

KINETICS AND ISOTHERMS OF LEAD (II) IONS ADSORPTION ONTO CHELATING SCHIFF BASE DERIVED FROM 2-HYDROXY-2-PHENYLACETOPHENONE AND 2-AMINO BENZOIC ACID

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ABSTRACT

The research is aimed at utilizing a Schiff base (SB) adsorbent derived from 2-aminobenzoic acid and Benzoin to study the kinetics and isotherms for adsorptive removal of lead (Pb^{2+}) from simulated wastewater samples as well as evaluate the dependence of adsorbent's adsorption capacity on select operational parameters (contact time and adsorbent dosage) using a batch adsorption technique. The SB was prepared by refluxing ethanolic solutions of Benzoin (0.01 mol) and 2-aminobenzoic acid (0.01 mol). The equilibrium adsorption data for lead (II) ions were fitted to Langmuir isotherm model. The maximum monolayer adsorption capacity of SB as obtained from Langmuir isotherm was 4.97 mg/g ($R^2 = 1.0000$) for contact time and 4.93 mg/g ($R^2 = 0.9999$) at 0.5g for adsorbent dose. Kinetics data correlated well with the pseudosecond order kinetic model ($R^2 = 0.9999$) more than with the pseudofirst order kinetic model ($R^2 = 0.9656$). Characterization of the Schiff's base by Fourier Transform Infrared Spectroscopy (FTIR) before and after adsorption of Pb^{2+} revealed that adsorption has taken place as evidenced by shifts and disappearances of important peaks in their spectra.

Keywords: Adsorption, Lead (II), Chelating, Schiff's base, Acid

INTRODUCTION

The continued pollution of aquatic environments resulting from the excessive release of dissolved hazardous heavy metals ions via untreated effluents and wastewater originating from divergent sources has attracted attention globally (Herica *et al.*, 2012). Industrial wastewater and untreated effluents from battery manufacturing, dyeing, electroplating, lumbering and wood processing, mining and mineral processing, metallurgical, organic chemicals, paint, pesticides, petroleum refining, pharmaceuticals, pigments, photographic film production, rubber and plastics, tanneries and textiles have been established to be the main anthropogenic point sources of pollution phenomenon by heavy metals (Tripathi and Ranjan, 2015; Ellchi and Tetsuo, 1984). In other words, rising levels of mobile and soluble heavy metal species is a serious problem, owing to their high degree of toxicity (Herica *et al.*, 2012). Pollution of downstream sources of water occurs by transportation of heavy metals through runoff from adjacent industrial sites (Lakherwal, 2014). Unlike organic pollutants, heavy metals are recalcitrant non-biodegradable species which undergo bioaccumulation and biomagnifications in organisms leading to numerous diseases, symptoms and disorders including high blood pressure, anaemia, lead poisoning, coma and many other well-documented abnormal manifestations (Orodu *et al.*, 2014; Yu, 2005). Most of the heavy metals discharged into the wastewater threaten human health due to their carcinogenic and mutagenic propensities (Srivastava *et al.*, 2006; Lakherwal, 2014). Some heavy metal ions of major environmental concern are cadmium (Cd^{2+}), lead (Pb^{2+}), mercury (Hg_2^{2+}), aluminium (Al^{3+}), cobalt (Co^{2+}), copper (Cu^{2+}), manganese (Mn^{2+}), nickel (Ni^{2+}) and zinc (Zn^{2+}) (Tripathi and Ranjan, 2015; Ellchi and Tetsuo, 1984). The removal of these toxic heavy metals from effluents and untreated wastewater before disposal has therefore become essentially pertinent to safeguard human health against their adverse and hazardous effects.

Various treatment methods have been employed to remove heavy metal ion pollutants from aqueous media such as chemical precipitation, coagulation, ion exchange, membrane separation, evaporation, and electrolysis and which are often expensive and ineffective especially in removing heavy metal ions from dilute solutions (Monier, 2012; Shiyani *et al.*, 2009). Of all the various treatment methods used for the removal of heavy metals from wastewater, adsorption techniques is one of the well-recognized

and most effective. Adsorption technique utilizing commercial activated carbons and synthetic ion exchangers is the most preferred approach in heavy metal ions removal from industrial effluents and untreated wastewaters especially when the metal ions are at trace concentration levels and high ionic selectivity is required (Monier, 2012). To economically meet the existing higher standards and wastewater reuse requirements in the physicochemical treatment of effluent and municipal wastewater, adsorption process is fundamental (Nordiana and Siti, 2013). The obvious disadvantages of this technique that offset its benefits are the rising costs of the commercial activated carbons and synthetic ion exchangers as well as its inadequate efficiency (Monier, 2012). The quest to produce cheaper and more available adsorbents motivated researchers, informing the recent upsurge in research for unconventional adsorbents of natural or synthetic origin.

