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Coal ash conversion into effective adsorbents for removal of heavy metals and dyes from wastewater

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Abstract

Fly ash was modified by hydrothermal treatment using NaOH solutions under various conditions for zeolite synthesis. The XRD patterns are presented. The results indicated that the samples obtained after treatment are much different. The XRD profiles revealed a number of new reflexes, suggesting a phase transformation probably occurred. Both heat treatment and chemical treatment increased the surface area and pore volume. It was found that zeolite P would be formed at the conditions of higher NaOH concentration and temperature. The treated fly ash was tested for adsorption of heavy metal ions and dyes in aqueous solution. It was shown that fly ash and the modified forms could effectively absorb heavy metals and methylene blue but not effectively adsorb rhodamine B. Modifying fly ash with NaOH solution would significantly enhance the adsorption capacity depending on the treatment temperature, time, and base concentration. The adsorption capacity of methylene blue would increases with pH of the dye solution and the sorption capacity of FA-NaOH could reach 5×10^{-5} mol/g. The adsorption isotherm could be described by the Langmuir and Freundlich isotherm equations. Removal of copper and nickel ions could also be achieved on those treated fly ash. The removal efficiency for copper and nickel ions could be from 30% to 90% depending on the initial concentrations. The increase in adsorption temperature will enhance the adsorption efficiency for both heavy metals. The pseudo second-order kinetics would be better for fitting the dynamic adsorption of Cu and Ni ions.

Keywords: Fly ash; Zeolite; Basic dyes; Heavy metals; Adsorption; Adsorption isotherm; Kinetics

1. Introduction

Industrial wastewater presents a challenge to conventional physico-chemical and biological treatment methods. Considering both volume discharged and effluent composition, the wastewater generated by the textile industry is rated as the most polluting among all industrial sectors. Heavy metals are also among the most important pollutants in source and treated water, and are becoming a severe public health problem due to the toxicity of some heavy metals. During the last few years, new and/or tighter regulations coupled with increased enforcement concerning wastewater discharges have been established in many countries. This challenge has prompted intensive research in new advanced treatment technologies, some of which currently making their way to full-scale installations. Among these processes, liquid-phase adsorption has been shown to be highly efficient for the removal of dyes and other organic matters as well as heavy metals from process or waste effluent.

At present, there is growing interest in using low cost materials for adsorption of dyes and heavy metals. In the past years, several investigations have been reported using industrial or agricultural wastes as adsorbents and the data show that those wastes exhibit more or less adsorption capacity [1–8].

Fly ash is a by-product producing during the combustion of coal in the electricity generation process. Disposal of fly ash has become an increasing economic and environmental burden. In Australia, fly ash production is around 8 Mt per year while the annual incorporation into cement and concrete accounts for only about 1 Mt. In the past several years, efforts are being made to explore other applications such as transformation into zeolite, potassium fertiliser and flue gas absorbent [9]. The production of zeolites using coal fly ash as a resource constitutes one important issue of waste management and many techniques have been investigated. The most common method involves a hydrothermal

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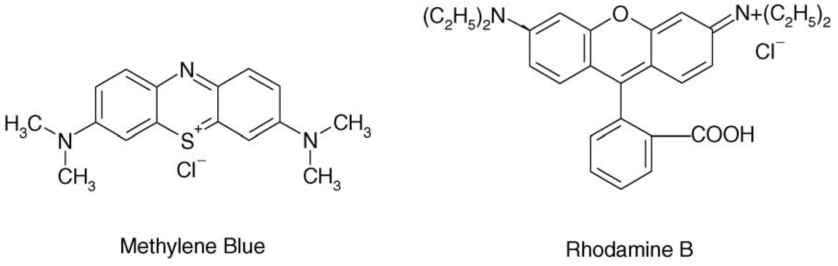

Methylene Blue

Rhodamine B

Fig. 1. Chemical structure of dyes.

process, whereby the fly ash is mixed with an alkali solution at different conditions of temperature, pressure and reaction time. Such a treatment process can lead to the formation of various types of zeolite including types P, A and X. The type of zeolite formed is a function of the temperature, pressure, concentration, aging period, pH, Al, Si and cation sources used during preparation.

We have investigated the adsorption of dye using fly ash and found that fly ash could be a cheaper adsorbent while still having higher adsorption capacity [10]. In this paper, we reported our investigation using hydrothermal treatment of fly ash to convert it to zeolite and the application in adsorption of dyes and heavy metals in aqueous solution.

2. Experimental

2.1. Adsorbent materials and chemicals

A raw fly ash (FA) sample was obtained from Muja Power Station in Western Australia. The chemical compositions of the fly ash are SiO_2 (55%), $A1_2O_3$ (29%), Fe_2O_3 (8.8%), CaO (1.6%) and MgO (1.0%). This fly ash was sieved under different particle size and the section with particle size less than 45 μm was obtained and used for hydrothermal treatment. Typically, 10 g of solid was mixed with 20 mL of NaOH solution with different concentrations and set at room temperature (FA-RT), 60 (FA-60), 100 (FA-100) and 140 °C (FA-140) in oven for 24 h. After that, the products were washed with distilled water and dried at 110 °C overnight.

