

Diffusion-Chemisorption and Pseudo-Second Order Kinetic Models for Heavy Metal Removal from Aqueous Solutions Using Modified and Unmodified Oil Palm Fruit Fibre

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Authors' contributions

The work was carried out in collaboration between all authors. Author AO designed the work, supervised the laboratory work and wrote the first draft of the manuscript. Authors CE and CA carried out the Laboratory work and generated all the data used and wrote a part of the manuscript. Authors JI and KU did the data interpretation and fine tuned the drafted manuscript. All authors read and approved the final manuscript.

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ABSTRACT

Aims: To investigate the kinetics of Cu(II), Pb(II), Ni(II) and Sn(II) absorption from aqueous solution using modified and unmodified oil palm fruit fibre using diffusion-chemisorption and pseudo-second order kinetic models.

Study Design: The use of oil palm fruit fibre as absorbent for heavy metals absorption from aqueous solution.

Place and Duration of Study: Physical Chemistry Laboratory, Department of Pure and Industrial

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Chemistry, Abia State University, Uturu, Abia State, Nigeria. The duration was about six (6) months.

Methodology: Majorly, instrumental methodology was used for the detection of the heavy metals. Batch absorption experiment for Cu(II), Pb(II), Ni(II) and Sn(II) were carried out for each absorbent (UOPF, 0.5MOPF and 1.0MOPF). Filtrations were done using whatman no 41-filter paper. The filtrate of each metal solution was analyzed using Shimadzu model 6650 Atomic Absorption Spectrophotometer.

Results: The results showed that chemical modification of the absorbent increased its absorption efficiency which followed the sequence; 1.0MOPF > 0.5MOPF > UOPF. The results also showed that Ni(II) was more removed than the other metals using the three absorbents. Diffusion-chemisorption and pseudo second order kinetic equations were used to model the sorption experimental data. The pseudo-second order model gave better fit to the sorption studies. This implies that the sorption process was of chemisorption mechanism. The initial sorption rate (h_0) was higher for Ni (II) ions with values of 0.484, 3.675 and 2.964 for UOPF, 0.5MOPF and 1.0MOPF respectively, and lowest for Sn (II) with values of 0.035, 0.111 and 0.828 for UOPF, 0.5MOPF and 1.0MOPF respectively.

Conclusion: The results of this study showed that oil palm fruit fibre can be efficiently used as a low-cost alternative for the removal of Cu(II), Pb(II), Ni(II) and Sn(II) from aqueous solutions.

Keywords: Absorption, diffusion-chemisorption, metal ions, oil palm fruit fiber, pseudo second order kinetics.

1. INTRODUCTION

Environmental pollution by heavy metals is now a major concern throughout the world. The increased use of metals and chemicals in industries has resulted in the generation of large quantities of effluents containing high levels of toxic heavy metals [1,2]. Among these heavy metals are Cu, Pb, Ni and Sn. These metals are notable for their wide environmental dispersion from various industrial activities. The presence of these metals in the environment is of major concern because of their toxicity and tendency to bioaccumulate in food chain even in relatively low concentration [1,3,4]. The problem of heavy metal pollution in the environment needs continuous monitoring and surveillance as these elements do not degrade and tend to biomagnify in man through the food chain. Hence, there is need to remove them from the ecosystems [3].

Following the need to remove heavy metals from industrial waste water effluents to reduce environmental pollution, various conventional methods have been developed over the years. These methods include chemical precipitation, ion exchange, oxidation and reduction, fixation or cementation and solvent extraction [5]. However, these techniques have disadvantages such as high capital and operational cost or treatment and disposal of the residual metal sludge [4]. Recently, attentions have been directed towards the use of agricultural by-product as a new

technology for removing toxic metals from industrial effluent [3]. Recent studies have shown that heavy metals can be removed using agricultural by-products [6-8]. Some of the agricultural by-products that have been studied includes tea residue [9]; waste egg shell [10]; maize tassel [11]; waste Eucalyptus charcoal [12]; palm shell activated carbon [13]; sawdust [14]; oil bean seed shell [15] *Luffa cylindrical* fibre [16]; cocoa pod husk [17]; boiler fly ash [18]; coconut fibre [19] and so on. The major advantages of agricultural by-products are that they are economical, eco-friendly, abundant, renewable and more efficient [4].

The modifications of the absorbent have been reported to enhance the sorption capacity of the absorbent [20]. Several types of modification have been reported such as urea and ferric Fe(III) oxyhydroxide modified sawdust [21]; NaOH modified sugar beet pulp [22]; NaOH modified rice husk ash [23]. Thiolation of absorbents have also been reported, such as sulphur-hydryl infused cellulose surface [24]; thiolated cassava waste [25]; and thiolation of coconut fibre [26,27].

Therefore, this research was aimed at investigating the kinetics of Cu(II), Pb(II), Ni(II) and Sn(II) absorption from aqueous solution using modified and unmodified oil palm fruit fibre using diffusion-chemisorption and pseudo-second order kinetic models.

