muntingiaceae), commonly known as Jamaican cherry or *kerukup siam* in Malaysia, is used traditionally to treat various ailments including gastrointestinal disorders. The current study is aimed to determine the gastroprotective activity and the possible underlying mechanism involving the modulation of biochemical compounds of ethyl acetate fraction (EAF) of *Muntingia calabura* methanolic leaves extract (MEMC). MEMC was fractionated to obtain EAF. EAF was investigated for possible gastroprotective property using ethanol-induced gastric ulcer rat model. Sprague Dawley rats were administered orally once daily with 8% Tween 80 (control), 100 mg/kg ranitidine, or EAF, in the doses of 100, 250, and 500 mg/kg, for seven days, followed by ulcer induction using absolute ethanol. The rats were euthanized; macroscopic observation of the stomach was done. The ulcer area (UA) was determined and the percentage protection afforded by EAF was calculated. Biochemical estimation including superoxide dismutase (SOD), glutathione (GSH), catalase (CAT) and malondialdehyde (MDA) levels in the ethanol-induced stomach tissue homogenate was determined. Pre-treatment of EAF significantly ( $p < 0.001$ ) attenuated the gastric lesions in rats' stomach in a dose-dependent manner. EAF treatment markedly ameliorated the SOD, GSH and CAT activity while attenuating MDA level, relative to the vehicle group. In conclusion, the protective effect of EAF on gastric ulceration in rats could be associated with the modulation of biochemical compounds that renders the antiperoxidative effect and causes improvement in the antioxidant status.

**Keywords:** *Muntingia calabura*, gastroprotection, mechanism of action, antiperoxidative, antioxidant

ENV-001

## DISPERSION OF NANOCELLULOSE CRYSTALS INTO NATURAL RUBBER BIONANOCOMPOSITES

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In the present study nanocellulose crystals (NCCs) were isolated from treated bagasse fibre (BF) by acid hydrolysis followed by separation using centrifugation, homogenization, ultra sonication and dialysis. The obtained natural nanofiller was homogenously dispersed in biopolymer natural rubber matrix composite. Transmission Electron Microscopy (TEM) images showed nanocrystals of rod shape morphology. Thermogravimetric Analysis (TGA) and Differential Thermogravimetry (DTG) profiles of raw bagasse fiber, pure bagasse cellulose and NCC, all had characteristic onset and decomposition temperatures indicating a change in the structure after chemical treatments. Particle size distribution measurements corroborated with the TEM and FESEM results and showed that the majority of the nano crystals were in the 100 – 300 nm range. Fourier Transform Infra-Red (FTIR) analysis showed functional group changes as the amorphous regions of the polymer were removed revealing the ordered crystalline portions. X-Ray Diffraction (XRD) and Crystallinity Index (CrI) calculations showed a steady increase in the Crystallinity from an amorphous to crystals fiber.

**Keyword:** Dispersion, Nanocellulose crystals, Natural rubber, Bionanocomposite

**IMPACTS OF CITRIC ACID ON THE PHYTOEXTRACTION OF ZINC (Zn) USING *SORGHUM BICOLOR L.M* PLANTS**

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Greenhouse hydroponic experiments were carried out to examine the impacts of citric acid on Zn uptake and phytoextraction potentials of sorghum (*sorghum bicolor L.M*). Two-week-old seedlings transplanted in hydroponic solutions were treated with different doses of Zn in the concentration range of 5, 25, 50, 100 and 200 mg/L alone or in combination with 10 mM citric acid. After 21 day of culture, the plants were harvested, separated into roots and shoots and then dried. Fresh and dry weights, Zn uptake, translocation factor (TF), bioconcentration factor (BCF), proline, ascorbate and pigment contents were determined. The results indicate that Zn uptake, fresh and dry weights, TF, BCF, proline and ascorbate contents were concentration dependent with more significant increase ( $P < 0.05$ ) after application of citric acid. Pigments and protein contents were however, severely decreased with increasing Zn concentrations and appreciated gradually with the addition of citric acid. Thus, citric acid efficiently increased phytoextractability of Zn and *Sorghum bicolor LM* was non-hyperaccumulator of Zn; but may be used for phytoremediation of Zn contaminated environments with assistance of citric acid.

**Keywords:** Citric acid, Hydroponic, Phytoextraction, Proline, *Sorghum bicolor L.M*, Zn.

**APPLIED OF A NEW BACTERIAL SPECIES ISOLATED FROM AN  
ACTIVATED SLUDGE FOR THE DENITRIFICATION TREATMENT  
OF THE WASTEWATER**

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Denitrification is the key reactions in the nitrogen cycle, which allowed converting the nitrate to atmospheric nitrogen, in a single step or by two successive steps through the intermediate nitrite. These reactions are much recommended in the wastewater treatment, under the action of several denitrifying bacteria. In this communication, we were developed a new denitrifying strain called *Proteus sp. S19*, isolated from an Algerian activated sludge. This strain was identified and incubated in a tube of broth containing nitrate. For the denitrification assay, we have incubated the strain *Proteus sp. S19* in a bioreactor contains a synthetic medium rich in nitrite ion, the determinations of residual and produced concentrations of different ions present in the medium before and after the denitrification process was effectuated. The bacterial density of the medium was increased during the denitrification process which was explained by the growth of the *Proteus sp. S19* strain in the medium with a denitrification rate of 90%.

**Keywords:** Nitrate, *Proteus sp S19*, Denitrification, Wastewater treatment.

**BRIQUETTING OF MILLET STALK AND SHEANUT SHELL AS A TOOL FOR SOLID WASTE MANAGEMENT AND ENERGY GENERATION FOR RURAL HOUSEHOLD IN NIGERIA**

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In an effort to reduce agro-wastes and provide a cheap and affordable alternative to firewood in rural household, this study was carried out to produce bio-briquettes from sheanut shell and millet stalk using a simple extruder briquetting machine. Different samples of briquettes were produced by blending varying proportions of the wastes in the ratio of 100:0; 80:20; 70:30; 60:40; 50:50 using cassava starch as binder. The result of the proximate analyses showed that sheanut shell briquettes had a moisture content of 2.50% and millet stalk 3.50%; ash content of 2.50% and 4.50%; volatile matter of 16.0% and 11.0% while fixed carbon content of 79.0% and 81.50% respectively. The viability test results showed that sheanut shell briquettes had a higher density 0.60g/cm<sup>3</sup> and compressive strength of 2600Mpa than the millet stalk briquette with 0.31g/cm<sup>3</sup> and 880MPa respectively. It took the sheanut shell briquettes 20 mins to boil 2 litres of water with calorific value of 7.50ms/kg while it took the millet stalk briquettes 22 mins to boil same quantity of water with calorific value of 6.88 MJ/kg. Also, the specific power output was 480.46 w/Kg for the sheanut shell briquettes and 435.52 w/Kg for the millet stalk briquette. A good biomass fuel was produced which could be used for cooking and ironing in rural household in Nigeria.

**Keywords:** Briquettes, Biomass, Sheanut Shell, Millet Stalk, Firewood.

## INSIGHTS INTO THE PERFORMANCE OF NITROGEN-DOPED POROUS CARBON DERIVED FROM BIOWASTES AS REDOX CATALYST FOR BISPHENOL A REMOVAL

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Redox oxidation technology based on peroxymonosulfate (PMS) activation is an emerging technology for water remediation. In this study, a series of N-doped porous carbons was prepared from various biowastes (spent coffee ground, saw dust, banana peel, orange peel and dried leaf) and used as redox catalyst for bisphenol A removal. The physicochemical properties of the catalysts prepared from different biowastes were investigated using various advanced characterisation instruments and the results indicated that the choice of biowaste influenced the intrinsic (extent of N doping, % graphitic N) and extrinsic (morphology, specific surface area) properties of the resultant N-doped porous carbon. It was found that the N-doped porous carbons prepared from spent coffee ground (PC-SC) and saw dust (PC-SD) have higher at.% graphitic N (44-46 at.%) and specific surface area ( $>400 \text{ m}^2 \text{ g}^{-1}$ ), and they performed significantly better ( $k_{app} = 0.072\text{-}0.087 \text{ min}^{-1}$ ) than those prepared from banana peel, orange peel and dried leaves ( $k_{app} < 0.046 \text{ min}^{-1}$ ). The dominant reactive oxygen species (ROS) was studied using the chemical scavenger (nitrobenzene, sodium azide and ethanol) technique and electron paramagnetic resonance spectroscopy indicating that the  $^1\text{O}_2$  was the dominant ROS in the catalytic system. The  $^1\text{O}_2$  was produced from the interaction between the graphitic N active site and PMS. Meanwhile, the BPA degradation mechanism was also investigated using the LC/MS/MS system and based on the detected BPA intermediates, the BPA degradation pathway is proposed. This study conspicuously shows that the porous carbon prepared from biowastes has a remarkable potential for application in redox catalysis. However, the right selection of biowaste is crucial to produce high performance porous carbon with enhanced catalytic activity.

**Keywords:** Peroxymonosulfate, biowaste, Singlet oxygen, Bisphenol A

**CONVERTING SAWDUST INTO HIGH PERFORMANCE REDOX  
CATALYST FOR ACID ORANGE 7 REMOVAL**

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The application of sawdust and urea as precursors to produce nitrogen-doped carbon (NC) catalyst for acid orange 7 (AO7) removals via peroxymonosulfate (PMS) activation was investigated. The catalyst was prepared using a facile pyrolysis method and the synthesis condition was varied. The surface chemistry, structural and morphological properties of the N-doped carbon catalysts prepared using different conditions were investigated in more detail using various characterization techniques. The surface morphology and microstructure of the catalysts were characterized by scanning electron microscopy indicating that the structure morphology of NC800-20 catalyst has rough surface texture due to increasing surface defects compare to C800. The crystalline structure of the catalysts was analyzed by XRD. The results indicated that sawdust has successfully thermolytic transform into carbonaceous amorphous material. The as-prepared metal-free NC800-20 catalysts show a superior efficiency (93% AO7 removal in 10 min) compared to undoped catalyst (52% AO7 removal in 10 min) in activating peroxymonosulfate to generate reactive radicals for degradation of acid orange 7. A thorough investigation in converting sawdust into a functional catalytic material is presented in this study. Overall, the results show that the sawdust can be converted to high-performance catalyst for redox catalysis.

**Keywords:** Lignocellulosic biomass waste, Sawdust, Peroxymonosulfate, Pyrolysis, N-doped Carbon based catalyst, Acid Orange 7.

**ADSORPTION CHARACTERIZATION OF Cu(II) IONS ONTO  
CHITOSAN BEADS AND CHITOSAN-ALGINATE BEADS:  
EQUILIBRIUM AND THERMODYNAMIC STUDIES**

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This study examined the adsorption of Cu (II) ions from aqueous solution onto chitosan beads and chitosan-alginate beads. Batch adsorption experiments were conducted to determine the optimum initial pH, agitation period and adsorbent dosage for the adsorption of Cu (II) ions. The adsorption capacity obtained based on the linearized Langmuir isotherm model for chitosan and chitosan-alginate beads in a single metal system were 75.76 and 70.42 mg/g, respectively. Meanwhile, the beads showed higher selectivity towards Cu (II) over Pb (II) ions in a binary metal system. Similar observation was also made during the displacement study, whereby both of the beads showed a higher displacement of Pb (II) ions in the presences of Cu (II) ions. Various thermodynamic parameters such as enthalpy ( $\Delta H^\circ$ ), Gibbs free energy ( $\Delta G^\circ$ ) and entropy ( $\Delta S^\circ$ ) changes were computed and the results showed that the adsorption of Cu(II) ions onto both of the beads were spontaneous and endothermic in nature. Based on the ion exchange study, the release of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^+$  and  $\text{Na}^+$  ions played an important role in the adsorption of Cu (II) ions by both of the beads but only at lower concentration of Cu (II) ions. Fourier transform infrared spectroscopy together with other spectroscopy analysis was used to elucidate the mechanism for the adsorption of Cu (II) ions onto chitosan beads and chitosan-alginate beads.

**Keywords:** Chitosan beads, chitosan-alginate beads, adsorption isotherm, thermodynamics.

**SURFACE WATER QUALITY: OCCURRENCE AND  
DETERMINATION OF ORGANOCHLORINE PESTICIDES (OCP) IN  
SUNGAI LANGAT**

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Rivers as surface water in Malaysia are recipients of effluents and wastewater and yet it is important water source for daily uses of some villagers living along the river. Endocrine disruptors such as pesticides can be found in river due to continuous discharge into it. The objectives of this research is to find out the occurrence and concentration of Organochlorine Pesticides (OCP) in Sungai Langat and also to see water quality parameters such as pH, dissolved oxygen (DO), Total Suspended Solid (TSS), Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD) and ammonia nitrogen (AN) affecting the river water quality. 12 stations in total including upstream to downstream of Sungai Langat and also tributary of Sungai Langat. The instrument used to find out concentration of OCP is Triple Quadrupole GCMS. The sources of OCP are mainly industrial effluents and also direct domestic discharges. Pesticides application is essential in protecting crops and increases their productivity. However, this causes the OCP residues to contaminate the crops and their surrounding environment that will eventually lead to exposure to human being along Sungai Langat.

**Keywords:** Endocrine disruptors, Surface water, Organochlorine Pesticides, water quality parameter.

## ARSENIC REMOVAL BY ACTIVATED CARBON FROM DURIAN HUSK

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The preparation of activated carbon from durian husk, variety D175 which also known as *Udang Merah* by chemical activation technique using zinc chloride (10%) as activating agent and its ability to remove arsenic in aqueous solutions were reported in this study. The proximate analysis of durian husk and the characterization of activated carbon using FTIR, SEM and TGA were evaluated. The influence of various factor such as weight of absorbent, arsenic concentration, contact time and pH were studied. The experimental data were analyzed by the Langmuir and Freundlich model of adsorption. Two simplified kinetic models including pseudo-first-order and pseudo-second-order equation were selected to follow the adsorption process. Result from the study suggest that activated carbon prepared from durian husk, D175 has potential to be a promising precursor for the production of activated carbon with the excellent adsorption capacity to remove arsenic in aqueous solution.

**Keywords:** Arsenic, Activated carbon and Durian husk.

**EFFECT OF WELDING SPEED ON WELD SEAM,  
MICROSTRUCTURE AND MICROHARDNESS IN LASER WELDING  
OF CARBON 45 STEEL**

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The present study is concerned with the effects of welding speed on the weld seam, microstructure and microhardness profile in laser welding of carbon 45 steel using IPG YLS-3000W Fiber Laser with Robot. The effects of welding speed (0.005m/s-0.015m/s) on weld width, penetration depth, microstructure, and microhardness were investigated. Before welding, the material surface (with thickness 4mm) was ground with sandpaper to remove oxide, increase the surface finish and reduce the reflection of laser; it was then cleaned with acetone to remove foreign material on the surface of the sample and to avoid the impact of welding effect. The material was then cut into two pieces evenly and then ground with sandpaper, cleaned with acetone, and finally welded. After laser welding of the sample, the welded sample was cut for grinding and scanning. The grinding was done using 180#, 240#, 320#, 400#, 500# and 600# grits of sandpapers. Al<sub>2</sub>O<sub>3</sub> polishing solution was used to polish the sample, 4% nitric acid solution was used in etching the sample, and then finally dried. The microstructure of the welded sample was observed under metallographic microscope. The welded sample was pushed around on the microscope to observe the microstructure of the weld zone, heat affected zone and the weld metal. The microhardness analysis of the sample was carried out using the HV-1000 microhardness tester. The results showed that both weld bead width and penetration depth increase with decrease in laser speed. For all the welding speed values the hardness value is normally distributed, and the weld hardness value is maximum in the heat affected zone and minimum in the matrix.

**Keywords:** Fiber Laser, Laser Welding, Microstructure, Microhardness, Welding Speed.

**WASTE EGG SHELL DERIVED CaO SUPPORTED BY W-Mo MIXED OXIDE BIFUNCTIONAL CATALYST FOR THE PRODUCTION OF BIODIESEL FROM HIGH FFA WASTE COOKING OIL: STOICHIOMETRIC EFFECT**

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Biodiesel is considered as one of the most promising biofuels produced from the vegetable oils. This is due to its environmental benign and availability of variety of feedstock. Biodiesel is generally produced by either esterification reaction of free fatty acid or transesterification reaction of triglycerides, using acid or base catalysts, respectively. The conventional method of producing biodiesel is by using homogeneous catalyst over the edible vegetable oil as feedstock to produce biodiesel and glycerol as the major by-products. However, this method is expensive and time consuming as it requires water washing to remove the liquid catalyst after the reaction, and the catalyst can only be used once. The current sustainable way of biodiesel production is through the utilization of heterogeneous catalysts on low grade vegetable oil, particularly non-edible oil. This would make the biodiesel production process low cost and easier. The catalyst could be used more than once and it is more environmentally friendly. In this work, the solid bifunctional tungsten molybdenum supported by calcium mixed oxide catalyst ( $W_x\text{-Mo}_y/\text{CaO}$ ,  $0.3 \leq x, 0.7 \leq y$ ) was used to produce biodiesel from high FFA waste cooking oil. The catalyst was produced using simple wet impregnation method. The prepared catalyst has successfully converted the high FFA waste cooking oil to biodiesel in a single step reaction process, owing to its acid-base bifunctional capacity. Presence of acid and basic sites of the catalyst are believed to promote the catalytic activity for the transesterification reaction of high FFA waste cooking oil. The stoichiometric effect of transition bimetallic oxide loading on acid-base properties, surface area and porosity of the catalyst were studied. The catalytic activity of the catalyst for transesterification reaction increases with the increase in the W molar weight ratio over Mo from 0.3 to 0.7. The  $W_{0.6}\text{-Mo}_{0.4}/\text{CaO}$  catalyst recorded the highest biodiesel yield of 96.2% under mild reaction conditions of 15:1 methanol to oil molar ratio, 70 °C reaction temperature, 2 wt.% catalyst loading and 2 h reaction time. The newly synthesized catalysts were characterised by X-ray diffraction (XRD), vapour pressure scanning electron microscope (VP-SEM) and temperature programmed desorption of CO<sub>2</sub> and NH<sub>3</sub>.

**Keywords:** Transesterification, Waste cooking oil, Heterogeneous bifunctional catalyst, Stoichiometry effect.

## METALATION OF CARBAZOLE AS A REVERSIBLE HYDROGEN STORAGE MATERIAL

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Heteroaromatic organic hydrides, e.g. carbazole, are prospectively interesting hydrogen storage materials owing to their high hydrogen capacities. However, the high thermodynamic dehydrogenation of carbazole has restrained it for both on-board and off-board applications. In our research work, we proposed a strategy on the thermodynamic alteration by substituting the H in -NH group of carbazole with alkali or alkaline earth metals. Upon this approach, the enthalpy of dehydrogenation reduced significantly, which corresponds with the enhanced electron delocalization from the N of carbazole to aromatic rings in carbazole. Theoretical calculations also showed that metal substitution have elongated the  $\alpha$  site of C-H bond of carbazolide, resembling activation of carbazolide alongside metal substitution. The hydrogenation and dehydrogenation process of different aspects were investigated such as using various noble metal catalysts and temperatures. From the results obtained, lithium carbazolide was capable to reversibly absorb and desorb hydrogen at 473 K (7 MPa) using ruthenium and 5 wt% palladium supported on carbon, respectively. As such, it is believable that this system could be a promising liquid organic hydrogen carrier (LOHC) for hydrogen storage.

**Keywords:** Thermodynamic alteration, Carbazole, Reversible hydrogen storage

**PREPARATION AND CHARACTERIZATION OF STARCH  
GRAFTED POLYMETHACRYLAMIDE USING AMMONIUM  
PERSULPHATE INITIATOR**

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In this study, polymethacrylamide (PMAA) was successfully grafted onto starch by simple free radical polymerization technique using ammonium persulphate (APS) initiator. Reaction conditions were examined. The highest percentage grafting of 352% was found to be best at 1:4 starch:methacrylamide weight ratio, 0.4 g APS and 90°C reaction temperature. The washed copolymers were characterized by spectroscopic and thermal analyses. Fourier transform infrared spectroscopy results showed the presence of PMAA peaks. Differential scanning electroscop (DSC) analysis revealed that there is improvement in glass transition temperature (T<sub>g</sub>) of polymethacrylamide (267°C). Thermogravimetric analyses (TGA) also showed that the prepared copolymer has improved thermal stability.