Lead (Pb) is a basic chemical element, which can combine with other substances to form lead compounds. A bluish-white metal or bright lustre, lead is very soft, highly malleable, and ductile, has a low melting point and is resistant to corrosion. These characteristics brought lead into common usage even in ancient times. In modern times manufacturers have used lead in a variety of products, some of which included; gasoline, paint, plumbing, fine crystal, electric cable insulation, storage batteries, ammunitions and insecticides. However, lead, which throughout history has been one of man's most useful and versatile metals, is one of man's oldest poisons. The Royal Society of Canada (1986) reported that human exposure to lead has harmful effects on kidney, central nervous and reproductive systems. Today, lead toxicity continuous to be a significant health problem, especially in children living in urban areas. Lead in drinking water can cause a variety of an adverse health effect in babies and children, exposure to lead in drinking water above the action level can result in delays in physical and mental development, along with slight deficits in attention span and learning abilities. Adults who have been taking lead-polluted water for many years can develop kidney problems or high blood pressure (Nordiana and Siti, 2013).

The synthesis of various Schiff's base-derived adsorbents used for adsorptive removal of lead and other heavy metal ions via chelation mechanism have been reported in the literature. They include Schiff's bases fabricated from 4-aminoantipyrine and 2-

methoxybenzaldehyde condensation (Chuan-Wei *et al.*, 2013), dialdehyde starch and aminopyrazine reaction (Salisu *et al.*, 2016), ditopic zwitterionic Schiff's base modified silica gel (Xu *et al.*, 2018), chitosan-crotonaldehyde Schiff's Base coated AZ91E alloy (Mohamed and Fekry, 2011), ϵ -caprolactone monomer and oxydianiline Schiff's base modified cotton fiber (Agathian *et al.*, 2018), UiO-66-Schiff's base (Yuan *et al.*, 2017), chitosan cinnamaldehyde Schiff's base (Tsaneva *et al.*, 2017) and Fe₃O₄-silica Schiff's base (Setoodekhah and Momeni, 2017). The aim of the current research work is to synthesize a chelating Schiff's base derived from 2-aminobenzoic acid and 2-hydroxy-2-phenylacetophenone (also called *benzoin*) and utilize the synthesized Schiff's base as an adsorbent in the study of adsorption isotherms and kinetics for adsorptive removal of lead ion (Pb²⁺) from synthetic wastewater, as well as evaluate the effects of contact time and adsorbent dosage on the adsorption process.

MATERIALS AND METHOD

Reagents

All the reagents used were of analytical reagent grade and were used without further purification. They include *Benzoin* (also called 2-hydroxy-2-phenylacetophenone), 2-aminobenzoic acid, Sodium acetate, Pb(NO₃)₂ as a source of Pb²⁺, distilled water and calcium chloride as desiccant.

Preparation of Schiff's base (SB)

To prepare a Schiff's base (SB), an ethanolic benzoin (0.01 mol) solution and 2-aminobenzoic acid (0.01 mol) were added into a 500 cm³ round bottom flask, followed by the addition of 3.0 g of anhydrous sodium acetate. The mixture was refluxed for an hour on a steam bath. To precipitate the grayish white product the content was poured into ice-cold water which was separated and

recrystallized from rectified spirit, filtered, washed with water, and dried in vacuum desiccators (Mahaptra *et al.*, 1977).

Preparation of Stock Solution for Calibration

A stock solution containing 1000 mg/L of lead (II) ions was prepared by transferring 1.5992 g of analytical-reagent grade lead (II) nitrate salt into 1000 mL volumetric flask, dissolving the solute in and topping the flask with distilled water, followed by gentle shaking to obtain a homogeneous solution (Osman 2013; Dada *et al.*, 2013). Measured volume aliquots of the stock solution were serially diluted accordingly to prepare different concentration levels of the lead (II) ions.

