CHEMICAL, MATHEMATICAL AND PHYSICAL SCIENCES

SYNTHESIS AND CHARACTERIZATION OF MOLECULARLY IMPRINTED POLYMER USING TRANS **OLEIC FATTY ACID AS TEMPLATE**

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Labelling of trans fatty acid (TFA) content in foods is mandatory in many countries. The impact of TFA content in food labels prompts analytical laboratories to address outstanding issues on trans fatty acid analysis. In this work, a molecularly imprinted polymer (MIP) using trans oleic fatty acid methyl ester (FAME) as template was prepared by precipitation polymerization method using methacrylic acid as functional monomer, trimethylolpropane methacrylateas cross-linking agent, 2,2-azobis (isobutyronitrile) as the radical initiator, and dichloromethane as porogen. A non-imprinted polymer (NIP) was also prepared. Template removal was done by soxhlet extraction using methanol-acetic acid (9:1 v/v) as the extraction solvent. The binding properties of trans oleic FAME imprinted polymers were evaluated in different solvent systems by equilibrium experiments. Scatchard plot analysis in heptanes revealed that there were two classes of binding sites populated in the imprinted polymers which indicated that the polymer possesses heterogeneous binding sites distribution. The stronger affinity binding type exhibits dissociation constant (K₄) 10 times smaller than that of the weaker type with binding capacity of 48.04 ugtrans oleic FAME/mg MIP. The resulting Freundlich isotherm further demonstrated the heterogeneity of the binding sites of the MIP, with heterogeneity index "a" equal to 0.4758. Polymer characterization was done by scanning electron microscopy (SEM) and Elemental analysis (Combustion Method) to support the imprinting and rebinding process of trans oleic acid. This MIP is a potential adsorbent material for the solidphase extraction (SPE) of trans oleic acid in food.

Keywords: Molecularly imprinted polymer, trans oleic acid, precipitation polymerization, Scatchard plot, Freundlich isotherm

ADSORPTION OF VOCS BY CARBONIZED OKRA STALKS

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Volatile organic compounds (VOCs) have become the focus of environmental health researches due to their role in indoor air pollution, called the sick building or sick house syndrome. The search for novel gas adsorbents has led to potential applications of lignocellulosics and waste biomass, such as kenaf, hay, peanut hulls, sawdust, and others. The okra stalk (Abelmoschus esculentus Moench syn. Hibiscus esculentus L.) was carbonized, activated, and tested for its potential in adsorbing VOCs specifically, formaldehyde, toluene, and xylene. Characterization of the okra stalk in its raw and carbonized form was done. Its ability to adsorb vapor, and mesoporosity and microporosity were also evaluated. VOC adsorption was determined by closed chamber method using a vaccuum desiccator. The concentration of VOC in the chamber was measured using Formaldemeter hTV and corresponding Gastec indicator tubes. Adsorption of gases particularly polar and non-polar VOCs onto carbonized and activated okra stalks was shown to be promising, particularly with formaldehyde adsorption, which may be due to the hydrophilicity of formaldehyde and the ability of carbonized okra to retain vapor. Further, the okra stalks adsorbed better than the commercial activated carbon. A higher rate of adsorption was observed from the non-activated samples which could indicate that the number of polar sites on the carbon surface decreased with activation. On the other hand, non-polar VOC adsorption (toluene and xylene) showed that okra stalks activated at 800! and 900! adsorbed better than its non-activated counterpart.

Keywords: VOC, adsorption, okra stalks, charcoal, formaldehyde

ANTI-ANGIOGENIC PROPERTY OF THE AQUEOUS EXTRACT OF Canarium ovatum LEAVES USING CAM ASSAY

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The aqueous leaf extract (ALE) of Canarium ovatum was evaluated for possible anti-angiogenic effect in the duck (Anas platyrynchos) embryos using chorio-allantoic membrane (CAM) assay. In the study, C. ovatum ALE at 1, 10 and 100 µg/mL was administered in ovo at day 3 of incubation using a modified windowing technique. Results revealed a significant inhibition in blood vessel formation in all the C. ovatum ALE treated samples in a dose dependent manner compared to the control group given with double distilled water only. The embryos treated with the highest dose (100 µg/mL) exhibited much reduction in primary blood vessel count and secondary blood vessel thickness resulting in relatively smaller embryos. The findings confirm the anti-angiogenic activity of *Canarium* ovatum. Further studies are warranted to determine its active components, the possible mechanism of action and therefore establish its potential as an antitumor agent.

Keywords: aqueous leaf extract, Canarium ovatum, anti-angiogenic, Anas platyrynchos, chorio-allantoic membrane assay

AN ELECTRONIC NOSE BASED ON CONDUCTING POLYMERS FOR THE DIFFERENTIATION OF PHILIPPINE COFFEE VARIETIES

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An electronic nose (e-nose) based on conducting polymers was developed to discriminate different varieties of Philippine coffee: Arabica, Excelsa, Liberica, and Robusta. Conducting polymers such as polyaniline and polypyrrole doped with various counter-ions were utilized as the molecular recognition element in the e-nose system. Thin-film of conducting polymers were electro-deposited through potentiostatic polymerization onto the gap between the two gold wires (diameter = 620 im; distance = 200 μm) set on a Teflon substrate and these conducting polymers were conditioned at a potential of 250 mV. The sensors response was recorded as the voltage produced by a linearized wheatstone bridge circuit. The sensors responded rapidly when exposed to the headspace of the coffee beans sample. The response exhibited good reversibility and reproducibility. The response of the sensor array in the e-nose system exhibited distinct patterns for each coffee variety. This was visualised by bar graph and radar plot. Chemometric analysis through pattern recognition techniques such as principal component analysis (PCA) and cluster analysis (CA) highlighted clusters for each coffee variety. This system offers a simple, low-cost and reliable method for the discrimination of Philippine coffee variety. It can also be applied for the authentication for coffee variety.