2. EXPERIMENTAL DETAILS

2.1 Materials

2.1.1 Reagents used and Synthetic wastewater

All reagents used were of analytical grade and were used as purchased without further purification. Double-distilled water was used in preparation of all sample solutions. Stock solutions of Cu(II), Pb(II), Ni(II) and Sn(II) ions were prepared (1000 mg/L) by dissolving the desired quantity of CuSO₄, PbSO₄·6H₂O, NiSO₄·6H₂O and SnCl₂ respectively in double-distilled water. The working concentrations of 50 mg/L were obtained by proper dilution of the stock solutions.

2.1.2 Absorbent

The oil palm fruit fibre was obtained from an oil palm mill in Umuire village in Umuahia, Abia State, Nigeria. The fibre was de-oiled by soaking it in hot distilled water and with detergent for 24 hrs. It was rinsed in hot distilled water to remove debris and air dried. The air-dried oil palm fruit fibre was milled and sieved through a 150 µm mesh [1].

2.2 Methods

2.2.1 Activation of absorbent

The sieved fibre absorbent was further soaked in excess 0.3 M HNO₃ solution for 24 hrs; it was then filtered through a whatman No 41-filter paper and rinsed with distilled water. The rinsed absorbent was later air dried for 24 hrs; the treatment of the absorbent with 0.3 M HNO₃ solution aids the removal of any debris or soluble biomolecules that might interact with metal ions during sorption. This process is called chemical activation of the oil palm fruit fibre [1].

2.2.2 Modification of absorbent

The air dried activated oil palm fruit fibre was weighed and divided into three parts, 50 g of the first part was left untreated and labeled unmodified oil palm fruit fibre (UOPF), 50 g of the second part labeled 0.5M modified oil palm fruit fibre (0.5MOPF) and the third part labeled 1.0M modified oil palm fruit fibre (1.0MOPF). They were acid treated by dissolving in excess 0.5 M and 1.0 M mercaptoacetic acid (HSCH₂COOH)

solution, respectively. The suspensions were stirred for 30 mins and left to stand for 24 hrs at 36°C. After 24 hrs the mixtures labeled 0.5MOPF and 1.0MOPF were filtered off using whatman No 41-filter paper, the residue in each filter paper was then soaked in 1.0 M hydroxylamine (NH₂OH) for 1 hr after which they were filtered using whatman No 41-filter paper and rinsed with deionized water. The washed residue of 0.5MOPF and 1.0MOPF were air dried. The three working absorbents were stored in air tight containers for further use in absorption experiment.

2.2.3 Absorption procedure

A modified absorption procedure of that used by Abia and Asuquo [1] was used. Batch absorption experiment for Cu(II), Pb(II), Ni(II) and Sn(II) were carried out for each absorbent (UOPF, 0.5MOPF and 1.0MOPF) using an initial metal concentrations 50 mg/L. 50 mL of standard solution of each metal solution was transferred into various 250 mL Erlenmeyer flask corked and labeled. Then 0.5 g of each absorbent was weighed into the different labeled flasks and agitated in a mechanical shaker for the different contact times (10 mins, 20 mins, 30 mins, 40 mins, 50 mins and 60 mins). After each agitation time, the contents of each flask were then filtered using whatman no 41-filter paper and the filtrate of each metal solution was analyzed using Shimadzu model 6650 Atomic Absorption Spectrophotometer. Blank solutions were also prepared and analyzed to determine the actual initial metal ion concentration and any absorption that may have occurred by the glass wares.

3. RESULTS AND DISCUSSION

3.1 Absorption Capacity

The amount of metal ions removed was calculated by:

$$\% \text{ Removal} = (C_i - C_e / C_i)100 \quad (1)$$

where C_i is the initial concentration, C_e is the final concentration of metal ions.

Sorption capacity 'q' was calculated using the formula:

$$q = (C_i - C_e / m)V \quad (2)$$

where V is the volume of the solution; C_i is the initial metal ion concentration; C_e is the final

