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Kinetic studies of copper ion adsorption on palm kernel fibre

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Abstract

Palm kernel fibre is a common agricultural waste in West Africa and its use as an adsorbent for the removal of copper ions from aqueous solution has been studied. Batch kinetics studies were carried out based on the assumption of the pseudo-second-order kinetic model, which was developed to predict the rate constant of adsorption, the equilibrium adsorption capacity and initial adsorption rate with the effect of initial copper concentration and reaction temperature. A comparison was made of the linear least-squares method and a trial-and-error non-linear method of the pseudo-second-order kinetic model for the adsorption of copper onto palm kernel fibre.

Keywords: Copper; Sorption; Palm kernel fibre; Pseudo-second-order; Non-linear

1. Introduction

The presence of copper in wastewater of several industries has contributed in no small way to pollution of ground and surface water. Treatment of wastewater containing copper could be achieved with settling as settleable metal hydroxides, activated carbon adsorption, ion exchange, reverse osmosis, electrochemical treatment, evaporation and biological methods. But these methods are not economical and do not exhibit high efficiency, especially at low concentrations therefore, the search for other cost effective and efficient methods for copper removal from aqueous solution is required.

The adsorptive removal of copper ions from aqueous solution using agricultural waste products has gained popularity in recent times. Some agricultural waste products that have been successfully applied as adsorbent for copper ions from aqueous solution [1] include seed of *Capsicum annum* [2], carrot residues [3], banana pith [4], soybean hulls, cottonseed hulls, rice straw, sugarcane bagasse [5], apple wastes [6], groundnut shells [7], grape stalks [8], wheat shell [9], tea waste [10], sunflower stalk [11] and tree fern [12]. Due to their natural abundance in the environment and their affinity for heavy metals, agricultural wastes remain a cheap and readily available source of materials that can be applied for heavy metal removal from aqueous solution. Agricultural wastes may therefore serve as an excellent alternative to activated carbon, which is quite unaffordable especially in developing countries.

Palm kernel fibre is an agricultural waste product in the production of palm oil from the palm kernel fruit. In West Africa, a number of large and medium scale palm oil producing industries exist, producing palm oil for international and local consumption and also for raw materials for other industries. Due to the high demand for palm oil, large amounts of wastes are generated annually. Over 15,600 tonnes of shell and fibre are generated annually and only about 5% of the wastes are sometimes burnt to generate heat for the boiler [13]. Agricultural waste products are complex materials containing lignin and cellulose as its major constituents. Chemical adsorption can occur by the polar functional groups of lignin, which include alcohols, aldehydes, ketones, acids phenolic hydroxides and ethers which act as chemical bonding agents [14]. Ho and Ofomaja [15,16,17,18] have shown that palm kernel fibre can be effectively applied as adsorbent for the removal of lead ion and anionic dye from aqueous solution.

This study is aimed at investigating the possibility of using palm kernel fibre as an efficient alternative adsorbent material for the adsorptive removal of copper ions from aqueous solution. The dynamic behaviour of copper ion adsorption onto palm

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kernel fibre was studied with the effect of initial copper concentration and temperature. A comparison was made of the linear least-squares method and non-linear method of the widely used pseudo-second-order kinetic model to the experimental adsorption of copper onto palm kernel fibre. A trial-and-error procedure was used for the non-linear method using the *solver* add-in with Microsoft's spreadsheet, Microsoft Excel.

2. Materials and methods

Palm kernel fibre used in this study was obtained from the Nigerian Institute for Oil Palm Research (N.I.F.O.R) Benin City, Nigeria. The palm kernel fibre was allowed to dry with the residual oil after processing for about 2 months. The raw fibre was dried in an oven at 80 °C for 6 h, ground and screened through a set of sieves to obtain particles of size 50–60 μm. The sieved fibre was steeped in 0.02 mol/dm^3 HCl overnight. The acid solution was filtered off and the fibre washed with distilled water until the pH of the wash becomes neutral. The fibre was dried at 80 °C for 24 h and stored in an airtight container. The stock solution of copper sulphate ($CuSO_4{\cdot}5H_2O$) was prepared by dissolving an accurately weighed amount of the salt in deionized water. The experimental solutions were prepared by diluting the stock solution with distilled water when necessary.