Keywords: electronic nose, conducting polymers, coffee, principal component analysis and cluster analysis

APPLICATION OF DIGITAL IMAGING COLORIMETRY TO THE DETERMINATION OF METAL ION **CONCENTRATION IN AQUEOUS SOLUTION**

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Digital imaging colorimetry (DIC) is an alternative method for quantitative measurements in analytical chemistry, wherein accessible digital devices and images that employ the RGB color space are used. In this study, DIC was employed to quantify Co(II), Ni(II) and Cu(II) in aqueous solution. The study was carried out using a digital camera designed in a fixed position over an improvised light box set-up. The acquired image was digitized on the RGB color space using Adobe Photoshop. Then, calibration curves for each of the metal ions was constructed by plotting the concentration of metal ion against the selected digitized color values under optimized conditions (i.e., camera distance and filter color). The highest linearity and sensitivity for each calibration curve was found to be associated with the digitized color value (R, G or B) complementary to the color of the metal ion solution. The dynamic linear range for each calibration curve was from 0.10 M to 0.60 M of the metal ion, and the linearity and sensitivity were 0.992 r² and -291.6 G-value/M for Co(II); 0.996 r² and -114.6 Rvalue/M for Ni(II); and 0.997 r² and -161.0 R-value/M for Cu(II). These calibration curves were comparable with those constructed using the conventional spectrophotometric method.

Keywords: Digital imaging colorimetry (DIC), colorimetric sensing, RGB color coordinate, Co(II), Ni(II), Cu(II), spectrophotometric method

AQUEOUS AND METAL-FREE ASYMMETRIC ALDOL REACTIONS ORGANOCATALYZED BY CHIRAL 2-PYRIDYLIMIDAZOLINES – A GREEN APPROACH TOWARDS STEREOSELECTIVE C-C BOND FORMATION

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Among the frontier challenges in the 21st century is the development of asymmetric reactions with excellent enantioselectivity and atom economy. After the birth of proline-catalyzed asymmetric carbon-carbon bond forming reactions, an explosion of various organocatalytic methodologies have been reported. Among the organocatalyst manifolds, the catalytic utility of 2imidazolines has been less explored. In this study, various 2pyridylimidazolines were screened for their catalytic activity towards direct asymmetric Aldol reaction. The chiral catalysts were synthesized through iodine-promoted oxidative condensation and cyclization of 2-pyridine carbaldehyde (or 2,6-pyridine dicarbaldehyde) with chiral 1,2-diamine derivatives in excellent yields. Various catalytic parameters were optimized to determine the best reaction condition, such as catalyst loading, temperature, reaction time, and solvent. The best conditions which gave excellent enantioinduction (up to 99:1 enantiomeric ratio) and yield were observed with brine as the solvent, a one hour reaction period at room temperature, and 10 mol% as the minimum catalyst load. Thus, our study demonstrates a green and sustainable approach en route the asymmetric construction of β-hydroxy carbonyl structures.

Keywords: organocatalysis, 2-pyridylimidazolines, Aldol reaction, asymmetric catalysis

BIOMOLECULAR AND CHROMATOGRAPHIC CHEMICAL FINGERPRINTING OF SECONDARY METABOLITES FROM Moringa oleifera Lam. OBTAINED THROUGH SUPERCRITICAL-CO, EXTRACTION

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Moringaoleiferais one of the most important food plants with potential nutraceutical value deserving complete characterization beyond phytochemical analysis. DNA-binding assay using a two-dimensional thin layer chromatography (2D-TLC) combined with Supercritical-CO, Extraction (SCE) can abbreviate the screening for target bioactive compounds by eliminating the use of voluminous toxic solvents and zeroing in on control of gene expression. The leaves, seeds, and roots of M. oleifera were extracted using Supercritical-CO₂ (SC-CO₂) with pressures of 10, 20, and 30 megaPascals (mPa). The extracts were screened for DNA binding property using two-dimensional thin layer chromatography (2D-TLC) in a solvent system of ethyl acetate and toluene (90:10 v/v). Extracts from seeds and roots at 10 mPa showed affinity to DNA with Rf ratios (Rf value without DNA/Rf value with DNA) of 0.647 and 0.789, while the seed extracts at 20 mPa gave an Rf ratio of 0.818. All extracts from the leaves showed the same organic compounds of oleic acid, erucic acid, ethylene/acrylic acid, and polyvinyl stearate as shown by Fourier-transform Infrared (FT-IR) spectroscopy. The seed and root extracts at various pressures displayed varied functional groups. The HPLC chromatograms of all extracts showed at least one peak at 15 minutes running time. Combined DNA-binding assay and SC-CO₂ extraction prove to be an efficient system for high throughput screening (HTS) for target bioactive compounds, with a high degree of purity as shown by HPLC chromatograms. The various organic compounds partially identified by FT-IR deserve further investigation with respect to their biological functions.

Keywords: 2D-TLC, SC-CO2, FT-IR, HPLC, Moringa oleifera

Citrus microcarpa PEEL EXTRACT AS ACTIVE INGREDIENT FOR LIQUID HAND WASH

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Some liquid hand wash products contain Citrus microcarpa (calamansi) juice and virgin coconut oil as active reagents added to SLES, CDEA, Dehyton AB 30, propylene glycol, salt, preservatives, and water. Because calamansi juice as an active ingredient increases the cost of production, its peel extracts can be investigated as a substitute. This study determined the acceptability of calamansi peel extract as a substitute active reagent of calamansi juice for liquid hand wash to improve the viscosity of the product and reduce production cost. A three-day extracted peel extract of calamansi was formulated with varying concentrations of 20, 40, 60, 80 and 100% into a liquid hand wash and subjected to sensory evaluation. The liquid hand wash with 40% peel extract was the most preferred concentration with "like very much" as to appearance and foamability and "like moderately" as to scent and viscosity. The product was effective based on total plate count in cfu/20cm² using a swab test. The calamansi peel can be incorporated to calamansi juice as an active reagent for liquid hand wash and a 40% solution of the three-day extract of calamansi peel is recommended.