Table 1
Surface area and pore volume of fly ash and derived materials

Sample	S_{BET} (m^2/g)	V (cc/g)	Major phase
FA	8.4	0.010	Quartz, mullite, and hematite
FA-1M-100	8.1	0.013	Quartz, mullite, and hematite
FA-3M-100	16.7	0.034	NaP1, quartz, mullite, and hematite
FA-5M-100	24.0	0.058	NaP1, quartz, mullite, and hematite
FA-5M-RT	9.7	0.014	Quartz, mullite, and hematite
FA-5M-60	12.4	0.027	NaP1, quartz, mullite, and hematite
FA-5M-140	25.7	0.055	NaP1, quartz, mullite, and hematite

Two metal ion solutions (Ni and Cu) were prepared using the $NiSO_4$ and $Cu(NO_3)_2$ (AR, AJAX) in distilled water. Two basic dyes, methylene blue (MB) and rhodamine B (RB), were obtained from AJAX Chemical. Their chemical structures are shown in Fig. 1. A stock solution with concentration at 10^{-4} M was prepared and the solutions for adsorption tests were prepared from the stock solution to the desired concentration.

2.2. Characterisation of adsorbents

Power XRD patterns were measured by a Rigaku Miniflex diffractometer with Co Kα radiations generated at 30 kV, 15 mA. Scattering patterns were collected from 1.5 to 80° with a scan time of 1 min per 2 steps.

Nitrogen adsorption–desorption isotherms were obtained using NOVA 1200 at the liquid nitrogen temperature (−196 °C). Powder samples were degassed to less than 5 mTorr at 200 °C for 4 h prior to analysis. Surface area calculations were made

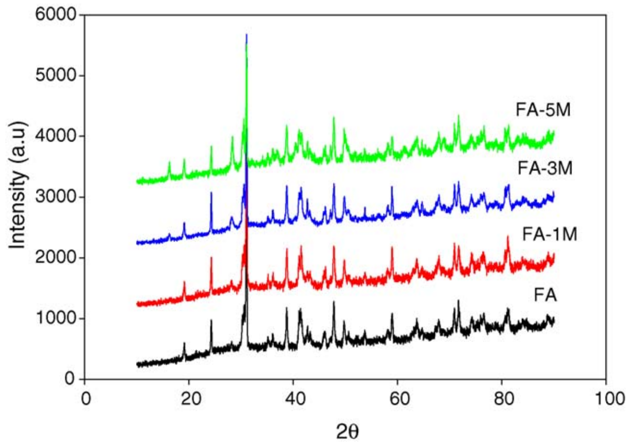

Fig. 2. XRD patterns of fly ash treated at different NaOH concentrations.

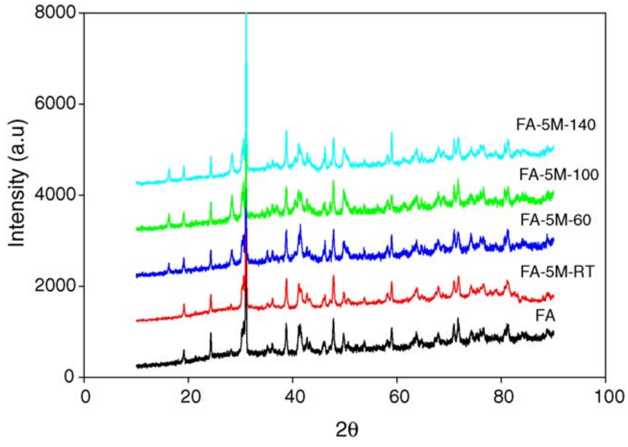

Fig. 3. XRD patterns of fly ash treated at different temperatures.

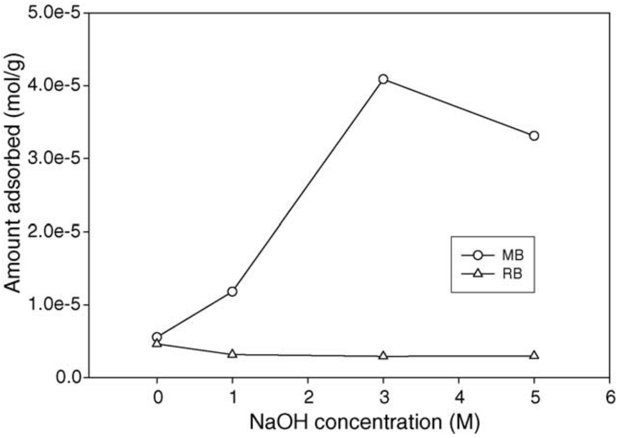

Fig. 4. Effect of NaOH concentration on dye adsorption on treated fly ash. Adsorption conditions: $T = 30\,^{\circ}\mathrm{C}$, pH 6.2.

using the BET equation. Pore size distributions were calculated from desorption branches of isotherms based on BJH method.

2.3. Sorption test

2.3.1. Dye adsorption

The adsorption of dyes was performed by shaking 0.02–0.1 g of solid in 100 mL of dye solution of varying concentration at 100 rpm for 72 h (Certomat R shaker from B. Braun). The determination of dyes was done spectrophotometrically on a Spectronic 20 Genesis Spectrophotometer (USA) by measuring absorbance at $\lambda_{\max}$ of 665 and 556 nm for methylene blue and rhodamine B, respectively. To investigate the effect of pH on adsorption, a series of dye solution were prepared by adjusting pH over a range of 2–11 using 1 M HNO_3 or NaOH solution. The pH of solutions was measured with a pH meter (Radiometer PHM250 ion Analyser).

2.3.2. Heavy metal ion adsorption

The adsorption of heavy metals was also conducted in batch experiments. Typically, 100 mL of solution containing different concentrations of Ni^{2+} and Cu^{2+} were poured into a 250 mL bottle with 0.05 g adsorbents and set at 100 rpm and different temperatures (30, 40 °C) for various time. After adsorption, solution was filtered and the concentrations of Ni and Cu in solution were determined by an Atomic Adsorption Spectrometer (SpectrAA110, Varian).