2.1. Characteristics of palm kernel fibre

The proximate composition of the palm kernel fibre was determined using the Association of Official Analytical Chemist [19]. The IR spectra of the copra meal sample were recorded using KBr wafers in conjunction with a Perkin-Elmer infrared spectrophotometer. KBr wafers were prepared by mixing a given sample with KBr crystals, the resulting mixture being ground to a fine powder and heated for 1 h at 373 K. Finally, the mixture was pressed into a KBr wafer under vacuum conditions and used as such for IR studies.

2.2. Effect of initial concentration

Kinetic experiments were carried out by agitating 100 ml of copper ion solution of concentration ranging from 50 to 250 mg/dm^3 with 1.0 g of palm kernel fibre in a 250 ml beaker at 299 K at an optimum pH of 5.01 and at a constant agitation speed of 200 rpm for 60 min. Samples (3.0 ml) were pipetted out at different time intervals, centrifuged and the concentration of copper analyzed using atomic absorption spectrophotometer Solar Series, Model 969.

2.3. Effect of temperature

A range of reaction temperatures (299, 309, 319, 329 and 339 K) were used and the flasks were agitated for 60 min. All contact investigations were performed in a 1 dm^3 flask. A 1.0 g sample of palm kernel fibre was added to 100 ml volume of copper ion solution set at pH 5.01 and agitated at 200 rpm for all the experiments. The experiments were carried out at initial copper ion concentration 250 mg/dm^3 for all the studies.

3. Results and discussion

3.1. Characteristics of palm kernel fibre

The percentage proximate composition of palm kernel fibre on dry weight basis was: carbohydrate 38.2%, lipid 9.00%, fibre 19.9%, ash 6.30% protein 14.8%, moisture 9.78%, Ca 0.63%, P 0.45% and Mg 0.29%. I.R. measurement for the palm kernel fibre showed the presence of the following groups: C=O (1680.1 cm^{-1}), COOH (3300–2500, 1110.8 cm^{-1}), –OH (3426.2 cm^{-1}), C=C (1675 cm^{-1}), C–N (1030–1230 cm^{-1}), N–H (1548.8 cm^{-1}) and NH_2 (3400–3500cm^{-1}).

3.2. Kinetic study

The pseudo-second-order kinetics may be expressed as [20,21]:

$$\frac{dq_t}{dt} = k(q_e - q_t)^2 \tag{1}$$

where k is the rate constant of adsorption (dm^3/mg min), q_e is the amount of copper ions adsorbed at equilibrium (mg/g) and q_t is the amount of copper ions adsorbed at time t (mg/g). Separating the variables in Eq. (1) gives:

$$\frac{dq_t}{(q_e - q_t)^2} = k\,dt \tag{2}$$

Integrating this for the boundary conditions $t=0$ to $t=t$ and $q_t=0$ to $q_t=q_t$ gives:

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + kt \tag{3}$$

which is the integrated rate law for a pseudo-second-order reaction. Eq. (3) can be rearranged to obtain:

$$q_t = \frac{t}{(1/kq_e^2) + (t/q_e)} \tag{4}$$

which has a linear form of:

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{1}{q_e}t \tag{5}$$

If the initial adsorption rate, as $h=q_t/t$ when t approach to 0, h, (mg/g min), is:

$$h = kq_e^2 \tag{6}$$

then Eqs. (4) and (5) become:

$$q_t = \frac{t}{(1/h) + (t/q_e)} \tag{7}$$

and

$$\frac{t}{q_t} = \frac{1}{h} + \frac{1}{q_e}t \tag{8}$$

The pseudo-second-order rate constants can be determined experimentally by plotting t/q_t against t. Although there are many factors which influence the adsorption capacity, including the initial adsorbate concentration, the reaction temperature, the solution pH value, the adsorbent particle size and dose and the nature of the adsorbate, a kinetic model is concerned only with the effect of observable parameters on the overall rate [22].