Keywords: Citrus microcarpa, peel, extract, hand wash, calamansi

DEVELOPMENT OF DRAWING INK FROM **CEPHALOPODS**

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The production of the squid ink, developed by the researchers of Cebu Technological University (CTU), Main Campus-Technology Research Center (TRC), promotes the utilization of ink from the sac of Cepaholopods, in particular squid, diamondback squid, and cuttlefish, with the objective of decreasing the cost of commercial ink. The squid ink using different species of cephalopods is superimposed with water, acid, glycerin, thinner and varnish. The developed squid ink from Thysanoteuthis rhombus with 25% squid pigment and 50% acid concentration has a grayish black color, slightly desirable solvent-like odor, ink dryness with a sharp irregular line, thick and dry ink consistency which is comparable with the commercial ink as to its color, dryness, and consistency. An improved ink formulation from sac of cuttlefish has light black color, moderately desirable solvent-like odor, sharp regular line, thin and dry ink consistency. All attributes significantly differ among ink sac from different species of Cephalopods based on Analysis of Variance and Duncan Multiple Range Tests at 5% level of significance. The drawing ink from squid had a density (0.98 g/ml) which is close to commercial ink, and is safe for use since the lead content was less than 3.00 ppm. The researchers refining the ink density towards wider application of the newly formulated ink from a natural source.

Keywords: cephalopods, ink sac, drawing ink, squid ink, ink density

CONTROLLED DRUG DELIVERY CONSTRUCTED USING INKJET PRINTING

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Controlled drug delivery promotes the best efficient therapeutic effect of a medication in a patient. It involves delivering the drug in the proper dosage at the proper time at the site where it is needed. Several approaches have been developed to achieve this goal, including oil-in-water emulsion solvent evaporation, wet spinning, and compression-heat moulding. However, these methods require tedious procedure and result in a big amount of chemical wastage. In this study, the feasibility of using inkjet printing (IJP) in distributing dexamethasone (DEXP) throughout biodegradable poly(lactic-co-glycolic acid) (PLGA) platforms was investigated. The printed structures were rendered three-dimensional (3D) by simply rolling the 2D. These were then investigated for use as sustained delivery reservoirs. Release studies showed a minimal initial burst release, with a sustained release over an extended time period. Empirical modelling showed a good agreement with experimental in vitro release data. In conclusion, ink jet printing can be used to produce spatially resolved patterns that provide control over the release profile obtained. The preparation technique offers a simple procedure with a minimal amount of material waste.

Keywords: inkjet printing, drug release, biodegradable polymer, dexamethasone, controlled drug delivery

BLOOD LEAD ANALYSIS BY ANODIC STRIPPING VOLTAMMETRY COUPLED WITH METAL-EXCHANGE REAGENT SAMPLE PRE-TREATMENT

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Lead is ubiquitous and diagnosis of exposure to this toxic metal can be challenging as symptoms of lead poisoning may mimic other disorders. There is a need for a more sensitive technique with fast turnaround time. Lead in human blood samples was analyzed by anodic stripping votammetry (ASV) following sample pretreatment with a metal-exchange reagent. The voltammetric analyzer used was E-Chem/Powerlab electrochemical system with thin mercury film, Ag/AgCl and platinum electrodes. The lead stripping peak appeared at the potential of -0.45 V in acetate buffer of pH 4.5. The optimum volume ratio of metal-exchange reagent to blood sample used was 2.9 mL: 100iL. Repeatable results were obtained in the analysis of blood spiked with lead (100-500 ig/L). The intra-batch and inter-batch coefficient of variance (CV) values were 7.8% and 12%, respectively. Linear responses (i vs. Pb conc.) were obtained for low Pb conc. range (100-500 ig/L) and high Pb concentration range (1.00 to 5.00 mg/L). The limit of detection (LOD) and the limit of quantitation (LOQ) were 9.00 ig/ L and 29.0 ig/L, respectively. The % recovery ranged from 109.3 to 129.0%. Results of ASV analysis of five human blood samples gave values ranging from 1.23-35 ìg/dL (vs. reference value of 20.0 ìg/dL). The use of a metal-exchange reagent in the study was shown to be a promising alternative sample pre-treatment method for the measurement of lead levels in human blood.

Keywords: lead, anodic stripping voltammetry, metal-exchange reagent, sample pre-treatment, blood

ENHANCED ELECTROCATALYTIC ACTIVITY OF PULSE DEPOSITED Pt PARTICLES DISPERSED ON PEDOT-MODIFIED Au ELECTRODE TOWARDS ETHANOLOXIDATION

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Poly(3,4-ethylenedioxythiophene) (PEDOT)-modified Au electrodes were electrochemically prepared via potentiodynamic polymerization using 0.01 M EDOT and 0.10 M HClO, on a Au substrate at a potential range from 0.0 to 1.10 V (vs. Ag/AgCl). The PEDOT-modified Au electrode was electrochemically characterized in a monomer-free 0.10M HClO₄ electrolyte. The surface morphology was probed using Scanning Electron Microscopy (SEM). Platinum particles dispersed on PEDOTmodified Au electrodes were prepared in two steps: (1) aqueous electropolymerization of EDOT, and (2) pulse deposition of platinum. Pulse deposition of Pt nanoparticles was carried out using the following optimized parameters: -30 mA/cm² of pulse current with 0.2 s on-time and 0.4 s offtime at 700 pulses. Electrocatalytic activity of the prepared Pt/PEDOT/Au electrode was evaluated towards ethanol oxidation using 1.0 M ethanol in $0.10 \text{ M H}_2\text{SO}_4$ electrolyte solution from E = 0.0 V to E = 0.90 V (vs. Ag/ AgCl) at a scan rate of 100 mV·s⁻¹. The CV profile of the prepared nanocomposite shows an anodic peak at E = +0.700V which is an indication of the presence of platinum. Examination of the surface morphology of the Pt nanoparticles (Pt NPs)/PEDOT/Au-modified electrode revealed welldispersed platinum particles on the polymer matrix with a diameter of less than 100 nm. Enhancement of the electrocatalytic activity towards ethanol oxidation was observed in pulse-deposited platinum particles compared to potentiodynamically-deposited platinum particles on PEDOT-modified Au electrode.

