

ISSN 2221-6413 (Print), ISSN 2223-2559 (Online)

Coden: PJSIB5 56(2) 59-124 (2013)

Pakistan Journal of Scientific and Industrial Research

Series A: Physical Sciences

Vol. 56, No.2, May-June, 2013



(for on-line access please visit web-site <http://www.pjsir.org>)

Published by
Scientific Information Centre
Pakistan Council of Scientific and Industrial Research
Karachi, Pakistan

Pakistan Journal of Scientific and Industrial Research

Series A: Physical Sciences

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Series A: Physical Sciences [ISSN 2221-6413 (Print); ISSN 2223-2559 (online)] (appearing as issues of January-February, May-June and September-October) and

Series B: Biological Sciences [ISSN 2221-6421 (Print); ISSN 2223-2567 (online)] (appearing as issues of March-April, July-August and November-December).

Each Series will appear three times in a year.

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Subscription rates (including handling and Air Mail postage): *Local:* Rs. 2000 per volume, single issue Rs. 350; *Foreign:* US\$ 400 per volume, single issue US\$ 70.

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Printed and Published by: PCSIR Scientific Information Centre, PCSIR Laboratories Campus, Shahrah-e-Dr. Salimuzzaman Siddiqui, Karachi-75280, Pakistan.

Editorial Address

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Pakistan Journal of Scientific and Industrial Research, PCSIR Scientific Information Centre

PCSIR Laboratories Campus, Shahrah-e-Dr. Salimuzzaman Siddiqui, Karachi-75280, Pakistan

Tel: 92-21-34651739-40, 34651741-43; Fax: 92-21-34651738; Web: <http://www.pjsir.org>, E-mail: info@pjsir.org

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Contents

Cyclic Voltammetric Studies of Thymoquinone with Iron (III) Farah Kishwar and Qamar-ul-Haq	59
Kinetic and Equilibrium Study of Adsorption of Di-Azo Dyes on Commercial Activated Carbon Emad Abdulilah Saleh Al-Hyali and Ra'ed Tariq Ghanim Al-Abady	70
A Study on the Production of Biodiesel from Used Frying Oil Mehmood Abbas, Farooq Ahmad, Adnan Skhawat Ali, Maqsood Ahmed, Muhammad Farhan, Syed Ahtisham Shabbir, Aqsa Iftikhar and Nazish Mohy-u-Din	86
Thermo-Kinetics Studies of Dye Removal with Kaolin from <i>Tectona grandis</i> and Indigo Dye Effluents Abayomi Olagundoye Adetuyi, Abdul-Akeem Alaba Mojibola and Jamiu Mosebolatan Jabar	93
Effect of Mining on Heavy Metal Concentration in Soils from the Vicinity of Itakpe Iron Ore Mine in Kogi State, Nigeria Christiana Omono Matthews-Amune and Samuel Kakulu	100
Hydrochemical Characterization and Quality Evaluation of Groundwater in Parts of the Basement Complex Area of Ekiti, Southwestern Nigeria Talabi Abel Ojo and Tijani Moshood Niyi	107
Short Communication	
Cationic Interference in Mercury Analysis by Cold Vapour Atomic Absorption Spectroscopy Rajkumar Dewani, Shaikh Mohiuddin, Iftikhar Imam Naqvi and Syeda Shadiah Masood	122

Cyclic Voltammetric Studies of Thymoquinone with Iron (III)

Farah Kishwar* and Qamar-ul-Haq

Department of Chemistry, Federal Urdu University of Arts, Science and Technology,
Gulshan-e-Iqbal Campus, Karachi-75300, Pakistan

(received November 28, 2012; revised February 11, 2013; accepted March 6, 2013)

Abstract. Complexation of thymoquinone an active ingredient of *Nigella sativa*, with Fe(III) has been analyzed using cyclic voltammetry. This electrochemical study was performed at glassy carbon as working electrode, platinum as auxiliary and saturated calomel as reference electrode. Whole work was performed at 25 ± 1 °C in aqueous medium using NaCl as supporting electrolyte. Present study reflects effects of scan rates, concentrations, ratios and successive cyclic scans on Fe(III)-thymoquinone complex. Results revealed that the complex shows quasi-reversible electron transfer process. E° of complex was found to be 0.271 ± 0.030 V whereas diffusion coefficient was 9.178×10^{-5} cm²s⁻¹. The values of transfer coefficients, α and β were also determined. The value of α was found to be 0.786 ± 0.01 - 0.923 ± 0.02 whereas value of β was found in the range of 0.813 ± 0.01 - 1.021 ± 0.01 . Calibration curve method with linear regression line confirms that cyclic voltammetry can be used for quantification of Fe(III)-thymoquinone complex for pharmaceutical assay.