In this study, the coefficient of determination, r^2, was used to test the best-fitting of the kinetic model to the experimental data:

$$r^2 = \frac{\sum (q_\mathrm{m} - \bar{q}_t)^2}{\sum (q_\mathrm{m} - \bar{q}_t)^2 + \sum (q_\mathrm{m} - q_t)^2}, \quad (9)$$

where q_m is amount of copper ion on the surface of the palm kernel fibre at any time, t, (mg/g) obtained from the pseudo-second-order kinetic model; q_t is the amount of copper ion on the surface of the palm kernel fibre at any time, t, (mg/g) obtained from experiment; and $\bar{q}_t$ is the average of q_t, (mg/g).

3.3. Linear regression method

Linear regressions are frequently used to determine the best-fitting kinetic models and the method of least-squares is used for finding parameters of the kinetic models. However, the pseudo-second-order kinetic model can be linearized as four different types (Table 1) and a simple linear regression will result in different parameter estimates [22–24]. The most popular linear form used is type 1. Figs. 1–4 show experimental data with linear equations of the four pseudo-second-order kinetic models obtained by using the linear method for the adsorption of copper onto palm kernel fibre at various initial copper concentrations. Similar patterns were obtained for the effect of temperature. Values of the pseudo-second-order kinetic model constant, k, the amount of copper ion adsorbed at equilibrium, q_e and the initial adsorption rate, h, are listed in Tables 2 and 3 for the adsorption of copper ion onto palm kernel fibre for different adsorption conditions. These values of the extremely high coefficient of determinations, r^2, obtained from all types, indicate that there is strong positive evidence that the copper adsorption onto palm kernel fibre follows the pseudo-second-order kinetic expression.

Table 1
Pseudo-second-order kinetic model linear forms

Type	Linear form	Plot	Parameters
1	$\frac{t}{q_t} = \frac{1}{kq_\mathrm{e}^2} + \frac{1}{q_\mathrm{e}}t$	t/q_t vs. t	q_e = 1/slope k = slope^2/intercept h = 1/intercept
2	$\frac{1}{q_t} = \left(\frac{1}{kq_\mathrm{e}^2}\right)\frac{1}{t} + \frac{1}{q_\mathrm{e}}$	$1/q_t$ vs. $1/t$	q_e = 1/intercept k = intercept^2/slope h = 1/slope
3	$q_t = q_\mathrm{e} - \left(\frac{1}{kq_\mathrm{e}}\right)\frac{q_t}{t}$	q_t vs. q_t/t	q_e = intercept k = −1/(intercept × slope) h = −intercept/slope
4	$\frac{q_t}{t} = kq_\mathrm{e}^2 - kq_\mathrm{e}q_t$	q_t/t vs. q_t	q_e = −intercept/slope k = slope^2/intercept h = intercept

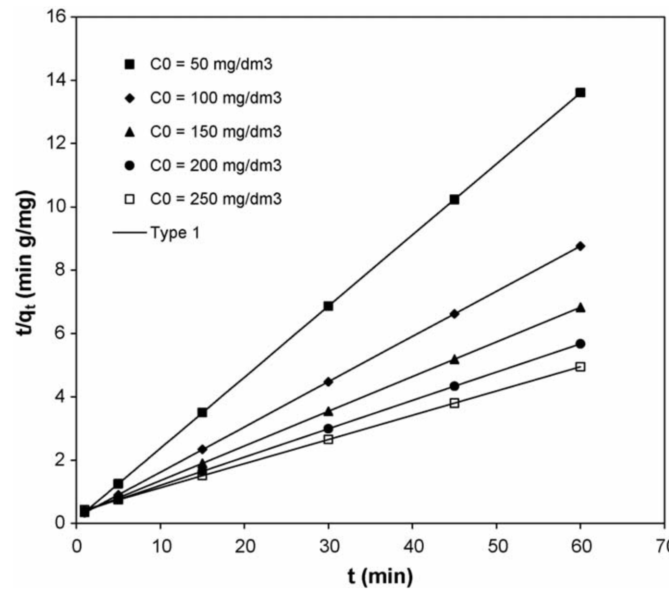

Fig. 1. Type-1 pseudo-second-order linear equations obtained by using the linear method for the adsorption of copper onto palm kernel fibre at various initial copper concentrations.