metal ion concentration; m is the dry weight of the bio sorbent. Fig. 1 represents the percentage removal versus contact time for Pb(II) ion onto UOPF, 0.5MOPF and 1.0MOPF. The graph shows that there was an increase for 10 mins for the three absorbents which slowed down at 20 mins and thereafter further increase in contact time had no significant change in the % Removal. Fig. 2 represent the percentage removal versus contact time for Cu(II) ion with the three absorbents (UOPF, 0.5MOPF and 1.0MOPF). From the graph the absorption of Cu(II) ion were rapid initially from 10 min, and later slowed down after which increase in time had no significant change in % removal due to exhaustion of absorption sites [28]. However, for absorption of Cu(II) onto 0.5MOPF, it slowed at 20 mins and leveled to 40 min and thereafter made a slight increase at 50 mins and 60 mins. Fig. 3 presents the % removal of Sn(II) with change in contact time onto the three absorbents, it could be seen that there was a rapid initial % removal of Sn(II) onto 1.0MOPF within 10 mins, which slowed down for the next 10 mins and then leveled out for Sn(II) absorption onto 0.5MOPF, the % removal from 10 to 20 mins was not rapid which became constant within 20 mins to 40 mins. Thereafter, there was an increase at 50 mins which declined at 60 mins. On Sn(II) absorption onto UOPF, the % removal was slow and reached a peak at 30 mins and thereafter declined at 40 mins, leveled out at 50 mins and later on had a slight increase at 60 mins. In figure 4 for Ni(II) absorption onto the three absorbents, there was an initial rapid removal of Ni(ii) in 1.0MOPF and 0.5MOPF within 10 mins. But for 1.0MOPF, it slowed down at 20 mins and thereafter became constant. And that of 0.5MOPF became constant after initial rapid removal with 10 mins. The % removal of Ni(II) using UOPF was not as rapid as was with 1.0MOPF and 0.5MOPF. There was a slow and steady removal for up to 20 mins which became constant thereafter for 30 mins to 60 mins. From the figures, the sequence of increase in percentage of each metal absorbent is 1.0MOPF > 0.5MOPF > UOPF. This may be attributed to the increase in the number of functional groups on the modified absorbents (0.5MOPF and 1.0MOPF) since chemical modification of an absorbent tends to increase the absorption capacity of the absorbent [29]. The modification of the absorbent using mercaptoacetic acid incorporates the Thiol (SH) group onto the matrix of the absorbent. Studies have shown that incorporation of the Thiol (SH) group enhances the binding capacity of the absorbent by

increasing the concentration and number of possible binding sites for metal ion sorption [1,25]. The initial faster rate of removal of each metal ion may be due to the availability of the uncovered surface area of the absorbents, since absorption kinetics depends on the surface area of the absorbents [30]. The flattening out of the curves after certain contact time shows that the absorbent surface was saturated and the rate of absorption then decreased and remained steady. Similar results have been reported [31-33]. This observation has been explained that increase in absorption and consequently the attainment of equilibrium may be due to limited mass transfer of the adsorbate molecules from the bulk liquid to the external surface of the absorbent [13] and the rate at which the adsorbate is transported from the exterior to the interior sites of the biosorbent particles [34]. This result can also be described as taking place in two steps: rapid absorption and slow absorption. Rapid absorption mainly occurs at the absorption sites on the external and/or in the porous channel of absorbent while slow absorption takes place on the intraparticle sites of the absorbent [35].

Figs. 5, 6, and 7 represent the % removal of the four metals against each of the three absorbents. In Fig. 5 for absorption onto UOPF, the % removal of Ni(II) and Pb(II) were rapid within 20 mins which became fairly constant thereafter up to 60 mins. For Sn(II), the % removal was slow and reached a peak at 30 mins, slightly declined at 40 mins and 50 mins and thereafter had a slight increase at 60 mins. % removal of Cu(II) was not as rapid as Ni(II) and Pb(II) but after 10 mins it became fairly constant. In Fig. 6 for the absorption onto 0.5MOPF, the % removal of Ni(II) was rapid within 10 mins after which change in contact time had no significant effect on the % removal. For Sn(II), the % removal was slow initially up to 20 mins. From 20 mins to 40 mins, it became fairly constant, slightly increases at 50 mins and then declined at 60 mins. For Cu(II) and Pb(II), there was a rapid increase within 10 mins which slowed down for the next 10 mins. For Cu(II), the removal slowed till 30 mins, then declined at 40 mins and increased slowly till 60 mins. However, for Pb(II), the removal became fairly constant after 10 mins. In Fig. 7 for absorption onto 1.0MOPF, the % removal of the four metals were rapid initially within 10 mins which slowed down for the next 10 mins after which further increase in contact time had no significant effect on the % removal. For Pb(II), the removal became fairly constant after 10 mins. However, in Figs. 5, 6, and 7, it could be

observed that Ni(II) was more removed by three absorbents. The fast-initial uptake may be due to the availability of uncovered surface area of the

absorbent [30]. The trend in the sorption capacity of the three absorbents for the metal ions is Ni > Pb > Cu > Sn.

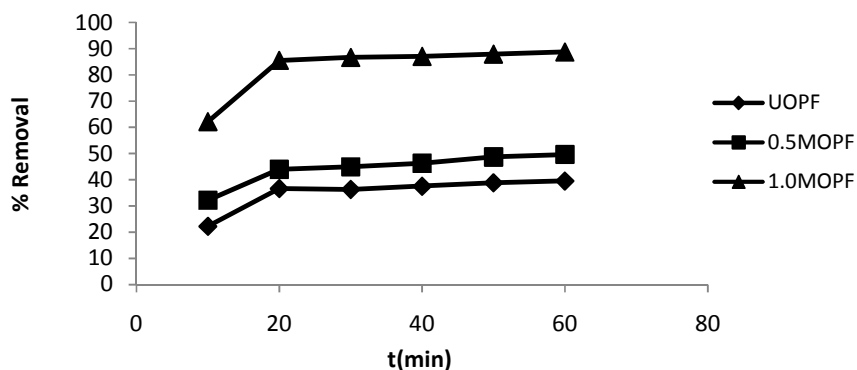


Fig. 1. % Removed versus contact time for Pb(II) ion absorption onto modified and unmodified oil palm fruit fibre