Adsorption Contact time Experiments

The batch adsorption experiments were conducted in 250 mL Erlenmeyer flasks with 50 mL of 10mg/L standard solution and equilibrated using a shaker. The amount of SB used throughout this research was 0.1 g for 30, 60, 90, 120, 150 and 180 minutes. All filtrates were analysed using atomic absorption spectrometer (*Bulk Scientific UPG 210*).

Amount of Adsorbent Variation (Dose)

The amounts of adsorbents were varied from 0.5, 1.0, 1.5 and 2.0g. 0.5g of the adsorbent was mixed with 50 cm³ of 10mg/L of lead solution and the mixture was shaken at 280 rpm for 90 minutes at 40°C. After the adsorption, the mixture was filtered through filter paper. The same method was applied to 1.0, 1.5 g and 2.0 g amount of adsorbent, lead concentration and the temperature constant. All filtrates were analysed using atomic absorption spectrometer as reported in Dada *et al.* (2013).

The amount of lead adsorption at equilibrium was calculated using the following equation:

$$\text{Adsorption capacity } (q_e) = \left(\frac{C_o - C_e}{W} \right) V \quad [1]$$

$$\% \text{Adsorption} = \left(\frac{C_o - C_e}{C_o} \right) \times 100 \quad [2]$$

where C_o is the initial concentration of lead(II) ions (mg/L), C_e is the final concentration of lead(II) ions (mg/L), V is the volume of lead(II) ions solution (L) and W is the weight of SB (g) used (Osman 2013).

RESULTS AND DISCUSSIONS

The FTIR spectrum of SB was shown in Figure 1. The peaks at 1680 and 1579 cm⁻¹ were attributed to the C=O group (conjugated

and the C=N bonds, respectively. The present of imine group, C=N confirmed that a Schiff's base was formed from the reaction between 2-aminobenzoic acid and benzoin. The peaks found at 1598 cm⁻¹ were due to the C=C bonds in the benzene rings of SB. The peaks at 3030 and 2933 cm⁻¹ were attributed to the C-H stretching. Meanwhile the =C-H stretching was evident at 3080 cm⁻¹. The broad peak at 3378 cm⁻¹ is due to O-H stretching.

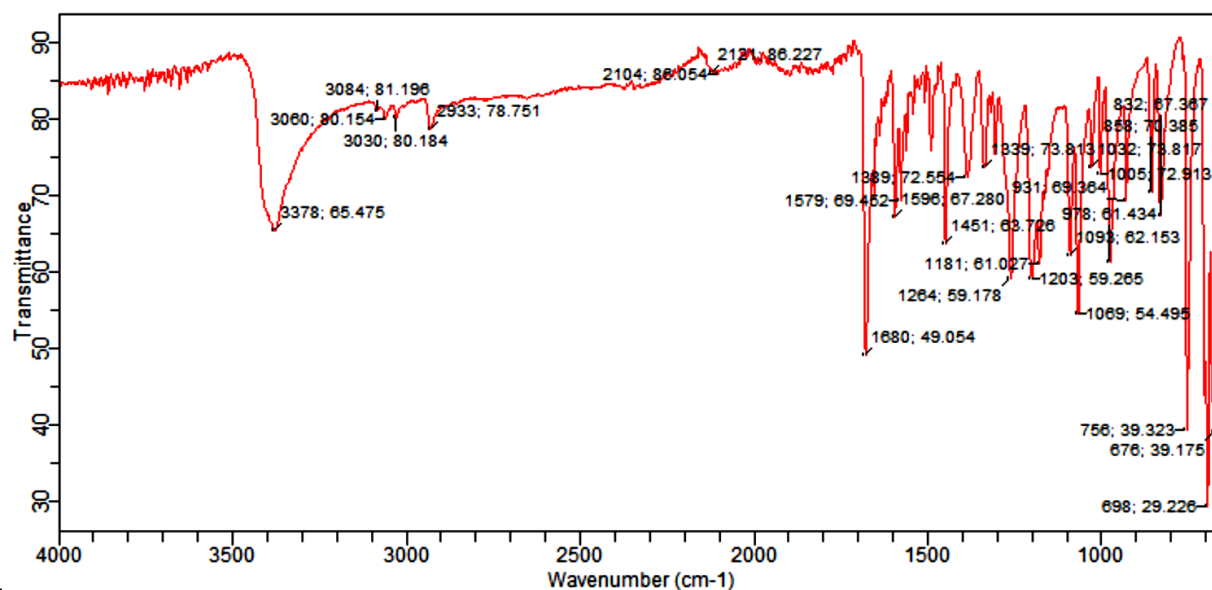


Figure 1: FTIR of the Schiff's base (SB)

The medium broad peak with two sharp peaks at 3478 cm⁻¹ could be due to the present of N-H molecule on the 2-aminobenzoic acid after the adsorption the absorption band was increased significantly Figure 2.