3.4. Non-linear method

It has been reported that transformations of non-linear pseudo-second-order kinetic models to linear forms implicitly alter their error structure and may also violate the error variance and normality assumptions of standard least-squares method [22]. Further analysis was performed by using the non-linear method, a trial-and-error procedure, which is applicable to computer operation, was developed to determine the pseudo-second-order rate parameters by optimization routine to maximize the coefficient of determination between experimental data

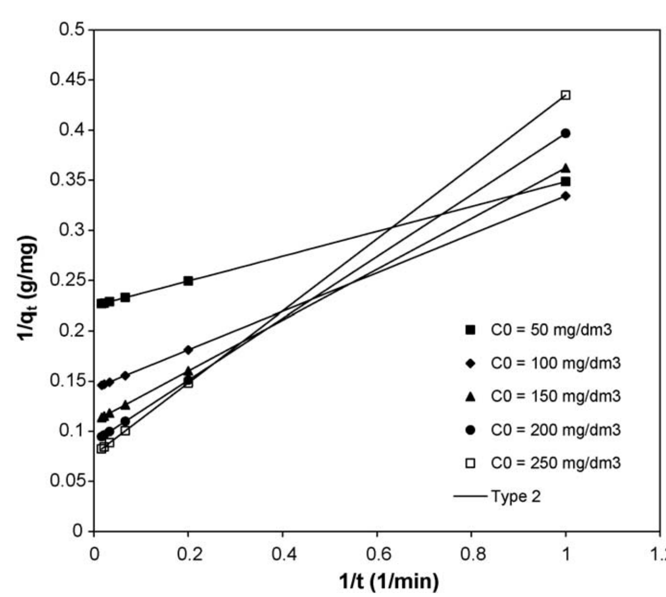

Fig. 2. Type-2 pseudo-second-order linear equations obtained by using the linear method for the adsorption of copper onto palm kernel fibre at various initial copper concentrations.

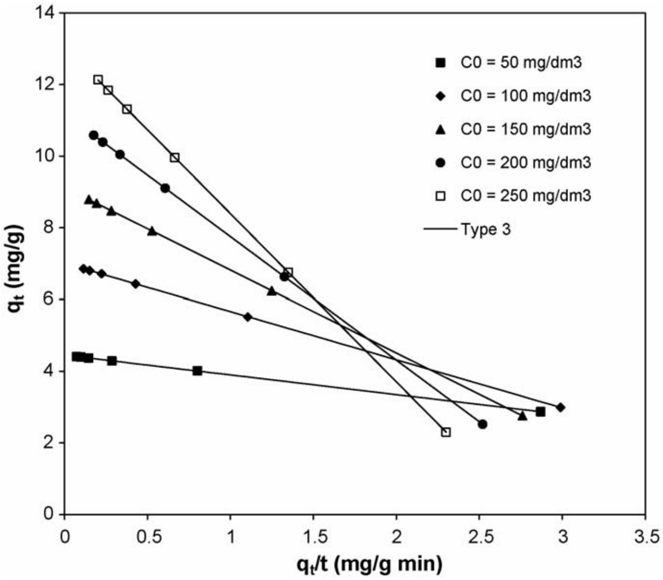

Fig. 3. Type-3 pseudo-second-order linear equations obtained by using the linear method for the adsorption of copper onto palm kernel fibre at various initial copper concentrations.