Keywords: poly(3,4-ethylenedioxythiophene), electropolymerization, Pt nanoparticles, pulse deposition, electrocatalysis

ENHANCED PHOTOCATALYTIC DECOLORIZATION OF MALACHITE GREEN USING RUTHENIUM – DOPED TITANIUM DIOXIDE NANOCRYSTALS UNDER VISIBLE LIGHT ILLUMINATION

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Ruthenium-doped Titanium dioxide (TiO₂) photocatalysts were successfully prepared using sol-gel method and structural characterization involved X-ray Diffractometry (XRD), Scanning Electron Microscopy and Energy Dispersive X-ray Analysis (SEM +EDX), and Fourier Transform Infrared Spectroscopy (FT-IR). SEM image analysis revealed the synthesized Ru-TiO₂ particles are highly aggregated and surfaces are clearly rough. The phase composition using XRD analysis revealed that anatase and rutile peaks were found in undoped TiO2 and only anatase peaks were present Ru-doped TiO₂ systems which were calcined at 500°C. Furthermore, an increase calcination temperature to 700°C promotes the transformation of anatase to rutile peaks. The Debye-Scherrer equation was used to estimate the crystallite size based from the diffractograms. The estimated values are 3.49 nm, 3.53 nm, 3.53 nm for 0.20, 0.50 and 0.80 mole % Rudoped TiO₂, respectively. The photocatalytic property of the Ru doped TiO₂ were tested against the decolorization of malachite green solution (MG). A plot ln Co/C versus time gave R2 values ranging from 0.9041 to 0.996 which suggests that the photodecolorization of MG using TiO₂ photocatalysts follows pseudo-first order kinetics. The photocatalytic activity of the catalysts was found to improve with the addition of ruthenium, from 20 % for undoped TiO₂ to 87 % for 0.8 mol % TiO₂. The maximum percent removal of MG dye was achieved using 1.50 g/L of catalyst loading for 0.8 mol % ruthenium added. The TiO₂ photocatalyst prepared at calcination temperature of 500°C showed the highest percent removal compared to those calcined at 300°C and 700°C. When the calcination temperature was increased the photocatalytic activity of the synthesized catalyst was found to decrease over time.

Keywords: Malachite green, Photodecolorization, X-ray Diffractometry, Titanium Dioxide, Ruthenium

EXTRACTION AND CHARACTERIZATION PHENOLIC ANTIOXIDANTS FROM CALAMANSI PEEL, COFFEE HUSK AND CACAO POD HUSK

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Phenolic compounds from calamansi peel, coffee husk and cacao pod husk were extracted using a cheap solvent. The extracts were evaluated for their antioxidant capacity by 2,2-diphenyl-1-picryl-hydrazyl (DPPH) radical scavenging assay and ferric ion reducing antioxidant power (FRAP) test. Antimicrobial property and High Performance Liquid Chromatography (HPLC) analyses were also conducted. The DPPH radical scavenging activity of the phenolic extracts (PE) were in the order: cacao husk PE > coffee husk PE > calamansi peel PE. Both extracts from cacao and coffee husks were better free radical scavengers than the synthetic reference antioxidants, butylated hydroxyl-anisole (BHA) and butylated hydroxyltoluene (BHT), but that from calamansi peel was not. The reducing power of the extracts, on the other hand, were in the order: coffee husk PE > cacao husk PE > calamansi peel PE. Based on DPPH assay and reducing power test, cacao and coffee husks appeared to be excellent sources of natural antioxidants but not calamansi peel. Phenolic extract from calamansi peel, however, exhibited better antimicrobial activity against some organisms than the two extracts. Results of HPLC analysis showed that per 200 mg powder, there was 0.8 ng caffeic acid in cacao pod husk; 0.3 ng caffeic acid and 0.9 ng chlorogenic in coffee husk; and 1.1 ng p-coumaric acid and 1.3 ng sinapic acid in calamansi peel.

Keywords: DPPH assay, FRAP test, phenolic compounds, antioxidants, calamansi

HYDROLOGICAL CHARACTERIZATION OF THE EASTERN SIDE OF BATAN, AKLAN AQUIFER

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Groundwater is constantly being recycled and replenished by rainfall. However, because of the uneven distribution of rain and the heavy use of water in some areas, some basins/aquifers are experiencing environmental stress. To delineate the ground water recharge areas for the eastern side of Batan, Aklan, Philippines, and to assess the vulnerability of the aquifer to pollution and sea water intrusion, measurements on the filed parameters, pH, conductivity, as well as, the eight major ions calcium, magnesium, sodium, potassium, chloride, nitrate, sulfate, and bicarbonate were made. Radioactivity of the water samples was also measured. Some geochemical processes were recognized in the Batan, Aklan basin, the most important of which are water-bedrock interaction and sea water intrusion. The groundwater in the eastern Batan, Aklan basin is predominantly of the calcium magnesium bicarbonate type. Two clusters of water type were found. Cluster 1 (Aby, Mandong Manukan, and Poblacion Magkawit) and Cluster 2 (Banica Hill, Banica Plain, Poblacion Acacia and Angas-1). Recharge in Cluster 1 becomes sodium chloride dominated due to possible impending brackish water intrusion. Recharge in the Cluster 2 does not show salinisation/ seawater intrusion and may indicate active recharge to the subsurface water. The overall quality of subsurface water in the eastern side of Batan, Aklan can be considered good and compliant with the limits set by the Philippine National Standards for Drinking Water for the physicochemical parameters, except for the Angas-1 and Angas creek waters which have very acidic pH. Both the gross alpha and beta activities in all the sites were within the regulatory limits for radioactivity in drinking water of the Department of Health.