Keywords: thymoquinone, iron (III), cyclic voltammetry, Quasi-reversible behaviour, Randles-Sevick equation

Kinetic and Equilibrium Study of Adsorption of Di-Azo Dyes on Commercial Activated Carbon

Emad Abdulilah Saleh Al-Hyali* and Ra'ed Tariq Ghanim Al-Abady
Chemistry Department, College of Education, University of Mosul, Mosul, Iraq

(received June 13, 2011; revised May 1, 2012; accepted July 16, 2012)

Abstract. This research work is concerned with studying the adsorption of a number of di-azo dyes on commercial activated carbon (CAC). The synthesized dyes vary in their structures by the central parts, which are either ortho, meta or para phenylene diamine. This variation affects the linearity of molecules, their spatial arrangement and electron movement throughout the molecule by resonance. Factors affecting adsorption process, such as the effect of contact time, initial concentration, pH of the adsorption medium, adsorbent dose, effect of solvent and temperature were studied. The results indicated that, the adsorption process is fast in the first 10 min, then gradually decreased with time and approaches maximum within 70-80 min for all the studied dyes. The increase of initial concentration and temperature decreased the adsorption efficiency. The results also shows that, the adsorption is found to be more efficient at low pH value. The increase of the adsorbent dose increases the adsorption efficiency and decreases its capacity. The variation of solvent (ethanol-water ratio) indicates that the decrease of dielectric constant lowers the adsorption efficiency. The study included application of three adsorption isotherms; Freundlich, Langmuir and Temkin on the experimental data of the studied systems. The results indicated that, Freundlich isotherm fits better the adsorption data. Kinetic analysis of the adsorption data was also conducted by employing 4 kinetic models; pseudo first order and pseudo second order, Elovich and intra particle diffusion equations. The results obtained conclude that, the studied systems follow the Pseudo second order model.

Keywords: kinetic study, equilibrium of adsorption, di-azo dyes, activated carbon

A Study on the Production of Biodiesel from Used Frying Oil

**Mehmood Abbas^a, Farooq Ahmad^{a*}, Adnan Skhawat Ali^a, Maqsood Ahmed^a, Muhammad Farhan^a,
Syed Ahtisham Shabbir^b, Aqsa Iftikhar^a and Nazish Mohy-u-Din^a**

^aSustainable Development Study Centre, GC University, Lahore, Pakistan

^bDirectorate General of Health, Lahore, Government of Punjab, Pakistan

(received February 28, 2012; revised December 29, 2012; accepted January 4, 2013)

Abstract. The study was carried out to utilize waste frying oil for biodiesel production because it is cheap, easily available and renewable raw material. The used frying oil was analyzed for water contents (0.43 %), iodine value (52), saponification value (205), free fatty acids (8.7 %) and acid value (0.8 mg KOH/g). Esterification and transesterification were conducted to convert free fatty acids and triglycerides to methyl ester (biodiesel), respectively. One-step and two-step transesterification reactions were carried out to measure the efficiency of these processes for biodiesel production. The biodiesel produced from used frying oil was examined for flash point (185 °C), kinematic viscosity (4.86 mm²/s) and specific gravity (0.884 g/mL) that were meeting the limits of ASTM and Thai standards. Hence, it was proved to be a useful technique for biodiesel production at commercial scale.

Keywords: biodiesel, esterification, transesterification, waste cooking oil, renewable raw material

Thermo-Kinetics Studies of Dye Removal with Kaolin from *Tectona grandis* and Indigo Dye Effluents

Abayomi Olagundoye Adetuyi*, Abdul-Akeem Alaba Mojibola and Jamiu Mosebolatan Jabar
Department of Industrial Chemistry, Federal University of Technology, P.M.B 704, Akure, Ondo State, Nigeria

(received May 15, 2012; revised August 28, 2012; accepted September 12, 2012))

Abstract. The kinetics and thermodynamic behaviour of dye molecules removed from *Tectona grandis* and indigo dye effluents using kaolin as the coagulant were studied and the results obtained were compared with the commercial alum (as standard). Comparison of data obtained from Langmuir and Freundlich isotherms indicated that the coagulation process was chemisorptions. The isotherm parameters of Langmuir isotherm such as coagulation capacity Q_0 ; coagulation energy intensity b ; and correlation coefficient R^2 , in this study were greater than that of Freundlich. The correlation coefficient of the pseudo-second-order kinetics was almost equal to unity and the values of $q_{e(\text{Cal.})}$ were of insignificant difference from the corresponding $q_{e(\text{exp.})}$ and these made the pseudo-second-order kinetics fitted well with the coagulation process. The thermodynamic parameters (ΔH and ΔS) obtained indicated that the coagulation process was endothermic and spontaneous, respectively. The negative values of ΔG shows the irreversible nature of the coagulation process and the correlation coefficients (R^2) closed to unity indicates the fitness of the coagulation thermodynamics with the experimental data.