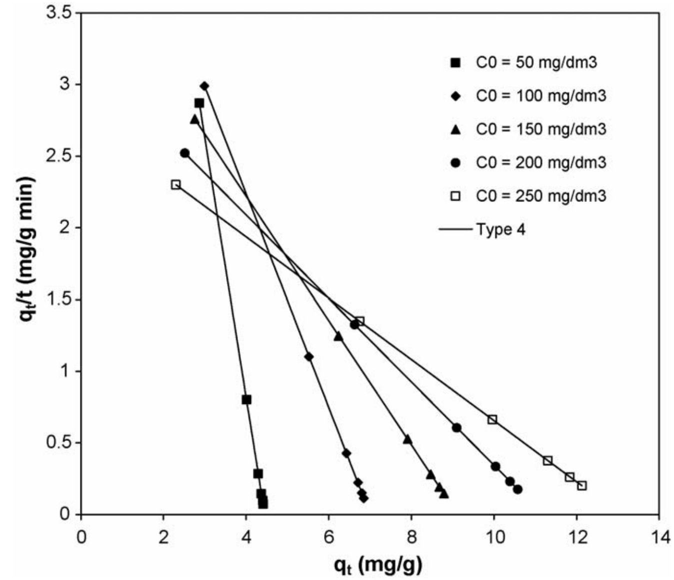

Fig. 4. Type-4 pseudo-second-order linear equations obtained by using the linear method for the adsorption of copper onto palm kernel fibre at various initial copper concentrations.

and pseudo-second-order model using the *solver* add-in with Microsoft's spreadsheet, Microsoft Excel [22]. The abilities of four linear pseudo-second-order equations to model the kinetic adsorption data were examined. In all cases, the results from the four pseudo-second-order linear equations were the same. Tables 4 and 5 show the pseudo-second-order rate parameters obtained by using the non-linear method for effect of initial copper concentration and temperature, respectively. By using the non-linear method, there is no problem with transformations of the non-linear pseudo-second-order rate equation to linear forms and also they had the same error structures [24]. All of the parameters obtained from both linear and non-linear method were nearly the same. It may be as a result of the perfect fitting of the linear models. When experimental data fitted the models well there is no difference between using linear or non-linear method.

3.5. *Effect of initial copper ion concentration*

The effect of initial copper ion concentration on the rate of copper ions uptake onto palm kernel fibre was studied using batch agitation in 250 ml beakers containing copper ion solutions of initial concentrations ranging from 50 to 250 mg/dm^3, agitated at 200 rpm and at 299 K. Fig. 5 shows the plot of experimental data points for the adsorption of copper ions onto palm kernel fibre as a function of time. It is clear that the amount of copper ions adsorbed at equilibrium increases with an increase in initial copper ion concentration. The removal of copper ions

Table 2
Pseudo-second-order kinetic parameters obtained by using the linear methods at different initial copper concentrations

Type	Parameters	50 (mg/dm^3)	100 (mg/dm^3)	150 (mg/dm^3)	200 (mg/dm^3)	250 (mg/dm^3)
1	q_e (mg/g)	4.452	7.003	9.127	11.19	13.08
	k (g/mg min)	0.4056	0.1068	0.04737	0.02602	0.01631
	h (mg/g min)	8.037	5.237	3.946	3.256	2.789
	r^2	1.000	1.000	1.000	1.000	1.000
2	q_e (mg/g)	4.451	7.005	9.123	11.19	13.07
	k (g/mg min)	0.4079	0.1063	0.04754	0.02599	0.01633
	h (mg/g min)	8.081	5.216	3.957	3.253	2.791
	r^2	1.000	1.000	1.000	1.000	1.000
3	q_e (mg/g)	4.451	7.005	9.124	11.19	13.07
	k (g/mg min)	0.4079	0.1063	0.04751	0.02599	0.01633
	h (mg/g min)	8.081	5.216	3.956	3.253	2.791
	r^2	1.000	1.000	1.000	1.000	1.000
4	q_e (mg/g)	4.451	7.005	9.124	11.19	13.07
	k (g/mg min)	0.4079	0.1063	0.04751	0.02599	0.01633
	h (mg/g min)	8.081	5.216	3.956	3.253	2.791
	r^2	1.000	1.000	1.000	1.000	1.000