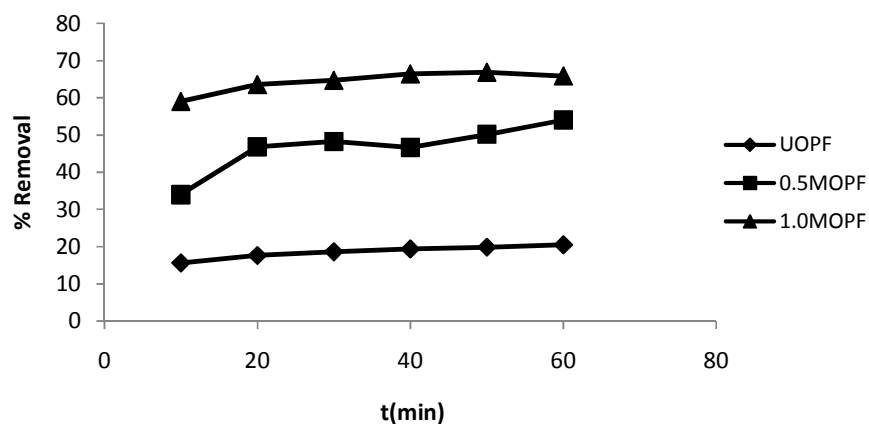


Fig. 2. % Removal versus contact time for Cu(II) ion absorption onto modified and unmodified oil palm fruit fibre

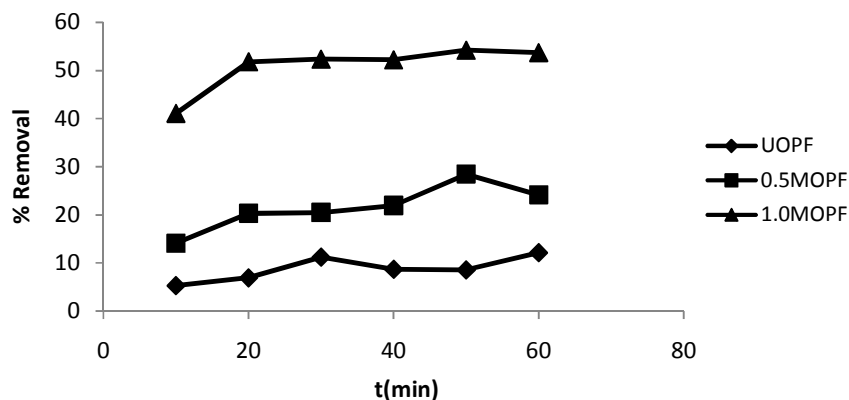


Fig. 3. % Removal versus contact time for Sn(II) ion absorption onto modified and unmodified oil palm fruit fibre

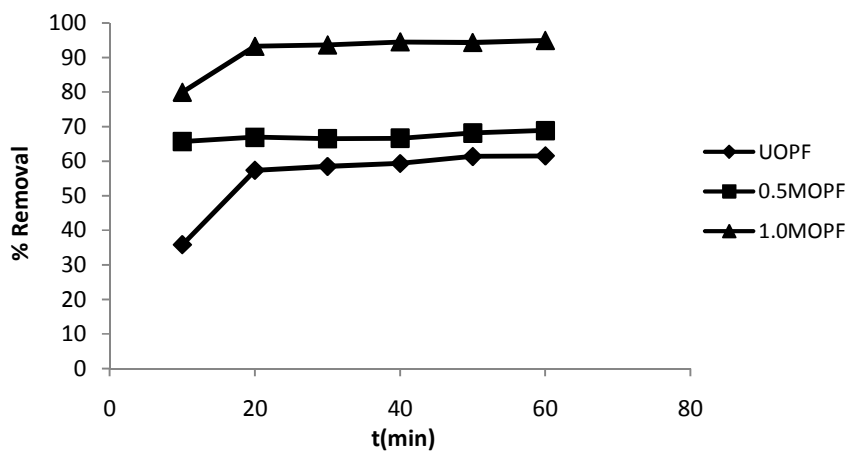


Fig. 4. % Removal versus contact time for Ni(II) ion absorption onto modified and unmodified oil palm fruit fibre

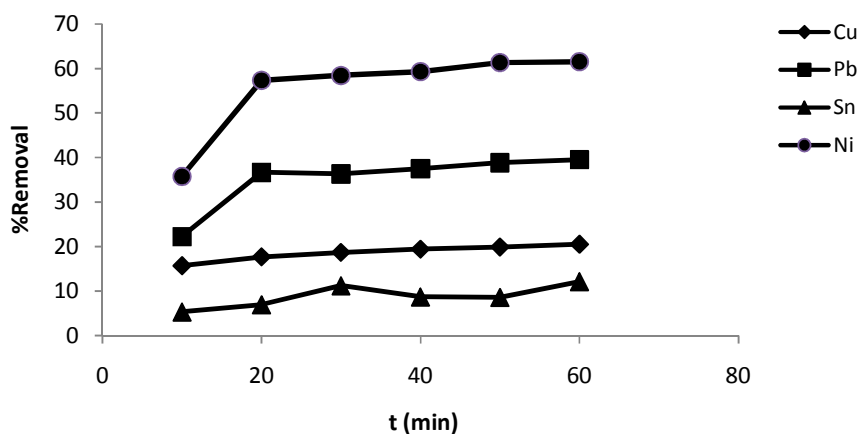


Fig. 5. % Removal of Cu(II), Pb(II), Ni(II) and Sn(II) versus contact time for UOPF