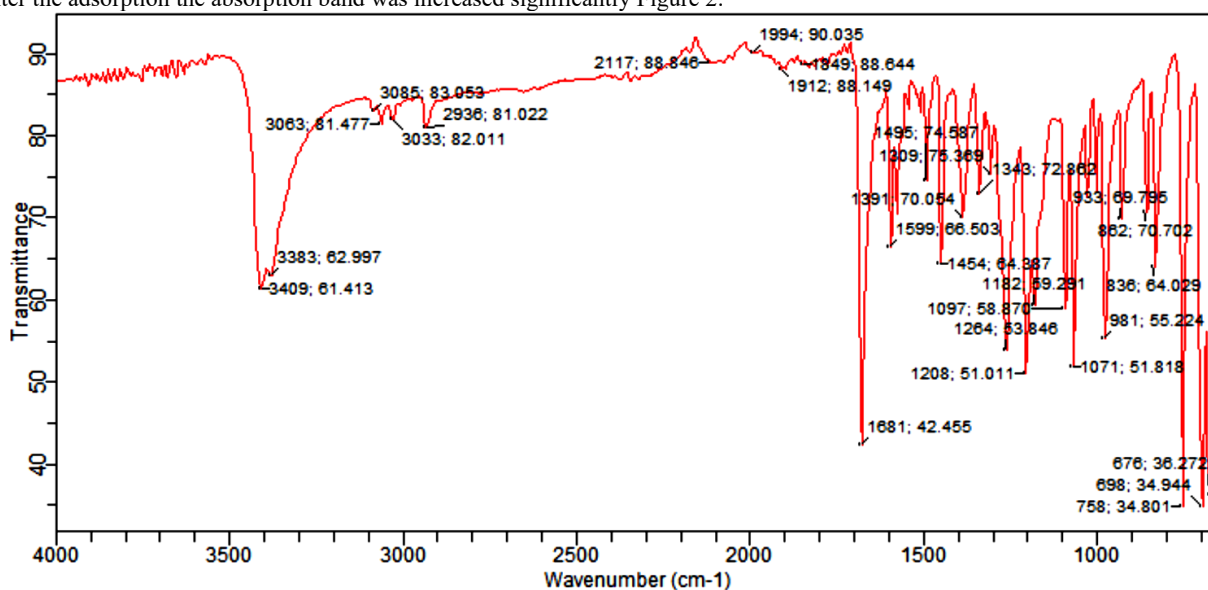


Figure 2: FTIR of the Schiff's base (SB) after adsorption

Effect of contact time on the removal of lead (II) ions by SB

The adsorption rate of lead (II) ions onto SB was investigated as it represented the amount of time required before the adsorption process becomes constant and equilibrium was reached. As shown in Figure 3, the adsorption rate was high and equilibrated at 90min and it decreased gradually until it reached 180 min with an adsorption percentage of 97.82%.