Table 3
Pseudo-second-order kinetic parameters obtained by using the linear methods at different temperatures

Type	Parameters	299 (K)	309 (K)	319 (K)	329 (K)	339 (K)
1	q_e (mg/g)	13.08	14.75	16.42	18.12	20.12
	k (g/mg min)	0.01631	0.02284	0.03066	0.03920	0.05204
	h (mg/g min)	2.789	4.971	8.266	12.87	21.07
	r^2	1.000	1.000	1.000	1.000	1.000
2	q_e (mg/g)	13.07	14.75	16.42	18.11	20.12
	k (g/mg min)	0.01633	0.02286	0.03057	0.03928	0.05212
	h (mg/g min)	2.791	4.974	8.247	12.89	21.10
	r^2	1.000	1.000	1.000	1.000	1.000
3	q_e (mg/g)	13.07	14.75	16.42	18.11	20.12
	k (g/mg min)	0.01633	0.02286	0.03058	0.03927	0.05212
	h (mg/g min)	2.791	4.974	8.249	12.89	21.10
	r^2	1.000	1.000	1.000	1.000	1.000
4	q_e (mg/g)	13.07	14.75	16.42	18.11	20.12
	k (g/mg min)	0.01633	0.02286	0.03058	0.03927	0.05212
	h (mg/g min)	2.791	4.974	8.249	12.89	21.10
	r^2	1.000	1.000	1.000	1.000	1.000

Table 4
Pseudo-second-order rate parameters obtained using the non-linear methods at different initial copper concentrations

Parameters	C_0 (mg/dm^3)				
	50	100	150	200	250
q_e (mg/g)	4.451	7.005	9.125	11.19	13.07
k (g/mg min)	0.4079	0.1063	0.04746	0.02601	0.01633
h (mg/g min)	8.080	5.215	3.952	3.255	2.791
r^2	1.000	1.000	1.000	1.000	1.000

increased from 4.451 to 13.07 mg/g when the initial copper ion concentration is increased from 50 to 250 mg/dm^3 at 299 K and pH 5.01. Tables 2 and 3 show a good compliance with the pseudo-second-order model and the coefficient of determinations for the linear and the non-linear methods were found to be 1.000 for all the systems (Tables 2–5). The q_e increased with increasing initial copper ion concentration, while the k and the h decreased with increasing initial copper ion concentration. For a chemical reaction controlled adsorption as described by the pseudo-second-order rate model, the pseudo-second-order rate constants will be independent of particle diameter and flow rate but will be dependent on concentration of ions in solution [21]. The corresponding linear plots of the values of q_e, h and k against initial copper ion concentration, C_0, were therefore regressed to obtain expressions these values in terms of the concentration parameters, respectively with extremely high coefficient of determinations ($r^2 = 1.000$) as shown:

$$q_e = 0.3245C_0^{0.6680} \tag{10}$$

$$k = 1.027 \times 10^3 C_0^{-1.997} \tag{11}$$

$$h = 1.082 \times 10^2 C_0^{-0.6612} \tag{12}$$

Table 5
Pseudo-second-order rate parameters obtained using the non-linear methods at different temperatures

Parameters	T (K)				
	299	309	319	329	339
q_e (mg/g)	13.07	14.75	16.42	18.11	20.12
k (g/mg min)	0.01633	0.02286	0.03059	0.03926	0.05213
h (mg/g min)	2.791	4.974	8.253	12.88	21.10
r^2	1.000	1.000	1.000	1.000	1.000