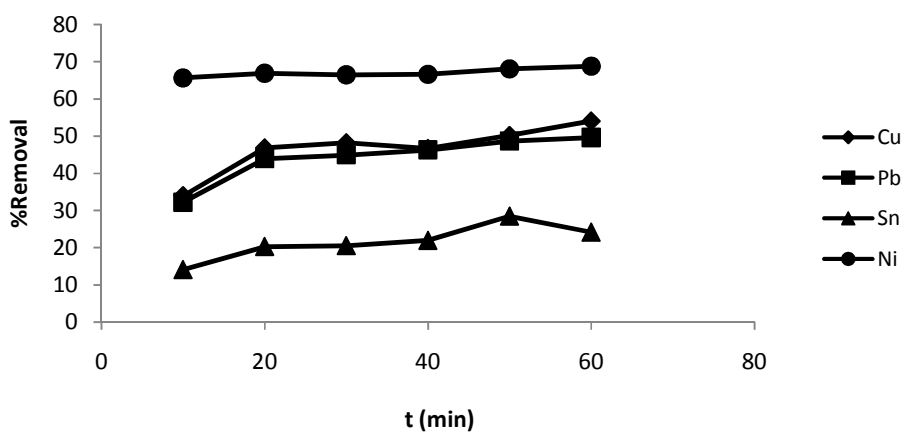


Fig. 6. % Removal of Cu(II), Pb(II), Ni(II) and Sn(II) versus contact time for 0.5MOPF

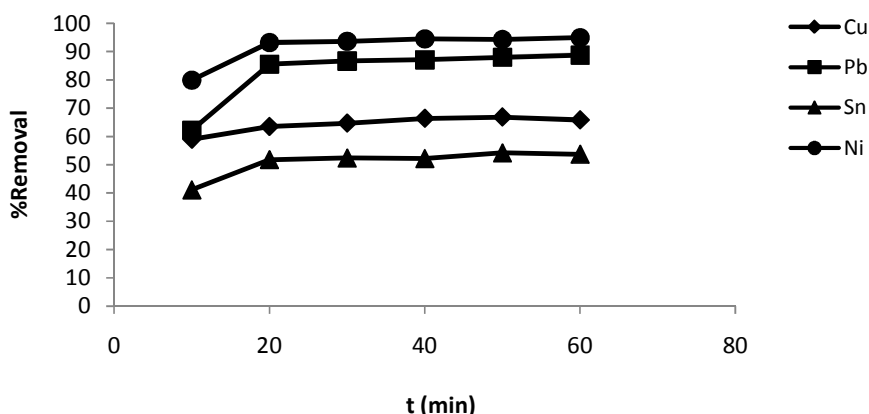


Fig. 7. % Removal of Cu(II), Pb(II), Ni(II) and Sn(II) versus contact time for 1.0MOPF

3.2 Absorption Kinetics

Absorption kinetics reveals the uptake rate of the solute, the residence time and it is an important aspect in evaluation of sorption as a unit operation [36,9]. A rapid kinetics will facilitate smaller reactors (lower retention time for effective metal uptake), whereas a slow rate of uptake will necessitate long column or series of columns to utilize maximum potential of the biosorbent [14]. The rate of sorption onto a sorbent surface depends upon a number of parameters such as structural properties of the sorbent, initial concentration of the sorbate and the interaction between the sorbate and the active sites of the sorbent [14].

The mechanism of sorption often involves chemical reaction between functional present on the absorbent surface and the metal ions or hydrolyzed species. This involves in most cases, formation of metal organic complexes or cation exchange reaction because of high cation exchange capacity of the absorbent [37]. Other possible mechanisms involve mass transport processes, bulk transport in the liquid phase, diffusion across the liquid film surrounding the absorbent particles and diffusion into micropores and macropores. The effect of external film diffusion on biosorption rate is assumed to be insignificant and ignored in many kinetic analyses, particularly when the bio sorbent is employed as a free suspension in a well agitated batch system. Therefore, the rate of absorption is usually measured by determining the change in concentration of the adsorbate in contact with the absorbent as a function of time [14]. The kinetics of Cu(II), Pb(II), Sn(II) and Ni(II) ions sorption onto modified and unmodified oil palm fruit fibre

was analyzed using diffusion-chemisorption kinetics and pseudo-second order kinetic models.

