

Application of Calcined Oysters Waste Shell as an Effective Low-Cost Heterogeneous Catalyst for Biodiesel Synthesis

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Biodiesel production via transesterification of palm olein oil with methanol using solid oxide catalyst derived from waste shell of Oyster (*Saccostrea commercialis*) was employed in this work. The Oyster shell was found to be the rich sources of calcium carbonate and transformed to CaO catalyst after calcinations at 900°C and activation at 600°C. The effect of the molar ratio of methanol to oil between 6:1 – 15:1, catalyst amount between 2-30 wt.%, and reaction time between 0.5-10 h were optimized for the transesterification at 65°C. Biodiesel production above 90% was achieved when carried out the transesterification by employing approximately 20 wt.% catalyst and 12:1 methanol to oil molar ratio at the reaction time of 5 h. The applicability of the present study that utilizes waste resources as catalyst in the production of a green biodiesel and the results indicate that the combusted oyster waste shell at high temperature can be used as economic catalyst for the biodiesel production.

Keywords Biodiesel; Transesterification; Oyster shell

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Optimization of Esterification and Transesterification of Waste Coconut Oil from Waste Water Pond for Community Biodiesel Production

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Waste coconut oil obtained from waste water pond contains high free fatty acid (FFA) which is not suitable to produce biodiesel via transesterification. The two steps of reaction (esterification and transesterification) were used to produce biodiesel from waste coconut oil. The conventional experiment of approximate condition for esterification and transesterification before response surface methodology (RSM) was applied. The result obtained from these experiments were selected in five level of each factor for RSM determination. The RSM was applied for investigating the experimental design. A five-level-four-factorial central composite design (CCD) using response surface methodology (RSM) was employed to optimize the process variables for minimizing the FFA of waste coconut oil and maximizing the waste coconut biodiesel (%) yield. The results from RSM showed that the optimization conditions in esterification step were 3.9% v/v of HCl catalyst amount, 79% v/v of methanol amount, 4 h of reaction time and 60°C temperature. This condition can reduce 92.95% of FFA content to 2.17%. In addition, the optimization conditions in transesterification step were 0.4% w/v of NaOH catalyst amount, 19% v/v of methanol amount, 60 min of reaction time and 60°C temperature. This condition gave 94.47% of methyl ester purity. The prepared waste coconut biodiesel met the community biodiesel standards.

Keywords Waste coconut oil; Biodiesel; Esterification; Transesterification; Response surface methodology

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Lepidocrocite Titanates as a New Basic Catalyst for the Ketonization of Long-Chain Fatty Acids

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Conventional basic catalysts for the ketonization of long-chain fatty acids suffer from drawbacks such as low surface area, limited chemical composition, and low thermal stability. In this work, layered titanates with lepidocrocite structure were studied as a new class of materials exhibiting basic property catalysing the ketonization of long-chain fatty acids. Taking $K_{0.8}Zn_{0.4}Ti_{1.6}O_4$ as an example, its basicity was investigated using CO_2 -temperature program desorption (CO_2 TPD). Its basic strength was compared with that of TiO_2 , MgO , and $Mg-Al$ mixed oxides. After that, $K_{0.8}Zn_{0.4}Ti_{1.6}O_4$ was employed as a catalyst in the ketonization of palmitic acid into palmitone. This reaction is of importance in the transformation of fatty acids in renewable feedstock into diesel fuel. It was found that $K_{0.8}Zn_{0.4}Ti_{1.6}O_4$ intercalated palmitic acid into the interlayer space, thus increasing the surface area active for catalysis. It also exhibited excellent thermal stability, retaining the crystal structure after the catalytic activity testing at $375^\circ C$. Moreover, other compositions ($K_{0.8}Li_{0.27}Ti_{1.73}O_4$ and $K_{0.9}Mg_{0.45}Ti_{1.55}O_4$) were made and their catalytic activity were investigated. All three compositions gave higher yields of palmitone than $Mg-Al$ mixed oxides under identical experimental conditions. Several advantages of lepidocrocite titanates, including the opening up of the interlayer space for catalysis, variable chemical compositions, and high thermal stability, make them a new interesting class of basic catalyst for the ketonization of long-chain fatty acids.