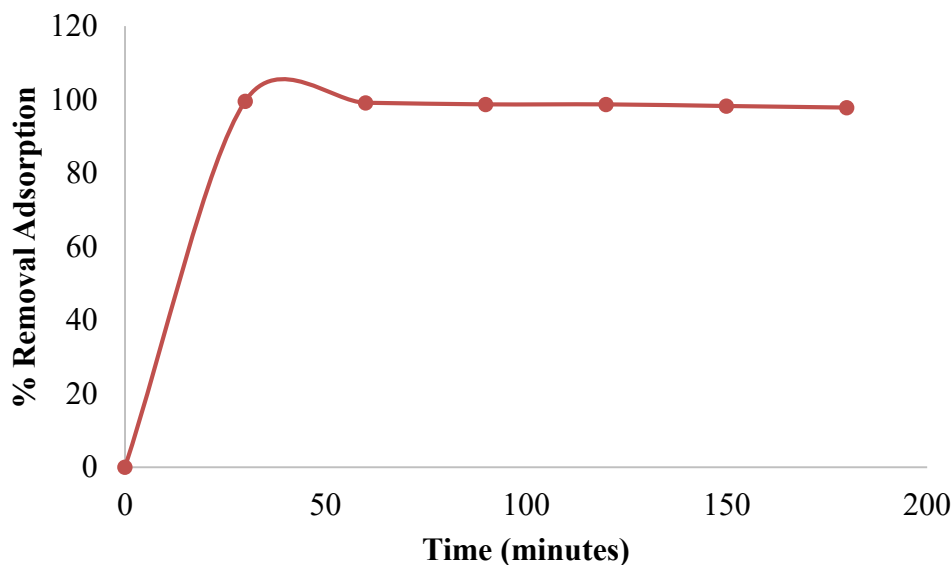


Figure 3: Effect of contact time on the removal of lead (II) ions by SB.

The high adsorption rate observed at initial stage could be due to the adsorption of lead (II) ions onto the external surface of the adsorbent. Meanwhile, the slower uptake rate observed after 90 min is due to the pore diffusion and the quick exhaustion of the available adsorption sites. The adsorption data were then analyzed with pseudo first- and pseudosecond order kinetic models. In correlation with previous work Borhade *et al.* (2015), got 95 % of the Pb^{2+} and Cd^{2+} were removed after 150 minutes at its equilibrium from the adsorbent surface. However, the removal of Ni^{2+} increased with contact time and reached a maximum at 150 min with the removal efficiency 60%. In Nordiana and Siti (2013) work from 120 minutes to 150 minutes and almost constant until 300 minutes with highest 97.84 % removal of lead ions. At that

time, it was showed the sufficient to achieved equilibrium and the adsorption did not change with further increase in contact time.

Effect of Adsorbent Dose

The dependence of adsorption of lead on the amount of Schiff's base was studied at temperature of 40°C by varying the amount of adsorbent (0.5, 1.0 1.5, and 2.0g). The result is shown Figure 4. The amount of lead adsorbed ($mg.g^{-1}$) was found to decrease with increasing amount of adsorbent. The amount of lead (II) ions adsorbed decrease from 4.93, 4.56, 4.88 and it increases at $4.93 g^{-1}$ at adsorbent amount of 0.5 to 2.0g respectively. Based on Table 3.4, an amount of 0.5g and 2.0 of the adsorbent was found to be significant to remove higher percentage of lead from the aqueous solution.

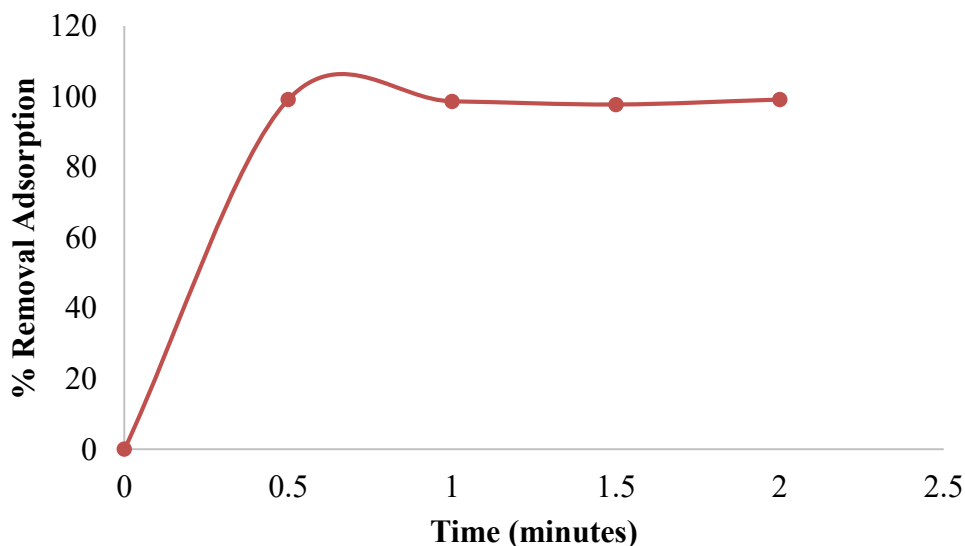


Figure 4: Effect of adsorbent dose on the removal of lead (II) ions by SB.