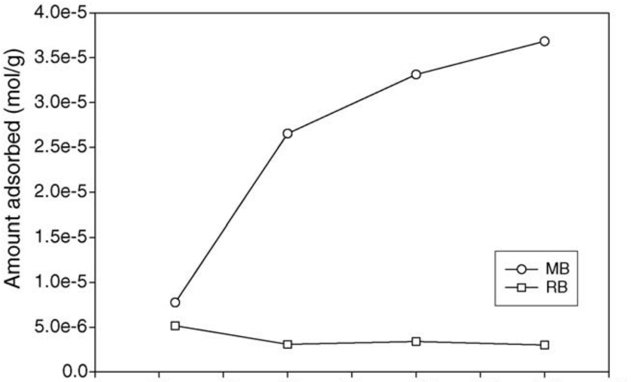

Fig. 5. Effect of treatment temperature on dye adsorption on treated fly ash. Adsorption conditions: $T = 30\,^{\circ}\mathrm{C}$, pH 6.2.

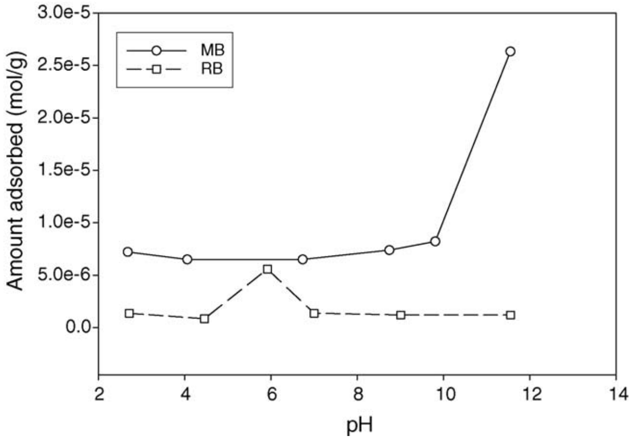

Fig. 6. Effect of solution pH on dye adsorption on fly ash at 30 °C.

3. Results and discussion

3.1. Characterisation of absorbents

The crystalline phases of fly ash and the hydrothermal treated fly ash were determined by XRD analyses. Fig. 2 shows the XRD patterns of fly ash and the samples treated at different NaOH concentration. The results indicate that the untreated fly ash is composed of quartz, mullite, and hematite. The fly ash treated at 1 M NaOH concentration shows similar XRD patterns as that of untreated fly ash. Hydrothermal treatment can produce a new phase, zeolite P only when the fly ash was treated at high concentrated NaOH (>3 M). BET surface area and pore volume of various fly ash and treated samples are given in Table 1. As seen that the surface areas of FA and FA treated at 1 M are the same. Hydrothermal treatment at higher NaOH concentration increases the surface area and pore volume, due to the transformation of some fly ash to zeolite P.

Fig. 3 presents the XRD patterns of fly ash and treated FA at different temperatures. Room temperature treatment of fly ash could not effectively convert fly ash to zeolite. The zeolite P will be formed at temperature higher than 60 °C. BET surface area and pore volume of the samples are also given in Table 1. It is seen that the surface area and pore volume increases with the increasing temperature. The surface area of fly ash can change from 8.4 to 25.7 m^2/g for untreated fly ash and FA-5M-140.

3.2. Dye adsorption studies

3.2.1. Effect of NaOH concentration

The effect of NaOH solution concentration on methylene blue and rhodamine B adsorption is displayed in Fig. 4. As seen that fly ash shows low adsorption of two dyes, around 6×10^{-6} mol/g. The increase in basic solution concentration in

Table 2
Comparison of adsorption isotherm models

Adsorbent	Dye	Langmuir			Freundlich		
		k (L/mol)	Q_0 (mol/g)	R^2	K (mol/g)	$1/n$	R^2
FA	MB	2.26×10^7	5.63×10^{-6}	0.958	5.79×10^{-6}	3.03×10^{-3}	0.958
FA-100		2.37×10^5	4.46×10^{-5}	0.961	3.81×10^{-3}	0.4198	0.994
FA-140		5.45×10^5	5.32×10^{-5}	0.966	5.16×10^{-3}	0.4047	0.984
FA	RB	2.86×10^7	5.13×10^{-6}	0.979	6.03×10^{-6}	0.0145	0.971
FA-140		1.51×10^6	3.98×10^{-6}	0.976	8.49×10^{-6}	0.0728	0.965

treatment of fly ash will significantly influence the adsorption capacity and methylene blue and rhodamine B present quite a different adsorption behaviour. With the increasing concentration, the adsorption capacity of methylene blue is increased. The adsorption capacity at 1, 3 and 5 M NaOH solution will be 1.2×10^{-5}, 4.2×10^{-5} and 3.5×10^{-5} mol/g at the equilibrium. However, for rhodamine B the adsorption capacity will be decreased when the fly ash was modified by NaOH solution, suggesting that zeolitisation of fly ash does not promote the adsorption though the surface area and pore volume are enhanced.