Keywords Basicity; Lepidocrocite; Titanate; Ketonization; Fatty acids

Furfural Production by Dehydration of Xylose with SO₃H-MCM-41 Catalyst

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The chemical from biomass have been interested and developed leading to green chemical industry. Carbohydrate especially sugar is one of important raw material for chemical production from biomass. It is derived from plant by decomposition of cellulosic compounds. Xylose is a major composition of hemicellulose that is interested in raw material in this work. Furfural, one of important chemical intermediate, could be produced by dehydration of xylose in the presence of acid catalyst. However, the catalytic activity and stability should be improved. In this research, the polarity of SO₃H-MCM-41 was modified and tested the catalytic activity in dehydration reaction of xylose. The physical and chemical properties of catalyst was characterized by X-ray diffraction (XRD), Transmission electron microscope (TEM), N₂ sorption, Fourier transform infrared (FT-IR) spectroscopy, Thermogravimetric analysis (TGA), and Elemental analysis. The XRD pattern and FT-IR spectrum confirm the hexagonal MCM-41 structure and the presence of sulfonic acid (SO₃H-), respectively. The acid capacity was determined by titration (1.40 mmol/g). The catalyst containing the part of non-polar (modified by MPMDS) gave the highest furfural yield (90%) at the condition of reaction temperature 155 °C and reaction time of 8 h.

Keywords Furfural; Xylose; Dehydration; SO₃H- MCM-41 catalyst

Effect of Alkaline Combined with Ultrasonic Pretreatment of Water Hyacinth (*Eichhornia crassipes*) on Reducing Sugar Concentration

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The effect of alkaline pretreatment of water hyacinth on reducing sugar concentration was investigated. Six different alkaline pretreatment methods with/without ultrasonic assistance were compared and determined for the best pretreatment method for reducing sugar production. It was found that alkaline combined with ultrasonic pretreatment was more effective in chemical degradation of water hyacinth than alkaline pretreatment. The best result was obtained when the sample was pretreated by 2%NaOH with ultrasonic assistance (ultrasonic twice for 30 minutes of each). The compositions of water hyacinth after pretreatment were 52.52±3.01% cellulose, 17.16±0.50% hemicellulose, and 3.32±0.11% lignin with reducing sugar of 323.13±3.57 mg/g dried sample. The Scanning Electron Microscopic (SEM) analysis exhibited significant morphological changes occurring in the biomasses after pretreatments. The effect of commercial cellulase (Accellerase® 1500) loading on reducing sugar concentration was also studied. The increase of enzyme loading from 250 to 550 CMC U/g dried biomass led to the increase in reducing sugar concentration from 255.40±14.30 to 330.81±2.92 mg/g dried sample. However, the higher enzyme loading (700 CMC U/ g dried biomass) did not improve the reducing sugar concentration. Therefore, alkaline combined with ultrasonic pretreatment of water hyacinth could be an efficient process for reducing sugar production.

Keywords Alkaline; Enzyme hydrolysis; Pretreatment; Ultrasonic; Water hyacinth

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Production and Characterisation of Activated Carbon from Synthetic-Fibre Industrial Sewage Sludge using Microwave Irradiation