Keywords: pH, groundwater, recharge, major ions, Aklan

MORPHOLOGICAL AND CHEMICAL PROPERTIES OF BIOLOGICAL APATITE POWDERS FROM 0.5M HYDROCHLORIC ACID (HCI) PRE-TREATED AND THERMALLY-TREATED TILAPIA BONES

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Hydroxyapatite (HAp) from biological apatite has been widely studied for its ability to replace broken bones and teeth. Numerous techniques can produce good quality HAp; however, these techniques are expensive. In this research, the production of biological HAp powders from 0.5M HCl pre-treated tilapia bones using thermal treatment is presented. Proper cleaning of bones was done before thermal treatment (700°Cd"Td"1000°C). Energy dispersive x-ray (EDX) spectroscopy and scanning electron microscopy (SEM) were used to elucidate the elemental and surface morphology, respectively. The characterization results confirmed the presence of HAp in all prepared samples. Better HAp crystals are observed compared with the untreated tilapia bones. As the annealing/calcination temperature of tilapia bones increased, the good quality of HAp crystal is formed as shown in SEM-EDX results. SEM micrographs depict the roundlike and rod-like shapes of HAp powders along with the coalescence of particles as calcination temperature increases which are better than untreated prepared samples. Furthermore, EDX results also confirm the calciumdeficient formation of HAp powders. However, formation of 0.5M HCl pre-treated HAp crystals is observed at 800°C as revealed by [Ca]/[P] ratio. These results conform to the properties and composition of biological apatites implying that the produced HAp powders have better quality than untreated samples and are now ready for further studies on its applications.

Keywords: HAp, apatite, biological apatite, SEM-EDX, thermal treatment

OPTIMIZATION STUDY ON THE DILUTE ACID PRETREATMENT AND ENZYME SACHARIFICATION OF THE MACROALGAE Sargassum spp. FOR ETHANOL PRODUCTION

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A promising alternative non-food feedstock for bioethanol is macroalgae, not only due to their fast growth rate and large biomass yield but also because the Philippines is already one of its major global commercial producers. This study focused on the optimization of the acid pretreatment and enzyme saccharification of the macroalgae Sargassum spp. using the Response Surface Methodology (RSM). The dried and ground Sargassum was treated with H₂SO₄ to final concentrations of 0 to 6.36% (w/v) at solid loading of 10% (w/w) at varying temperatures (111 to 129 °C) and reaction time (0.16 to 1.84 hr). The acid-treated biomass was subjected to enzyme saccharification using cellulase and cellobiase at pH 4.8 at a temperature of 50 °C and 100 rpm in a water bath shaker for 48 hrs. The total reducing sugars and glucose of the samples were determined by the DNS (dinitrosalicylic acid) method and high performance liquid chromatography, respectively. Results showed that the effects of temperature, acid concentration and reaction time on glucose released were statistically significant (p < 0.05) with the increase in acid concentration from 0.1 % (w/v) to 1.0 % (w/v) to be the least significant. For maximum reducing sugar and glucose yields, the predictive model provided the optimum conditions of 3.36 to 4.15% (w/v) H₂SO₄, 115 °C and 1.44-1.50 hrs. This optimal pretreatment conditions, as well as the conditions employed in enzyme saccharification, are relatively milder compared to that of terrestrial biomass.

Keywords: acid pretreatment, bioethanol, enzyme saccharification, macroalgae, Sargassum

PRODUCTION OF PLANT OILS AND METHYLESTERS USING THE SUPERCRITICAL FLUID EXTRACTION METHOD

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The oil from jatropha seeds and grated coconut meat can be effectively extracted by supercritical carbon dioxide. Extractions were conducted using a SCFE equipment, Model: 46-19360-60Hz equipped with Super pressure Compressor 6000 PSI Motor Driven. The extraction temperature and extraction pressure significantly affected the extraction rate of SFE. The fatty acid profile of jatropha oil was composed of oleic, linoleic, palmitic and stearic fatty acids, while the fatty acid profile of coconut oil was composed of caprylic, capric, lauric, myristic, and palmitic acid, with lauric acid as the most predominant. Methyl esters can be produced by *in situ* supercritical methanol transesterification directly from the jatropha seeds and grated coconut. Operating conditions were: reaction temperature/ pressure (70 °C/7000-8000 psi). Ratio of methanol to weight of ground jatropha seeds was 1:2. As the temperature increased, the crude biodiesel and FAMEs yields also increased. The method is a single-step process, where the usual oil extraction process is avoided. In addition, because this process does not require a catalyst as in the usual method, the purification of products after transesterification reaction is much simpler. Therefore, this new process can offer an alternative way to convert jatropha seeds and coconut meat directly to methyl esters by a simpler and shorter production process eliminating waste products. The use of supercritical fluid extraction (SCFE) has gained attention in the production of oils and methyl esters due to the use of carbon dioxide supercritical fluid solvent. It is cheaper in price, a nonflammable compound, and O₂-free, thus protecting samples against any oxidative degradation.

Keywords: Supercritical fluid extraction, jatropha oil, methyl ester, supercritical methanol, transesterification

PRODUCTION AND ENZYMATIC MODIFICATION OF FIBER FROM CACAO POD HUSK AND ITS UTILIZATION AS BAKERY INGREDIENT

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The cacao processing industry generates 10 tons of husk (fresh weight) from each ton of dry cacao seeds which present serious disposal problem to the cocoa industry. Its utilization as source of dietary fiber was the objective of this investigation. On a dry matter basis, the dietary fiber components consist of about 13% hemicelluloses, 11% cellulose, and 24% lignin. Analysis of functional properties showed that fiber from cacao skin has more absorptive power in both water and oil, higher swelling power and starch solubility. Glucose dialysis retardation index (GDRI) analysis of fibers showed comparable values with the fiber-rich powders from asparagus by-products, rice bran, guar gum and psyllium powder. Enzymatic treatments involving cellulases, xylanases and pectinases showed changes in the functional properties, total phenolic content, and GDRI of enzymaticallymodified fibers. Utilization in cookies and cupcake formulations showed acceptable sensory qualities up to 20% flour substitution with cacao husk flour. Increase from 10% to 20% resulted to increase in crude fiber content by about 36% in cookies and 67% in cupcakes. Increase in total polyphenols by 57% was also observed.