Nordiana and Siti (2013) observed the adsorption of Pb (II) ions to increase linearly as the amount of adsorbent is gradually increased from 1 g to 3 g and remained constant from 3 g to 5 g. The maximum adsorption is obtained at the adsorbent dose of 3 g with 97.73 % removal of Pb(II) ions where a further increase in the quantity of the adsorbent had no more effect to the adsorption rate.

Therefore, the study indicated that 3 g of the adsorbent is sufficient to adsorb the maximum amount of Pb(II) ions. For the 1 g to 3 g of adsorbent, the increase in adsorption was due to the availability of more adsorbing sites at high doses. It has been suggested that electrostatic interactions between the adsorbent and metal ions in solution can be a significant factor in the sorption process. In this

connection, the higher is the adsorbent dosage in suspension the higher will be the amount of metal ions retained by a sorbent unit at a given metal concentration, unless the adsorbent reaches saturation.

Kinetics studies

The adsorption kinetics of metal ions is important for designing the adsorption system and is required for determining the optimum

operating conditions for a full scale batch process. Many adsorption kinetic models have been applied to understand the adsorption kinetics and the rate-limiting step. Among all, pseudo first- and pseudosecond order models are the most commonly used to study the adsorption kinetics of heavy metal ions (Chuan-Wei *et al.*, 2013). The pseudofirst order kinetic model is given in its linear form as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \tag{3}$$

where q_e is the amount of lead(II) ions adsorbed at equilibrium (mg/g), q_t is amount of lead(II) ions adsorbed at time t (mg/g) and k_1 is the pseudofirst order kinetic constant (min^{-1}). Linear plot of $\log(q_e - q_t)$ against t for the adsorption of lead (II) ions onto SB was shown in Figure 5. In many cases, the pseudofirst order kinetic model does not fit well to the whole range of contact time. It would only be applicable over the initial stage of the adsorption process (Chuan-Wei *et al.*, 2013).

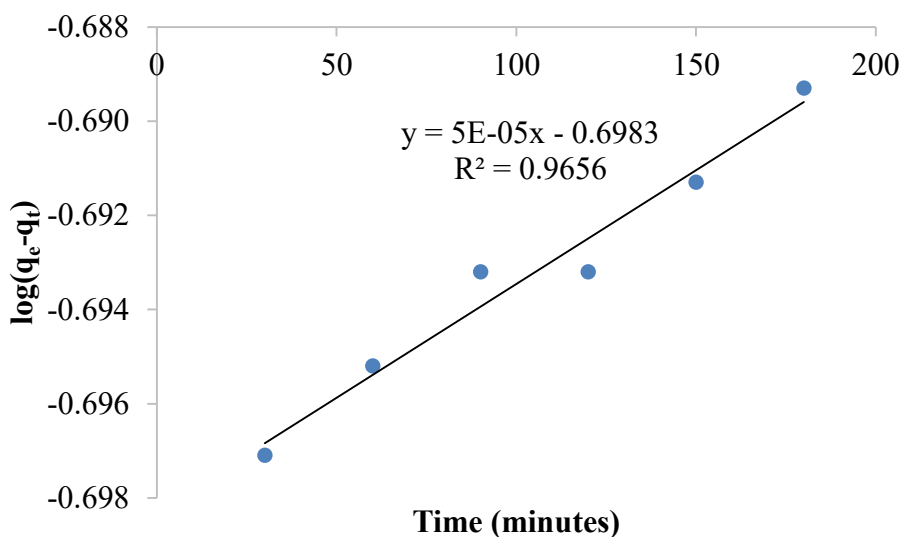


Figure 5: Pseudofirst order plots for the removal of lead(II) ions with SB.

The pseudosecond order kinetic model is based on the assumption that the rate-limiting step in the adsorption of heavy metal ions is chemisorption involving the valence force through the sharing or exchange of electrons between the adsorbate and the adsorbent (Chuan-Wei *et al.*, 2013). This kinetic model is represented as:

$$\frac{t}{q_t} = \frac{1}{h} + \frac{t}{q_e} \tag{4}$$

where $h = k_2 q_e^2$, q_e and q_t are the amount of lead(II) ions adsorbed at equilibrium and at time t (mg/g) respectively and k_2 is the pseudosecond order kinetic constant (g/mg·min). Figure 6 shows the linear plots of t/q_t against t .