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The main purpose of this research was to produce and characterise activated carbon generated from synthetic-fibre industrial sewage sludge using microwave irradiation as an energy source. Raw sewage sludge, obtained from the wastewater treatment plant of the TTL Industries Public Company, with the initial moisture content between 15 and 35 wt% was pyrolysed using microwave irradiation with the microwave powers of 480, 640, and 800 W for 30 min. Straw was added to the sludge in the amounts of 7.5 and 15.0 wt% in order to examine the ability of straw on adsorbing microwave irradiation. The resulting activated carbon was characterized for its solid yield, iodine number, porosity, and proximate analysis. The adsorption kinetics of the activated carbon were also examined using the model wastewater containing methyl orange with the concentration 0.1-1.0 g/L. It was found that the solid yield was in range of 44.6-59.9 wt%. An increase in microwave power and initial moisture content of the raw sludge resulted in a decrease in solid yield. The addition of straw had a negligible effect on the solid yield. The iodine number of the resulting activated carbon ranged from the lowest value of 40.7 to the highest value of 123.0 mg/g of activated carbon. The results from the iodine number tests were in accordance with those from the determination of the porosity of activated carbon using a scanning electron microscope (SEM). For the proximate analysis of the resulting activated carbon, it was found that when the microwave power increased, the remaining amount of moisture and volatile matter in the activated carbon decreased substantially. The analysis of adsorption isotherms of the resulting activated carbon revealed that the experimental data fitted well with the Langmuir isotherm.

Keywords Activated carbon; Sewage sludge; Microwave irradiation; Adsorption kinetics

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Effect of Iron Filings' Sizes on Reactive Blue Dye Degradation Using Fenton and Fenton-like processes

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High concentrations (250-3000 mg/L) of commercial reactive blue dye degradation were studied using conventional Fenton (Fe^{2+}) and Fenton-like (Fe^0) processes. Iron filings with the sizes of 75-90 μm , 38-45 μm , <38 μm and mixed sizes were used as Fe^0 , while $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ was used for Fe^{2+} . Effect of pH (2.5, 3.0, 3.5, 4.0, 4.5) and mole ratio of $\text{Fe}:\text{H}_2\text{O}_2$ were investigated for both Fenton and Fenton-like processes. It was found that the optimum pH was 2.5 in every condition but the optimum mole ratio of $\text{Fe}:\text{H}_2\text{O}_2$ was varied, depending on the initial dye concentrations. After 180 minutes of Fenton's reactions, dye degradations of 98.9-99.75% were found from Fenton-like process, while those of 99.4-99.97% were found from the traditional Fenton. The smaller the iron filings' sizes, the better the efficiency. At low dye concentrations (250-400 mg/L), Fenton and Fenton-like processes yielded similar sludge volumes (0-0.6 ml/L) after adjusting pH to 7. However, at higher dye concentration (750-1000 mg/L), traditional Fenton produced 150-180 ml/L of sludge volumes, whereas Fenton-like produced only 0.2-90 ml/L. In terms of iron cost, traditional Fenton requires 2224 mg/L of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ which costs 1.42 baht/L while the iron filings, from the Fenton-like process needs 447 mg/L which costs 0.09 baht/L for the same 3000 mg/L dye concentration treatment. Therefore, Fenton-like process with iron filings is better alternative for dye degradation in terms of cost and sludge treatment.

Keywords Fenton Process, Fenton-like Process, Iron Filings, Reactive Blue Dye

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Preparation of Silica from Rice Husk Ash as a Sorbent Material for THMs Removal

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Presently, the rice husk is used as a renewable energy source, causing the rice husk ash (RHA) to become a product of energy production. This research focus on adding value to RHA developing it into a sorbent material for THMs removal to improve water quality. This research, comprised of two steps being preparation of the adsorbent by sol-gel process and functionalization of 3-aminopropyl triethoxysilane on the silica surface. Comparison had been made between raw rice husk ash sample with and without heat treatment of 850°C for 3 hours. The average pore volume and specific surface area were determined by Brunauer-Emmett-Teller (BET) technique. It was shown that the synthesized sol-gel from calcined rice husk ash had higher total pore volume and surface area compared with the sol-gel prepared from raw rice husk ash. The sol-gel silica were further characterized by Fourier Transform Infrared Spectroscopy (FTIR). It was found that IR peak for Si-O-Si, Si-OH, Si-H, and OH were obtained for both sol-gel.