Keywords: cacao pod husk, dietary fiber, enzymatic treatment, GDRI, polyphenols

DETERMINATION OF FOLIC ACID IN FORTIFIED FOODS AND FOLATE IN SOME PHILIPPINE VEGETABLES BASED ON THE DEVELOPED AND VALIDATED METHODS OF ANALYSES

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Microbiological assay has been regarded as the reference method in the analysis of folates and folic acid (FA) in foods, but trends in analytical chemistry towards simple and less time-consuming analytical methods set forth other reliable type of analysis. High-performance liquid chromatographic analysis with UV detection at 280 nm was developed and validated to determine the FA content of fortified foods. The analytical method involves FA extraction with phosphate buffer or amylase treatment after buffer extraction (for samples with carbohydrate bound-FA) and analysis with HPLC. A limit of detection of 0.0524ppm, limit of quantitation of 0.1747ppm, and an average correlation coefficient (r) of 0.998 were obtained during in-house method validation. The repeatability and intermediate precision were also evaluated based on relative standard deviation and found to be 3.91% and 10.97%, respectively. Recovery of the test results was established at 81% to 97%, while the accuracy of the method was verified using SRM 1849. The validation parameters obtained indicated that the method is fit for use in the determination of folic acid in food. Fifteen fortified food samples available in the market were analyzed using the validated method and revealed that the folic acid contents of most of the samples tested were lower than those indicated in their nutritional labels. On the other hand, 5-CH₃-THFA, 5-CHO-THFA and THFA in seven green leafy vegetables were also assessed through extraction by tripleenzyme digestion with amylase, protease, and conjugase but only 5-CHO-THFA and THFA were successfully quantified by HPLC. Extracts of alugbati, malunggay, and swamp cabbage were subjected to LC-MS analysis for reevaluation but no mass fragments similar to standards were obtained.

Keywords: foliates, folic acid, reversed-phase high performance liquid chromatography, in-house method validation, fortified foods

PUTATIVE ALLERGENS IN GMO VERSUS CONVENTIONALLY-BRED RICE

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This study used in silico approach to screen nascent allergens in GMO and conventionally-bred rice. The protein sequences that were analyzed were taken from published data from various studies on GMO and conventionally-bred rice. To determine allergenicity of the proteins, allergen databases and algorithms, such as Allermatch, Algored and Appel were used. The analysis revealed the following putative allergenic proteins for the GMO rice, namely: cysteine proteinase precursor, putative germin A, glycosyl hydrolases, and subtilisin-like serine proteinase, an unknown protein, and late embryogenesis abundant for the conventionally-bred rice. The proteins that computationally showed allergenicity are related to stress and defense response, metabolism, and storage and degradation of proteins.

Keywords: allergens, GMO, bioinformatics, food safety assessment

RADIOLOGICALAND PHYSICO-CHEMICAL APPRAISAL OF AKLAN RIVER

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In this study, gammametric counting and gross alpha-beta measurement by Liquid Scintillation Spectrometry (LSC) were used to mark out regions with impending elevated concentrations of minerals/pollutants in the offshoot streams of the Aklan River. The study was done in line with the establishment of a baseline data of geo-environmental pollutants and mineral resources in the province of Aklan, and to set up action limits for soil, sediment, and water contamination using the local baseline. Seven tributaries were selected as sampling points. Streambed sediments were collected from the 7 sites and were analyzed for gamma emitting radionuclides using a high purity Germanium (HPGe) detector; surface water from all of the five sites were analyzed for bicarbonate ions by titrimetry; pH and conductivity by selective electrodes; and radioactivity by Wallac 1414 Liquid Scintillation Counting; and microbial load by Sim Plate method. None of the watercourses exceeded the regulatory limits set by the Philippine National Standards for Drinking Water for conductivity, pH, and bicarbonate parameters for surface waters. As far as radioactivity is concerned, all of the sites' water samples gave total alpha activity of less than the detection limit, LLD, (LLD= 0.03 Bq/L), which was way below the drinking water regulatory limit of 0.1 Bq/L for alpha emitters. All the samples exhibited beta activities of less than LLD (LLD= 0.3 Bq/L), which was also way below the drinking water regulatory limit of 1.0 Bq/L for beta emitters. In terms of radiogenic nuclides in sediments, all the sediments had Cs-137, Sr-90, and I-131 concentrations of less than 3 Bq/K. Microbial load was elevated at site 7 with approximately 738 populations per plate during a post monsoon sampling.

Keywords: Aklan River, gross alpha-beta, gamma, drinking water, water contamination

RAPID DETERMINATION OF FIVE ARTIFICIAL SWEETENERS IN BEVERAGES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

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Artificial sweeteners are increasingly used in a wide range of commercial beverages. Most of these sweeteners have a maximum allowable concentration prescribed by international and local standards. Ensuring proper implementation of existing regulations requires reliable quantitative analytical methods to measure sweeteners in beverage matrices. Here, we present the results of a single-laboratory validation of two methods for the rapid determination of five authorized artificial sweeteners in beverages using high performance liquid chromatography with ultraviolet and refractive index detection. The procedure involved extraction of acesulfame-K (ACS-K), aspartame (ASP), neotame (NEO), saccharin (SAC), and sucralose (SUC) with a buffer solution followed by chromatographic analysis. Samples containing ACS-K, ASP, NEO, and SAC were analyzed using HPLC-UV with gradient elution at 220 nm while separation of SUC was achieved by HPLC-RI. The calibration curve was linear in the concentration ranges of: 50 to 500 ig/mL ACS-K, 80 to 800 ig/mL ASP, 15 to 150 ig/mL NEO and SAC, and 100 to 1,000 ig/mL SUC. The limits of detection (and quantification) for ACS-K, ASP, NEO, SAC, and SUC were 8.2 (27.3) ig/mL, 13.0 (43.5) ig/mL, 2.8 (9.5) ig/ mL, 3.8 (12.5) ig/mL, and 5.5 (18.4) ig/mL, respectively. Repeatability and intermediate precision tests showed relative standard deviations and Horrat ratios generally lower than the maximum acceptable values. Analysis of reference materials yielded results that fell well within the acceptable concentration range. Recoveries from samples spiked at low, medium and high concentrations ranged from 99 to 109 percent. The developed inhouse methods were found suitable for rapid quantitative determination of five artificial sweeteners in beverages.