3.6. Effect of reaction temperature

The temperature dependence of copper ions adsorption onto palm kernel fibre was studied with a constant initial copper ion concentration of 250 mg/dm^3 at 200 rpm and palm kernel fibre dose 10 g/dm^3 at various temperature of adsorption reaction. Fig. 6 shows the effect of contact time on the adsorption of

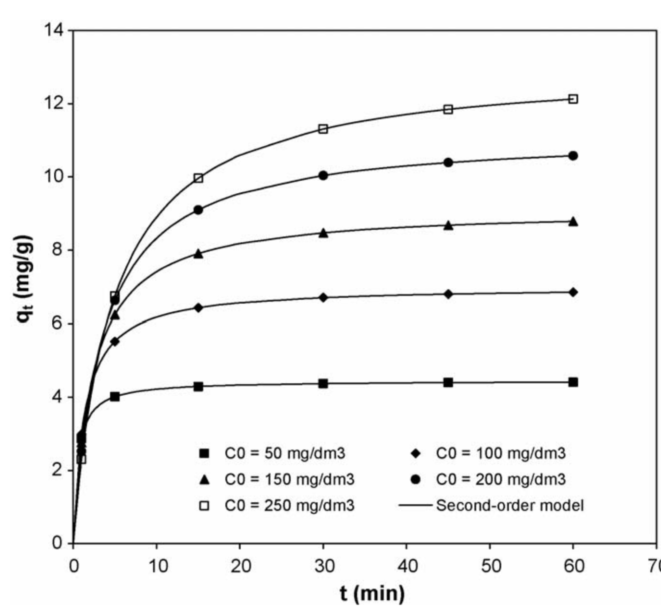

Fig. 5. Effect of initial copper concentration on uptake of copper ions onto palm kernel fibre.

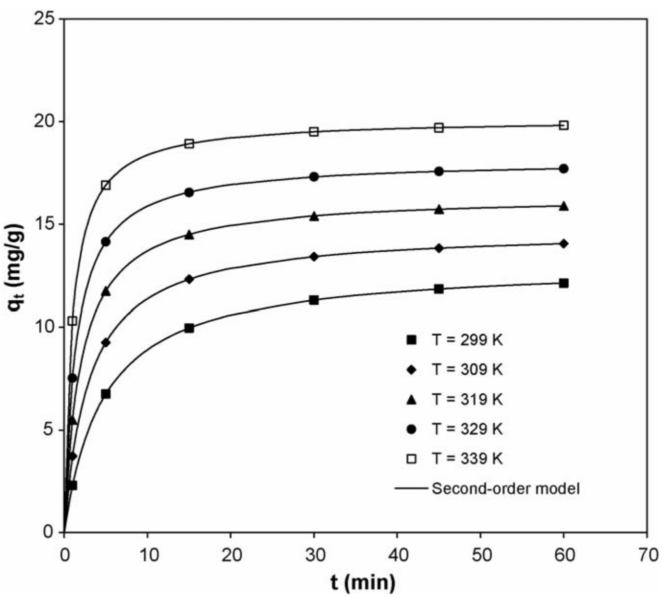

Fig. 6. Effect of temperature on adsorption capacity.

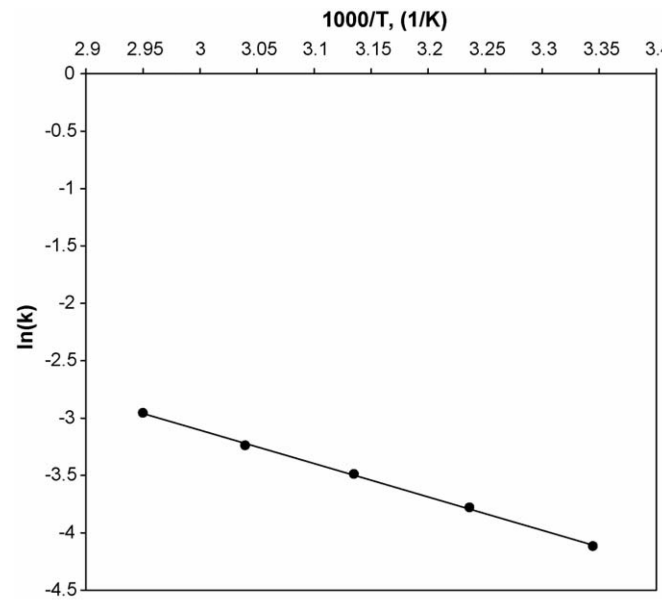

Fig. 7. Linear relationship between pseudo-second-order rate constant, k and reciprocal of temperature.