Keywords THMs; Rice husk ash; Adsorption

Preparation of Biosorbent from Pineapple Peels: Pretreatment with NaOH

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This study presents the result on enhanced surface area of pineapple peels by alkaline pretreatment compared with non-pretreated samples. The pineapple peels were crushed and sieved to particle size of 200 mesh. Pretreatment with NaOH was carried out by variation of contact time (1, 6, 12, 24 h), NaOH concentration (0.5, 1.0, 2.0%)(w/v) and reaction temperature (30, 50, 100°C). The FTIR spectra bands of pineapple peels observed at 3373.34 and 2920.69 cm^{-1} represent -OH and aliphatic C-H group and the peak at 1634.38 cm^{-1} corresponds to C=O stretching. Experimental results showed that alkaline pretreatment can improve the internal surface area of pineapple peels as seen from an increase in Methylene blue number from 125.35 mg/g of non-pretreated samples to 548 mg/g of pretreated samples with 2% (w/v) NaOH at 50°C for 24 h. In addition, an increase in Iodine number was also observed in similar trend. Result from Scanning electron microscope (SEM) showed that pretreated samples have roughness and more fibrous than non-pretreated pineapple peels.

Keywords Agricultural waste; Pineapple peels; NaOH pretreatment; Biosorbent

17- α Methyltestosterone Removal by *Salvinia molesta* Based Bioremediation Treatment System

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The aim of this study was to explore the potential ability of *Salvinia molesta* for remediating 17- α Methyltestosterone (MT) from wastewater. MT is a kind of Endocrine Disrupting Compounds (EDCs). Exposing to this compound may cause disturbance to natural organisms. The remediation treatment system was conducted in batch process which was containing synthetic wastewater. The potential of this plant for treating MT wastewater in a remediation system was observed. A high performance liquid chromatographic (HPLC) method using ultraviolet (UV) detection has been used to analyze the samples. During the pre-test, it was found that *Salvinia molesta* was able to remove MT to 0.03 mg.l⁻¹ within 7 days. For kinetic studies, the MT sorption was fitted with Pseudo-second order with the rate constant (k) of 1.60 g.mg⁻¹.day⁻¹. Based on this result, it is likely to say that *Salvinia molesta* can be used to phyto-remediate MT from wastewater. Further investigations on factor affecting the removal efficiency involve several variables such as, weights of plant, concentrations of compound, and wavelengths of exposure-light.

Keywords 17- α Methyltestosterone; *Salvinia molesta*; Remediation; High performance liquid chromatographic; Kinetic studies

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Determination of 17 α -Methyltestosterone using Bismuth Film Modified Screen-Printed Carbon Nanotube Electrode

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17 α -Methyltestosterone (MT) is a synthetically produced anabolic and androgenic steroid hormone. Thus, it is commonly used to promote both muscle growth and the development of male sexual characters in both humans and animals. In medical treatment, MT and other synthetic forms of testosterone have been applied as hormone supplement in human with testosterone deficiency. In Thailand, MT is widely used in aquaculture practices. Treatment of tilapia fry with MT-impregnated food to produce all-male populations has become a common aquaculture practice. The left-over of the food becomes major environmental concerns as they can affect the biological activity of non-target organisms. Determination of MT and its derivatives in samples having high environmental matrices require several steps of sample preparation, i.e. filtration, extraction, clean-up and elution. In recent years, a great effort has been made to develop new analytical methodologies to achieve lower detection levels. This study aimed to develop a new modified electrochemical sensor to determine MT in water samples. Electrochemical sensor has an alternative detection due to its high sensitivity, low-cost and portability. In this study, the modified electrode was developed to determine MT using adsorptive stripping voltammetry. The bismuth film was deposited *in-situ* on a screen-printed carbon nanotube electrode. Optimization of this technique was carried out by varying; deposition potential, deposition time and concentration of bismuth. It was found that the optimal condition for MT detection was 6 mg/L of bismuth, 90 s for deposition time and -0.5 V vs. Ag/AgCl for deposition potential. MT was undetectable on screen-printed carbon nanotube electrode and it was found that the reduction peak at 0.78 V on bismuth film modified screen-printed carbon nanotube electrode.

Keywords 17 α -Methyltestosterone; Stripping voltammetry; Carbon nanotube electrode.