**Keywords:** Starch, Methacrylamide, Free-radical Polymerization, Ammonium persulphate,  
Biomaterials

**ULTRASMALL CS-ALMCM-41 BASIC CATALYSTS: EFFECTS OF ALUMINUM ADDITION ON THEIR PHYSICO-CHEMICAL AND CATALYTIC PROPERTIES**

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In this study, ultra-small MCM-41 mesoporous solids (<30 nm) are synthesized using colloidal silica and cesium hydroxide as cheap silica source and mineralizer, respectively. Four samples with a SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio of 5, 20, 30 and ∞ (designated as CsM-5, CsM-20, CsM-30 and CsM-∞, respectively) are prepared. The effects of SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio on the formation, physico-chemical and catalytic properties of CsMCM-41 nanoparticles are studied. The results show that incorporation of high content of Al atoms into the framework reduces the ordering of the mesostructure, and hence reducing the porosity of the solids. By using CsOH mineralizer, the morphology and the size of CsMCM-41 also changed from 3-D nanoball (CsM-5: 29 nm) to 2-D nanosphere (CsM-20: 27 nm) and finally to 2-D nanosphere with hollow and very thin wall (CsM-30: 25 nm, CsM-∞: 20 nm) when Al content increased. The basicity of CsMCM-41 nanoparticles was also affected by the Al and Cs contents and hence, their catalytic behavior was studied in aldol condensation of heptanal with benzaldehyde enhanced with microwave heating. CsM-20 is the best catalyst among the samples studied with 87.2% conversion and 78.4% selective to jasminaldehyde were achieved under optimum reaction condition.

**Keywords:** Ultrasmall Cs-MCM-41, Nanoparticles, Mesoporous material, Incorporation of aluminum, Aldol condensation.

## APPLICATION ON CARBON DIOXIDE (CO<sub>2</sub>) ADSORPTION OF FERRITE BASED SORBENTS

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The synthesis of BaFe<sub>12</sub>O<sub>19</sub> and Ba<sub>2</sub>NiFe<sub>18</sub>O<sub>23</sub> prepared via sol-gel method followed by calcination at 900 °C was investigated. Both catalysts were characterized by X-Ray Diffraction (XRD), TGA, Fourier-Transform Infrared Spectroscopy (FTIR) and N<sub>2</sub> Adsorption-Desorption Isotherm. Ferrite based adsorbents is a potential sorbent due to their high adsorption capacities. XRD revealed that high calcination temperature is beneficial for the formation of catalysts. Metal oxide has been used widely as adsorbent for carbon dioxide (CO<sub>2</sub>) due its low cost, high porous structure, environmentally friendly nature and high surface area. In this study, the performance of BaFe<sub>12</sub>O<sub>19</sub> and Ba<sub>2</sub>NiFe<sub>18</sub>O<sub>23</sub> as adsorbents of CO<sub>2</sub> was investigated. During the adsorption study, it was found that Ba<sub>2</sub>NiFe<sub>18</sub>O<sub>23</sub> had the highest adsorption capacity of 10.54 mg CO<sub>2</sub> /g compared to BaFe<sub>12</sub>O<sub>19</sub> with 8.66 mg CO<sub>2</sub> /g.

**Keywords:** Metal oxide, Adsorption, Carbon Dioxide, Sol-gel.

**ADSORPTION OF 4-CHLOROPHENOL IN AQUEOUS SOLUTION BY SODIUM ALGINATE/NANOCELLULOSE BEADS**

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The increasing use of chlorophenols in chemical, petrochemical, plastic, leather, paint, pharmaceutical, and steel industries leads to environmental pollution because of their high toxicity, higher solubility and low biodegradability. Most of the industrial adsorbents are in the form of porous macro-sized particles leading to a reduction in the adsorption capacity and rate due to diffusion within the particles. Moreover, the cost of adsorption technology is mainly associated with the adsorbent, a proper adsorbent should be selected to realise an economically viable treatment. Thus, the present study exploited oil palm fronds to isolate cellulose in the preparation of sodium alginate/nanocellulose beads using calcium chloride as the ionic crosslinking agent. Synthesised beads were characterised employing FT-IR, BET, DSC, TGA, and SEM analyses. FT-IR study indicated that nanocellulose effectively bonded to sodium alginate. SEM images revealed that the prepared beads have a spherical morphology and a rough surface which permits efficient adsorption. The thermal stability of prepared samples was identified through TGA and DSC studies. N<sub>2</sub>-adsorption studies affirmed that the prepared beads have high porosity and large specific surface area. The adsorption process of formulated beads followed the Langmuir adsorption isotherm accompanying high adsorption capacities, while it was evident that the solution pH and temperature influenced the adsorption efficiency. Strong adsorbability makes sodium alginate/nanocellulose beads as potential bio-based adsorbing materials for remedy of water bodies contaminated with chlorophenols.

**Keywords:** Nanocellulose, Beads, Chlorophenols, adsorption, oil palm fronds.

## PREPARATION AND CHARACTERIZATION OF MODIFIED COCONUT HUSK LIGNIN-PHENOL-GLYOXAL (LPG) AS GREEN WOOD ADHESIVES

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In wood industries, phenolic resin has become a great concern in human health, environmental and economy issues. The most commonly resins produced are mainly from petroleum-based phenol and formaldehyde. The used of formaldehyde in phenol-formaldehyde (PF) resin has become a major concern since it is classified as toxic and carcinogenic material. As for phenol, many studies have done by using lignin as greener substitution of phenol due to its similar structure of hydroxyl groups. In this study, lignin was extracted from coconut husk (CH) by using alkaline pulping process (Kraft and soda) and organosolv pulping. The isolated lignins were characterized using complementary analyses (FTIR, <sup>1</sup>H, <sup>13</sup>C, <sup>31</sup>P, HSQC NMR, GPC, CHNS, TGA and DSC) in order to study the chemical and physical properties of lignins. For the production of green wood adhesive, glyoxal (non-toxic non-volatile) has been used as an alternative aldehyde in order to replace formaldehyde. The different concentrations of each type of lignin substitution (10 %, 30 % and 50 %) were prepared in lignin-phenol-glyoxal (LPG) resins. The resulted wood adhesives were studied for their viscosity, gel time and solid content. The performance of resulted wood adhesives were further characterized in terms of mechanical test (tensile strength) and internal bonding. In this study, Kraft-lignin-phenol-glyoxal (KLPG), soda-lignin-phenol-glyoxal (SLPG) and organosolv-lignin-phenol-glyoxal (OLPG) showed an improvement in adhesive strength compared to phenol-glyoxal (PG) and phenol-formaldehyde (PF) adhesives. In mechanical test, the PG adhesives with lignin substitution showed the higher tensile strength around 70 MPa with an increased about 20 % from the control PG (50 MPa) and PF adhesive (45 MPa).

**Keywords:** Lignin, Coconut husk, Glyoxal, Green wood adhesive.

**ENHANCEMENT OF POLY (VINYL) ALCOHOL USING  
DELAMINATED LAYERED DOUBLE HYDROXIDE FOR THE  
FORMULATION OF MECHANICALLY STRONG NANOCOMPOSITE  
HYDROGEL FOR WOUND DRESSING APPLICATION**

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A new type of nanocomposite hydrogel (NCH) has been prepared by crosslinking PVA with delaminated layered double hydroxides (LDH). The LDH was first intercalated with sodium dodecyl sulfate (SDS) to facilitate delamination. The intercalated LDH were synthesized via co-precipitation followed by subsequent hydrothermal treatment and delamination in DMSO. The mechanical properties of PVA-NCH with 0.1 wt% LDH were significantly enhanced. The tensile strength of PVA-NCH containing 0.1 wt% LDH was remarkably increased by three-fold, while the value of Young's modulus was recorded to be the highest compared to other ratios. The morphologies and microstructures of the PVA-NCH were further investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier Transform infrared (FTIR) and X-ray diffraction (XRD). TEM image showed the LDH was delaminated into single nanosheets. It was demonstrated that the delaminated LDH played an important role in increasing physical cross-linking with PVA macromolecules to form strong hydrogel through hydrogen bonding. The formulation of the strong nanocomposite hydrogel can be applied in the wound dressing application.

**Keywords:** Nanocomposite hydrogel, Polyvinyl alcohol, Layered double hydroxide, intercalation, Hydrothermal treatment, Delamination, Nanosheets, Mechanical properties.

**CHARACTERIZATIONS OF CELLULOSE NANOCRYSTAL DERIVED FROM OIL PALM FROND AND ITS APPLICATION AS A GREEN REINFORCING AGENT FOR CORROSION PROTECTION OF THE EPOXY Zn-RICH COATING ON MILD STEEL**

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In Malaysia, oil palm fronds (OPF) have contributed 70 % (w/w) of total biomass from oil palm plantation. Recently, it has been reported that OPF can be utilized to extract cellulose which has an excellent potential and functions in various commercial applications. In this study, cellulose nanocrystal (CNC) was prepared by using OPF pulp from autohydrolysis pretreatment via acid hydrolysis. The CNC produced were characterized using transform-infrared spectroscopy (FTIR), scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDX), Transmission electron microscopy (TEM), Brunauer-Emmet-Teller (BET) analysis, thermogravimetric analysis (TGA), Differential scanning calorimetry (DSC), Gel permeation chromatography (GPC), X-ray diffraction (XRD) and CP/MAS <sup>13</sup>C NMR analysis. The average length and width for the CNC obtained from TEM were 92.58 nm and 7.19 nm respectively. A series of epoxy Zn-rich nanocomposite with different CNC weight percentage were prepared as coatings on mild steel. The corrosion protection performance of prepared coatings on mild steel immersed in 3.5 % (w/w) NaCl solution were investigated by open circuit potential (OCP), electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PD) analysis. The prepared coatings also undergone salt spray for 24 hours to study the corrosion diffusion effect. The result revealed that, addition of 0.5 wt % of CNC in epoxy-Zn rich formulation is an optimum weight based on IE % obtained, which was 99 %. This proved that, introduction of CNC into the epoxy-Zn rich formulation help to enhance the coating to give better corrosion protection for mild steel.

**Keywords:** Oil palm frond, Cellulose Nanocrystal, Epoxy-Zn rich, Coating, Corrosion resistance

**PHYSICAL AND CHEMICAL MODIFICATION ON NATURAL RUBBER/POLY-3-HYDROXYBUTYRATE BLENDS BY GRAFTING METHOD**

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In the present study, the biopolymer blend of natural rubber (NR) and poly-3-hydroxybutyrate (PHB) was performed using solvent casting technique. However, the interfacial adhesion between NR and PHB blend was poor. To improve these systems, reactive blending such as grafting of NR with PHB was utilized using benzoyl peroxide as radical initiator. The grafting reaction was characterized in depth by ID and 2D Nuclear Magnetic Resonance (NMR) and further confirmed using Fourier transform infrared spectroscopy (FTIR). The new and additional peak observed in the NMR spectrum supports the grafting reaction between NR and PHB. The interfacial adhesion of the grafted polymers was enhanced in comparison with the blended polymers. This can be proven by Differential Scanning Calorimetry (DSC) analysis, when a single glass transition temperature (T<sub>g</sub>) value was obtained compared to two T<sub>g</sub> values for NR/PHB blend. In addition, the thermal stability between NR/PHB blending and grafting were investigated using thermogravimetric analysis (TGA). Both systems show that the thermal stability was improved when compared to PHB and NR alone.

**Keywords:** Biopolymer blend, Grafting, Interfacial adhesion, Natural rubber, Poly-3-hydroxybutyrate (PHB).

**CO- AND NI-CATALYZED DEHYDROGENATION OF HYDRAZINE BORANE FOR HYDROGEN STORAGE**

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Hydrazine borane (denoted as HB,  $N_2H_4BH_3$ ), having hydrogen content as high as 15.4 wt%, which is greater than the 2025 target of US DOE has been considered as promising hydrogen storage material. Despite of its high  $H_2$  capacity, its thermolytic decomposition is an exothermic process and it releases unwanted byproducts such as borazine ( $N_2H_4$ ) and ammonia ( $NH_3$ ), which in turn cause adverse effect to the fuel cell catalyst. Moreover, HB melts prior to its dehydrogenation, resulting in serious sample foaming. Finding a reactive and selective catalyst to improve the dehydrogenation properties is thus crucial. A “co-precipitation” method was applied to introduce Co- and Ni-based catalysts to HB. The synthesized Co- and Ni-catalysed HB were then characterized by TGA, DSC, XRD, XPS, TPD, TPD-MS, FT-IR, NMR and TEM. Our preliminary results showed collective improvements in the catalyzed dehydrogenation process, such as lowered dehydrogenation temperatures, enhanced rate of dehydrogenation, suppression of sample foaming and by-products formation.

**Keywords:** Chemical hydrogen storage, Hydrazine borane, Catalysts, Thermolysis

## INFLUENCE OF CATALYST SUPPORT ON THE HYDROGENATION OF 9-ETHYLCARBAZOLE

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In this piece of research, the catalytic performances on the hydrogenation reaction of organic hydride, 9-ethylcarbazole were studied. Ruthenium metal was used as the main catalyst with titanium dioxide as a support. The hydrogenation of 9-ethylcarbazole via Ru/TiO<sub>2</sub> showed good hydrogen intake despite lower rates of reaction as it required longer time to complete. The modification of supports with difference in the ratios of zinc oxide to titanium dioxide, Ru/Zn<sub>(x)</sub>Ti<sub>(1-x)</sub>O<sub>2</sub> were introduced to this research to evaluate the possible improvement in the rate of hydrogenation reactions. The supports were synthesized using sol-gel method and were dispersed with ruthenium metal with the addition of sodium borohydride as the reducing agent. The characterization of the synthesized catalysts using UV-Vis, XRD, TEM, and BET were applied. The hydrogenation reactions of 9-ethylcarbazole were then performed via Ru/TiO<sub>2</sub> catalyst and in comparison with the modified supports, Ru/Zn<sub>(x)</sub>Ti<sub>(1-x)</sub>O<sub>2</sub> to study the catalytic performances. The preliminary results showed that the modification in supports with zinc oxide gave a positive trend in the rate of reaction. The kinetics of hydrogen absorption on 9-ethylcarbazole were greatly improved as the amount of zinc oxide in support increased while showing depletion in the hydrogen storage capacity.

**Keywords:** Hydrogen storage, Catalytic hydrogenation, 9-ethylcarbazole, Ruthenium catalyst.

## **BIOPLASTICS CELLULOSE FILMS IN THE PREPARATION AND PROPERTIES FOR SUSTAINABLE DEVELOPMENT MATERIALS**

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Bioplastic cellulose is an attractive material to alternatively conventional petroleum-based plastics. Currently, the researchers are in progress to bioplastics using as sourced corn starch and sugarcane starch. As sustainable issue, it would be resourceful to use cellulose or hemicellulose extracted from waste biomasses which are not competitive with edible food. Cellulose is environmentally friendly polymer which is renewable and advantageous in high strength and biodegradability. Also, non-allergy to human is on favorable properties for several applications. The objective of this study is to prepare bioplastic cellulose films from the celluloses, extracted from plant wastes and cotton without chemical modification. Here, celluloses were extracted from oil palm empty fruit bunch (OPEFB) and oil palm frond (OPF). Both OPEFB and OPF are abundant in Malaysia and Indonesia and facing waste management problem at the present time. Therefore, this work focuses on the application of bioplastic celluloses, sourced from OPEFB and OPF, respectively. First, 1wt% of cellulose solutions from each cellulose sources were prepared using DMAc/ 6wt% LiCl solution according our previous reports. Then, the respective cellulose hydrogels were prepared using phase separation gelation in ethanol atmosphere at room temperature. Finally, the obtained cellulose hydrogels were compressed by hot press to be dried cellulose films under 1 kg/cm<sup>2</sup> pressure at 80-150 °C. The resultantly dried films were transparency with crystallinity. The tensile strength and water swelling of the cellulose films were compared at different compressing temperatures and times for the resultant cellulose films. Cellulose films with about 16µm thickness were obtained at 100°C and obtained high tensile strength of 34, 46 and 55 N/cm<sup>2</sup> for their samples compressively heated at 80, 100 and 150°C, respectively. The crystallinity of the films increased with the increase of the heating temperature. But, decreasing the water swelling property of the films was observed, as the crystallinity increased.

**Keywords:** Bioplastic, Biomass resource, Cellulose, Regeneration

**PREPARATION OF HALLOYSITE NANOTUBES (HNT)-SUPPORTED LANTHANUM AND THEIR APPLICATIONS FOR THE CATALYTIC DEGRADATION OF CHITOSAN (CS)**

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The catalytic degradation of chitosan (CS) using halloysite nanotubes-supported lanthanum (HNT-La) has been studied. The HNT-La catalysts with different amounts of La (4.3, 5.6 and 16.9 wt. %) were prepared and the catalytic degradation of CS was then carried out under different reaction conditions, i.e. amount of La, degradation time and temperature. The catalysts and the degraded CS samples were then characterized by X-ray diffraction (XRD), photoluminescence (PL) spectroscopy, scanning electron microscopy coupled with energy dispersive X-ray spectrometry (SEM/EDX), Fourier Transform Infrared (FT-IR) spectroscopy, viscometry measurements, thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). FT-IR showed that the degraded CS samples retained their structures, however their degree of deacetylation (DD) increases with La amount and degradation time due to cleavage of peptide-like acetoamido (NHCOCH<sub>3</sub>) groups into free amine groups and loss of crystallinity. Similarly, the viscosities and calculated average molecular weight,  $M_v$ , also showed similar trends as a result of chain scission of the CS backbone. TGA results revealed that the degraded CS samples underwent degradation more readily than CS due to their lower thermal stabilities than CS. The appearance of the glass transition temperatures,  $T_g$  of the degraded CS samples at very low temperatures in the DSC curves indicated their low molecular weight and low DD.

**Keywords:** Chitosan, halloysite nanotubes, Lanthanum, Degradation, Molecular weight, Degree of deacetylation.

**IMPACT STRENGTH, WATER ABSORPTION AND MECHANICAL PROPERTIES OF HDPE-WASTE FILLED WITH BAMBARA NUT SHELL POWDER AND EGGSHELL POWDER (BNSP/ESP)**

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Over the past years, bio-fillers are used in the production of biodegradable polymer composites that can replace the non-biodegradable synthetic polymers that poses serious threat to the environment. In this work, Bambara nut shell powder (BNSP) and eggshell powder (ESP) were incorporated to reinforce high-density polyethylene (HDPE)-waste, resulting in the formation of a biodegradable hybrid composite. The impact strength, water absorption and mechanical properties of the neat HDPE-waste and the hybrid composite of the HDPE-waste/BNSP/ESP were then investigated. Characterizations of the composites were accomplished using Fourier transformed infrared spectroscopy (FT-IR), Differential scanning calorimetry (DSC) and Thermogravimetric analysis (TGA). The addition of these bio-fillers showed a significant increase in the impact strength, water absorption, elongation at break and flexural modulus of the hybrid composite compared to the neat HDPE-waste. The two bio-fillers were successfully used as reinforcement for the HDPE-waste and could offer new opportunity in the production of new materials for advanced application.

**Keywords:** HDPE-waste, Bambara nut shell powder, Eggshell powder, Bio-filler, Hybrid composite, Mechanical properties.

**DEGRADATION OF METHYLENE BLUE USING BIOSYNTHEZED  
ZINC OXIDE PHOTOCATALYST:  
EFFECT OF SYNTHESIS PARAMETERS**

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Biological reducing agents are being explored extensively to minimize the effects of toxic chemicals used in the fabrication of nanoparticles. The green synthesis route is found to be simple, cost-effective, energy-saving, environment-friendly, less time consuming, and does not require any hazardous chemicals. The present study reports a green approach to synthesize zinc oxide (ZnO) nanoparticles by using banana peel (BP) extract as reducing and capping agents. The aim of the study is to optimize the synthesis parameters involved by evaluating the photocatalytic degradation of methylene blue (MB) under visible light irradiation. The effect of pH was first investigated. The synthesized nanoparticles were characterized and the XRD patterns showed that the purity of the synthesized ZnO nanoparticles were dependent on the pH of the synthesis conditions. The average crystal sizes of the synthesized nanoparticles determined by the Debye-Scherrer equation were in the range of 30-80 nm. The surface morphologies observed using SEM also revealed different ZnO morphologies depending on the pH of the synthesis conditions. The significance of other synthesis parameters such as the amount of the BP extract, concentration of zinc acetate dihydrate, reaction time, and temperature were also investigated and optimized. According to the results, the optimum conditions for the synthesis of ZnO nanoparticles were pH=12, 1 mL BP extract, 0.02 M zinc acetate dihydrate, 3 hours reaction time, and temperature 60 °C. The synthesized ZnO nanoparticles exhibited good photocatalytic activity on the degradation of MB. Hence, a simple and effective green approach for synthesis of ZnO nanoparticles with an excellent photocatalytic performance is reported in this study.