copper ions from aqueous solution at different reaction temperatures. The results show that with increase in reaction temperature from 299 to 339 K, the adsorption capacity of copper ions after 60 min of contact increased. The fact that the adsorption of copper ions is favored by temperature indicates that the mobility of the copper ions increases with a rise in the temperature. It can also be said that reaction of copper ions and surface functional groups is enhanced by increased temperature of reaction. This trend suggests that a chemisorption reaction or an activated adsorption between copper ions and functional groups on palm kernel fibre surface involving valences forces through sharing or exchange of electrons between palm kernel fibre and copper ions occurs [20]. Increase in temperature will also lead to increase in the amount adsorbed at equilibrium for a chemisorption reaction.

It has been reported that non-linear method could be a better way to obtain the kinetic parameters [24]. Table 5 shows the adsorption rate constant, k, initial adsorption rate, h and equilibrium adsorption capacity, q_e, as a function of solution temperature. Thus, on increasing the temperature from 299 to 339 K, q_e and h are increased from 13.07 to 20.12 mg/g and from 2.791 to 21.10 mg/g min, respectively. The logarithmic plots of h and q_e versus temperature were made and the plots where found to give straight lines whose linear regression, r^2 are given as 0.9990 and 0.9994. Mathematical expressions were therefore drawn relating the initial adsorption rate, h and the equilibrium capacity, q_e and reaction temperature as follows:

$$h = 1.308 \times 10^{-39} T^{15.93} \qquad (13)$$

and

$$q_e = 9.435 \times 10^{-8} T^{3.403} \qquad (14)$$

The values of rate constant, k, were found to increase from 0.01633 to 0.05213 g/mg min, for an increase in the solution temperature of 299 to 339 K. There is a linear relationship between the k and temperature with coefficient of determination of 0.9992 (Fig. 7). The adsorption rate constant is usually expressed as a function of solution temperature by the relationship:

$$k = k_0 \exp\left(\frac{-E}{RT}\right) \qquad (15)$$

where k is the rate constant of adsorption (g/mg min), k_0 the temperature independent factor (g/mg min), E the activation energy of adsorption (kJ/mol), R the gas constant (8.314 J/mol K) and T the solution temperature (K).

Therefore, the relationship between k and T can be represented in an Arrhenius form as:

$$k = 2.723 \times 10^{2} \exp\left(\frac{-24.14}{8.314T}\right) \qquad (16)$$

From this equation, the rate constant for adsorption, k_0, is 2.723×10^2 g/mg min and the activation energy for adsorption, E, is 24.14 kJ/mol, outside the range of values of 8–22 kJ/mol for diffusion-controlled processes [25]. The rate-controlling step in the process can be characterized in part by its activation energy. Therefore, since the adsorption increased with temperature, which implies the uptake is endothermic in nature and activation energy is higher than 22 kJ/mol, then one can say that chemisorption process significant in the adsorption of copper ions onto palm kernel fibre.

4. Conclusions

Palm kernel fibre has been shown to have a fairly high capacity for the removal of copper ions from solution. It was also revealed that the copper ions/palm kernel fibre interaction is endothermic with an activation energy higher than 22 kJ/mol, indicating that adsorption is chemical in nature. The adsorption equilibrium capacity, the adsorption rate constant and the initial adsorption rate are function of the initial copper concentration and the reaction temperature. Both linear and non-linear method could be a way to obtain the kinetic parameters when fitting experimental data and kinetic model are in a high correlation.

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