A diffusion chemisorption model as developed by Sutherland [38], was used to model the sorption experimental data. Sutherland's diffusion chemisorption model is an empirical equation and it is usually used to simulate sorption of heavy metals onto heterogeneous surfaces. The equation is given as:

$$t^{0.5}/q_t = (1/q_e) * t^{0.5} + 1/k_{DC} \quad (3)$$

where q_t is amount adsorbed at time t , q_e is the equilibrium amount adsorbed and k_{DC} is the rate constant for diffusion chemisorption model. According to Sutherland [38], the initial slope of the experimental kinetic curve is found empirically to be a function of the diffusion-chemisorption constants k_{DC} and equilibrium sorption capacity q_e . The following relationship is obtained by assuming a linear region as $t \rightarrow 0$. The initial rate is represented by the empirical relationship [39].

$$k_i = k_{DC}^2 / q_e \quad (4)$$

where k_i is the initial sorption rate (mg/g-min). A plot of $t^{0.5}/q_t$ versus $t^{0.5}$ for the diffusion chemisorption model is shown in Fig. 8 for UOPF, Fig. 9 for 0.5MOPF and Fig. 10 for 1.0MOPF. It could be seen that the plots gave very good straight lines for the metal ions. The values of k_{DC} and q_e were obtained from the intercepts and slopes of the respective plots and are shown in Table 1. It could be seen that the values of q_e were highest for Pb(II) onto 1.0MOPF (52.91 mg/g) and lowest for Cu(II) onto UOPF (10.83 mg/g).

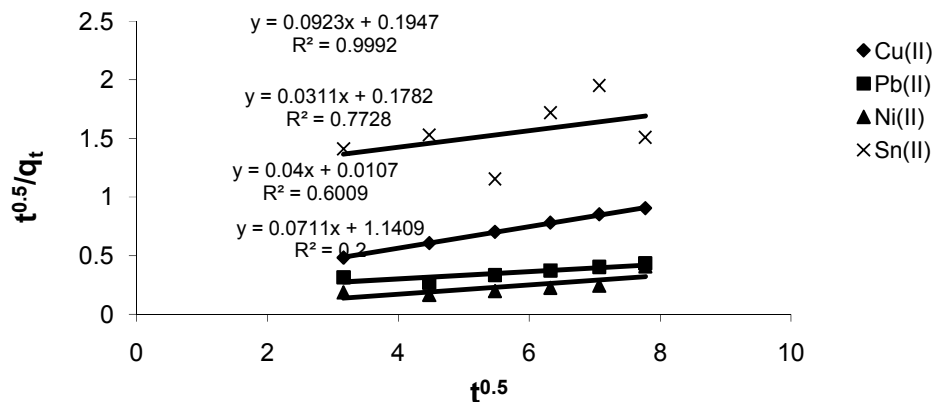


Fig. 8. Diffusion-chemisorption plot for Cu(II), Ni(II), Pb(II) and Sn(II) absorption onto UOPF

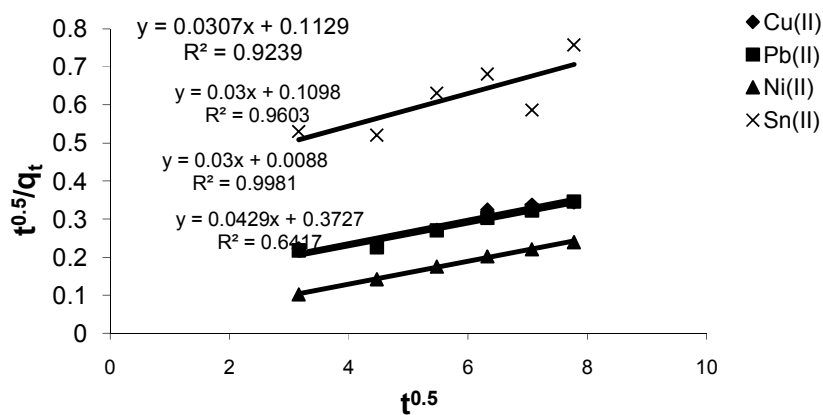


Fig. 9. Diffusion-chemisorption plot for Cu(II), Ni(II), Pb(II) and Sn(II) absorption onto 0.5MOPF

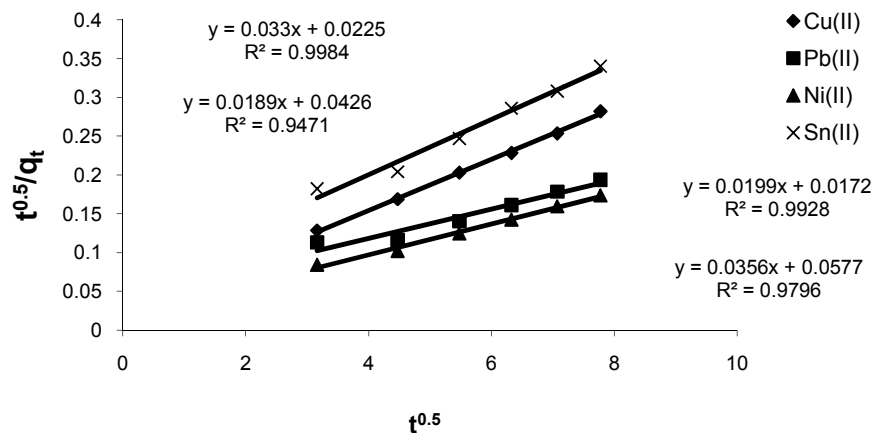


Fig. 10. Diffusion-Chemisorption plot for Cu(II), Ni(II), Pb(II) and Sn(II) absorption onto 1.0MOPF