**Keywords:** Green synthesis, Zinc oxide nanoparticles, Photocatalytic degradation, Methylene Blue.

**A NEW ROUTE FOR THE PREPARATION OF LIQUID EPOXIDIZED  
NATURAL RUBBER (LENR)**

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A new route of the production of Liquid Epoxidized Natural Rubber (LENR) was effectively performed in the presence of metal supported on halloysite nanotubes (HNT) catalyst in depolymerization of Epoxidized Natural Rubber (ENR). The effect of different metal salt supported on HNT was investigated. As much as 5 wt % of nickel (II) and copper (II) were suspended on HNT for 4 hours before drying in an oven overnight. The characteristic of each catalyst was investigated using Scanning electron microscopy (SEM), Energy Dispersive X-ray (EDX), photo-luminescence (PL) and Fourier-transform Infrared Spectroscopy (FTIR). FTIR results show that the metal ion was physically mixed with HNT. Each catalyst was used to study the catalytic depolymerization of ENR at 200 °C for 4 hours of reaction time. The catalytic depolymerization activity was determined by gel permeation chromatography (GPC). Results show a reduction of average molecular weight ( $M_w$ ) from 284,113 of purified ENR to 19,419.5 in the presence of 0.1 g  $Cu^{2+}$ /HNT is the best among all catalysts. The structure of the depolymerized product and the percentage of epoxide ring opening were further investigated using Nuclear Magnetic Resonance spectroscopies (NMR) and FTIR.

**Keywords:** Liquid Epoxidized Natural Rubber (LENR), Ni salt, Cu salt, Halloysite Nanotubes (HNT), Epoxidized Natural Rubber (ENR-50)

**ENHANCING ANTICORROSION PROPERTIES AND WATER REPELLENCY OF A TANNIN DOPED HYBRID SOL-GEL COATING ON MILD STEEL IN 0.5 M HCL MEDIUM**

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This study, proposed an eco-friendly sol-gel coating on mild steel surfaces with a double property of corrosion resistance and hydrophobicity in 0.5 M HCl. The featured protective tannin methanol extract (TME) and 70% acetone extract (TAE) doped hybrid sol-gel coating is prepared using tetraethoxysilane (TEOS) and 3-glycidoxypropyltrimethoxysilane (GPTMS) as organic-inorganic precursors. Understanding into the protection mechanisms of these hybrids doped coatings on the mild steel electrodes has been described using electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization measurements (PD). The hydrophobicity was determined *via* water contact angle meanwhile the coating and surface morphology were determined by using scanning electron microscopy. Result from electrochemical measurements revealed that the mixture of tannin doped sol-gel coating resulted in the highest corrosion resistance (IE = 93%). The higher protective property of this coating matrix is related to high water contact angle ( $\theta = 104^\circ$ ) values. SEM analyses of surface morphology revealed continued protection of corrosion with better performance obtained for the coating matrices due to their effective film possessions against the diffusion of destructive species as well as the hydrophobic nature of the doped sol-gel matrix. These result built-up a new outlook for the design and carrying out of innovative coatings in applications of high performance.

**Keywords:** Corrosion, Tannins, Sol-gel, SEM, Hydrophobic.

**CHEMICAL SCREENING OF AGARWOOD OIL SAMPLES  
AVAILABLE IN CAPITAL OF MALAYSIA MARKET: AN  
IMPORTANT ASPECT IN THE AUTHENTICATION PROCESS  
AND QUALITY**

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Agarwood oil is well known as a luxury source of essential oil that is widely applied as a main ingredient in perfumery industry. In fact, agarwood has been long consumed during traditional ceremony and even in medicinal purpose due to its effective therapeutic characteristic. Considering agarwood as an endangered species with exceptionally high price, adulteration of agarwood oil practice is the best option since sometimes ago. The authenticity of agarwood oil becomes the biggest concern from commercial value aspect and health consideration. The study of agarwood composition and formation has been increasing for the purpose of its better quality. Yet, no standard method for agarwood oil classification has been amended which is considered as one of global issues. Current work focuses on studying the quality of various types of agarwood oil sold in market in capital of Malaysia, Kuala Lumpur. For this purpose, chemical composition was analyzed by using chromatography analysis. Comparison of the oils was also done with the pure agarwood oil extracted in the laboratory and oil from local manufacturer agarwood industry. Discover in this study, compounds dihydrokaranone, guaia-1(10),11-dien-15-oic acid, p-vinylguaiacol, p-vinylphenol, selina-3,11-dien-14-oic acid and sinenofuranol were only found in market samples while p-methylanisol, 1,5-diphenyl-3-pentanone, 2-hydroxyguaia-1(10),11-dien-oic acid, eudesmol, guaia-1(10),11-dien-9-one and pentadecic acid were only identified in local trader samples.

**Keywords:** Agarwood oil, Gas chromatography, Chromatogram, hydrodistillation.

**PRODUCTION OF OIL PALM (ELAEIS GUINEENSIS) FRONDS  
LIGNIN DERIVED NON-TOXIC ALDEHYDE FOR ECO-FRIENDLY  
WOOD ADHESIVE**

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Lignocellulosic materials can significantly contribute to the development of eco-friendly wood adhesives. In this work, glyoxal-phenolic resins for plywood were prepared using organosolv lignin, which was isolated from black liquor recovered from organosolv pulping of oil palm fronds (OPF) and considered to be an alternative to phenol. Glyoxal, which is a dialdehyde obtained from several natural resources, was used as substitute for formaldehyde. The structure of organosolv lignin and the resins were characterized by FTIR and NMR, and for thermal stability by TGA and DSC. The resins were further studied for their viscosity, pH, solids content and gel times. The resins performance as wood adhesive was further established from mechanical test in terms of tensile strength and modulus of elasticity (MOE) to obtain the optimum ratios of organosolv lignin, which replaces phenol in organosolv lignin phenol glyoxal (OLPG) resins. The adhesive composition having 50% (w/w) of phenol substituted by organosolv lignin, termed as 50% OLPG showed highest adhesive strength compared to phenol formaldehyde (PF) commercial adhesive.

**Keywords:** Oil palm fronds, Organosolv lignin, Glyoxal, Wood adhesive.

**SOLID STATE SYNTHESIS AND CHARACTERIZATION OF Cu(II)  
AND Ni(II) POLYNUCLEAR METAL COMPLEXES WITH LIGAND  
DERIVED FROM 3-METHOXY-4-HYDROXY BENZALDEHYDE AND  
L-LEUCINE**

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Two polynuclear metal complexes with general formula  $[M^{II}_3L_4(H_2O)_9].4.5H_2O$  and  $[M^{II}_3L_3(H_2O)_9].5H_2O$  for Cu(II) and Ni(II) ions respectively, have been synthesized via the one step solid state synthesis with ligand derived from 3-methoxy-4-hydroxy benzaldehyde and L-Leucine. The compounds were characterized by elemental analysis, ESI-Mass spectrometry, infrared spectroscopy, thermogravimetric analysis (TGA) and conductivity measurements. Data from experimental results showed that the ligand binds to two metal centers in a Tridentate (O N O) manner through the imine nitrogen, phenolic and carboxylic oxygen respectively. Antimicrobial activities of the ligand and the coordination polymers were investigated and found active against some selected bacterial and fungal species.

**Keywords:** Solid–solid reaction, Polynuclear metal complexes, Mass spectrometry, Thermal decomposition, Antimicrobial activity.

**SYNTHESIS AND CHARACTERIZATION OF MoO<sub>x</sub> THIN FILM ON  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> (0001) SINGLE CRYSTAL****<sup>1,2</sup>Ibrahim Garba Wawata, <sup>2,3</sup>Michael Bowker**

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Catalysts surface remain a crucial arena in catalysis, where the interplay between reactants molecules in the presence of desired active sites results in selective products in most industrial processes. However, industrial heterogeneous based catalysts are difficult to characterize after post reaction using modern spectroscopic techniques, due to surface alteration and contamination. These have been concern of scientists in the field of catalysis, surface science and reaction engineering, leading to the emergence of model catalysis. In this study, Mo oxides thin films were synthesized on single crystal Alumina ( $\alpha$  Al<sub>2</sub>O<sub>3</sub>-0001) via low cost wet chemical deposition method. These catalysts were characterized using Light microscopy, Atomic force microscopy (AFM), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS) and X-Ray diffraction (XRD) analysis. The MoO<sub>3</sub> films revealing inhomogeneous crystalline MoO<sub>3</sub> islands with varying sizes and shapes at different coverage, which is concentration and thickness dependant. However, AFM analysis reveals the films consisting of large and small crystalline island of thickness between 2 – 10  $\mu$ , and 60 – 250 nm at low coverage. In addition, Raman spectroscopic studies indicates formation of crystalline polymeric MoO<sub>3</sub> species at high coverage, with presence of both dioxo (O=)<sub>2</sub> MoO<sub>2</sub> and polymeric MoO<sub>3</sub> species at low coverage. XPS analyses of the films confirm the presence of Mo 3d and Al 2p in their high oxidation states, for films calcined at 500 °C. Moreover, the XRD reveal phases attributed to Al<sub>2</sub>O<sub>3</sub> 0001 and Mo oxides phases. These model catalysts will found application in operando and in-situ monitoring of selective oxidation reactions.

**Keywords:** Heterogeneous, Catalysis, Thin Film, MoO<sub>x</sub>, Wet chemical deposition

**SYNTHESIS AND CHARACTERIZATION OF 7-HYDROXYCOUMARIN CONTAINING NEW TYPE OF PHTHALOCYANINE COMPOUNDS AND INVESTIGATION OF THERMAL AND ELECTROCHEMICAL PROPERTIES**

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In this research, phthalonitrile was synthesized from the reaction of 7-hydroxycoumarin with 3-nitrophthalonitrile in DMF. Cyclotetramerization of phthalonitrile in the presence of metal salts, Ni(II), Co(II), Zn(II), Cu(II), Fe(II)) gave peripherally substituted metal phthalocyanine complexes. The structures of the synthesized compounds were characterized by a combination of microanalysis, <sup>1</sup>H NMR, UV-Vis, and FT-IR spectrophotometry. Solubility of metal phthalocyanine complexes was found to be very low in common laboratory solvent. The electronic spectrum of NiPc, CuPc, and ZnPc in DMF, showing intense Q absorption at 690 nm. The Q band absorptions were observed at 678 nm, for CoPc, and FePc. The thermal stability of the phthalocyanine derivatives was checked by T.G.A. It was, therefore, concluded that the metal phthalocyanines prepared in this study showed suitably high thermal stability and can be used for various applications.

**Keywords:** Phthalocyanine, 3-Phthalonitrile, electrochemistry, Thermal analysis.

**EFFECT OF LIGHT IRRADIATION TYPE ON PHOTOCATALYTIC DEGRADATION OF 2-CHLOROPHENOL USING MESOPOROUS TITANIA NANOPARTICLES**

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In this research, remarkable impact of varied light irradiation was studied on photocatalytic degradation of 2-chlorophenol (2-CP) using mesoporous titania nanoparticles (MTN) which was prepared via microwave-assisted method. The reaction was carried out for 150 min under UV-A, UV-C, sunlight and visible light using a batch reactor. The catalyst was characterized by XRD, UV-Vis DRS, BET, FESEM and HRTEM. The photodegradation of 2-CP under sunlight has exhibited the best performance with 100% followed by UV-A, visible light and UV-C for degradation of 96%, 52% and 45%, respectively. This indicated that even the band gap of MTN (3.2 eV) remains unchanged after surface modification, but the catalyst was succeeded to be activated under visible light region with significant degradation due to abundant of sites defect which highly contributed in light-absorption modification.

**Keywords:** Light irradiation, Photocatalytic degradation, Mesoporous titania nanoparticles, 2-chlorophenol.

**SYNTHESIS OF COPPER-BASED METAL ORGANIC FRAMEWORK  
INCORPORATED WITH GRAPHENE FOR NON-ENZYMATIC  
DETECTION OF H<sub>2</sub>O<sub>2</sub>**

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This work reports on the synthesis of a copper-based metal-organic framework (Cu-MOF) incorporated with graphene oxide (Cu-BTC-MOF/GO) (BTC=1,3,5-benzene-tricarboxylate and GO=graphene oxide) using the reflux method. The results of X-ray diffraction and Fourier transform infrared spectroscopy confirmed the formation of Cu-BTC-MOF in the presence of GO nanosheets through the reflux process. The modification of indium tin oxide (ITO) electrode was carried out by drop-casting of Cu-BTC-MOF/GO composite and electrochemical reduction of GO to electrochemically reduce graphene oxide (ErGO). Owing to the synergetic effects of Cu-BTC-MOF and graphene, the Cu-BTC-MOF/ErGO/ITO electrode exhibited notable electrocatalytic activity for the reduction of H<sub>2</sub>O<sub>2</sub>, leading to a non-enzymatic electrochemical sensor.

**Keywords:** Metal organic framework, Graphene, Hydrogen peroxide, Electrochemical sensor

**SYNTHESIS AND CHARACTERIZATION OF ZnO/LTO FOR THE  
REMOVAL OF METHYLENE BLUE UNDER VISIBLE LIGHT  
IRRADIATION**

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Lithium titanates (LTO) prepared *via* hydrothermal method was impregnated with different percentage weight of ZnO via wetness impregnation. The catalysts were labeled as  $x$ ZnO/LTO ( $x = 1$  wt%, 3wt% and 5wt %). The catalysts were applied in the photocatalytic degradation of MB under visible light irradiation. The catalysts exhibited type IV isotherm with H4 hysteresis loop. The SEM and HRTEM images show the presence of cubic crystals of ZnO on the surface of LTO. The band gap energy of the catalysts reduced to the range of 3.04-3.07 eV after impregnation. For 10 ppm MB solution, the highest removal of MB (95.30%) was achieved using 0.075g 3wt% ZnO/LTO at pH 6. Efficient removal of total organic carbon can be observed for the first 3 hours but eventually, the concentration increased slightly at the end of the reaction. In the presence of ZnO, the separation of the catalyst after reaction improved and the adsorbed MB can be easily removed using distilled water and acetone. The catalyst was recycled 3 times without significant loss in its photocatalytic ability. Based on the scavenger studies, the most reactive species that involve in the reaction was hydroxyl radicals followed by electrons and holes.

**Keywords:** ZnO/LTO, visible light, Methylene blue, Hydrothermal, Wetness impregnation, Photocatalytic degradation

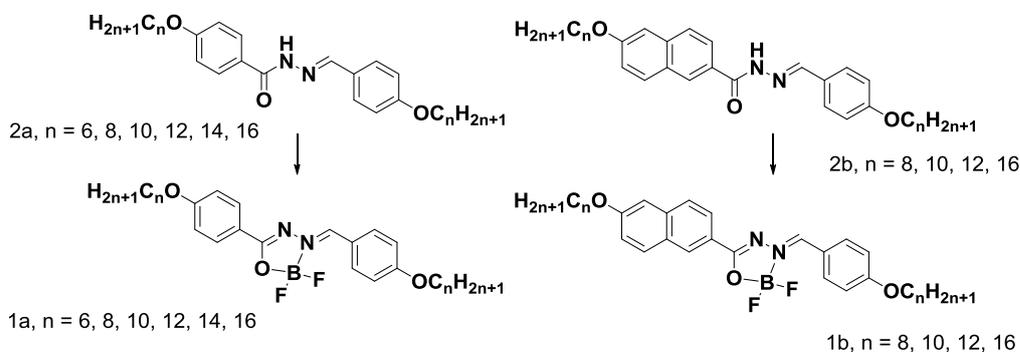
## ROD-LIKE MESOGENIC BORON DIFLUORIDE COMPLEXES DERIVED FROM SCHIFF BASES

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In recent years, boron difluoride (BF<sub>2</sub>) complexes have been intensively investigated to explore their applications as potential materials used in diverse research fields. Many boron complexes with novel structures have been reported and their optical properties were also investigated. Most of boron complexes have a coordination number of three or four, and three types of coordination modes containing [N,O], [O,O], [N,N], have long been known for boron difluoride. Among these structures, [N,O]-BF<sub>2</sub> are the most studied since they show novel photophysical and chemical properties. In this work, one new series of boron difluoride complexes **1a** and **1b** derived from enamine **2a** and **2b** was reported. All compounds were characterized by <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy, elementary analysis, polarized optical microscope, and differential scanning calorimeter and X-ray diffraction methods. Both **2a** and **2b** were not mesogenic. In contrast, both **1a** and **1b** exhibited smectic and/or nematic mesogenic properties.



**Keywords:** Liquid crystal, Smectic, Nematic phases, Borondifluoride, Schiff bases

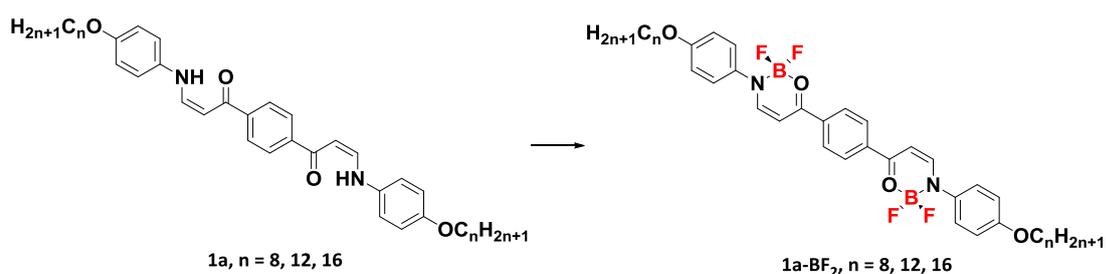
## ROD-LIKE MESOGENIC BIS (BORONDIFLUORIDE) COMPLEXES DERIVED FROM $\beta$ -ENAMINOKETONES

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Complexes or adducts containing borondifluoride ( $\text{BF}_2$ ) have received much attention during the past decades. Most of these known  $\text{BF}_2$  complexes were [O,O]-chelated and less common were [N,O]-coordinated. In contrast, rarer complexes coordinated to [N,N]-chelating ligands, such as  $\beta$ -diketimines and formazanates were known. Even thus, only a few  $\text{BF}_2$  complexes were known to complex with N,O-chelating ligands, such as quinolone-b-ketones. These  $\text{BF}_2$ -[N,O] complexes often showed interesting photophysical and photochemical properties, which might have applications in OLED, OFET, LCs and others. In this work, a new series of bis(borondifluoride) complexes **1a-BF<sub>2</sub>** derived from  $\beta$ -enaminoketones **1a** was prepared, characterized and studied. All compounds were characterized by  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopy, elementary analysis, polarized optical microscope, differential scanning calorimeter and their structures confirmed by X-ray diffraction methods. Both **1a** and **1a-BF<sub>2</sub>** show smectic phases.



**Keywords:** liquid crystal, borondifluoride,  $\beta$ -enaminoketones, smectic phases.

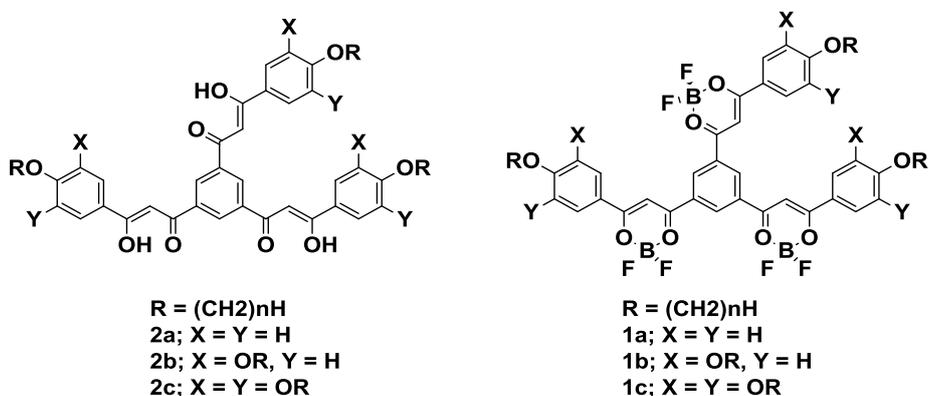
## FLUORESCENT MESOGENIC TRIS (BORONDIFLUORIDE) COMPLEXES DERIVED FROM HEXAKETONATES

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During the past years, borondifluoride ( $\text{BF}_2$ ) complexes have been continuously reported. Many known borondifluoride complexes as fluorescent materials applied in many areas; such as biological imaging, molecular probes, electroluminescent devices, photosensitizers and others were prepared and investigated. However, among them only a few examples were truly mesogenic. Most of these mesogenic mono- and bis(borondifluoride) complexes. Nevertheless tris(boron difluoride) mesogen has never been reported. In this work, a new series of columnar borondifluoride complexes, containing a central 1,3,5-benzene core substituted with three  $\beta$ -diketonate with three, six and nine terminal alkoxy groups and is described. All compounds were characterized by  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopy, polarized optical microscope (POM) and differential scanning calorimeter (DSC), and their structures were confirmed by X-ray diffraction (XRD) methods. Both compounds **2b** and **1b-c** formed hexagonal columnar phases.