Kinetic Study of the Pyrolysis of Municipal Solid Waste (MSW) using Microwave Irradiation

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This research studied the chemical kinetics of the pyrolysis of cylindrical municipal solid waste (MSW) pellets, 1.0 cm in diameter and 1.0 cm in length, using microwave irradiation, with the microwave power of 800 W and the pyrolysis time of 5-25 min. Proximate analysis, ultimate analysis, and heating value (HV) of MSW, both before and after pyrolysis, were also determined. The effects of the initial moisture content of MSW, which was in the range of 0-30 wt%, and of the amount of charcoal powder added to enhance the absorption of microwave irradiation, ranging from 20-40 wt%, on the pyrolysed MSW and on the kinetics of the pyrolysis of MSW were investigated. It was found that the pyrolysis temperature was within the range of 200-500 °C and that when the pyrolysis time increased, it resulted in the decreases of the remaining amounts of moisture and volatile matter, but caused the remaining amount of fixed carbon to increase. The remaining amount of ash was negligibly affected by the pyrolysis time. After pyrolysis, the percentage of the remaining carbon (C) increased from 40% to 60-88%. On the contrary, the percentages of hydrogen (H) and oxygen (O) decreased from ~7% to ~2-5% and from ~51% to ~9-37%, respectively. After pyrolysis, the heating value of MSW slightly increased from 20.0 to 21.4-22.8 MJ/kg. For the analysis of the chemical kinetics of the pyrolysis of MSW, it was found that the order of the reaction (n) was in the range of 1.2-1.3 and the rate constant (k) was within the range of 1.9 to 23.9. The activation energy (E_a) of the MSW pyrolysis was found to be approximately 170 kJ/mol.

Keywords Municipal solid waste (MSW); Chemical kinetics; Pyrolysis; Microwave irradiation

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Copper Ion Detection by Amine-Functionalized Silica Monolith

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Amine-functionalized silica monolith was prepared by co-condensation between tetraethylorthosilicate and N-[3-(trimethoxysilyl)propyl]ethylenediamine. The mixture between ethanol, H₂O, tetraethylorthosilicate, N-[3-(trimethoxysilyl)propyl]ethylenediamine and 1 M HCl was stirred for 1 hour at room temperature to obtain a clear silica sol. 1.2 g of the sol was poured into plastic vial with the paraffin cover and left for 2 days for the polymerization process to obtain the gel. The disc-shape gel was dried in the oven at 50 °C for 10 hours. The silica monolith obtained was weighed and analyzed by IR spectroscopy and BET method. The disc-shape silica monoliths were further used for copper sensing studies. The functionalized monoliths were attempted with three different concentrations of CuCl₂ (0.001, 0.01 and 0.1 M) at four different pHs (2, 3, 4 and 5). The monoliths were left in 25 mL of Cu²⁺ solutions for 6 hrs to allow equilibrium absorption and the visible absorption at the wavelength of 579 nm of the obtained blue monoliths was measured. The blue color indicates the complex formation between Cu²⁺ and the amine groups of the functionalized silica monolith. The absorbance of the monoliths after copper ion adsorption depends on the concentration and pH. A higher absorbance of the monolith was obtained at a higher concentration of copper ion and lower pH. The interference ions (Cd²⁺ and Ni²⁺) were also studied. Cd²⁺ and Ni²⁺ did not significantly change the visible absorption at 579 nm of the monolith.