The pseudosecond order kinetic model gave higher correlation ($R^2 = 0.9999$) than the pseudofirst order kinetic model ($R^2 = 0.9656$). Hence, the pseudosecond order is more appropriate to represent the adsorption data of lead (II) ions onto SB. The poor fit observed for pseudofirst order kinetic model could be due to a time lag caused by a boundary layer or the external resistance controlling at the beginning of the adsorption process (Chuan-Wei *et al.*, 2013).

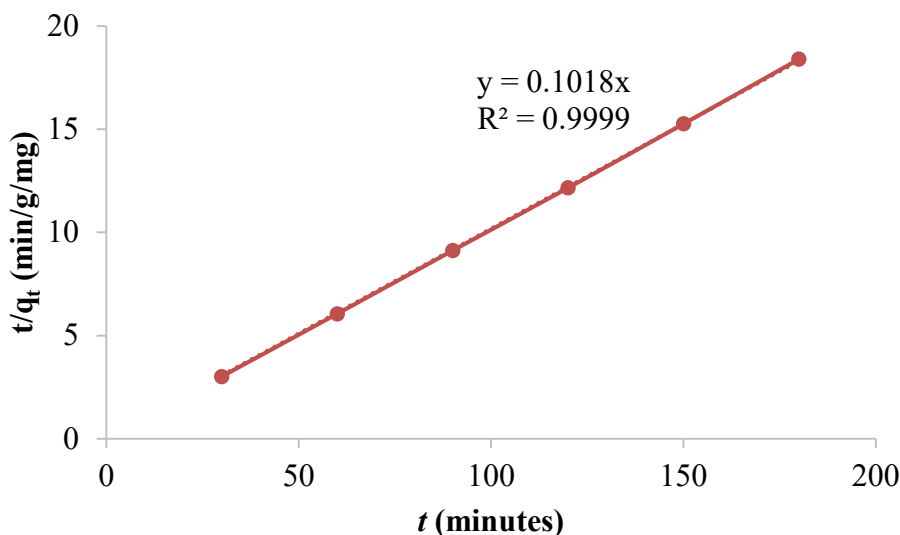


Figure 6: Pseudo-second order plots for the removal of lead (II) ions with Schiff's base (SB).

The work correlates well with the work of Zare-Dorabei *et al.* (2016). The adsorption kinetic studies for evaluating the mechanism of adsorption process showed a good fit to the pseudo-second order and the work of Wang *et al.* (2016). Using gelation with alginate used as selective removal of heavy metals (Pb²⁺, Cd²⁺, Cu²⁺) also followed pseudo-second order. According to Shen *et al.* (2015), using g-C₃N₄ as adsorbent for heavy metals from aqueous solution their adsorption kinetics of Pb (II), Cu (II) followed pseudo-second order.

Isotherm Studies

The maximum adsorption capacity of Schiff's base for lead was investigated over a range of lead concentrations. Figures 7 and 8 show the plot of adsorption capacity, C_e/q_e (mg g⁻¹) versus the equilibrium concentration of lead ions in the solution, C_e (mg L⁻¹). The amount of lead adsorbed was found to increase with increasing lead concentration until the maximum adsorption capacity was achieved. The distribution of metal ions between liquid and solid

phases is generally described using Langmuir (Langmuir, 1916) adsorption isotherm equation. The Langmuir equation assumes uniform energies of adsorption onto the adsorbent surface and no transformation of adsorbate in the plane of the surface. The Langmuir adsorption isotherm is given as:

$$\frac{C_e}{q_e} = \frac{1}{KV_m} + \frac{C_e}{V_m} \tag{5}$$

where C_e is the equilibrium Pb²⁺ concentration (mg. L⁻¹), q_e is the amount lead adsorbed at equilibrium (mg. g⁻¹), V_m is the monolayer adsorption capacity (mg. g⁻¹) and K is the Langmuir equilibrium constant (L. mg⁻¹). Figure 7 and 8 shows the Langmuir adsorption isotherm obtained by plotting C_e/q_e versus C_e. Based on the value obtained from isothermal model, it is clear that the Langmuir equation has best fitted for the adsorption of lead onto Schiff's base.