XRD and BET surface area of samples have shown that zeolitisation of fly ash can occur at high treated temperature and high NaOH concentration, resulting in the increase in surface area and pore volume. This will favour the increase of methylene blue adsorption on the solids. In addition, conversion of fly ash to zeolite changes the surface properties of solid adsorbents by increasing the cation exchange capacity, which also favours the adsorption of methylene blue on treated fly ashes. The decrease in adsorption of rhodamine B is due to the different chemical structure and property of the dye. The interaction between solid and dye influences the adsorption behaviour. It has been found that pH of solid slurry of fly ash shows low value less than 6, however, the NaOH treated fly ash samples present higher pH values greater than 9 and increases with NaOH concentration and treatment temperature. This indicates that surface properties of raw fly ash and treated fly ashes are different. Conversion of fly ash to zeolite increase the negative charge. Thus, the presence of an acidic group in the dye prevents the adsorption on basic solid

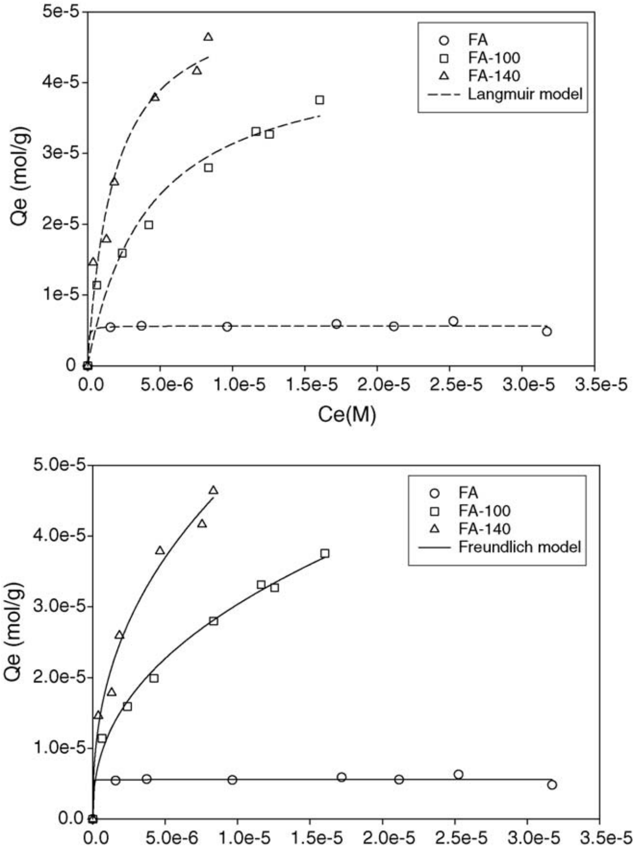

Fig. 7. Adsorption isotherms of methylene blue on various fly ashes. Adsorption conditions: $T = 30\,^{\circ}\mathrm{C}$, pH 6.2.

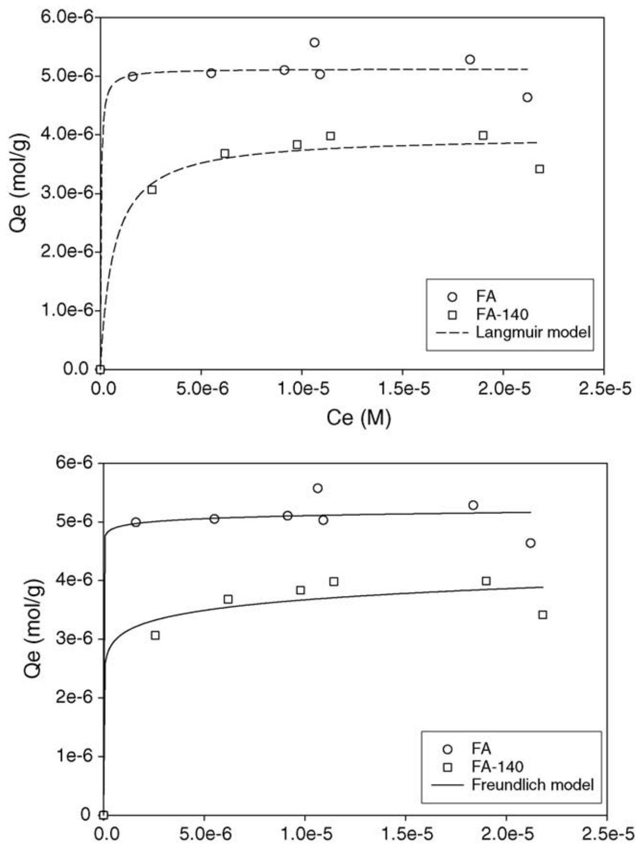

Fig. 8. Adsorption isotherms of rhodamine B on fly ash samples. Adsorption conditions: $T = 30\,^{\circ}\mathrm{C}$, pH 6.2.

surface, resulting in the decrease in adsorption of rhodamine B. The above results also suggest that dye adsorption depends not only the surface area of adsorbent but also the chemical property of dye. For methylene blue, the adsorption has a close relation with surface area of solid but the chemical property of rhodamine B plays an important role for the adsorption.

3.2.2. *Effect of treatment temperature*

Fig. 5 illustrates the effect of treatment temperature on dye adsorption. As shown that high temperature treatment will favour the adsorption of methylene blue but reduce the adsorption of rhodamine B. At the treatment temperature of 140 °C the adsorption of methylene blue can increase to 3.6×10^{-5} mol/g while the adsorption of rhodamine B will slightly decrease to 4×10^{-6} mol/g. XRD results have shown that more fly ash will be converted to zeolite NaP. Due to the conversion of fly ash to zeolite at higher temperatures, the surface functional groups are changed from positive charge to negative charge, resulted in the decrease in adsorption of rhodamine B.