Keywords Copper ion sensor; Amine-functionalized silica monolith; Optical sensor

The Application of Isotope Geochemistry on the Groundwater Dynamic Study of Limestone Aquifers in Saraburi – Lopburi Karst Area, Central Thailand

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Saraburi - Lopburi Area is Thailand's largest area of limestone production for commercial uses contributed by the Permian Saraburi Group which the limestone exposed as the beautiful "Karst Topography". The groundwater (GW) system in karst area is characterized by the soluble rocks, usually form caves and underground conduits, which developing unique GW turbulent flows and rapid vertical infiltration, make the aquifer change in very dynamic and tend to be complex, high heterogeneity and associated with vast of springs. The stable and radiogenic isotope geochemistry analysis was widely applied on the karst GW dynamic especially in the study of seasonal changes, GW-surface water interaction and recharge mechanism assessment for the GW modeling and conservation. The water samples were collected from 60 GW production wells in grid sampling including the daily precipitation, surface water and springs cover the high GW exploitation of 600 km² area in Saraburi – Lopburi province in post-, pre- and monsoon seasons during 2013-2014. The stable isotope ratio data ($\delta^2\text{H}/\delta^{18}\text{O}$) were analyzed by laser absorption spectroscopy instruments. The reservoir sample shows clearly evaporation effect ($\delta^{18}\text{O} > 5.0$) especially in pre-monsoon season that the fingerprints can be used to trace some GW with the interaction of local SW recharged. The daily precipitation in 2013 of 48 rainwater samples can be represented in $\delta^2\text{H}/\delta^{18}\text{O}$ relationship of the Local Meteoric Water Line (LMWL, $y = 7.4819x + 3.2578$) which clearly variation by seasonal effects can be accounted. The GW with $\delta^{18}\text{O}$ value ranged over -7.0‰ and rapidly response to precipitation sources indicating the recharge zone. While the GW in discharge zone show enrich values ($\delta^{18}\text{O} > 7.0$) and less fluctuated along seasonal change. Tritium content, which was analyzed by LSC counting with electrolysis enrichment method, in GW samples ranging from 0 – 1.7 Tritium Unit (TU) and relatively high tritium content (>1.0 TU) in the recharge zone that are close to the surface water and rain water contents (1.5-2.0 TU) indicate relatively short flow path comparing to the GW in discharge zone.

Keywords Isotope; Geochemistry; Groundwater dynamic; Limestone

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Dual Optical Hg²⁺-Selective Fluorometric Chemosensors Based on Rhodamine 6G

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New “naked-eye” indicators and fluorescent chemosensors based on 2-[3-(2-aminoethylsulfanyl)propylsulfanyl] ethanamine covalently bound to one and two units of rhodamine-6G moieties (RG-1 and RG-2) were designed and synthesised for highly sensitive and selective detection of Hg²⁺. Their sensing behaviors toward metal ions were investigated by UV/Vis and fluorescence spectroscopy. The binding to Hg²⁺ was observed through both fluorescence enhancement and a chromogenic change (from colorless to pink) from the spirolactam ring-opening process of rhodamine 6G. Especially, RG-1 exhibited the reversible behavior and revealed a very high selectivity in the presence of competitive ions, particularly Cu²⁺, Ag⁺, Pb²⁺, Ca²⁺, Cd²⁺, Co²⁺, Fe²⁺, Mn²⁺, Na⁺, Ni²⁺, K⁺, Ba²⁺, Li⁺ and Zn²⁺, with a low detection limit of 1.7 ppb toward Hg²⁺.

Keywords Chemosensors; Mercury sensor; Hg²⁺-selectivity

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Trace Metals Levels in Human Urine from Kaduna Metropolis

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Samples of human urine, a biological specimen were collected from volunteers in Kaduna metropolis, Nigeria. The concentrations of the four metals (zinc, cobalt, manganese and copper) were determined using Atomic Absorption Spectroscopy. The mean and standard deviation for the four metals were found as follows: Zn ($7.746 \pm 3.981 \mu\text{g/L}$), Co ($2.866 \pm 1.291 \mu\text{g/L}$), Mn ($5.480 \pm 2.179 \mu\text{g/L}$) and Cu ($11.236 \pm 6.146 \mu\text{g/L}$) respectively. These results were compared to standards for normal human levels obtained from Agency for Toxic Substances and Disease Registry (ATSDR) (2013) for these metals in human urine and all were found to be higher than the normal human levels. Several factors could be attributed to the high concentration levels of these metals.

Keywords Atomic absorption spectroscopy; ATSDR; Kaduna metropolis; Metals; Urine

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