**Keywords:** Liquid crystal, Diketonate, Columnar phases, Borondifluoride.

# IRON OXIDE BASED CATALYST DOPED WITH TRANSITION METAL FOR OXIDATIVE DESULFURIZATION OF MODEL DIESEL FUEL

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The catalytic oxidative desulfurization (Cat-ODS) process has been introduced as a new technology to achieve ultra-low sulphur levels in diesel fuels. The alumina supported iron base catalysts doped with cobalt were prepared by wet impregnation method. In this study, the performance of the alumina supported iron base catalyst was investigated in Cat-ODS of model diesel. The desulfurization was investigated using tert-butyl hydroperoxide (TBHP) as an oxidizing agent and N, N-dimethylformamide as an extraction solvent. By using other metals such as Co, Cu and Ni in the Cat-ODS, some efforts have been made to improve the Fe/Al<sub>2</sub>O<sub>3</sub> based catalyst. The results illustrated the catalytic activity of alumina supported Fe catalyst decreased in the order of: Co/Fe-Al<sub>2</sub>O<sub>3</sub> > Ni/Fe-Al<sub>2</sub>O<sub>3</sub> > Cu/Fe-Al<sub>2</sub>O<sub>3</sub>. Thus, Co was considered as the best dopant for the Iron-based catalyst. Next, different dopant ratio of Co doped with Fe based, Co/Fe-Al<sub>2</sub>O<sub>3</sub> (90:10) and Co/Fe-Al<sub>2</sub>O<sub>3</sub> (80:20) were prepared at 400°C. The catalytic activity decrease as dopant ratio increase. Catalyst with 10 wt% of Co successfully removed 96% of thiophene, 100% of DBT and 92% of 4,6-DMDBT in model diesel compared to the 20 wt% of cobalt that only removes 85% of thiophene, 80% of DBT and 68% of 4,6-DMDBT. Further investigation, catalyst that calcined at 400°C give higher sulphur removal compared to the catalyst calcined at 500°C. X-ray diffraction analysis (XRD) result showed that Fe/Co-Al<sub>2</sub>O<sub>3</sub> (90:10) prepared at 400°C was amorphous, while micrograph of the field emission scanning electron microscopy (FESEM) illustrated an inhomogeneous distribution of various particle sizes. The energy dispersive X-ray analysis (EDX) result has confirmed the presence of Fe and Co in all of prepared catalysts.

**Keywords:** Catalytic oxidative desulfurization, Iron oxide, Diesel

INO-011

# METAKAOLIN-BASED GEOPOLYMER MADE OF AMMONIA ACTIVATOR USING MICROWAVE CURE

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Geopolymer is a new material having silica-alumina framework and has excellent engineering properties and eco-friendly processing in the fabrication. Recently, geopolymer attracts a lot of attention in sustainable issues. So, there are many researchers who have reported geopolymers for alternative cement materials and adsorbents as well as similar zeolite. Thus, geopolymer is applied for many kinds of practical uses in chemical fields. As disadvantage, geopolymer normally needs to use high concentration of NaOH solution for geopolymerization as an alkaline activator and tends to remain unreacted form in the matrix. Thus if unreacted NaOH remains such excess base is dissolvable for water from geopolymer bulk and then the activator easily remains porous voids. Therefore, it caused decreasing physical strength of geopolymer. So, we have tried to search new alkali activator which replaces with NaOH. In the present work, geopolymer was prepared by using ammonia activator instead of NaOH because gaseous or liquid alkaline is easy to remove after geopolymerization. In experiments, metakaolin, ammonia and sodium silicate were used as raw materials and alkaline activator mixed the weight of sodium silicate to ammonia as fixed ratio of 1. The solid to liquid weight ratio was 0.45 for geopolymer. And then, it was mixed well for 10 min. Then, geopolymer paste cast in 35×35×35 cm<sup>3</sup> mold and cured by microwave for 5min at 200, 500 700W and 24h at 80°C in heating oven that microwaving method could be shorter in the curing time than the oven.[1] So the geopolymer cured at 500-700W was good mechanical strength in compressive strength. In contrast, the samples cured at 80°C and 200W were obtained very weak strength as compared with those of the 500 and 700W cases. So higher power of the microwave affected increasing mechanical strength in ammonia activator geopolymer.

**Keywords:** Geopolymer, Metakaolin, Ammonia activator, Microwave

INO-012

# COMPARATIVE STUDIES OF ELECTROPLATED NICKEL SULPHIDE FILM ON INDIUM-TIN OXIDE AND COPPER SUBSTRATES

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This work presented comparative studies of electroplated nickel sulphide (Ni-S) film on indium tin oxide (ITO) and copper substrate. The Ni-S film was galvanostatically (~5 mA) deposited on the substrates. The Ni-S film was revealed to have a granular-like morphology, which homogeneously deposited on the substrates. Cyclic voltammetry was used to evaluate the stability of the Ni-S film under redox conditions. In general, Ni-S/ITO sample exhibited excellent electrochemical stability throughout various scan rates and repeated cycles (~ 50); while on Ni-S/Cu the onset of Cu<sup>2+</sup>/Cu redox peaks were observed aside from that of [Fe(CN)<sub>6</sub>]<sup>3-/4-</sup>. It should be noted that the redox peaks for Cu<sup>2+</sup>/Cu were significantly small in magnitude compare to that of [Fe(CN)<sub>6</sub>]<sup>3-/4-</sup>, thus indicating its potential role as a corrosion resistant layer.

**Keywords:** Nickel Sulphide, Electroplating, Cyclic voltammetry

INO-013

# DINUCLEAR SILVER (I) COMPLEXES BEARING BIS- AND TETRA- N-HETEROCYCLIC CARBENE LIGAND: SYNTHESIS, CRYSTAL ANALYSES AND ANTIBACTERIAL ACTIVITIES

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The synthesis, characterization, crystal analyses and antibacterial studies of four bisbenzimidazolium salts (1-4) and four tetrabenzimidazolium salts (5-8) as *N*-heterocyclic carbene (NHC) precursors to their dinuclear Ag(I) complexes (Ag1-Ag8) were reported. Salts 1-4 were synthesized through simple addition reaction between *n*-alkylbenzimidazole (alkyl = ethyl, propyl, butyl, benzyl) with 1,2-dibromoethane in 2:1 molar ratio. On the other hand, salts 5-8 were synthesized through the reaction of 3-(2-bromoethyl)-1-alkylbenzimidazole bromide (alkyl = ethyl, propyl, butyl, benzyl) with 1,2-butylbisbenzimidazole in 2:1 ratio. Furthermore, Ag1-Ag8 were synthesized *via in-situ* deprotonation of respective salts with Ag<sub>2</sub>O. The syntheses of all compounds were proved by elemental analyses, FTIR, and <sup>1</sup>H- and <sup>13</sup>C-NMR. As expected, Ag1-Ag4 present as dinuclear Ag(I)-NHC complexes (L<sub>2</sub>Ag<sub>2</sub>·2PF<sub>6</sub>) after undergo metalation with molar ratio of salt:Ag<sub>2</sub>O is 2:1. Surprisingly, Ag5-Ag8 did not present as tetranuclear Ag(I)-NHC complexes as expected even after undergo metalation with molar ratio of salt:Ag<sub>2</sub>O, 4:1, but as dinuclear Ag(I)-NHC complexes with general formula of LAg<sub>2</sub>·2PF<sub>6</sub>. The crystal study of Ag4 and Ag7 using X-ray crystallography study proved the formation of two different structures of dinuclear Ag(I)-NHC complexes. All the benzimidazolium 1-8 showed no antibacterial activities while all Ag(I)-NHC Ag1-Ag8 show medium activities against *E. coli* (ATCC 25922) and *S. aureus* (ATCC 12600) compared to the standard antibiotic drug, Amoxicillin.

**Keywords:** Bisbenzimidazolium, Tetrabenzimidazolium, Ag(I)-NHC complexes, Antibacterial.

**EFFECT OF MALEIC ANHYDRIDE MODIFICATION OF  
POLY(LACTIC ACID) ON THE TENSILE PROPERTIES AND  
DIMENSIONAL STABILITY OF KENAF FIBER BIOCOMPOSITE.**

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The present work reported on the fabrication of the Maleic Anhydride (MA) modified Poly(lactic acid)(PLA)-based biocomposite reinforced kenaf fiber through melt-blending and compression molding approaches, and probe their performances in terms of dimensional stability and tensile properties. The results clearly showed that tensile properties of the pure PLA appeared to be slightly higher compared to that of MA-modified PLA. Perhaps, due to the hydrophilic behavior of the MA, thus making the MA modified PLA to absorb more moisture and swell up compared to the pure PLA. The kenaf incorporated unmodified PLA biocomposite recorded the least tensile properties as well as poorest dimensional stability, possibly due to poor interfacial adhesion between the kenaf fiber and the PLA. A considerable rise in the tensile properties were noted, and enhancement in the dimensional stability upon incorporating kenaf fiber into MA-modified PLA, indicating an improvement in the interfacial bonding resulting from MA modification of the PLA. This research revealed that modification of PLA using MA is potentially promising in fabricating better and stronger polymer biocomposites for different applications.

**Keywords:** Polymer, Biocomposite, Poly(lactic acid), Natural fiber, Tensile properties.

**CHEMISTRY AND BIO-SAFETY OF SYNTHESIZED ORGANIC FERTILIZER PRODUCED FROM NEEM (*AZADIRACHTA INDICA*) SEEDS**

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*Organic* fertilizers are fertilizers derived from animal matter, animal excreta (*manure*), human excreta, and vegetable matter. Naturally occurring *organic* fertilizers include animal wastes from meat processing, *manure*, slurry etc. This research was carried out to study the production, bio-safety and the chemistry of organic fertilizer from neem seeds, rice husk, blood meal, bone meal, and calcium carbonate; by mixing and blending using a mixer and hammer mill. roximate analysis was carried out using standard procedures to determine the plant's nutritive value of the formulated organic fertilizer for the presence of nitrogen by Khadjel distillation method, phosphorus by Bray No.1 method, Potassium by flame spectrophotometer, Organic Carbon by Wakley- Black dichromate method and other micronutrients (Sodium, Magnesium, and Calcium). Nitrogen which is required by plants for the production of chlorophyll which is necessary for the plant to synthesis its food using sunlight; it is the major component of amino acids, the major constituents of protein. Phosphorus is primarily used for the growth and repair of body cells and tissues. Potassium is required by plants to maintain the turgor pressure of the cell by keeping the cell strong. The result from the analysis indicated following based on the N.P.K content in each. Formulation 1 (N=4810mg/kg, P=4.76mg/kg, K=1166.7mg/kg), Formulation 2 (N= 7140mg/kg, P=19.83mg/kg, K = 1250mg/kg, Formulation 3 (N= 4060mg/kg, P=19.39mg/kg, K=600mg/kg), Formulation 4 (N=7700mg/kg,P=19.28mg/kg, K = 650mg/kg) and Formulation 5 ( N= 148400mg/kg,P= 20.10mg/kg, K=600mg/kg). The result shows that formulation 5 had the highest nitrogen content (N=148400mg/kg) while formulation 3 had the lowest nitrogen content (N=40600mg/kg) which is due to the increase in proportion of blood meal, poultry litters and neem seeds. This suggests that organic fertilizer could be a better substitute to conventional inorganic fertilizer as a soil conditioner and good plant manure when applied based on the soil nutrient requirements.

**Key words:** Manure, Neem seed, Organic, Production, Farmers, Fertilizer, Soil.

**CLEAVAGE OF  $\beta$ -ETHER LINKAGES DURING ORGANOSOLV AND STEAM EXPLOSION PRETREATMENTS**

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Two different mechanisms have been proposed for the lignin deconstruction during biomass pretreatment: (1) an acid-catalyzed hydrolytic route involving a carbonium ion on the  $\beta$  position of the lateral chain and (2) an homolytic mechanism with a  $\beta$ -O-4 radical cleavage and subsequent coupling reactions. Taking into account the complexity of the lignin polymer and of its chemistry, the distinction between these two degradation pathways is tricky and the results or discussion from literature are sometime confusing. In this paper we have studied two different pulping processes (organosolv and steam explosion). Steam explosion lignin (SEL) and ethanol organosolv lignin (EOL) were extracted and subjected to a structural characterization by 2D HSQC NMR, <sup>31</sup>P NMR, SEC and compared to Milled Wood Lignin (MWL). In both cases, a strong decrease in the  $\beta$ -O-4 content is observed with increasing treatment severity. It has been observed that mixed reactions of hydrolysis and homolysis are involved but the SEL is mainly cleaved homolytically, favouring recondensation through radical coupling even at low reaction severity.

**Keywords:**  $\beta$ -Ether linkages, Steam Explosion

**ISOLATION AND CHARACTERIZATION OF TRITERPENES FROM  
PETROLEUM ETHER AND ETHYL ACETATE EXTRACTS OF BARK  
OF THE *ZIZIPHUS SPINA CHRISTI* (L) DESF.**

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*Ziziphus spina christi* (L) Desf. is a plant used in Nigerian folk medicine for the treatment of syphilis and cancer. It also has antihelminthic and antidiarrhetic properties. Through flash column chromatographic (FCC) techniques, two compounds have been isolated from petroleum ether and ethyl acetate extracts of the bark of *Ziziphus spina christi*. These compounds were characterized using <sup>1</sup>HNMR, <sup>13</sup>CNMR, HSQC and IR Spectroscopic studies. The results for <sup>1</sup>HNMR of compound Z<sub>1</sub> include  $\delta_H$  (ppm) 4.70 (1H, br. s) 4.58 (1H, dd, J=11.1, Hz), 1.60 (1H, br, s) and 1.28 (1H, br. s). The spectrum also showed signals between  $\delta_H$  (ppm) 0.73, 0.80, 0.95, 0.96, 1.60 and 1.62 which are the characteristics of methyl (CH<sub>3</sub>) protons and Other signals due to methylene (CH<sub>2</sub>) protons, while <sup>1</sup>HNMR of Compound Z<sub>2</sub> :include  $\delta_H$  (ppm) 12.02 (1H, br.s), is characteristic of the hydrogen of the hydroxyl group of a carboxylic acid,  $\delta_H$  (ppm) 4.67 (1H,s) and 4.54 (1H,s), which are the singlet hydrogens characteristic of alkene. Several clusters of signals occurred between the range of  $\delta_H$  (ppm) 0.8 to 1.1, are characteristic of methyl hydrogens (CH<sub>3</sub>) and others between 1.1 to 1.5 are characteristic of methylene (CH<sub>2</sub>) protons. These two compounds are: betulin (lup-20(29)-en-3 $\beta$ , 28-diol) and betulinic acid (3 $\beta$ -hydroxy-lup-20(29)-en-28-oic acid).

**Keywords:** *Ziziphus spina christi*, Bark, Isolation, Ethyl acetate, Triterpenes

**DESIGN, SYNTHESIS AND CYTOTOXIC EVALUATION OF O-CARBOXAMIDO STILBENE ANALOGUES**

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Resveratrol, a prominent stilbenoid-type compound that employ a wide range of biological activities, such as anti-carcinogenic, antioxidant, anti-inflammatory and anti-tumor. Resveratrol is also known as a favorable chemo preventive agent for inhibiting carcinogenesis processes that target kinases, cyclooxygenases, ribonucleotide reductase and DNA polymerases. A total of 10 stilbenes analogues with an amide moiety were synthesized and their cytotoxic effects on a series of human cancer cell lines are reported. In this study, the stilbenes have been synthesized by palladium-catalyzed Heck coupling between 3,5-dimethoxystyrene with amide precursor under basic condition. All the synthesized compounds were characterized using Fourier Transform Infrared spectroscopy (FT-IR), <sup>1</sup>H and <sup>13</sup>C Nuclear Magnetic Resonance spectroscopy (NMR) and CHN elemental analyses.

**Keywords:** *O*-carboxamido stilbenes, amido stilbenes, Heck coupling, cytotoxic effects

## APPROCHES TOWARD THE SYNTHESIS OF KINGIANINS

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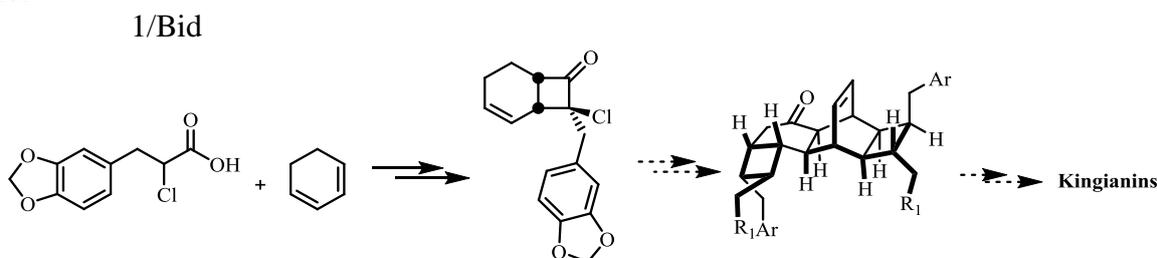
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The kingianins were a pentacyclic polyketides isolated previously from Malaysia *Endiandra kingiana* Gamble (Lauraceae). These compounds exhibited potency as a modulating agent between Bcl-xL/Bak and Mcl-1/Bid, which prompted its chemical investigation. The pentacyclic kingianin skeleton was formed by Diels-Alder reaction between two monomers having a bicyclo [4.2.0] octadiene backbone formed by a stereospecific electrocyclicization of polyenes. The research was focusing on construction of bicyclo [4.2.0] octadiene monomer using [2+2] ketene cycloaddition approach at the early stage of the synthesis. One of the main advantages of such a strategy is the rapid assembly of the carbon skeleton of kingianins, thus maximizing the chances for good overall yields of the final products. So far, an efficient synthesis of the bicyclo [4.2.0] octene backbone was successfully achieved. The approaches to synthesize this backbone starting from [2+2] cycloaddition of the cyclohexadienes to functionalized ketenes followed by functionalization of substituent at C-7 and C-8 positions with the correct relative configuration were described. From these approaches, several compounds were identified as the key intermediates. This key step of the synthesis provided an access to the kingianins skeleton.

**Keywords:** Kingianins, Pentacyclic polyketides, [2+2] ketene cycloaddition, Bcl-xL/Bak, Mcl-



**SCREENING FOR PHYTOCHEMICAL, ANTIBACTERIAL AND FREE-RADICAL  
SCAVENGING ACTIVITIES OF EXTRACTS FROM THE STEM BARK OF  
*NAUCLEA POBEGUINII*.**

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*Nauclea pobeguinii* belongs to the Rubiaceace family. It is widely used in traditional medicine in the treatment of intestinal worms, abdominal pains, sexual asthenia and gonorrhoea. This study examined the phytochemicals, antibacterial activities and antioxidant potential of the ethyl acetate and ethanol stem bark crude extracts of *Nauclea pobeguinii* from Nigeria. Phytochemical screening, antibacterial activities and antioxidant potential studies and free radical scavenging activity of the extracts were carried out using standard methods. Phytochemical screening of the ethyl acetate and ethanol extracts of the plant parts revealed the presence of saponins, alkaloids, tannins, phenols and steroids. Flavonoids are present in the ethyl acetate extracts while saponins are absent in ethyl acetate extract. Antibacterial studies of ethanol extracts revealed inhibitory activity on *Escherichia coli*, *Staphylococcus aureus*, *Serratiamarcescens*, *Klebsiella pneumonia* and *Pseudomonas spp* with zone of inhibitions ranging from 10-20mm while ethyl acetate extract only active on *Escherichia coli*. Both extracts showed good radical scavenging activity ranging from 1.5 to 3.5µg/ml when compared with the standard Quercetin (35.0µg/ml).