3.2.3. *Effect of pH on the dye adsorption*

Fig. 6 displays the variation of adsorption for two dyes at different pH of solution on fly ash. For methylene blue, the adsorption shows a slight increase at lower pH and sharply increases when the pH is higher than 9. For rhodamine B, the adsorption is also lower while shows a significant increase at pH 6. The effect of pH on dye adsorption can be explained by considering a zero point of charge of the fly ash. Above the zero point of charge the negative charge density on the surface of the fly ash increases, which favours the sorption of basic dyes [5].

The figure also shows that FA always has a high adsorption for methylene blue than rhodamine B, which is probably due to the chemical structure. The molecular size of rhodamine B (Fig. 1) is much larger than methylene blue prohibited it into the small pores of fly ash.

3.2.4. *Adsorption isotherms*

For solid–liquid system, adsorption isotherm is important in description of adsorption behaviour. In this work, we select two important isotherms, Langmuir and Freundlich isotherms.

The Langmuir isotherm takes an assumption that the adsorption occurs at specific homogeneous sites within the adsorbent. The generalised Langmuir isotherm can be written in the form

$$Q_e = \frac{kQ_0C_e}{1 + kC_e} \tag{1}$$

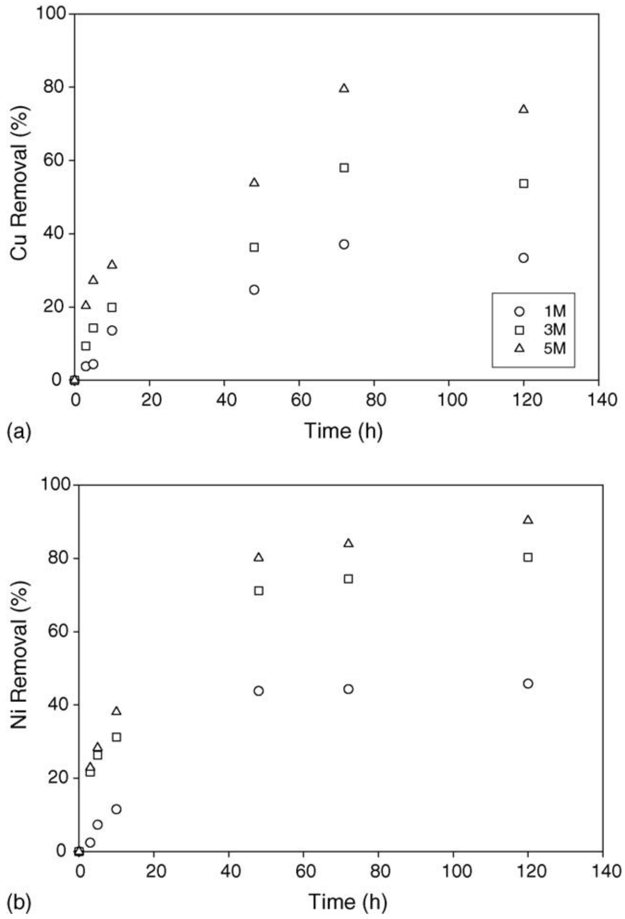

Fig. 9. Dynamic adsorption of metals on FA treated at different NaOH concentration: (a) Cu, (b) Ni. Adsorption conditions: $T = 40$ °C, pH 6.2.

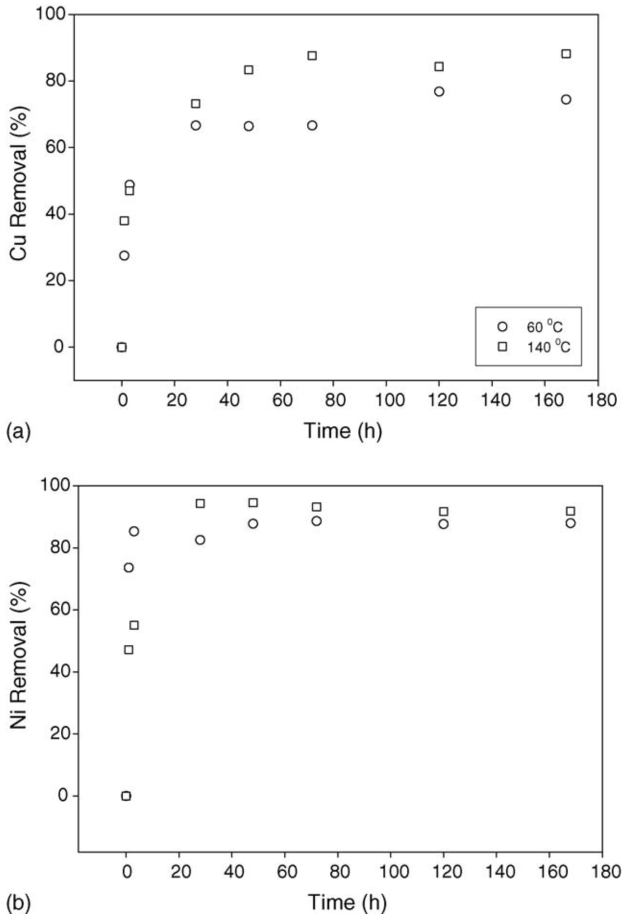

Fig. 10. Dynamic adsorption of metals on FA treated at different temperatures: (a) Cu, (b) Ni. Adsorption conditions: $T = 40$ °C, pH 6.2.

Q_e is the adsorbed amount of the dye (mol/g), C_e is the equilibrium concentration of the dye in solution (M), Q_0 is the monolayer adsorption capacity (mol/g) and k is the constant related to the free energy of adsorption (L/mol).