Keywords: artificial sweetener, beverage, high performance liquid chromatography, buffer extraction, method validation

SYNTHESIS AND CHARACTERIZATION OF HYPERBRANCHED AND STAR-SHAPE COPOLYMERS OF POLY(N-VINYLCARBAZOLE) USING A RAFT INIMER

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New materials with complex architectures, such as hyperbranched and star-shape copolymers, continue to be an interesting field of polymer science due to their intriguing properties and complicated synthetic routes. While many researchers have reported the successful synthesis of this type of polymers, there remains a need to develop simpler synthetic procedures that would produce these materials. This study introduces a facile synthesis of star-shape copolymers of poly(N-vinylcarbazole) [PVK] through a two-step process. The first involves the synthesis of a hyperbranched PVK core by polymerizing N-vinylcarbazole with 2-(methacryloyloxy)ethyl 4-cyano-4-(phenylcarbonothioylthio) pentanoate (CPP-HEMA) inimer through Reversible Addition Fragmentation Chain Transfer (RAFT) living radical polymerization. The inimer serves both as an initiator and as a branching point. The second step involves the copolymerization of the hyperbranched PVK core with another monomer N-isopropylacrylamide (NIPAM) using RAFT. The hyperbranched core in this step serves as macroinitiator from which polymer arms consisting of NIPAM would emanate. Successful synthesis of the star-shape polymer was confirmed through ¹H-NMR, Gel Permeation Chromatography, Fluorescence Spectrophotometry, Thermogravimetric Analysis and FT-IR Spectroscopy. The degree of branching for the hyperbranched structure was computed to be 0.65 and the number of polymer arms that emanated from the core was ascertained as 45 per hyperbranched core. Surface morphologies and aggregation patterns of the copolymer in different solvents like were also studied and correlated.

Keywords: hyperbranched, star-shape polymer, RAFT, inimer, PVK

SOLVENT-FREE SONOCHEMICAL SYNTHESIS AND ANTIFUNGALACTIVITY OF 1-ALKYL-3-METHYLIMIDAZOLIUM BROMIDE [RMIM]Br **IONIC LIQUIDS**

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Opportunistic fungal infections pose a continuous threat to human health and life. In recent years, there has been a considerable increase in occurrence and severity of candidiasis, an infection caused by the fungus Candida albicans. Moreover, the widespread and increased use of antifungal drugs has resulted in the development of resistance to these drugs. This has given rise to the challenge of developing novel and promising antifungal agents for clinical use. Azole-based drugs inhibit the enzyme associated in ergosterol synthesis and eventually hinder fungal growth. This study explored the one-pot synthesis of a series of 1-alkyl-3methylimidazolium bromide [RMIM]Br ionic liquids through a sonochemically-mediated reaction of 1-methylimidazole and alkyl bromides (RBr) under solvent-free conditions. High product yields were obtained for all syntheses (>94%) under mild conditions (2-5 hours at 20-30°C). The success of the synthetic method was confirmed through ¹H-NMR, ¹³C-NMR and FT-IR spectroscopy. All products were tested for antifungal activity against C. albicans with clotrimazole and water as positive and negative controls, respectively. At a definite concentration, ILs having C₄, C₆, C₈, C₁₂, and C₁₆ alkyl chain lengths exhibited antifungal activity with antimicrobial indices of 0.1, 0.2, 1.5, 2.3, and 2.5 against C. albicans, respectively. No inhibition activity was observed for [C₂MIM]Br. The results showed that an increase in the alkyl chain length corresponds to an increase in the antifungal activity of the ionic liquids.

Keywords: ionic liquid, solvent-free, sonochemistry, antifungal agent, Candida albicans, medicinal chemistry

BISPHENOL A SENSOR BASED ON **ELECTROPOLYMERIZED MOLECULARLY IMPRINTED POLYMER (E-MIP)**

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The application of electrochemical impedance spectroscopy in designing a chemical receptor using electropolymerizable terthiophene and carbazole monomers for the imprinting of bisphenol A, a known endocrine disrupting chemical has been demonstrated. The copolymers of bifunctional monomers of -COOH from the carbazole derivative and -OH functional group from terthiophene were found to possess good molecular recognition properties than when these respective monomers were singly electropolymerized. As in any electropolymerized films for sensing applications, a compromise of thickness and efficient formation of pre polymer complexes must be achieved in order to ensure maximum rebinding of the template molecules. Cyclic voltammetry offers a simple means of depositing sensor films directly onto substrate surfaces while the Electrochemical Impedance Spectroscopy (EIS) technique provides a versatile means of measuring the amount of template bound to the polymer matrix. In this study, the EIS has been demonstrated to give extensive information related to the permeability and thickness of the polymer material deposited on the surface that may used in advancing technologies relating to sensing via reversible surfaces through electric potential control and to development of dynamic surfaces for advanced sensing technology. The E-MIP sensor may have advantages in environmental monitoring of bisphenol A in aqueous analyte/pollutant samples

Keywords: sensing, imprinting, electropolymerization, EIS, copolymer film, bisphenol A

A NEW METHOD FOR CALCULATING THE ZEROS OF UNILATERAL POLYNOMIALS OVER THE **QUATERNIONS AND OCTONIONS**

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This paper presents a new method for calculating the zeros of the unilateral quaternionic (octonionic) polynomial

$$P_n(x) = \sum_{j=0}^n a_j x^j, a_n \neq 0,$$

with quaternionic (octonionic) coefficients a_i . It consists of forming the real polynomial

$$Q_{2n}(x) = \sum_{i=0}^{n} \sum_{j=0}^{n} \bar{a}_i a_j x^{i+j} = \sum_{k=0}^{n} b_k x^k,$$

where \bar{a}_i is the conjugate of a_i and expressing $P_n(x)$ in polar form as

$$P_n(x) = \sum_{k=0}^{n} a_k r^k (\cos k\theta + I\sin k\theta), a_n \neq 0$$

in which I is a generalized quaternion (octonion) imaginary unit $(I^2 = -1)$. Let us denote by $Zero(P_n)$ and $Zero(Q_{2n})$ the respective zero-sets of $P_n(x)$ and $Q_{2n}(x)$. Now, the results may be stated as follows: (1) If $x_h \in Zero(Q_{2n})$ is real, then $x_h \in Zero(P_n)$ (a real isolated zero); (2) If $x_h \in Zero(Q_{2n})$ is complex such that $x_h, \bar{x}_h \in Zero(P_n)$, then $\tilde{x}_h = q^{-1}x_hq \in Zero(P_n)$ for every non-real $q \neq 0$ (a non-real spherical zero); and (3) If x_h is complex such that x_h , $\bar{x}_h \notin Zero(P_n)$, then $\tilde{x}_h \in Zero(P_n)$ is a non-real isolated zero of $P_n(x)$, where \tilde{x}_h is:

$$\widetilde{x}_h = \left[\sum_{k=0}^n a_k r_h^{k-1} \sin k\theta_h\right]^{-1} \left[\sum_{k=0}^n a_k r_h^k \sin (k-1)\theta_h\right].$$