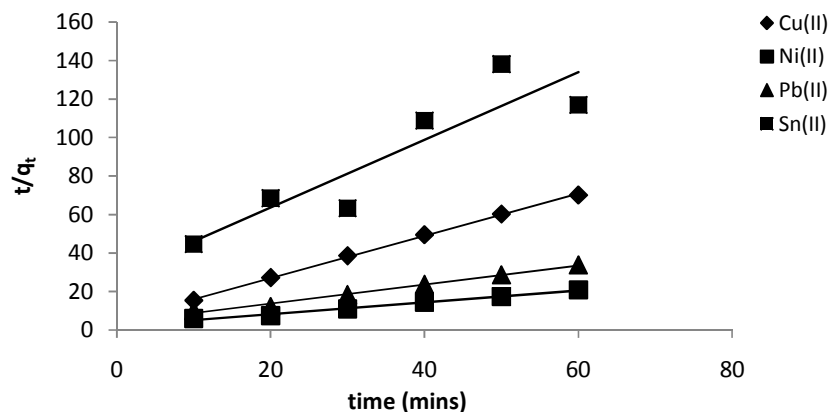


Fig. 11. Pseudo-Second order plot for Cu(II), Ni(II), Pb(II) and Sn(II) absorption onto OPF

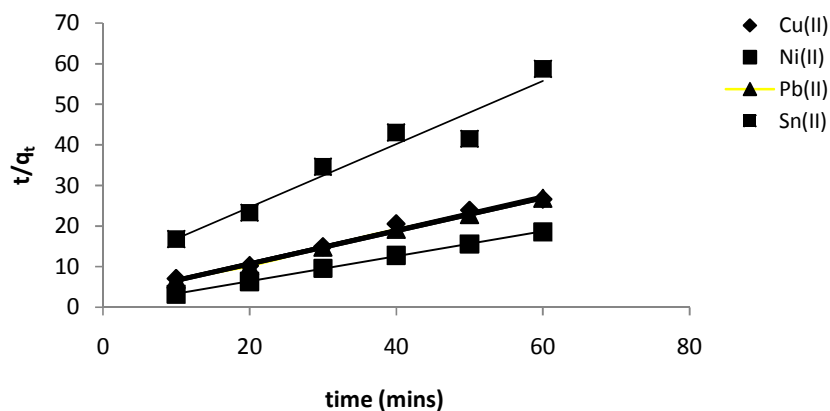


Fig. 12. Pseudo-Second order plot for Cu(II), Ni(II), Pb(II) and Sn(II) absorption onto 0.5MOPF

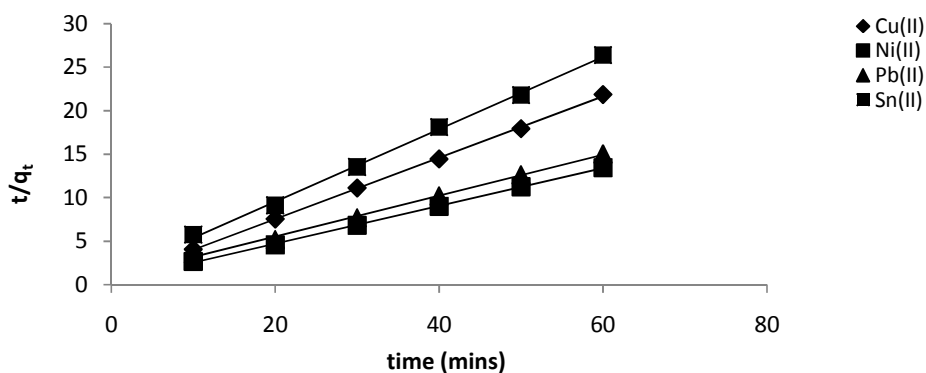


Fig. 13. Pseudo-Second order plot for Cu(II), Ni(II), Pb(II) and Sn(II) absorption onto 1.0MOPF

Table 1. Diffusion Chemisorption constants for Cu(II), Ni(II), Pb(II) and Sn(II) absorption onto modified and unmodified oil palm fruit fibre

| Absorbent | k_{DC} | | | | q_e | | | | k_i | | | |
|-----------|----------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| | Sn(II) | Cu(II) | Pb(II) | Ni(II) | Sn(II) | Cu(II) | Pb(II) | Ni(II) | Sn(II) | Cu(II) | Pb(II) | Ni(II) |
| UOPF | 0.877 | 5.136 | 5.612 | 93.46 | 14.07 | 10.83 | 32.15 | 25.00 | 0.055 | 2.436 | 0.980 | 349.4 |
| 0.5MOPF | 2.683 | 8.857 | 9.108 | 113.6 | 23.81 | 32.57 | 33.33 | 33.33 | 0.302 | 2.409 | 2.489 | 387.4 |
| 1.0MOPF | 17.33 | 44.44 | 23.47 | 58.14 | 28.09 | 30.30 | 52.91 | 50.25 | 10.69 | 65.19 | 10.42 | 67.27 |