The Freundlich isotherm is an empirical equation employed to describe heterogeneous systems. The Freundlich equation is expressed

$$Q_e = KC_e^{1/n} \tag{2}$$

where K and n are Freundlich adsorption isotherm constants, being indicative of the extent of the adsorption and the degree of non-linearity between solution concentration and adsorption, respectively.

Figs. 7 and 8 show a comparison of adsorption isotherms of methylene blue and rhodamine B for curve fitting of the experimental results with above two adsorption isotherms on fly ash and treated samples. The model parameters from all isotherms obtained from nonlinear regression are presented in Table 2. As seen that the Langmuir and Freundlich models are all good for the simulation of experimental data. The correlation coefficients for the Freundlich model and the Langmuir model are much similar for all cases.

3.3. Heavy metal adsorption

3.3.1. Effect of NaOH concentration

The dynamic adsorption of Ni and Cu ions on treated fly ash at different NaOH concentrations is shown in Fig. 9. As shown, the removal of Ni^{2+} and Cu^{2+} by treated fly ashes increases with increasing time up to 120 h. Higher concentration of NaOH treatment results in higher removal efficiency of metals due to the conversion of some fly ash to zeolite and the increased surface area and pore volume. In general, treated fly ash shows higher adsorption capacity for Ni than that for Cu. In case of Cu, the removal efficiency will increase from 40% to 75% when the treated NaOH concentration is changed from 1 to 5 M. For Ni, the removal efficiencies are 43% and 90% at the NaOH concentration of 1 and 5 M, respectively.

3.3.2. Effect of treatment temperature

Fig. 10 illustrates the effect of treatment temperature on the adsorption capacity in heavy metals. One can see that higher treatment temperature also favours the adsorption capacity due to the increase in surface area and pore volume, the same as the adsorption of methylene blue. For Cu, the removal efficiencies achieve at 70% and 90% at the temperature of 60 and 140 °C, respectively. For Ni, the difference is not so significant, but the

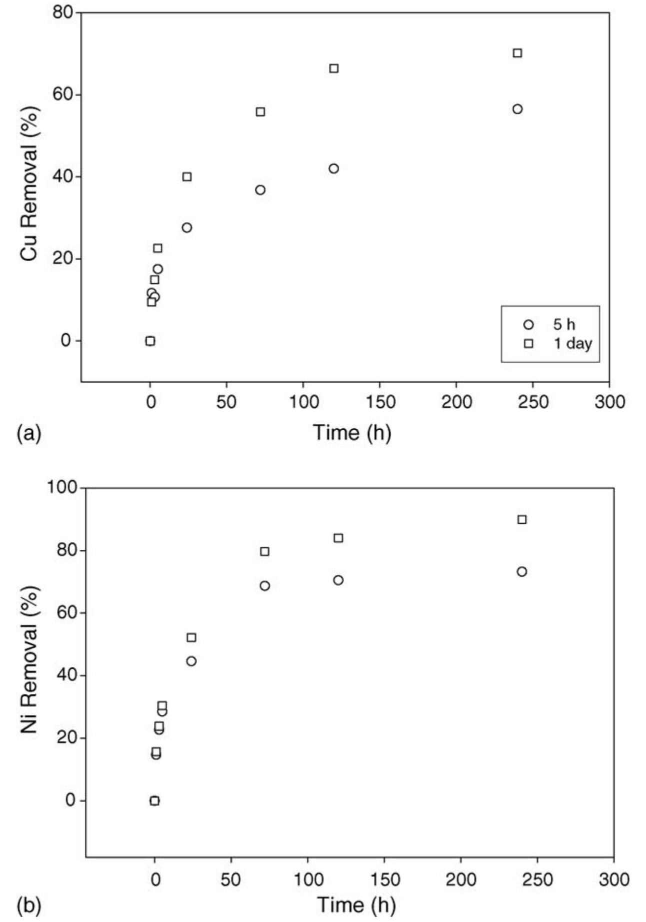

Fig. 11. Dynamic adsorption of metals on FA treated at different time: (a) Cu, (b) Ni. Adsorption conditions: $T = 40$ °C, pH 6.2.

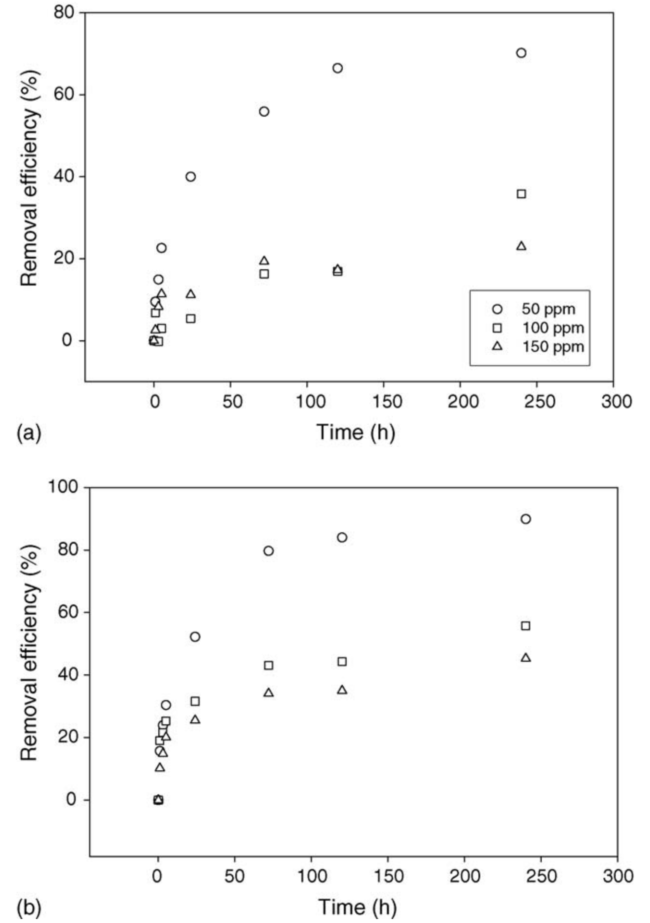

Fig. 12. Effect of initial concentration on metal removal: (a) Cu, (b) Ni. Adsorption conditions: $T = 40$ °C, pH 6.2.