Keywords. coagulant, effluents, kinetics, endothermic, thermodynamics

Effect of Mining on Heavy Metal Concentration in Soils from the Vicinity of Itakpe Iron Ore Mine in Kogi State, Nigeria

Christiana Omono Matthews-Amune* and Samuel Kakulu

Department of Chemistry, University of Abuja, Abuja, Nigeria

(received June 6, 2012; revised August 31, 2012; accepted September 12, 2012)

Abstract. The effects of mining on soils from Itakpe iron ore mining area in Kogi State, Nigeria were studied through the determination of the heavy metals (Cd, Cu, Mg, Ni, Pb and Zn) using flame atomic absorption spectroscopy. Soil samples were collected during the dry and rainy seasons. Significant levels of heavy metals were found. Median topsoil concentrations (0-15 cm) for Cd, Cu, Mg, Ni, Pb and Zn were 0.16 ± 0.02 , 0.15 ± 0.03 , 0.04 ± 0.03 , 0.1 ± 0.02 , 0.07 ± 0.01 , 0.04 ± 0.04 $\mu\text{g/g}$, respectively. The heavy metal concentrations of control soil were relatively lower than those in the Itakpe mining environment soil and within levels of total metal contamination in the normal soil content intervals and maximum allowable limits of heavy metals in soils. Correlations analysis shows that heavy metals were closely correlated with each other except for Pb, indicating the studied metals are from the same pollutant resource. This shows, mining as contributing to the metallic levels in the Itakpe mining site.

Keywords: mining, heavy metals, soil, pollution

Hydrochemical Characterization and Quality Evaluation of Groundwater in Parts of the Basement Complex Area of Ekiti, Southwestern Nigeria

Talabi Abel Ojo^{a*} and Tijani Moshood Niyi^b

^aDepartment of Geology, Ekiti State University, Ado-Ekiti, Nigeria

^bDepartment of Geology, University of Ibadan, Ibadan, Nigeria

(received August 8, 2011; revised March 29, 2012; accepted April 25, 2012)

Abstract. Well water (88 samples) were collected across various bedrock units in the basement terrain of Ekiti area, Southwestern Nigeria. They were subjected to *in-situ* physico-chemical measurement and hydrochemical analyses using ICP-OES and ion-chromatography methods for cations and anions, respectively. To understand the water quality and utilisation aspects of groundwater, chemical indices like sodium %, sodium adsorption ratio (SAR), Wilcox diagram and salinity diagram were constructed based on the analytical results. The results show pH values ranging between 6.0-7.8 and total hardness (TH) 3.2-508.7 mg/L. Major cations concentrations were in the order of $\text{Ca}^{2+} > \text{K}^+ > \text{Na}^+ > \text{Mg}^{2+}$ with average values of 28.5, 26.8, 24.2 and 7.9 mg/L, respectively while that of the anions were $\text{HCO}_3^- > \text{Cl}^- > \text{SO}_4^{2-} > \text{NO}_3^-$ with average values of 118.7, 54.2, 23.8 and 0.92 mg/L. The main hydrochemical facies being Ca-HCO₃ waters. The ionic orders of abundance varied in different rocks of the study area. These concentration trends show a low total dissolved solids (130-1544 $\mu\text{S}/\text{cm}$) indicating a low water-rock interaction due to low residence time which is an indication of CO₂ dominated infiltration recharge with limited migratory history typical of the shallow basement terrain in the study area. Quality assessment revealed a potable groundwater system with chemical parameters within the acceptable limits of the WHO and SON drinking water standards with exception of Fe, Mn and Pb in a couple of locations. Also, the estimated SAR alongside TDS revealed a shallow groundwater system suitable for irrigation purposes.

Keywords: basement, sodium adsorption ratio, salinity, water-rock interaction, irrigation

Short Communication

Cationic Interference in Mercury Analysis by Cold Vapour Atomic Absorption Spectroscopy

Rajkumar Dewani*^a, Shaikh Mohiuddin^b, Iftikhar Imam Naqvi^b and Syeda Shadia Masood^b

^aLeather Research Center, PCSIR, D-102, SITE, South Avenue, Karachi-75700, Pakistan

^bDepartment of Chemistry, University of Karachi, Karachi-75270, Pakistan

(received March 5, 2012; revised August 1, 2012; accepted August 5, 2012)

Abstract. The cationic interference in the analysis of mercury at trace level by cold vapour atomic absorption spectroscopy (CVAAS) has been investigated. Different cations (Na^+ , K^+ , Fe^{2+} , Zn^{2+} , Cr^{3+} , Co^{2+} , Ni^{2+} and Cu^{2+}) of varying concentrations are examined as possible interferents in the analysis of mercury. Na^+ , Fe^{2+} , Zn^{2+} and K^+ did not show any affect due to their presence in the detection of mercury while Co^{2+} , Ni^{2+} , Cu^{2+} and Cr^{3+} had a marked influence on mercury recovery and can be suggested as possible interfering agents for this method of Hg analysis.

Keywords: mercury analysis, cold vapour technique, cationic interference