Keywords: quaternions, octonions, isolated zeros, spherical zeros, zerosets, alternativity, and unilateral quaternionic (octonionic) polynomials

IDEALS OF AG**-GROUPOID

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A groupoid is a set associated with a binary operation and a groupoid S satisfying the *left invertive law*: (ab)c = (cb)a for all a, b, $c \hat{I} S$ is called an AG-groupoid. If, in addition, an AG-groupoid S satisfies a(bc) = b(ac)for all a, b, $c \hat{I} S$, it is called an AG^{**} -groupoid. In this paper, some properties of the ideals in an AG^{**} -groupoid are discussed. More particularly, the quasi-ideals, bi-ideals, interior ideals, and ideals in an intra-regular AG^{**} groupoid are investigated. The properties of ideals in an AG-groupoid necessitate the existence of a left identity. Every AG-groupoid with left identity is an AG^{**} -groupoid and not every AG^{**} -groupoid contains a left identity. Results on the ideals of an AG^{**} -groupoid without left identity that are parallel to those for AG-groupoid hold.

Let S be an AG^{**} -groupoid. Then the following hold:

- 1. If I is an ideal of S, then I is a bi-ideal (quasi-ideal, interior ideal) of S.
- 2. An idempotent subset I of S is a right ideal, if and only if I is an interior ideal of S.

Keywords: groupoid, AG-groupoid, AG^{**} -groupoid, quasi-ideals, bi-ideals, interior ideals, intra-regular ideals, left invertive law

STRONG CONVERGENCE OF AN ITERATIVE SEQUENCE TO A FIXED POINT OF NONEXPANSIVE MAPPING

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Let K be a closed convex subset of a real uniformly smooth Banach space X and $T: K \to K$ be nonexpansive mapping. We denote the set of fixed points of T by Fix(T). Here we assume that $Fix(T) \neq \phi$. For a fixed integer m we consider an iterative sequence (x_n) defined by $x_{n+1} = \alpha_n^1 u_1 + \alpha_n^2 u_2 + \dots + \alpha_n^m u_m + \beta_n x_n + \gamma_n T(x_n),$ where $x_0, u_1, u_2, \dots u_m \in K$ and $(\alpha_n^1), (\alpha_n^2), \dots (\alpha_n^m), (\beta_n), (\gamma_n)$ are sequences in the interval (0,1).

In this paper, we will show under some conditions the strong convergence of the above iterative sequence to a fixed point of T.

Keywords: strong convergence, nonexpansive mapping, fixed point, iterative sequence, convex set, Banach space

THE AVERAGE OF THE mTH POWER OF L_m NORMS OF ZERO-i POLYNOMIALS ON THE BOUNDARY OF THE UNIT DISC

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Let $n \ge 0$ be any integer and let

$$\Gamma_n = \left\{ p(z) = \sum_{j=0}^n a_j z^j : z = e^{i\theta}, \ a_j = 0,1 \ \forall \ 0 \le j \le n \right\}$$

$$Y_n = \left\{ p(z) = \sum_{j=0}^n a_j z^j : z = e^{i\theta}, \ a_j = 0, 1 \ \forall \ 0 \le j < n, \ a_n = j \right\}$$

be the collection of zero-i polynomials over $\mathbb C$ of degree less than or equal to n and of degree exactly equal to n, respectively. We denote

to be the Lm norm of any polynomial p(z) and $\gamma_n(m)$ and $\kappa_n(m)$ to be the average of the mth power of Lm norms in Γ_n and Y_n , respectively. It is known that the cardinality of Γ_n is 2^{n+1} and the cardinality of Y_n is 2^n . Hence $\gamma_n(m)$ and $\kappa_n(m)$ is given by:

$$\gamma_n(m) = \frac{1}{2^{n+1}} \sum_{p \in \Gamma_n} ||p||_m^m, \qquad \kappa_n(m) = \frac{1}{2^n} \sum_{p \in Y_n} ||p||_m^m$$

In this paper we derived the exact formula for γ_n and κ_n for various values of m.

Keywords: zero-i polynomials, norm, average, degree of polynomial, cardinality

THE AVERAGE OF THE NORM OF THE mth ORDER DERIVATIVE LITTLEWOOD POLYNOMIAL

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Let $n \ge 0$ be any integer and

$$\mathfrak{L}_n = \left\{ P(z) : P(z) = \sum_{j=0}^n a_j z^j \text{ where } a_j = \pm 1 \right\}$$

be the set of polynomials of degree equal to n. The elements of the set \mathfrak{L}_n are restricted polynomials called Littlewood polynomials.

Let $P(z) \in \mathfrak{L}_n$, we denote $\left\|P^{(m)}(z)\right\|_{L^{k}}^{k}$ to be the kth power of the L_k norm of the mth order derivative littlewood polynomial on the boundary of the unit disc and $\xi_n(k)$ be the average of the kth power of the L_k norms of the mth order derivative Littlewood polynomial over \mathfrak{L}_n . Then $\xi_n(k)$ is given by:

$$\xi_n(k) = \frac{1}{2^{n+1}} \sum_{P(z) \in \mathfrak{L}_n} \left\| P^{(m)}(z) \right\|_k^k$$

In this research, the author determined the exact formula for $\, \xi_n(k) \,$ for k equal to 6.

Keywords: Littlewood polynomials, mth order derivative, L_p -space, norm, average.