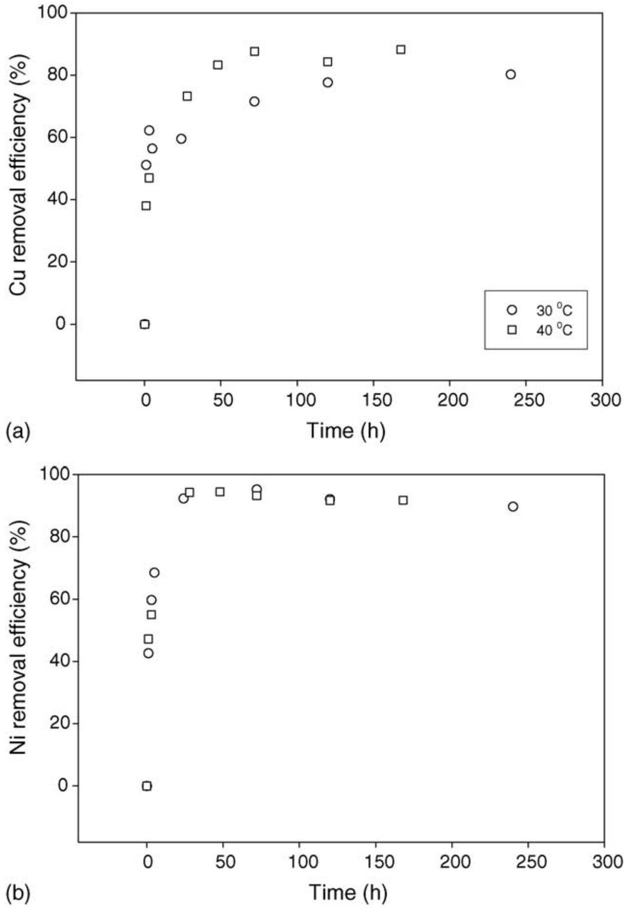

Fig. 13. Effect of adsorption temperature on metal removal: (a) Cu, (b) Ni, pH 6.2.

removal efficiency at 140 °C is still larger than that at 60 °C treatment, all values higher than 90%.

3.3.3. *Effect of treatment time*

The treated time also influences the adsorption capacity of the treated fly ash. Fig. 11 presents the dynamic adsorption of Cu and Ni on fly ash treated at 5 and 24 h. As seen that the removal efficiencies for Cu and Ni are all increased at longer treatment time. The removal efficiency for Ni will increase from 70% to 90% while it will increase from 58% to 70% for Cu.

3.3.4. *Effect of initial concentration of metal ions*

The initial metal concentration will influence the adsorption behaviour. The removal of Ni and Cu decreased with an increase of the metal ion concentration in the solution (Fig. 12). With the increase in the initial concentration of Ni and Cu from 50 to 150 mg/L, respectively, the percentage removal is decreased from 90 to 45 for Ni and 70 to 22 for Cu. The higher metal uptake at low concentration is attributed to the availability of greater surface area with active centres on the adsorbent for lesser amounts of adsorbate ions. These results are similar to the investigations reported by other researchers [11–13]. Panday et al. [11] found that the removal of Cu is highly concentration dependent and that the removal of Cu by fly ash decreases from 100 to 89.69% by increasing the Cu concentration. Bayat [13] reported that the removal of Ni increased with an increase of the metal ion concentration in the solution while the removal of Cu decreased slightly for fly ash.

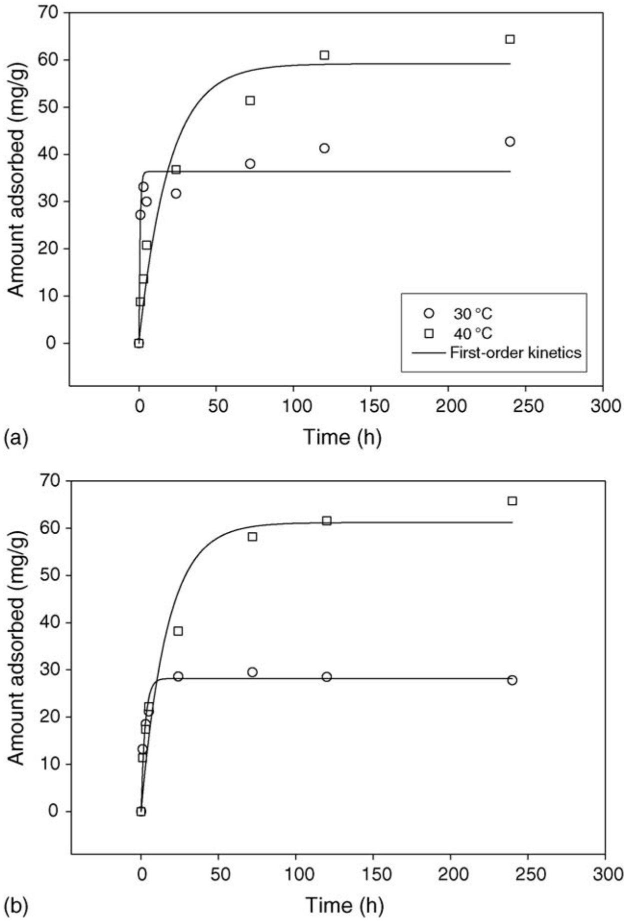

Fig. 14. The pseudo first-order kinetics of metal adsorption on treated fly ash at different temperatures: (a) Cu, (b) Ni.