Table 2. Pseudo-second order parameters for Cu(II), Ni(II), Pb(II) and Sn(II) absorption onto modified and unmodified oil palm fruit fibre

| Absorbent | K_{2ads} | | | | q_e | | | | h_0 | | | |
|-----------|------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| | Sn(II) | Cu(II) | Pb(II) | Ni(II) | Sn(II) | Cu(II) | Pb(II) | Ni(II) | Sn(II) | Cu(II) | Pb(II) | Ni(II) |
| UOPF | 0.109 | 0.240 | 0.066 | 0.045 | 0.568 | 0.911 | 2.018 | 3.265 | 0.035 | 0.199 | 0.268 | 0.484 |
| 0.5MOPF | 0.067 | 0.062 | 0.065 | 0.347 | 1.283 | 2.423 | 2.459 | 3.254 | 0.111 | 0.361 | 0.392 | 3.675 |
| 1.0MOPF | 0.143 | 0.257 | 0.065 | 0.141 | 2.403 | 2.835 | 4.266 | 4.589 | 0.828 | 2.065 | 1.186 | 2.964 |

Table 3. Values of R^2 for Cu(II), Ni(II), Pb(II) and Sn(II) absorption onto modified and unmodified oil palm fruit fibre

| Absorbents | Diffusion-Chemisorption model | | | | Pseudo-second order model | | | |
|------------|-------------------------------|--------|--------|--------|---------------------------|--------|--------|--------|
| | Sn(II) | Cu(II) | Pb(II) | Ni(II) | Sn(II) | Cu(II) | Pb(II) | Ni(II) |
| UOPF | 0.200 | 0.999 | 0.772 | 0.600 | 0.817 | 0.999 | 0.990 | 0.991 |
| 0.5MOPF | 0.641 | 0.923 | 0.960 | 0.998 | 0.941 | 0.988 | 0.998 | 0.999 |
| 1.0MOPF | 0.979 | 0.998 | 0.947 | 0.992 | 0.998 | 0.999 | 0.996 | 0.999 |

The experimental data was also analyzed using pseudo-second order kinetics model [40]. This equation is given as

$$t/q = 1/(K_{2ads} \cdot q_e^2) + t/q_e \quad (5)$$

where K_{2ads} is the constant of pseudo-second order kinetics ($\text{g.mg}^{-1}.\text{min}^{-1}$) and t is time in mins. The linear plots of t/q versus t using equation 5 for Pb(II), Cu(II), Sn(II) and Ni(II) absorption onto UOPF, 0.5MOPF and 1.0MOPF are shown in Figs. 11, 12, and 13 respectively. It could be seen that straight lines were obtained for the plots. The values of t/q were higher for UOPF followed by those for 0.5MOPF and then for 1.0MOPF. The pseudo-second order kinetic constant (K_{2ads}) and the equilibrium sorption capacity (q_e) values were calculated from the slopes and intercepts of the respective plots. The values obtained are shown in Table 2. The values of K_{2ads} were found to range from 0.045 for Ni(II) on UOPF to 0.347 for Ni(II) on 0.5MOPF but had no definite trend for the metal ions. The values of q_e generally increased with increase in modification and were higher for Ni(II), followed by Pb(II), followed by Cu(II) and then by Sn(II). The initial sorption rate h_0 was found to range from value of 0.035 to 3.675. The pseudo-second order model was developed on the assumption that chemisorption is the operative reaction mechanism [39].

In order to compare the validity of each model more efficiently, the coefficient of determination R^2 using Microsoft excel 2003 package was used. The R^2 values are shown in Table 3. Generally, the R^2 values were higher for the pseudo-second order model than the diffusion-chemisorption model. This means that the pseudo-second order model gave a better fit to the sorption process than the diffusion-chemisorption model. The good fit of pseudo-second order model to sorption systems have been reported [41,14,35,42]. Dizadji et al. [9] reported that initial absorption rate increased with increase in initial concentration and the rate constant decreased with increase in initial metal ion concentration.

4. CONCLUSION

This study demonstrated that oil palm fruit fibre can be used as absorbent for heavy metals absorption. Chemical treatment of the absorbent improved its absorption efficiency. 1.0MOPF showed the highest % removal for the heavy metals followed by 0.5MOPF and UOPF

respectively. The trend in the sorption capacity of the three absorbents for the metal ions was observed to be Ni > Pb > Cu > Sn. The kinetics of absorption of these heavy metals was found to be rapid, where equilibrium absorption could be attained within 20 mins. The sorption rates were found to follow pseudo-second order kinetics. The results of this study showed that oil palm fruit fibre can be efficiently used as a low-cost alternative for the removal of Cu(II), Pb(II), Ni(II) and Sn(II) from aqueous solutions.

CONSENT

It is not applicable.

ETHICAL APPROVAL

It is not applicable.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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