**Keywords:** *Nauclea pobeguinii*, Antioxidant, Phytochemical, Free radical, Antibacteria, Rubiaceae

**COMPLEXATION OF COPPER (II) ION WITH RED *CAPSICUM FRUTESCENS* EXTRACT IN ETHANOL**

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The *Capsicum* fruits (Cf) contain capsaicinoids which are amide- and hydroxyl-containing compounds. These groups show promising interest in binding with metal ions to form metal-ligand complexes. In this study, oven dried red Cf was extracted using 95% ethanol to afford a viscous brownish red oil with 12% yield. It was soluble in acetone, ethanol, acetonitrile and, hexane but insoluble in water. It was then complexed with Cu (II) ion in ethanol followed by characterization using thin layer chromatography and UV-Vis spectrometry. The fern green Cu (II)-Cf extract complex yielded 0.0727 g and it was insoluble in water and in organic solvents. The  $R_f$  values of the Cf extract, Cu (II) ion, and complex in acetone were 0.830, 0.620, and 0.00, respectively showing different migration rates. The  $\lambda_{max}$  values observed for the Cf extract, Cu (NO<sub>3</sub>)<sub>2</sub>, and complex were 520 nm, 740 nm, and 430 nm, respectively. The Cu(II) ion concentration left in the filtrate was 0.4380% suggesting that the binding of the Cf extract (0.80%) and Cu(II) (0.80%) ion was 2:1 ratio. The result suggests that Cf extract served as a potential ligand to bind with Cu (II) ion in solution.

**Keywords:** *Capsicum frutescens*, Extraction, Copper (II) ion, Binding, Characterization

**PHOTOLUMINESCENT ESTER OLIGOMERS CONTAINING  
CURCUMIN DYE UNITS: A SELECTIVE FLUORESCENT  
“TURN OFF” CHEMOSENSOR FOR FERRIC IONS**

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Ester oligomers were synthesized via polycondensation of curcumin with aromatic diacid which were characterized by elemental analysis, <sup>1</sup>H NMR, <sup>13</sup>C NMR, FT-IR and TGA. The onset of thermal degradation was 280 °C and ester oligomers with longer alkyl chain showed lower thermal stability. Photoluminescence property was examined by using UV-Vis and fluorescence spectroscopies. The ester oligomers exhibited green emission under UV irradiation. Selective fluorescence quenching occurred upon the addition of Fe<sup>3+</sup> over other heavy metal cations. The result suggesting potential of this curcumin-based ester oligomers as chemosensor for Fe<sup>3+</sup> ions.

**Keywords:** Photoluminescence, Oligomers, Thermal stability, Chemosensor

ORG-011

**SYNTHESIS OF A POLYMOF BASED ON A POLYMER  
LIGAND POSSESSING A 1,4- BENZENEDICARBOXYLIC  
ACID**

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This study reports the successful preparation of a polyMOF using a polymer ligand possessing a 1, 4-benzenedicarboxylic acid ligand in its side chains. Although the preparation of various metal-organic frameworks (MOFs) has been reported, the construction of MOFs using a polymer ligand (polyMOF) remains a developing field<sup>2</sup>. Here we present a new molecular design of a polyMOF consisting of a polymer ligand possessing a ligand moiety in the side chain. The structure of the resulting polyMOF, which was obtained from a polymer ligand and Zn (NO<sub>3</sub>)<sub>2</sub>, was confirmed by powder X-ray diffraction, and was found to consist of a MOF-like structure in the polymer. Microscopy images show that the polyMOF was constructed of crystalline structures despite its film form<sup>2</sup>.

**Keywords:** Metal-Organic Frameworks, Poly MOF, Self-assembly.

**CATALYTIC BIOMASS CONVERSION INTO METHYL  
LEVULINATE**

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In this study, microwave assisted synthesis of methyl levulinate (ML) was extensively studied by using glucose as model compound at different temperatures (130-170 °C), reaction times (10-50 mins) and catalyst loadings (0.6-1.2 wt%). Later the optimum parameter was applied to core oil palm trunk (COPT) sap. The synthesis was carried out in methanol catalyzed by aluminum sulfate  $Al_2(SO_4)_3$ . The results from gas chromatography-mass spectrometry (GC-MS) and high-performance liquid chromatography (HPLC) analysis indicated that the optimum parameter for ML synthesis was at 170°C, 50 min with 1.2 wt %  $Al_2(SO_4)_3$ . At this optimum reaction conditions, the highest glucose conversion (99.14%) and ML yield (68.40%) with low intermediate product of methyl glucoside (MGO) yield (9.8%) were achieved. Analysis of variance (ANOVA) showed the conversion of glucose was significantly affected by catalyst loading ( $P < 0.05$ ), meanwhile ML yield was significantly influenced by all three parameters; catalyst loading, reaction time and temperature ( $P < 0.001$ ). The GC-MS analysis showed methanolysis of COPT sap produced high ML yield (68.35 %) while glucose standard produced 63.96% ML. Thus, COPT sap has the potential to be converted into ML which is strategically green, efficient and economic.

**Keywords:**  $Al_2(SO_4)_3$ , HPLC, Esterification, Methanolysis

## SYNTHESIS AND CHARACTERIZATION OF POLYPYRROLE-POLYETHYLENIMINE NANOCOMPOSITES AND ITS APPLICATION OF NICKEL IONS REMOVAL FROM AQUEOUS SOLUTION

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A novel conducting polymer-based adsorbent, polypyrrole-polyethyleneimine (PPy-PEI) has been successfully prepared using  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{FeCl}_3$  (anhydrous) and  $(\text{NH}_2)_2\text{S}_2\text{O}_8$  as oxidants with an aim to remove nickel ions from aqueous solution. Polyethyleneimine (PEI) was added to form the nano composite in order to add more active sites for chelating with nickel ions. The removal of nickel ions from aqueous solution has been observed to improve from 42% using Ppy to a higher efficiency of 99.8% using Ppy-PEI composite in a batch equilibrium system. The optimum adsorption dosage has been found as 0.08 g at a pH of 4.5 with a contact time of 60 mins at room temperature. The ATR-FTIR, FESEM, XRD, BET and EDX provide the evidence of nickel ions adsorption by Ppy-PEI nanocomposite. TGA and DSC were used to study the thermal behaviour of the composite. The BET surface area and average particle size of prepared PPy powder was  $10.27 \text{ m}^2/\text{g}$  and  $\sim 18\text{-}34 \text{ nm}$ , respectively. The GPC result shows the molecular weight as 1595 and polymer dispersion index, PDI as 1.21, respectively. The values of  $Q_{\text{max}}$  and  $k_L$  were  $1.905 \text{ mg/g}$  and  $5.61 \text{ to } 6.26 \text{ L/g}$  respectively. The adsorption data has been well fitted to Freundlich isotherm model and followed the pseudo-first order kinetic model. As an effective adsorbent for the removal of nickel ions from aqueous solution, the adsorbent can be reused for several times with more than 82% at the 3<sup>rd</sup> cycle. This new conducting polymer-based adsorbent offers the promise to be commercially used in near future.

**Keywords:** Polypyrrole-polyethyleneimine; Nickel, Nanocomposite, Freundlich model, Pseudo-second order model.

**ADSORPTION ISOTHERM AND KINETICS FOR CARBON DIOXIDE CAPTURE USING BISMUTH (III) OXIDE IMPREGNATED ON ACTIVATED CARBON**

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Carbon dioxide (CO<sub>2</sub>) concentration level in the atmosphere has been increasing day by day due to the uncontrolled burning, development and deforestation. In this work, bismuth (III) oxide (Bi<sub>2</sub>O<sub>3</sub>) impregnated on activated carbon (AC) composites were prepared using different Bi<sub>2</sub>O<sub>3</sub> loading and their adsorption capacities were investigated. The results were compared to that of treated AC that were treated via chemical activation method. The samples were characterized using *X-ray diffraction* (XRD), Fourier transform infrared spectroscopy (FTIR), N<sub>2</sub> adsorption-desorption isotherm, and scanning electron microscope/energy dispersive X-Ray analyzer (SEM/EDX) and thermogravimetric analysis (TGA). The XRD pattern and SEM/EDX were confirmed the presence and well-dispersed of Bi<sub>2</sub>O<sub>3</sub> on the AC surfaces. The stability, adsorption and regeneration of CO<sub>2</sub> were analyzed at 30, 40 and 50 °C by using TGA. The 0.1Bi/AC by using non-chemical activation AC shows most efficient adsorbent at ambient condition of 30 °C with CO<sub>2</sub> adsorption capacity of 58.7108 mg CO<sub>2</sub>/g adsorbent. Although BET surface area of 0.1Bi/AC reduces (783.25 m<sup>2</sup>/g) compared to AC only, the Bi<sub>2</sub>O<sub>3</sub> loading noticeable enhance the CO<sub>2</sub> chemisorption. The adsorption kinetic indicating that chemisorption dominates the adsorption process as the data fits well the pseudo-second-order kinetic model with R<sup>2</sup> value of 0.999. The CO<sub>2</sub> capture capacity is consistent over 5 cycles with slightly higher than AC only ascribed to the chemical adsorption by interactions of carbonate species with Bi<sub>2</sub>O<sub>3</sub> besides the physisorption on AC. This features exhibits a potential for large scales application.

**Keywords:** CO<sub>2</sub> capture, Adsorption kinetics, Adsorption isotherm, Activated carbon, Bismuth (III) oxide

## MECHANISM OF SPIRO-HETEROCYCLE FORMATION: DENSITY FUNCTIONAL THEORY APPROACH

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Literature is hugely deficient in theoretical kinetic and thermodynamic data on formation mechanisms of some bioactive spiro-heterocycles. Formation of 1'-((((aminoxy)carbonyl)amino)methyl)-2'-(1-hydroxynaphthalen-2-yl)-2'-methyl-1',2',5',6',7',7a'-hexahydrospiro[indoline-3,3'pyrrolo[1,2-a]imidazol]-2-one was theoretically investigated using ab initio method. For more accurate result, density functional theory at B3LYP with 6-311+G\*\* basis set was employed. There were bond length alterations (C9–N10, C9–C12, C13–N10 and C8–N10) as the interaction between the reacting species progressed through the stationary points to the product. The HOMO–LUMO energy gap suggested that pyrrolidine-2-carboxylic acid ( $\Delta E = -6.27$  eV) acted as Lewis acid while indolin-2,3-dione ( $\Delta E = -3.90$  eV) acted as a Lewis base for the intermediate formation step. The formation of Van der Waals 1 (VWC 1) step leading to the formation of first Van der Waals complex was computed to exothermic ( $-72.95$  kJmol<sup>-1</sup>) and slightly endergonic ( $+1.78$  kJmol<sup>-1</sup>), while the transition step leading to the formation of transition state 1 has the rate constant, k, value of  $1.87 \times 10^{-1}$  dm<sup>3</sup>mol<sup>-1</sup>s<sup>-1</sup>. From the mechanism of the reaction, the rate of formation of the product was found to be dependent on the isatin, [A] and pyrrolidine-2-carboxylic acid, [B] concentration by the expression  $\frac{d[P]}{dt} = \frac{k_2^2 k_2' k_1'}{k_2' k_1 (k_{-2}' + k_2)}$  [A][B]. The study has shown alterations in bond lengths that occurred as the initial reacting species undergo transformations through intermediates and transition states to the final product. Through molecular energy theory (HOMO–LUMO energy gap), the study identified the electrophile and the nucleophile responsible for the actual reaction spark. In addition, the study accounted for the energy requirement of each of the steps involved in the transformation. Furthermore, the reaction rate-determining step was identified, and the reaction mechanism established. The data obtained for the formation mechanism of 1'-((((aminoxy)carbonyl)amino)methyl)-2'-(1-hydroxynaphthalen-2-yl)-2'-methyl-1',2',5',6',7',7a'-hexahydrospiro[indoline-3,3'pyrrolo[1,2-a]imidazol]-2-one could serve as guide for other important bioactive molecules.

**Keywords:** Pyrrolidine-2-carboxylic acid, Kinetics, Transition state, Van der Waals complex.

**MICROWAVE SYNTHESIS OF NANOSIZED ZEOLITE K-W AS  
SOLID BASE CATALYST FOR NITROALDOL HENRY REACTION  
OF VANILLIN WITH NITROETHANE**

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The upscale production of nanosized zeolite W remains challenging as it requires long crystallization time and the use of expensive organic template to direct the formation of zeolite framework structure. The aim of this work is to study the synthesis of potassium zeolite W nanocrystals with MER topology under microwave condition without the use of organic template. With microwave heating, K-W nanocrystals were obtained within 12 min at 180 °C. The spectroscopy and microscopy studies confirmed the importance of alkaline synthesis condition and K<sup>+</sup> cations in the selective formation of K-MER zeolite. The K-MER nanocrystals were tested as a solid base catalyst in Henry reaction of nitroethane with vanillin under microwave irradiation. The K-MER nanocatalyst demonstrated high catalytic performance and recyclability, giving vanillin conversion (86.5%) in 300 min of reaction at 190 °C, with no significant loss even after five reaction cycles.

**Keywords:** K-MER; Zeolite nanocatalyst; Template-free synthesis; Microwave heating; Henry reaction of vanillin with nitroethane

**ORGANOTEMPLATE-FREE CS-ABW NANOZEOLITE AS HIGHLY REACTIVE AND RECYCLABLE CATALYST FOR NITROALDOL REACTION BETWEEN BENZALDEHYDE AND NITROETHANE**

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Zeolite Cs-ABW nanocatalyst has been hydrothermally synthesized without using any harmful organic template. The zeolite nanocrystal with very high solid yield (81.88%) can be obtained within 60 min and at 180 °C (~20 atm); a condition which is much faster and gentle than the existing synthesis methods. The orthorhombic shaped Cs-ABW nanocrystals (mean size of 32 nm) have high aluminum content (Si/Al ratio = 1.14) and possess high basicity. The nanocrystals also exhibit better catalytic activity than conventional homogeneous catalysts in Henry (nitroaldol) reaction of benzaldehyde with nitroethane under microwave heating condition. The nanocatalyst does not suffer from coking and leaching problems. It can be reused without loss of reactivity even after five consecutive runs under the described reaction conditions.

**Keywords:** Sol gel chemistry, Microporous materials, Crystal growth, Crystal structure, Catalytic properties

**SYNTHESIS, CHARACTERIZATIONS AND APPLICATION OF  
METALLIC BI SELF-DOPED BIOBR COMPOSITES FOR  
PHOTOCATALYTIC DEGRADATION OF CIPROFLOXACIN USING  
INDOOR FLUORESCENT LIGHT ILLUMINATION**

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The photocatalytic degradation of ciprofloxacin antibiotic under 25 W indoor fluorescent light illumination using pristine BiOBr and Bi/BiOBr composites has been studied. BiOBr was initially synthesized at room-temperature using polyethylene glycol 400 (PEG400) as morphology control agent. Subsequently, metallic Bi self-doped BiOBr composites were formed via *in-situ* reduction at room-temperature using different concentrations of NaBH<sub>4</sub>, with BiOBr as template. Various characterization techniques, including x-ray diffraction (XRD), field emission scanning electron microscope (FESEM), attenuated total reflectance fourier transform infrared (ATR-FTIR), Brunauer-Emmett-Teller (BET) surface area, UV-vis diffuse reflectance spectra (UV-Vis DRS), photoluminescence (PL), high-resolution transmission electron microscope (HRTEM) and x-ray photoelectron spectroscopy (XPS) were employed to confirm the successful distribution of metallic Bi on the surface of BiOBr. The experimental results revealed that, the presence and amount of metallic Bi have great impact on the photocatalytic performance of BiOBr, with the optimized performance recorded using Bi/BiOBr-20 composite photocatalyst. Compared to pristine BiOBr with degradation rate constant of 0.00614 min<sup>-1</sup>, Bi/BiOBr-20 composite photocatalyst has a degradation rate constant of 0.02913 min<sup>-1</sup>. Such enhanced performance has been attributed to the efficient separation of photogenerated charge carriers and improved visible light absorption, with holes (h<sup>+</sup>) and superoxide radical anions (•O<sub>2</sub><sup>-</sup>) being the main active species. Finally, the Bi/BiOBr-20 composite photocatalyst was found to be stable and reusable, and a possible mechanism on the basis of the experimental results has been proposed.

**Keywords:** Metallic Bi, BiOBr, In-situ reduction, Ciprofloxacin, Indoor fluorescent light illumination

## GREEN HYDROCARBON PRODUCTION OVER NiO NANOPARTICLES SUPPORTED ON HIERARCHICAL ZEOLITE Y

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Global awareness about energy and environmental deterioration due to the excess consumption of fossil fuel has motivated researchers to seek an alternative for fossil fuel. Deoxygenation, an effective process to convert non-edible into green hydrocarbon which having same properties as petroleum fuel. Usually, this process is performed by using supported precious metal catalysts with hydrogen gas supply. Considering the expensive price of the noble metals, nickel based catalyst is more affordable, and having comparable performance to the precious metal-based catalyst. Besides, the usage of hydrogen gas would increase the carbon footprint as 9 kg of CO<sub>2</sub> are being generated per one kg H<sub>2</sub> produced from fossil fuel. From our previous studies, nanosized Y synthesized under mild synthesis condition have a positive impact on the deoxygenation activity as well as the product selectivity in the hydrogen free condition. In this study, we further investigated the potential of zeolite Y by creating uniform mesoporosity within zeolite Y nanocrystals by adding small amount of organosilane surfactant during synthesis step. After that, a series of nickel oxide (NiO) supported on the hierarchical zeolite Y with various Ni loading (5, 10, 15 and 20 wt% Ni) were prepared by deposition and precipitation method. The prepared catalysts were characterized by XRD, BET, FTIR, XRF, and NH<sub>3</sub>-TPD. The resultant Ni catalysts improved the deoxygenation performance of triolein at a reaction temperature at 380 °C in a semi batch reactor under solvent-free and hydrogen-free condition. The generation of uniform mesopore within the nanosized zeolite Y crystals have achieved better deoxygenation performance as compared to their nanosized counterparts. The incorporation of NiO onto the hierarchical zeolite Y has further improved deoxygenation performance and the product selectivity. Among the catalysts, 10 wt% NiO-hierarchical zeolite Y achieved highest conversion (76%) and highest hydrocarbon selectivity (99%) after reacted at 380 °C for 30 min. This enhanced performance was attributed to the synergistic effect between small NiO particle size (~ 5 nm) and uniform mesoporosity (~15 nm). This study has revealed that NiO supported on the hierarchical zeolite Y is a promising catalyst in the development of sustainable biofuel from the non-edible oil.

**Keywords:** Green Hydrocarbon, Triolein, NiO, Hierarchical Zeolite Y, Mesoporosity

**A GREEN SOLVOTHERMAL APPROACH FOR THE SYNTHESIS AND CHARACTERIZATIONS OF NICKEL NANOPARTICLES**

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Magnetic nanoparticles of Nickel have the unique potential of being employed in various industrial applications, such as in magnetic devices, catalysis, sensor development, optical electronics and biomedical applications. However traditional synthetic protocols of Nickel nanoparticles often involve the use of costly, corrosive and environmentally harmful chemicals. In this work, a one-step, green solvothermal synthetic approach, using environmentally friendly, and cost-effective materials has been adopted for the synthesis of Nickel nanoparticles. Nickel chloride was employed as precursor for the synthesis, ethylene glycol as solvent, sodium acetate as stabilizing agent and finally, polyethylene glycol as capping agent. The effect of temperature of reaction, precursor concentration, reaction time and the pH of the mixture, were investigated to obtain the optimal synthetic conditions. The synthesized Nickel nanoparticles were characterized with the aid of Ultra Violet Visible spectroscopy (UV-Vis), Fourier Transform InfraRed spectroscopy (FT-IR), X-Ray diffraction analysis (XRD), Brunauer-Emmett-Teller analysis (BET) and High-Resolution Transmission Electron Microscopy (HR-TEM). The surface plasmon resonance of Nickel nanoparticles was detected at 265 nm by UV-Visible spectroscopy. The presence of organic functional groups such as RCH<sub>2</sub> at 2945 cm<sup>-1</sup>, C-O at 1024 cm<sup>-1</sup> and Ni-(OH)<sub>2</sub> at 3293 cm<sup>-1</sup> was obtained by FT-IR spectroscopy. The X-ray diffraction analysis of the Nickel nanoparticles showed a cubic phase in the range of 25-100 nm, the HR-TEM obtained size and morphology of the Nickel nanoparticles was found to be 20-100 nm. The BET adsorption analysis indicated a large specific surface area of the Nickel nanoparticles, predisposing the nanoparticles as good candidates for catalysis, sensor development and as magnetic material for industrial applications.