Table 3
Parameters for kinetic models

Temp (°C)	Metal	Experimental, q_e (mg/g)	First-order kinetics			Second-order kinetics		
			k_1 (1/h)	q_e (mg/g)	R^2	k_2 (g/mg h)	q_e (mg/g)	R^2
30	Cu	42.0	1.30	36.36	0.893	5.79×10^{-3}	42.99	0.998
40		64.0	0.051	59.19	0.951	1.08×10^{-3}	67.38	0.996
30	Ni	28.0	0.38	28.18	0.961	0.4678	28.05	0.999
40		65.8	0.059	61.20	0.943	1.325×10^{-3}	68.26	0.998

3.3.5. *Effect of adsorption temperature*

The adsorption studies were also carried out at two different temperatures: 30 and 40 °C, as shown in Fig. 13. It is seen that the removal efficiency increases with the increasing temperature, indicating that the diffusion of metal ions on fly ash is endothermic reaction. For Cu, the removal efficiency is increased from 80% at 30 °C to 90% at 40 °C while for Ni the removal efficiency shows a slight increase at 40 °C compared with the value at 30 °C.

The kinetics of adsorption of Cu and Ni on fly ash was calculated using the Lagergren first-order and pseudo-second order rate equations as modified by Ho for the determination of the rate constant [14,15]. The Lagergren kinetics first-order and the pseudo second-order equations have been most widely used for the adsorption of an adsorbate from an aqueous solution, which are expressed by Eqs. (3) and (4)

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

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{4}$$

where, q_e and q_t are the amount of metal ions adsorbed per unit mass of the adsorbent (in mg/g) at equilibrium time and time t, respectively, and k_1 and k_2 are the rate constant for the first- and second-order kinetics. Due to the unknown q_e, it is better to use the following equation instead of Eq. (3) for curve-fitting of Lagergren kinetic first-order reaction.

$$q_t = q_e(1 - e^{-k_1 t}) \tag{5}$$

Figs. 14 and 15 show the curve-fitting plots of the Lagergren first-order and pseudo second-order plots, respectively, and the parameters obtained for the two models are presented in Table 3. As shown that the pseudo first-order and the pseudo second-order models will be good in fitting the experimental data. The data of equilibrium adsorption from the second-order kinetics will be close to the experimental data and regression coefficients also indicate that the pseudo second-order will show better simulation results.

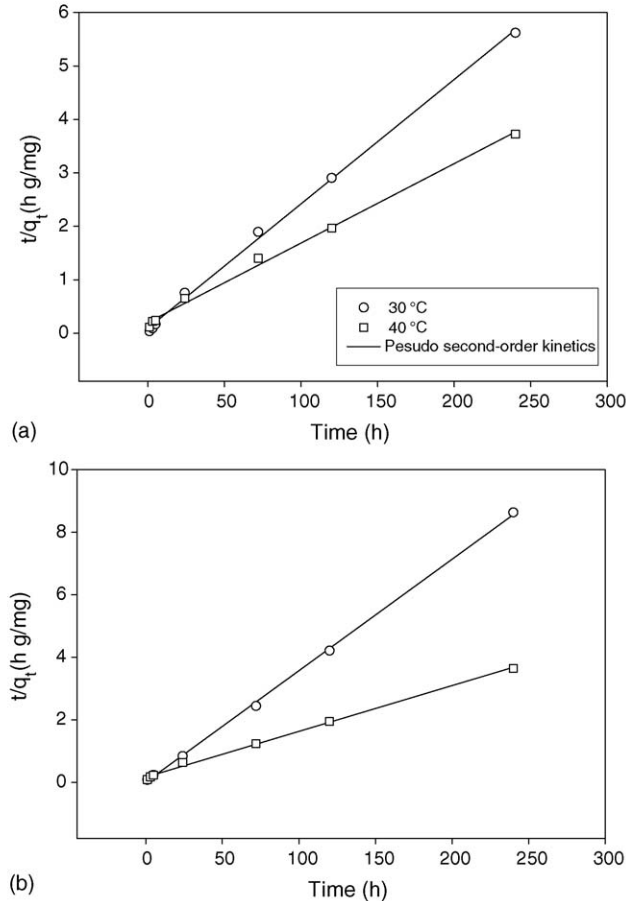

Fig. 15. The pseudo second-order kinetics of metal adsorption on treated fly ash at different temperatures: (a) Cu, (b) Ni.

4. Conclusion

Fly ash can be converted to zeolite P by hydrothermal treatment in NaOH solution. Higher NaOH concentration and temperature will favour the production of zeolite P. The treated fly ashes are much effective for adsorption of methylene blue but less adsorption in rhodamine B. The treatment parameters such as NaOH concentration, temperature and time will affect the adsorption capacity. The adsorption isotherm, Langmuir and Freundlich models, will well fit to the experimental data. For the heavy metals such as Cu and Ni, the treated fly ashes also show effective adsorption with higher capacity for Ni than Cu. The kinetics of adsorption of heavy metals will be described by pseudo first-order and pseudo second-order models but the pseudo second-order kinetics will be better.

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