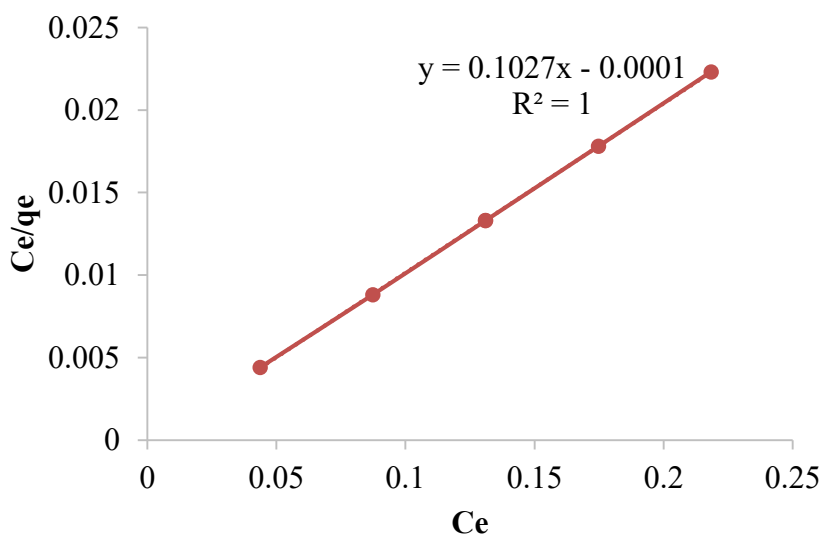


Figure 7: Langmuir adsorption isotherm for the adsorption of lead(II) ions onto Schiff's base (SB) for contact time

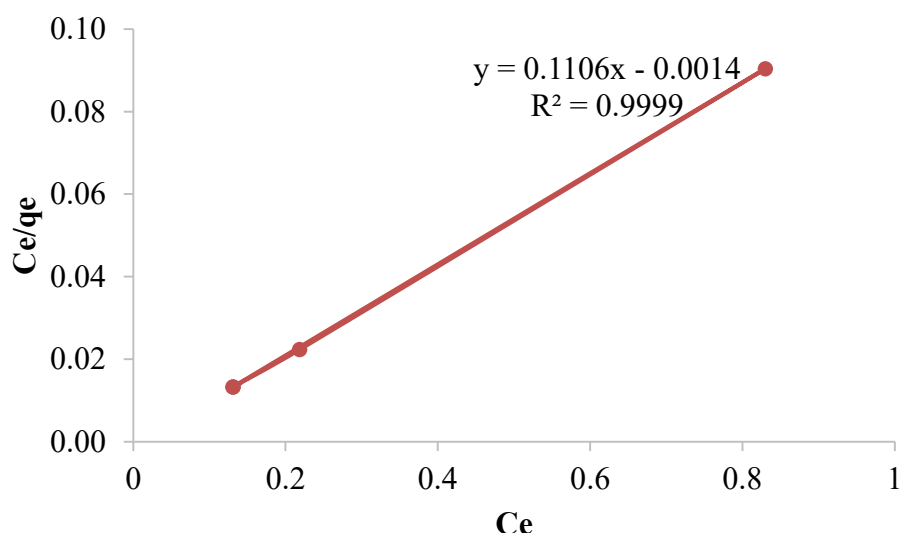


Figure 8: Langmuir adsorption isotherm for the adsorption of lead (II) ions onto SB for adsorbent dose

The results correlated to the work of Borhade *et al* (2015) which fitted Langmuir models well using experimentally obtained data for the adsorption of Pb^{2+} , Cd^{2+} , and Ni^{2+} and also the work of Wang *et al.*; (2016), the adsorption isotherms were better fitted by the Langmuir equation. According to Rouholar *et al.* (2016), the adsorption equilibrium isotherms for evaluating the mechanism of adsorption process showed a good fit to the Langmuir model.

CONCLUSION

This research work shows that Schiff's base (SB) synthesised from 2-aminobenzoic acid and Benzoin is a potentially effective synthetic adsorbent for the removal lead (II) ions from their simulated wastewater under neutral pH conditions. The adsorption process was established to be monolayer due to an excellent fitting of the adsorption data with Langmuir isotherm plots; the adsorption capacity is found to be dependent on contact time and dosage of the Schiff's base (SB) adsorbent used in the batch process; and has chemisorption mechanism to be the rate-determining step as suggested by better fitting of the kinetic data with the pseudosecond order kinetic model. Occurrence of shifts and disappearances of important peaks in the FTIR spectra of the Schiff's base before and after adsorption corroborated the occurrence of Pb^{2+} adsorption onto the synthetic Schiff's base.

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