**Keywords:** Nickel, Nanoparticle, Synthesis; Solvothermal, Green.

**SYNTHESIS AND CHARACTERIZATION OF ZnO QUANTUM DOTS  
MODIFIED MESOPOROUS TiO<sub>2</sub> FOR PHOTOOXIDATION OF  
TETRACYCLINE**

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Modification of TiO<sub>2</sub> with quantum dots (QDs) is an effective strategy to improve its photocatalytic performance under visible light irradiation. In this research, mesoporous TiO<sub>2</sub> (m-TiO<sub>2</sub>) was incorporated with 3%, 5%, 8% and 10% ZnO QDs via sol-gel method. The photocatalysts (*x*ZnO QDs/m-TiO<sub>2</sub>; *x*= 3%, 5%, 8% and 10%) were characterized using XRD, AFM, HRTEM, FESEM, UV-Vis DRS, BET, and PL analysis. The structural analysis revealed that ZnO QDs were uniformly distributed on the surface of m-TiO<sub>2</sub>. The average crystallite sizes of the photocatalysts calculated using the Debye–Scherrer equation were 12.23 nm, 10.04 nm, 9.85 nm, 9.54 nm and 8.49 nm for pristine m-TiO<sub>2</sub>, 3%-, 5%-, 8%- and 10%- ZnO QDs/m-TiO<sub>2</sub>, respectively. The photocatalytic activity of the photocatalysts was tested in photooxidation of tetracycline (TC) under visible light irradiation. In just 90 min, 96.8% of TC (40 mg/L) was photooxidized when 8%ZnO QDs/m-TiO<sub>2</sub> was used. Such enhanced photocatalytic activity is attributed to the formation of a heterojunction between the two semiconductors, which favors the separation of photogenerated electron-hole pairs in the photocatalyst. Another reason is due to size-dependent quantum confinement effect, which leads to the displacement of the conduction band potentials of ZnO QDs to more negative energy values compared to TiO<sub>2</sub>. The kinetics of the photooxidation process is explained in terms of the Langmuir-Hinshelwood model. The values of the kinetic rate constant (*k*) of the photooxidation were 8.3 x 10<sup>-3</sup> min<sup>-1</sup> and 19.9 x 10<sup>-3</sup> min<sup>-1</sup> for pristine TiO<sub>2</sub> and 8%ZnO QDs/m-TiO<sub>2</sub> photocatalysts, respectively.

**Key words:** Quantum dots, Zinc oxide, Titanium dioxide, Photooxidation, Tetracycline.

**SYNTHESIS OF LTL-TYPE ZEOLITES WITH DIFFERENT MORPHOLOGIES USING BAMBOO LEAF ASH AND THEIR USE AS CATALYSTS FOR PRODUCING BIOFUEL (2-ACETYL-5-METHYLFURAN)**

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Microporous zeolites LTL with different crystal morphologies and sizes were successfully synthesized using bamboo leaf silica ash (BLA). Various characterization techniques were used to study the physicochemical properties of the samples. The FESEM analysis showed that short rod, cylindrical, needle-like and nano-sized crystals with aspect ratio varying from 0.9 – 4.0 could be synthesized by simply tuning the water content and heating time. Furthermore, the effects of zeolite morphology on the catalytic acylation of 2-methylfuran with acetic anhydride were investigated. It was shown that the catalytic performance of zeolites not only depended on the morphology, but also the acidity (number of acid sites and acid strength), porosity and accessible of acid sites.

**Keywords:** Zeolite LTL, Acid catalyst, Morphology, 2-methylfuran, Acetic anhydride.

**DISSOCIATIVE ELECTRON TRANSFER VIA  $n \rightarrow \pi^+$  INTERACTION  
IN ISOLATED TYROSINE-CONTAINING MOLECULAR PEPTIDE  
RADICAL CATIONS**<sup>1</sup>W. K Tang, <sup>2</sup>X. Y. Mu, <sup>2</sup>I. K. Chu, <sup>1</sup>C. K. Siu<sup>1</sup>*Department of Chemistry, City University of Hong Kong, Tat Chee Avenue, Kowloon Tong, Hong Kong SAR, China*<sup>2</sup>*Department of Chemistry, The University of Hong Kong, Pokfulam, Hong Kong SAR, China*

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Electron hopping mechanism involving amino acid residues with low ionization potential as redox-active sites is of significance to understand the electron transport over long distance in protein. Upon one-electron oxidation in tyrosine residue,  $\pi$ -radical cation (electron hole) at its phenol side chain is generated. Subsequent electron transfer from vicinal electron donor to this  $\pi$ -electron hole completes the elementary step of electron migration. Herein, we demonstrate this elementary process at the molecular level with tyrosine-containing molecular peptide radical cations  $[M]^+$  generated from one-electron oxidative dissociation of ternary copper(II) complexes  $[Cu^{II}(\text{dien})M]^{2+}$  in the gas phase. Collision-induced dissociation (CID) of  $[FYGG]^+$  results unusual cleavage of the  $C\alpha-C\beta$  bond at the side chain of the N-terminal residue. This reaction channel can be attributed to the radical-cation character at their N-terminal amino group ( $NH_2^+$ ) resulting from a  $n \rightarrow \pi^+$  interaction from the nonbonding electron of  $NH_2$  ( $n$ ) to the  $\pi$ -electron hole at the Tyr side chain ( $\pi^+$ ). Density functional theory (DFT) studies and infrared multiple-photon dissociation (IRMPD) action spectroscopy reveals the establishment of this  $n \rightarrow \pi^+$  interaction featuring a characteristic  $NH_2$  scissor at  $1577\text{ cm}^{-1}$ . Potential energy scans and spin density analyses revealed that this  $n \rightarrow \pi^+$  interaction facilitates the dissociative electron transfer from the  $C\alpha-C\beta$  bond to the radical cation of Tyr side chain via  $NH_2$  as the relay station. Potential energy surface (PES) of the dissociative ET of the N-terminal  $C\alpha-C\beta$  bond shows that the dissociative ET is energetically unfavorable with increasing number of Gly spacers separating the Phe and Tyr residues and in accord with CID experiments for tri-, tetra-, penta- and hexa-peptide models. This work suggests that the occurrence of the side-chain cleavage of the N-terminal residue in tyrosine-containing  $[M]^+$  is an indicator for the formation of reactive conformers favoring the  $n \rightarrow \pi^+$  interaction between the N-terminal  $NH_2$  and the radical cationic side chain of Tyr; providing some three-dimensional information on the folding of peptide chains in the gas phase.

**Keywords:** Electron transfer, Radical peptide cation,  $n \rightarrow \pi^+$  interaction, Relay station

## THE EFFECT OF (Fe, Co and Ni) DOPANTS ON THE STRUCTURAL AND GAS SENSING PROPERTIES OF TIN OXIDE

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Transition elements and noble metals have been used traditionally as dopant in metal oxide to improve the performance especially in optical, semiconductor and sensor applications. Tin oxide (SnO<sub>2</sub>) nanostructures are found to be very promising gas sensing materials due to their inherent advantages such as wide band gap (3.6 eV at 300 K) and high electron mobility (100 - 200 cm<sup>2</sup> V<sup>-1</sup> S<sup>-1</sup>). In this study, pure and doped ((ferum (Fe), cobalt (Co) and nickel (Ni)) SnO<sub>2</sub> were synthesised *via* hydrothermal method. The effect of dopants on crystallinity and structural properties were analysed using various techniques, namely Fourier Transform Infrared (FTIR), X-ray diffraction (XRD), Ultraviolet- Visible Diffuse Reflectance Spectroscopy (UV-Vis DRS), X-ray Photoelectron Spectroscopy (XPS), Scanning Electron Microscopy (SEM) and High Resolution Transmission Electron Microscopy (HRTEM). The gas sensing test was carried out using 1000 ppm ethanol gas at different temperatures (200 – 450°C). The finding showed that the all three dopants did not significantly affect the crystal structure (rutile tetragonal) and the morphology of nanostructure (rods). However, the average diameter of nanorods decreased after the doping process, where Fe-doped SnO<sub>2</sub> (Fe: SnO<sub>2</sub>) exhibited the smallest average diameter (4.4 ± 0.56 nm) (five times smaller than SnO<sub>2</sub>), possibly due to the ionic radii of Fe<sup>3+</sup> much smaller than the other dopants (Co and Ni). Hence, the substitution of Sn<sup>4+</sup> ions (ionic radii, 0.69 Å) in SnO<sub>2</sub> crystal lattice with Fe<sup>3+</sup> ions reduced the diameter of SnO<sub>2</sub> nanorods considerably. The bandgap energy of SnO<sub>2</sub> also found to be reduced after the doping process due to the interaction between the *d* electrons of the dopant metals (Fe, Co and Ni) with conduction band of SnO<sub>2</sub>. Among the three dopants, Ni: SnO<sub>2</sub> exhibited the highest ethanol gas sensing response,  $R_0/R_g$  1.4 x 10<sup>4</sup> (13 times higher than SnO<sub>2</sub>), while Fe: SnO<sub>2</sub> recorded the lowest  $R_0/R_g$  8.4 x 10<sup>2</sup>. Overall, it was proposed that the presence of chemisorbed oxygen, oxygen vacancies, the catalytic properties of dopant and the large surface to volume ratio influence the gas sensing performance of the as-synthesised SnO<sub>2</sub> sample.

**Keywords:** Tin oxide, Fe, Co and Ni dopants, Nanorods, Hydrothermal.

**SYNTHESIS AND CHARACTERIZATION OF CMC/BiFeO<sub>3</sub>/PoPD  
NANOCOMPOSITE FILM FOR PHOTODEGRADATION OF  
ANIONIC DYE**

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Recently, there is a greater emphasis on ‘greener’ route for an inexpensive and environmentally benign material as catalyst support. In line with this objective, bismuth ferrite (BiFeO<sub>3</sub>) was synthesized by biotemplating method while poly-*o*-phenylenediamine (PoPD) was synthesized by oxidative polymerization using distilled water as a medium. The impregnation of BiFeO<sub>3</sub>/PoPD on CMC was successfully prepared by casting method. The photocatalytic efficiency of nanocomposite was investigated for the removal of methyl orange (MO) under direct sunlight irradiation. The prepared CMC/BiFeO<sub>3</sub>/PoPD nanocomposite was characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), thermogravimetric analysis (TGA), fourier transform infrared (FTIR) spectroscopy and N<sub>2</sub> adsorption analysis. XRD pattern revealed inclusion of BiFeO<sub>3</sub> and PoPD within CMC polymer medium. The effect of catalyst dosage, reaction time, and initial concentration of dye towards degradation performance of methyl orange were elucidated. The integration of PoPD to CMC/BiFeO<sub>3</sub> showed remarkable catalytic efficiency as compared to unmodified CMC/BiFeO<sub>3</sub> nanocomposite. This work could provide valuable information on the design of polymer modified semiconductor with more excellent properties and have potential industrial waste water treatment.

**Keywords:** Photocatalyst, Nanocomposite, Carboxymethyl cellulose, Bismuth ferrite, Poly-*o*-phenylenediamine, Methyl orange.

**EFFECTS OF FUNCTIONAL GROUPS OF IMIDAZOLIUM  
TEMPLATES ON CRYSTALLIZATION AND MORPHOLOGICAL  
PROFILES OF SAPO-5**

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1-propyl-2,3-dimethylimidazolium ([pdmIm]<sup>+</sup>), 1-benzyl-2,3-dimethylimidazolium ([bzmIm]<sup>+</sup>) and 3-(2,3-dihydroxypropyl)-1,2-dimethylimidazolium ([dhpdm]<sup>+</sup>) cations as structure-directing agents (SDA) for the synthesis of SAPO-5 microporous materials were reported. The effects of these SDAs on the crystallinity, crystal size, solid yield and morphology of the products were investigated. The results indicated that the crystallinity depended on the SDA's functional group where polar [pdmIm]<sup>+</sup> SDA bearing diol group had the fastest crystallization rate with highest solid yield. Thermogravimetry results also revealed that selection of SDA with proper polarity is vital to obtain SAPO-5 with high porosity ( $S_{\text{BET}}$ ,  $S_{\text{mic}}$ ,  $S_{\text{ext}}$  and  $V_{\text{total}}$ ) as high polar SDA exhibits strong interaction with the AFI framework that will affect the template removal during calcination.

**Keywords:** SAPO-5, Microporous material, hydrothermal synthesis, Imidazolium, Structure directing agents, Functional group

**INCORPORATION OF CHLOROTHALONIL INTO POLYLACTIC ACID-CO-POLYETHYLENE GLYCOL NANOPARTICLES FOR RUBBERWOOD PRESERVATION TREATMENT**

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Chlorothalonil (CTL), a hydrophobic organic biocide was incorporated into polylactic acid-co-polyethylene glycol (PLA-PEG) nanoparticles to develop a water-based formulation for rubberwood preservation treatment. The characterisation results showed that CTL was successfully incorporated into PLA-PEG nanoparticles. The average hydrodynamic particle size and polydispersity index of CTL-PLA-PEG nanoparticles were  $12.8 \pm 0.18$  nm and  $0.344 \pm 0.006$ , respectively. The % weight of CTL in nanoparticles was  $20.4 \pm 0.12$  % w/w whereas CTL loading efficiency was  $65.9 \pm 0.38$  %. Rubberwood (*Hevea brasiliensis*) specimens were treated with 0.025% w/v aqueous suspension of nanoparticles by employing vacuum-pressure treatment. The pressure and duration of treatment were reduced by half without compromising the quality. The treatability and chemical retention achieved were  $307.5 \pm 4.33$  L m<sup>-3</sup> and  $8.18 \pm 0.083$  kg/m<sup>3</sup>, respectively. Biological efficacy performances of treated wood were carried out by exposing to subterranean termite (*Coptotermes curvignathus*) and brown rot fungi (*Gloeophyllum trabeum*) in accelerated condition. The weight loss of wooden blocks that attributed to termite attack was reduced from  $9.7 \pm 0.28$  % to  $3.6 \pm 0.20$  %. In addition, the nanoparticles also reduced the weight loss attributed to fungi from  $37.1 \pm 0.11$  % to  $11.4 \pm 0.37$ . In short, the biological efficacy test has proven that the treatment with low concentration of CTL-PLA-PEG nanoparticles were sufficient to enhance the resistance of wood against decay fungi and termite.

**Keywords:** Wood preservative, Nanocarrier, Nanoprecipitation, Polylactic acid-co-polyethylene glycol, Chlorothalonil, Rubberwood

**SYNTHESIS AND CHARACTERIZATION OF GOLD  
NANOPARTICLE USING MODIFIED TURKEVICH METHOD**

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Gold nanoparticles (AuNPs) notably known to possess unique physicochemical properties such as size and geometry-related optoelectronic properties, large surface-to-volume ratio, high biocompatibility, and low toxicity. Due to its flexibility for functionalization, the AuNPs were prepared using a modified Turkevich method with lower precursor concentrations compared to previous studies to develop a functionalized AuNP for biochemical compound detection. Characterization of the synthesized AuNPs was performed using Fourier transform infrared (FTIR) spectroscopy, UV-vis spectroscopy and dynamic light scattering (DLS) analysis. The citrate capping of AuNPs was verified using FTIR spectroscopy. Via UV-vis spectroscopy, the size of AuNP was calculated at  $10.35 \pm 2.98$  nm and the AuNP retained its stability after a month. However, DLS analysis shown high hydrodynamic diameter ( $1.95 \pm 6.8$   $\mu\text{m}$ ), suggested due to the agglomeration of AuNP. Henceforth, AuNP synthesized will be used in the development of chemical sensor for biochemical compounds.

**Keywords:** Gold nanoparticles, Synthesis and characterization, Turkevich method, Surface plasmon resonance, Stability test

**CAMPBOR TREE (*DRYOBALANOPS AROMATICA*) BARK EXTRACTS  
AS GREEN INHIBITORS OF MILD STEEL CORROSION**

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The corrosion inhibition effect of Camphor tree (*Dryobalanops aromatica*) bark methanol extract (ME) and water extract (WE) on mild steel in 0.5 M HCl was investigated by potentiodynamic polarization, electrochemical impedance spectroscopy, energy-dispersive X-ray and scanning electron microscopy methods. The inhibition efficiencies of 80.3% and 76.3% were achieved with 1000 ppm ME and WE respectively. The polarization studies revealed that both extracts of Camphor tree bark studied, are mix-type inhibitors. The Nyquist plots showed that increase in concentration of the extracts, increases charge transfer resistance and decreases double layer capacitance. Both ME and WE of Camphor tree bark obey Langmuir adsorption isotherm. The energy-dispersive X-ray and scanning electron microscopy analyses revealed that the extracts have corrosion inhibition potential.

**Keywords:** *Dryobalanops aromatica*, Corrosion inhibition, Mild steel, Potentiodynamic polarization, Electrochemical impedance spectroscopy.

**INTERFACE GROWTH OF MONOLAYER MoS<sub>2</sub>**

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Atomically thin layers of transition metal dichalcogenides (TMDs), MX<sub>2</sub> (M= Mo, W; X=S, Se), are drawing much attention recently for their unique electronic and optoelectronic properties owned. Different types of TMDs sheets have been commonly prepared on surface of a substrate, which inevitably exposes them to various potential threats during storage and device fabrication. Herein, we describe an unconventional growth of monolayer MoS<sub>2</sub> sheet directly at the interface of Au and SiO<sub>2</sub> by using alkali metal-assisted chemical vapor deposition (CVD) method. The MoS<sub>2</sub> layer was synthesized underneath deposited Au, as observed using cross-sectional transmission electron microscope. Signature MoS<sub>2</sub> peaks were recorded using optical characterizations, providing an unambiguous confirmation on its identity. MoS<sub>2</sub> grains were found to nucleate progressively from the Au-SiO<sub>2</sub> edge towards the inner region, suggesting growth via interface diffusion mechanism. The MoS<sub>2</sub> grown this way can be easily incorporated into a functional field-effect transistor device at predetermined location with desired geometry. This provides a novel means for the synthesis of TMDs, which can be possibly extended to other two dimensional materials as well. In addition, it could be useful for the position-specific patterning and growth, which facilitates sophisticated device integration.

**Keywords:** Transition metal dichalcogenides, MoS<sub>2</sub>, CVD, growth.

P-001

## SYNTHESIS AND CHARACTERIZATION OF NEW STILBENOID DERIVATIVES AS POTENTIAL VASODILATOR

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### ABSTRACT

The development of anti-hypertensive drug has become a major concern in pharmacological and medical fields as hypertension is always related to the other chronic diseases such as stroke and other coronary diseases. Stilbenoids are a class of plant phenolics containing C6-C2-C6 unit in their structures. They share a common backbone stilbene structure but differ in the type and position of substituents on the ring. Resveratrol is one of the stilbenoid that possesses three hydroxyl groups of 3, 4' and 5 positions on the *trans*-stilbene structure, had been reported for its potency to act as vasodilator that able to work directly on the muscles in blood vessel wall by dilating and preventing the tightening of the blood vessel. In this study, a series of new stilbenoid derivatives with different position and number of alkoxy group have been synthesized using Wittig reaction method with commercially available dihydroxybenzoic acids and hydroxybenzaldehydes, the *trans*- isomers were then isolated and purified using column chromatography technique. The chemical structures of synthesized stilbenoid derivatives were characterized by FT-IR, <sup>1</sup>H NMR and <sup>13</sup>C NMR spectroscopy.

**Keywords:** Stilbenoids, Wittig reaction, Resveratrol, Vasodilator, Anti-hypertensive

**EXTRACTION OF ANTHOCYANIN FROM *HIBISCUS SABDARIFFA*  
FOR THE DEVELOPMENT OF A CORN STARCH- GLYCEROL  
BASED pH INDICATOR**

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Food packaging featuring with colorimetric pH indicator is among one of the reliable intelligent packaging systems. In the present study, such a colorimetry pH indicator was prepared from corn starch and glycerol in the presence of natural dye which was extracted from roselle calyxes (*Hibiscus sabdariffa*). This inspiration arose from the sensitivity of the anthocyanin in different pH environments which could be utilized to detect food spoilage. Both corn starch and glycerol act as polymer and plasticizer, respectively thus creating solid matrices to immobilize the anthocyanin within the film. Fourier transform infrared (FT-IR) and ultraviolet-visible (UV-Vis) spectra showed the entire film ingredient compatible towards each other and the interaction between the film mixture with anthocyanin increase the stability of anthocyanin towards light. The film colour variation was measured by using chromameter after immersing in acidic and basic solution with different pH values. An application test also conducted on spoiled milk and meat to observe its potential in becoming a sensor for detecting food spoilage. The result shows the film's colour turn to pink when immersed in spoiled milk and green when in contact with rotten meat thus proving that milk turned acidic when spoiled and meat become alkaline when rotten. Therefore, combination of starch, glycerol and anthocyanin could make up a sensitive colorimetric pH indicator in food freshness evaluation.

**Keywords:** Anthocyanin, Roselle, Plasticizer, Film, Sensor.

**A SELECTIVE 'TURN-OFF' FLUORESCENT CHEMOSENSOR FOR COLORIMETRIC SENSING OF Fe<sup>3+</sup> ION IN AN AQUEOUS MEDIUM**

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A poly 1-Aminonaphthalene (1-PAN), has been synthesized via chemical oxidative polymerization and was characterized by different spectroscopic methods. The 1-PAN was then applied as a chemosensor for detection of heavy metal ions. The chemosensor shows a high selectivity and sensitivity 'turn-off' fluorescence response towards Fe<sup>3+</sup> ion. Moreover, a significant colour changes from purple to yellow can be observed by naked eyes upon addition of Fe<sup>3+</sup> ion, compared to other metal ions. A good linear response for the chemosensor was found to be 0 - 50 mg L<sup>-1</sup> and with a detection limit of 8.02 mg L<sup>-1</sup>.

**Keywords:** 1-Aminonaphthalene; Fe<sup>3+</sup> chemosensor, fluorescence analysis; colorimetric

**OBSERVATION OF CARBON NANOTUBES LOCALLY IMPLANTED  
IN MICE: IMPLICATIONS FOR TISSUE REGENERATION****<sup>1</sup>E. Hirata, <sup>2</sup>M. Yudasaka, <sup>1</sup>Y. Maeda, <sup>2</sup>T. Tanaka, <sup>2</sup>H. Kataura and A. Yokoyama <sup>1</sup>**<sup>1</sup> *Graduate School of Dental Medicine, Hokkaido University, Sapporo 060-8586, Japan*<sup>2</sup> *Nanomaterials Research Institute (NMRI), National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba Central, Tsukuba 305-8565 Japan.*

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Carbon nanotubes (CNTs) are used in various functional materials and products, predominantly in engineering but also in basic research. The high biocompatibility of CNTs makes them attractive for use in bioapplications. We previously reported that CNT-coated collagen sponges have a favorable biocompatibility profile for bone. Biodistribution is an important factor of *in vivo* behavior when evaluating the biosafety of nanomaterials by analytical methods. Many researchers have performed intravenous administration of SWCNTs to examine their kinetics and toxicity. In this study, single-walled CNTs (SWCNTs) were implanted between the periosteum and parietal bone of mice. The *in vivo* kinetics of the implanted SWCNTs were observed by near-infrared (NIR) fluorescence imaging and transmission electron microscopy (TEM). Fluorescence was observed in the cranial region, and its intensity gradually decreased over 24 hours. After 56 days, fluorescence was weakly, but clearly, observed at the implanted area. Fluorescence was not observed in other organs, including the liver, spleen, and lung, by whole-body imaging. TEM observation revealed that the SWCNTs were localized in vacuoles such as lysosomes. These findings suggest that locally implanted SWCNTs remain at the site of implantation and do not migrate in detectable quantities to other organs. We expect that this proof-of-concept for the biodistribution of SWCNTs after local implantation will promote future studies for application of CNTs to biomaterials.

**Keywords:** Carbon nanotubes, Biocompatibility, Biodistribution, Near-infrared (NIR) fluorescence imaging.

**EFFECT OF Ni<sub>2</sub>O<sub>3</sub> ADDITION ON YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub>(Ni<sub>2</sub>O<sub>3</sub>)<sub>x</sub>  
SUPERCONDUCTOR****<sup>1</sup>A. N. Jannah and <sup>2</sup>I. Mariana.**

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This study was carried out to investigate the effect of addition of nano Ni<sub>2</sub>O<sub>3</sub> on the electrical properties and on critical current density ( $J_c$ ) of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub>(Ni<sub>2</sub>O<sub>3</sub>)<sub>x</sub> superconductor. The precursor was prepared by solid state reaction method. The samples were prepared by each with weight percentage of cobalt oxide of  $x = 0.05, 0.10$  and  $0.15$ . The effect of nano Ni<sub>2</sub>O<sub>3</sub> has been investigated using Van Der Pauw method, X-ray diffraction method, scanning electron microscopy, transition temperature and critical current density  $J_c$  measurements. The four-point probe method using the  $1 \mu\text{V}/\text{cm}$  criterion was used to measure the critical temperature ( $T_c$ ) between 50 K and 300 K and transport critical current density ( $J_c$ ) between 30 K and 77 K. The  $T_c$  measurement showed that the critical temperatures of samples were decreased as the composition of Ni<sub>2</sub>O<sub>3</sub> increased. The highest  $J_c$  was observed in the  $x = 0.15$  wt. % Ni<sub>2</sub>O<sub>3</sub> sample. Van Der Pauw results showed that all samples were in electric conduction (metal) and XRD showed all samples have orthorhombic structure. The grain morphology of all the added samples was almost similar, except for minor variations in texture and porosity.

**Keywords:** Critical current density, Orthorombic, Ni<sub>2</sub>O<sub>3</sub>, YBCO.

## SYNTHESIS AND CHARACTERIZATION OF NITROGEN-DOPED Sr<sub>4</sub>Nb<sub>2</sub>O<sub>9</sub> PHOTOCATALYST BY MECHANOCHEMICAL METHOD

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Perovskite-type oxynitride often exhibit intense colors and have potential for applications such as visible light driven photocatalyst. Wu et al. reported that nitrogen-doped Sr<sub>4</sub>Nb<sub>2</sub>O<sub>9</sub>, which was known as perovskite-type oxynitride, demonstrated high oxygen generation efficiency for water splitting under visible light irradiation. However, these oxynitride have been synthesized by the direct nitridation method which required very severe reaction conditions such as high temperature (900-1100°C) and under ammonia flow for long time (e.g., 10 h). Therefore, a safer and more convenient synthesis method is required. As an alternative candidate, mechanochemical method has attracted attention. This method does not need to treat sample at high temperature under ammonia flow for long time to dope nitrogen. Instead, solid materials such as CO(NH<sub>2</sub>)<sub>2</sub> can be utilized as nitrogen sources. Therefore, in this study, we attempted to develop a more safer and simple synthesis method of nitrogen-doped Sr<sub>4</sub>Nb<sub>2</sub>O<sub>9</sub> by using the mechanochemical method. At first, Sr<sub>4</sub>Nb<sub>2</sub>O<sub>9</sub> was synthesized by a solid-state reaction method at 1000°C for 9 h in air. Then, nitrogen-doped Sr<sub>4</sub>Nb<sub>2</sub>O<sub>9</sub> was synthesized by the mechanochemical treatment. In the experiment, a planetary ball mill (Frisch, P-7) was used to grind Sr<sub>4</sub>Nb<sub>2</sub>O<sub>9</sub> and CO(NH<sub>2</sub>)<sub>2</sub> (15wt%) at a speed of 700 rpm for 1 hour, and then the obtained samples were heated at 400°C in the air for 1 hour to remove the unreacted CO(NH<sub>2</sub>)<sub>2</sub>. As a result of measuring an X-ray diffraction pattern, it showed that the structures of both before and after the mechanochemical treated samples were Sr<sub>4</sub>Nb<sub>2</sub>O<sub>9</sub>. However, the peak of the mechanochemical treated sample shifted to a higher angle by 0.02° than the original. In addition, the absorption spectrum was shifted to the visible light region. These results demonstrated that nitrogen-doped Sr<sub>4</sub>Nb<sub>2</sub>O<sub>9</sub> was successfully synthesized by using the mechanochemical method with much safer reaction condition than the conventional method.

**Keywords:** Mechanochemical method, Visible light photocatalyst, Nitrogen-doping

## PREPARATION OF AMINE MODIFIED BIMODAL MESOPOROUS SILICA PARTICLES FOR CO<sub>2</sub> SEPARATION

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Bimodal mesoporous silica (BMS), which had two different sizes of mesopores, have drawn much attention recently because it exhibited unique diffusion properties in the pores. For this reason, when BMS is modified to have affinity with CO<sub>2</sub>, it is expected that high CO<sub>2</sub> adsorption capacity is obtained by small pores and fast adsorption rate is achieved by large pores. Therefore, we prepared various kinds of amine modified BMS, and the effects of pore structure of BMS samples and the type of introduced amine on CO<sub>2</sub> adsorption/desorption properties were examined in this study. In the experiments, BMS were prepared by co-condensation of two different silica precursors. Tetraethylorthosilicate (TEOS) and triblock copolymer Pluronic 123 (P123) were used as silica precursors and templating agent, respectively. Polymethylhydrosiloxane (PMHS) was not only used as silica precursors but also as template for large pores. First, various BMS samples were prepared with different PMHS concentration, and the pore structures of the resulting samples were investigated. The results showed that three types of mesoporous silica with different pore structures (monomodal and bimodal) were successfully prepared and it was found that the pore diameter and the pore volume could be controlled by changing PMHS concentrations. To introduce amine groups to the BMS samples, 3-Aminopropyltriethoxysilane (APTES), 3-(2-Aminoethoxylamino) propyltriethoxysilane (AEAPTES), 3-[2-(2Aminoethylamino) ethylamino] propyltrimethoxysilane (AEAEAPTES) were used. Then, CO<sub>2</sub> adsorption experiment was carried out for all the amine modified samples. The results showed that by applying BMS for CO<sub>2</sub> adsorption, it was found that high CO<sub>2</sub> adsorption capacity was achieved by small pores, and high utilization efficiency of the introduced amine groups within short time (30 min) was achieved by the contribution of large pores.

**Keywords:** CO<sub>2</sub> adsorption, Bimodal Mesoporous Silica, Amine modification

**EFFECT OF POWER FRQUENCY ON VARIOUS VOLATILE ORGANIC COMPOUNDS (VOCs) DECOMPOSITION USING NON-THERMAL PLASMA REACTOR COMBINED WITH CERAMIC FILTER**

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In our previous work, we developed a new type electrode system for gas treatment in which non-thermal plasma and a ceramic filter were integrated, and applied it for VOCs treatment. Configuration of the reactor is as follows; a copper wire electrode is wound in a coil form on the surface of a tubular porous ceramic filter as a discharge electrode. Another copper rod electrode, which is insulated by a quartz tube, is inserted inside the tube, and is used as a counter electrode. The ceramic tube is made of alumina. In this study, in order to investigate the effect of both power frequency and chemical structure of VOC on the decomposition efficiency, discharge decomposition experiment of various VOCs was carried out using our non-thermal plasma reactor. In the experiment, trichloroethylene (TCE), n-hexane, benzene, n-pentane, 1-hexene, 1-chlorohexane were used as model VOCs. The power frequencies used in the experiment were 0.5, 1, 3, and 5 kHz. As a result, in the case of 1-hexene and TCE, very high decomposition efficiency was shown, and at the same time, the decomposition efficiency was significantly influenced by the power frequency. On the other hand, the decompositions of 1-chlorohexane, n-hexane, n-pentane and benzene were influenced by the power frequency slightly, and the decomposition efficiency was remarkably lower than the case of 1-hexene and TCE. Furthermore, when the decomposition efficiency of n-hexane was compared to 1-chlorohexane, which has almost the same structure as n-hexane and just replacing one hydrogen to chlorine, the efficiency in 1-chlorohexane decomposition was higher at any frequency. In addition, when the decomposition efficiency was compared between n-hexane and n-pentane, no significant difference was observed. It indicates that only one carbon number difference does not affect discharge decomposition largely in the range of this study.

**Keywords:** Non-thermal Plasma, VOCs decomposition, Power Frequency.

**EFFECT OF PROPERTY OF CERAMIC FILTER ON NONTHERMAL  
PLASMA REACTOR FOR TRICHLOROETHYLENE  
DECOMPOSITION**

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Emission control of volatile organic compounds (VOCs) has been conducted in various countries because VOCs is one of the causes of air pollution such as the photochemical oxidants and suspended particulate matter (SPM). Therefore, the VOCs treatment with non-thermal equilibrium atmospheric pressure plasma has attracted attention recently, because this VOC removal reactor has simple structure that enable us to treat VOCs with low cost and less space. In our laboratory, we have been developing the new type VOC removal reactor, named SPCP reactor, and have been evaluating the decomposition characteristics of VOCs using the reactor. This reactor combines a ceramic filter and the SPCP (surface corona discharge induced plasma chemical process) method, which is a gas phase reaction method using discharge plasma. The SPCP reactor is characterized by high contact efficiency between plasma and VOCs, since the process gas passes through the ceramic filter on which plasma is generated. In this study, we examined the effect of the property of the ceramic filter and the power supply frequency condition on VOCs decomposition. In the experiment, trichloroethylene (TCE) was used as a model VOC pollutant. TCE decomposition experiments were performed using three types of ceramic filters having different pore diameters and inner and outer diameters. As a result, in the case of the experiment which was performed using a ceramic filter with 1.2  $\mu\text{m}$  pore diameter and power supply frequency of 0.2 kHz, highest decomposition efficiency of TCE was obtained. At this time, SED (Specific Electronic Density) in TCE 90% decomposition was 87.1 J / L, and it was the highest efficiency we achieved in our laboratory without using catalyst.

Keywords: Non-thermal plasma, VOC decomposition, Ceramic filter.

## N DOPED GRAPHENE QUANTUM DOTS/TITANIUM DIOXIDE NANOCOMPOSITES AS PHOTOTHERMAL AGENTS IN PHOTODYNAMIC THERAPY (PDT)

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Malignant tumours owe the high mortality rates which accounts for over 8 million deaths annually. Much effort has been devoted to identify an efficient approach for treating early-stage and locally advanced breast cancer. Among the emerging cancer therapy methods, photodynamic therapy (PDT) surpasses the traditional methods such as surgery, chemo- and radiation therapy because it is non-invasive in nature, has fewer side effects, causes negligible drug resistance and has low systemic toxicity. The effective use of PDT depends on the type of photothermal agents employed since the cancerous cells are locally killed by reactive oxygen species (ROS) produced by the photothermal agent under illumination and in the presence of oxygen. Some of the limitations of existing activatable organic photothermal agents such as porphyrin, phthalocyanines and bacteriochlorin derivatives are poor water dispersibility, photostability and their inability to be absorbed in the region (>700 nm). In terms of photostability and water dispersibility, semiconductor photothermal agents such as porphyrin derivatives are superior to existing PDT agents, however it suffers from low ROS-generation efficiency. N doped graphene quantum dots (N-GQD) have promising applications in bioimaging and biosensing particularly due to their photoluminescence effect. It has huge advantages of having large surface-area, broad and size-dependend absorption and emissions, good solubility, exceptional biocompatibility and low toxicity which make it good PDT agents. Besides, titanium dioxide nanoparticles (TiO<sub>2</sub>) are non-toxic to mammalian cells and their photocatalytic traits make them potential PDT agents. However, their applications for PDT is limited due to their wide bandgap (~3.2eV) which generates the ROS in the presence of ultraviolet light. UV light has poor penetration into biological tissues, besides damaging DNA. Thus, in this study N-GQDs/TiO<sub>2</sub> nanocomposites are developed as novel to activate TiO<sub>2</sub> NPs *in vivo* to generate ROS by modifying bandgap to the visible and/or NIR. The latter can achieve active targeting to tumour cells without harming healthy tissues and it also has hydrophilic properties which increases the cellular uptakes. Effective and improvised photothermal agent is needed for the treatment to save more lives.

**Keywords:** N-GQD/TiO<sub>2</sub> nanocomposites, photodynamic therapy, activatable photothermal agents, semiconductor quantum dot, reactive oxygen species

**DEVELOPMENT OF NOVEL UPCONVERSION NANOPARTICLES  
FUNCTIONALIZED WITH AMPHIPHILIC POLYMERS FOR  
DETECTION OF ALPRENOLOL**

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Upconversion nanoparticles (UCNPs) are an unique type of photoluminescence (PL) material in which lower energy excitation is converted into higher-energy emission via multi-photon absorption processes. In this research, novel amphiphilic copolymers were synthesized to act as functionalizing agents for NaLuGdF<sub>4</sub>:Yb<sup>3+</sup>/Er<sup>3+</sup> UCNPs. The UCNPs coated with amphiphilic copolymers were characterized by using FTIR, <sup>1</sup>HNMR, SEM, TEM, UV, and PL instruments. These materials were used as optical sensors to detect Alprenolol which is used for the treatment of high blood pressure. Our developed sensors displayed good analytical performance such as low limits of detection and good linearity. Furthermore, the sensors were applied to determine the level of high blood pressure drugs in human urine and serum samples with good recoveries.

**Keywords:** Upconversion nanoparticles, Amphiphilic copolymers, Optical sensors, Alprenolol, Human serum samples, Human urine samples

## NEW TURN-ON FLUORESCENT SENSOR FOR HIGHLY SENSITIVE DETECTION OF SPERMINE BASED ON AMPHIPHILIC POLYTHIOPHENE NANOHYBRIDS

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New fluorescent sensors based on CdTe quantum dots (P1QDs and P2QDs) coated with amphiphilic polythiophenes were developed for the detection of spermine. The emission of the P1QDs and P2QDs is found to be quenched in the presence of heparin by electron transfer mechanism through electrostatic and hydrogen bonding interactions. Upon the addition of spermine, heparin has been removed from the surface of P1QDs and P2QDs due to the strong electrostatic interactions between spermine and heparin, which showed critical fluorescence recovery of the sensors. Under optimum conditions, both P1QDs and P2QDs sensors were shown excellent analytical performance for spermine with the limits of detection (LOD) of 2.90 nmol L<sup>-1</sup> and good linearity ( $R^2=0.9957$ ) within 0.05-15  $\mu\text{mol L}^{-1}$  and LOD of 1.66 nmol L<sup>-1</sup> and good linearity ( $R^2=0.9928$ ) within 1-12  $\mu\text{mol L}^{-1}$ , respectively. The P1QDs and P2QDs sensors were shown a high selectivity to spermine over common interfering substances. Furthermore, the proposed method was successfully utilized in serum samples with gratifying results.

**Keywords:** Amphiphilic polythiophene nanohybrids, Spermine, Fluorescent sensors, Serum samples, Electron transfer

**A COLORMETRIC SENSOR FOR DETECTION OF HEXAVALENT CHROMIUM BASED ON GRAPHENE OXIDE**

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Graphene oxide (GO) was applied as a peroxidase mimetic catalyst to detect hexavalent chromium (Cr(VI)). The detection based on the color change of tetramethyl benzidine (TMB), which was catalyzed by GO. The color of the probe will be turned “OFF” and “ON” by adding 8-hydroxyquinoline (8-HQ) and Cr(VI), respectively. The mechanism could be explained due to the chelation reaction between 8-HQ and Cr(VI). The color change can be observed by naked eyes or UV-vis spectrophotometer. Under the optimal experimental conditions, the prepared probe exhibited high selectivity, limit of detection (6 nM), and large linear range. This study was applied successfully for Cr(VI) detection in real water.

**Keywords:** hexavalent chromium; chelate; graphene oxide; mimetic catalyst; naked eyes.

**EXTRINSIC-INTRINSIC APOPTOSIS SIGNAL PATHWAY OF A  
NOVEL DIVALENT NICKEL COMPLEX WITH S-BENZYL-  
DITHIOCARBAZATE LIGAND**

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A novel nickel (II) complex was synthesized and characterized by means of various physical, chemical and spectroscopic process. The X-ray single crystal diffraction analysis indicated two independent close comparable bischelated square planar complexes of Tran's configuration, where dithiocarbazate ligand coordinated via *N, S*-donor set. The bis(dithiocarbazato)nickel(II) complex is able to inhibit Ehrlich Ascites Carcinoma (EAC) cells expansion by 51.81%, with 0.3 mg/kg/day body weight dose administered intraperitoneally for five successive days in Swiss Webster mice. After LD<sub>50</sub> (1000 mg/kg) determination, the dose was adjusted as 50 mg/kg and upon administration, inhibition improved to 75.75%. The inhibitory effect of the complex was 26.6-76.4% against EAC cells (concentration 31.25-500 µg/ml) as determined by MTT colorimetric assay. Optical and Fluorescence microscopic techniques were used to determine the apoptotic cell morphological change. The expression pattern of apoptosis regulatory genes (increased expression of P<sup>53</sup>, Bax, Cas-8, Cas-9, Cas-3, cyt-C, TNFα and decreased expression of Bcl-2 gene) were observed in the cells treated with the nickel (II) complex for five consecutive days. Also, we studied the effect in silico of the synthesized nickel (II) complex and of a standard drug, Bleomycin. Molecular docking coupled with molecular dynamics simulation studies were used for both drugs with the aim to support experimental approach and to investigate whether our synthesized compound exhibits chemo-preventive effect towards targeting apoptotic genes. Both experimental and computational work uncover that the nickel (II) complex inhibit EAC cells growth successfully, suggesting a potential new approach for cancer treatment.

**Keywords:** Nickel (II) complex, Dithiocarbazate, EAC cells, Apoptosis, DNA fragmentation.

## **CHARACTERIZATION OF ZNO DOPED AG SYNTHESIZED BY LOW-TEMPERATURE AQUEOUS CHEMICAL BATH METHOD**

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In this work, the characterization of synthesis ZnO/Ag nanocomposite has been studied. In this study the seed layer ZnO 100 nm was done by RF sputtering on the glass substrate, following the ZnO-Ag thin film was deposited with different concentration of Ag (0, 1, 3 and 5%). The thin films preparation was conducted through the chemical path method and then annealing at 400 C° for one hour. The characteristics were studied using X-ray diffraction (XRD), FE-SEM Field Emission scanning electron micrographs (FE-SEM), Energy-dispersive X-ray spectroscopy (EDX) and UV–vis spectroscopy. The XRD analysis shows polycrystalline with a hexagonal wurtzite structure and the crystallite size decreasing with increase the Ag content. The morphology of as-grown ZnO thin films was investigated using (FE-SEM), the results show variant morphology, nanowires on ZnO pure and hexagonal flakes by increasing Ag. The nanowires have diameter 30 nm with length 3 $\mu$ m and the nanoflakes have diameter 500 nm. The elemental composition via EDX analysis showed the ZnO/Ag nanocomposites successfully. The optical properties study showed that the transmittance decreased by increasing Ag content and the energy band gap was decreased from  $E_g=3.28$  eV, to 3.26 eV.

**Keywords:** Nanocomposite, Zinc oxide, Characterization, Crystallite size, Energy band gap.

## **RAPID AND SENSITIVE FLUORESCENT PROBE FOR THE DETERMINATION OF ENVIRONMENTAL POLLUTANT USING MOLECULARLY IMPRINTED POLYMER NANOHYBRIDS**

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One of the emerging issues is the occurrence of pharmaceutical compounds and their metabolites in the environmental water treatment. Carbamazepine (CBZ) is one of the most pharmaceutical residues that have been frequently detected in the environmental water. In this work, molecularly imprinted polymer coated upconversion particles (UCPs@MIP) using the internal visible emission from UCPs upon excitation with a 980-nm light source has been synthesized as a fluorescent probe for the detection of CBZ in the environmental water. Compared to the direct light, the emission from UCPs is weak. Thus, polymerization is confined to the close proximity of the UCPs and the thickness of MIP shell can be tuned by changing the polymerization time. The  $\pi$ - $\pi$  stacking, electrostatic and hydrogen bond between CBZ and UCPs@MIP can efficiently enhance the selective recognition force. Under optimum conditions, the results of the fluorescence quenching analysis showed excellent sensitivity (LOD, 28.5 pM) with good linear ranges of 0.5 nM to 500  $\mu$ M for CBZ and a correlation coefficient of 0.992. Moreover, the practical application of the developed sensor for CBZ detection in tap and river water samples has been successfully described with satisfactory recoveries of 98.24-104.23% and RSD less than 5%. Interestingly, the synthesized UCPs@MIP revealed high selectivity over several structural analogues. This work offers a novel facile strategy to synthesize a UCPs@MIP fluorescent probe with selective, rapid and accurate sensing of CBZ in the environmental water.

**Keywords:** Molecular imprinting polymers, UCPs, Carbamazepine (CBZ), River water.

**MURAYYA KOENIGII AS GREEN CORROSION INHIBITORS FOR MILD STEEL IN CO<sub>2</sub> SATURATED 3.5 % NaCl MEDIUM**

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Green corrosion inhibitor is one of the recent emerging remedy to prevent the oil and gas pipeline corrosion. In this study, the leave extract of *Murraya koenigii* (Curry leaf) had been tested as corrosion inhibitor for mild steel corrosion in CO<sub>2</sub> saturated 3.5% NaCl medium. Standard corrosion techniques viz., weight loss and electrochemical technique (potentiodynamic polarization) were employed to study the corrosion inhibition properties. The changes of surface morphology on mild steel specimens before and after corrosion studies were screened through scanning electron microscopy (SEM) combined with energy dispersive X-Ray analyzer (EDX). In addition, Fourier Transform Infrared (FTIR) Spectroscopy was used to characterize the leave extract and the corrosion protective film formed over mild steel surface. Results shown that *Murraya koenigii* leave extract successfully reduce the corrosion rate through adsorption process which found follows the Langmuir adsorption isotherm.

**Keywords:** Green corrosion inhibitor, Mild steel, *Murraya koenigii*, Potentiodynamic polarization, SEM

## METAL-FREE SYNTHESIS AND HEPATOPROTECTIVE EFFECTS OF CARBAZOLE-3-CARBOXYLIC ACIDS

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Carbazoles are a large group of natural and synthetic tricyclic heteroaromatic alkaloids. Carbazoles and their derivatives are of great interest in the research fields of synthetic chemistry and medicinal chemistry, due to their tricyclic structure and widespread pharmacological properties. For instance, naturally occurring carbazole-3-carboxylic acid alkaloids, for example mukoeic acid, isomukonidine, clausine K, and pityriazole, exhibit promising biological activities. Inspired by their significance, new carbazole-3-carboxylic acid derivatives have been designed and synthesized from carbazole-3-carboxyaldehydes *via* metal-free protocol. Oxidation of carbazole-3-carboxyaldehydes promoted by a 70% aqueous solution of *tert*-butylhydroperoxide provides straightforward access to the corresponding carbazole-3-carboxylic acids in good yields with good functional group compatibility. Evaluation of hepatoprotective effects of the new carbazole-3-carboxylic acids against H<sub>2</sub>O<sub>2</sub>, a known hepatotoxin, in a human liver cell line (HepG2) were carried out. The findings showed that pre-treatment of HepG2 cells with carbazole-3-carboxylic acids prior to exposure of H<sub>2</sub>O<sub>2</sub> effectively increased cell viability. Similarly, co-exposure to H<sub>2</sub>O<sub>2</sub> and carbazole-3-carboxylic acids was also found effective. Their protective effect seems to be mediated by inhibiting intracellular ROS generation. In summary, a metal-free, simple, and inexpensive method for oxidation of carbazole aldehydes to furnish the corresponding acids was accomplished. Additionally, utilization of these compounds as hepatoprotective agents in HepG2 cell culture model against H<sub>2</sub>O<sub>2</sub>-induced hepatotoxicity was effective on pre- and co-treatment conditions. Therefore, the new carbazole-3-carboxylic acids could be promising as potential therapeutic agents for liver diseases.

**Keywords:** Carbazole-3-carboxylic acid, oxidation, *tert*-butylhydroperoxide, hepatoprotective effects, H<sub>2</sub>O<sub>2</sub>, HepG2 cells.

## STUDY ON TOTAL SUSPENDED SOLID IN LANGAT RIVER BASIN

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Total suspended solid is one of the parameter to determine water quality. Increasing value of suspended solid can cause deterioration of river water quality. High levels of total suspended solids will increase water temperatures and decrease dissolved oxygen (DO) levels. Total suspended solid is a solid particle that larger than 2 microns that suspended in water and can be measured from lab analysis. Preservation of samples with refrigerate at 4°C before lab analysis are important to minimize microbiological decomposition of solids. The analysis must be performed within seven (7) days to get the accurate result. It must be measured using filtration method according to American Public Health Association (APHA). This study shows results of total suspended solid from upstream to downstream of Langat river basin. Langat river is one of the largest raw water sources in Selangor and faced various problems due to natural or human activity along the basin. The present result range from 0-200mg/L demonstrated the potential negative impacts of water quality deterioration in Langat river basin. The total suspended solids in Langat river basin can be related to human activity and landuse activity. TSS value in Langat river basin is still within the limits and considered to be safe for human use and also aquatic organisms in river.

**Keywords:** Solids, Total suspended solid, Water quality, River.

**REPEATABILITY AND REPRODUCIBILITY IN BISPHENOL-A AND  
BISPHENOL-S IDENTIFICATION VIA TRIPLE QUADRUPOLE  
LIQUID CHROMATOGRAPHY MASS SPECTROMETRY**

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An analytical method was developed for the accurate determination of Bisphenol-A (BPA) and Bisphenol-S (BPS) in drinking water using triple quadrupole Liquid Chromatography Mass Spectrometry (LCMS/MS) system. A mixture standard solution of BPA and BPS with the concentration of 5 ppb were spiked in the drinking water and analysed using LCM/MS in the negative ion mode without further clean up or extraction steps. The chromatographic separation was achieved on reverse phase ZORBAX Eclipse Plus C18 Rapid Resolution HD, 2.1x50mm 1.8-Micron using gradient elution mobile phase of 90% Acetonitrile and 5mM of Ammonium Acetate as a buffer. The separation operates at the flow of 0.4 mL/min and runtime 11 min. BPA were detected at  $m/z$  227.1  $\rightarrow$  212 and  $m/z$  227.1  $\rightarrow$  133 while BPS were detected at  $m/z$  249.02  $\rightarrow$  184,  $m/z$  249.02  $\rightarrow$  155.9,  $m/z$  249.02  $\rightarrow$  108 and  $m/z$  249.02  $\rightarrow$  92. Repeatability and reproducibility studies showed that the LCMS/MS method is a reliable and reproducible, which can provide less than 20% relative standard deviation of method precision for analysis and 90-120 % of recovery respectively. Thus, this method was successfully applied to the trace determination of BPA and BPS in drinking water matrix using LCMS/MS.

**Keywords:** Bisphenol-A; Bisphenol-S; triple quadrupole liquid chromatography mass spectrometry; repeatability; reproducibility.

## TURBIDITY REMOVAL OF POLYACRYLAMIDE-GRAFTED EGG WHITE

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Polyacrylamide grafted egg white (Egg-g-PAM) has been successfully synthesized by microwave-assisted technique using ceric ammonium nitrate (CAN) as an accelerator. The grafting process was optimized at various acrylamide wt%, CAN concentration and microwave irradiation time. The optimized Egg-g-PAM and egg white (control) were characterized by Fourier transform infrared (FTIR) spectroscopy, Field emission scanning electron microscopy (FESEM) analysis, elemental analysis, thermogravimetric analysis (TGA), x-ray diffractometry (XRD) and point of zero charge (pHzpc). The % of turbidity removal for optimized Egg-g-PAM, alum and egg white were determined on a 0.25 wt% of kaolin suspension using standard 'jar test' procedure. The optimized PAM-g-Egg with 56.2% of grafting efficiency (GE) exhibited 95.55 % of turbidity removal at pH 7 and 2 g/dl dosage. The optimized Egg-g-PAM showed greater turbidity removal than that of alum (94.58%) and egg white (19.89%).

**Keywords:** Albumin, graft copolymer, microwave-assisted technique, engineered flocculant, coagulant

**RECEPTOR DESIGN AND SYNTHESIS BASED ON ELECTRON DEFICIENT 3,5-DINITROBENZAMIDE GROUP PENDANT WITH LEUCINE AND TRYPTOPHAN RESIDUE FOR CONCENTRATION DEPENDENT FLUORIDE ANIONS RECOGNITION**

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The role of H-bonding and anion- $\pi$  interaction in anion recognition specifically fluoride ( $F^-$ ) anion has recently got immense attention, given the implications in chemistry, biology and environment. In this connection, wide variety of synthetic and natural receptors containing amide, urea, pyrrole and indole has been explored as anion- receptors where the main force of interaction was found to be H-bonding. However, the elucidation of the halide binding site in *haloalkane dehalogenase* provided further impetus on exploration of the nature of interactions between indole (i.e. tryptophan) and the halide anions which illuminated the hitherto unknown chemistry of tryptophan in binding and recognition of halide anions. Most of these  $F^-$  receptors were found to be colorimetric. These colorimetric anion-receptors could be further developed as a sensor targeted for a specific anion like  $F^-$  or other anions which is an important application in the field of supramolecular chemistry. However, the importance of anion- $\pi$  interaction involving the complexation of anions with electron deficient aromatic groups has recently got immense attention. On the basis of this background we sought to know, whether the interaction of  $F^-$  anions with electron deficient 3,5-dinitrobenzamide system could be influenced by electron rich tryptophan residue and thereby synthesized a 3,5- dinitrobenzamide-based receptor incorporated with tryptophan, **R1** and the other with leucine for comparative study. The interactions of  $F^-$  anions with 3,5-dinitrobenzamide derived leucine and tryptophan receptors, **R1** and **R2** respectively produced dramatic concentration-dependent chromogenic effects which could be monitored using UV-visible spectroscopy. <sup>1</sup>H NMR spectroscopy revealed that the coordination of  $F^-$  anions to **R2** was driven by H- bonding and anion- $\pi$  interactions involving the tryptophan residue and 3,5-dinitrobenzamide group. In addition, we have developed a chromogenic chemo sensor for  $F^-$  anions from **R2**, immobilized in polystyrene film, which was compatible in aqueous solutions.

**Keyword:** Fluoride, Tryptophan, Indole, Anion recognition

**GENETIC VARIATION OF MGMT, XRCC1 AND XPD GENES  
AND LIFE STYLE HABITS ACT AS POTENTIAL RISKS FOR  
OESOPHAGEAL CARCINOMA IN THE NORTH EAST  
INDIAN POPULATION**

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In India the highest incidence of Esophageal cancer (ECA) has been reported from Assam in the North-east India. We conducted a case-control study among individuals diagnosed with ECA and healthy controls and its association with lifestyle. SNPs of DNA repair gene XRCC1, XPD and MGMT were determined by PCR-RFLP and HRM (High Resolution Melting) technique. In XRCC1 Arg399Gln codon the Arg/Gln genotype [OR=1.21, Chi sq=0.43, p<0.05] and Gln/Gln [OR=0.0, Chi sq=7.13, p<0.05] were significantly correlated with ECA. For Arg280His, Arg/His [OR=5.04, Chi sq=19.38, p<0.05] and His/His [OR=12.09, Chi sq=21.62, p<0.05] were observed to have association with ECA. In XPD Lys751Gln the Gln/Gln variant share positive association [OR=0.45 at 95%CI, Chi sq=4.26, p<0.05] whereas the Lys/Gln genotype showed no association between cases and controls. XPD Asp312Asn polymorphism showed statistically significant associations in Asp/Asn [OR = 0.22 at 95% CI, Chi sq= 9.68, p<0.05] and for Asn/Asn [OR=0.22 at 95% CI, Chi sq=12.5, p<0.05]. In MGMT rs7087131 polymorphism, the heterozygous variant (G/A) [OR=1.6 at 95%CI, Chi sq=2.68, p=0.1] and homozygous variant (A/A) did not have significant association with ECA. In case of MGMT rs7087131, variant genotype GA+AA were less in alcohol consumers (32.3%) than non-consumers (48.7%); (86%); smokers (27%) non-smokers (76%); in betel quid consumers (38.6%) than non-consumers (84.5%); in tobacco consumers (25%) than non-consumers (69%). In conclusion, our study supports the idea that polymorphisms in DNA repair genes, XRCC1 codon 399, 280 and XPD codon 751 and 312 may be associated with ECA risk in Northeast India. Lifestyle risk factors, such as smoking, alcohol, betel quid and tobacco has positive association with ECA. Our results suggest that the XRCC1 Arg399Gln, Arg280His and XPD Lys751Gln, Asp312Asn variant genotypes are associated with reduced risk of ECA in this high-risk Northeast Indian population.

**Keyword:** SNPs, MGMT, XPD, XRCC

**SYNTHESIS, CHARACTERIZATION AND DETERMINATION OF  
MESOPHASE TRANSITION AND FIRE RETARDANT PROPERTIES  
OF HEXASUBSTITUTED CYCLOTRIPHOSPHAZENE MOLECULES  
WITH SCHIFF BASE AND AMIDE LINKING UNITS**

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Nucleophilic substitution reaction between 4-hydroxybenzaldehyde and hexachlorocyclotriphosphazene, HCCP formed hexakis (oxy-4-benzaldehyde) cyclotriphosphazene, **1**. Intermediates **2a-e** was formed from the alkylation reaction of methyl 4-hydroxybenzoate with alkylated bromide (heptyl, nonyl, decyl, dodecyl and tetradecyl) which further reduced to form benzoic acid. Further reaction of **2a-e** and other substituted benzoic acid (OH, COOH, Cl and NO<sub>2</sub>) formed **3a-i** which then reduced to give subsequent amine **4a-i**. Condensation reaction between **1** and **4a-i** yielded new hexasubstituted cyclotriphosphazene compounds, **5a-i** having Schiff base and amide linking units. Compound **5j** was formed from the reduction of **5i**. All the intermediates and compounds were characterized using Fourier Transform Infrared spectroscopy (FT-IR), <sup>1</sup>H and <sup>13</sup>C Nuclear Magnetic Resonance spectroscopy (NMR) and CHN elemental analysis. Mesophase texture of these compounds were determined using Polarised Optical Microscope (POM) and their mesophase transition were further confirmed using Differential Scanning Calorimetry (DSC). Only compounds **5a-e** exhibited smectic A phase while other intermediates and final compounds are found to be non-mesogenic with no liquid crystal behaviour. The study on the application of fire retardant properties of the hexasubstituted cyclotriphosphazene compounds was done using Limiting Oxygen Index (LOI) testing. In this study, the polyester resin has been used as a matrix for moulding and the LOI value of pure polyester resin was 22.53%. The LOI value was increased to 24.71% when the polyester resin was incorporated with 1 wt% of hexachlorocyclotriphosphazene (HCCP), indicating that HCCP has a good fire retardant properties. Generally, all the final compounds showed a positive results and the highest LOI value was belonged to compound **5i** with 28.53%.

**Keywords:** Schiff base, amide, cyclotriphosphazene, mesophase, fire retardant.

**REMOVAL OF METHYLENE BLUE DYE FROM AQUEOUS SOLUTION USING DRIED *PANDANUS HELICOPUS* LEAVES**

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*Pandanus helicopus* or locally known as *rasau*, is a fast growing species of freshwater swamp which is abundant in Chini and Bera Lake. From observation, this species is normally chopped down to make ways for boating activities or burnt in Chini Lake. The plant waste/ biomass is disposed on-site which is an eye-sore. Hence, study was carried out to investigate the effectiveness of dried *Pandanus helicopus* leaves (DPHL) in the removal of methylene blue (MB) dye. The effects of initial MB concentration, adsorbent dosage, contact time, initial pH and adsorbent particle size were investigated. It was found that MB removal decreased with the increase of initial concentration at fixed amount of adsorbent. MB uptake increased with when adsorbent dosage increased whereas MB removal reached equilibrium after 6 hrs. MB removal efficiency increased with the increase of initial pH value in adsorbate from acidic to alkaline condition whereas MB uptake varied at different adsorbent particle sizes. Isotherm, kinetic and adsorption capacity of the DPHL on MB removal were also determined in this study. In conclusion, DPHL is potential to remove pollutants such as dye.

**Keywords:** *Pandanus helicopus*, Methylene blue removal, Isotherm, kinetic, Adsorption capacity