



## Removal of lead and zinc ions from water by low cost adsorbents

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### ABSTRACT

In this study, activated carbon, kaolin, bentonite, blast furnace slag and fly ash were used as adsorbent with a particle size between 100 mesh and 200 mesh to remove the lead and zinc ions from water. The concentration of the solutions prepared was in the range of 50–100 mg/L for lead and zinc for single and binary systems which are diluted as required for batch experiments. The effect of contact time, pH and adsorbent dosage on removal of lead and zinc by adsorption was investigated. The equilibrium time was found to be 30 min for activated carbon and 3 h for kaolin, bentonite, blast furnace slag and fly ash. The most effective pH value for lead and zinc removal was 6 for activated carbon. pH value did not effect lead and zinc removal significantly for other adsorbents. Adsorbent doses were varied from 5 g/L to 20 g/L for both lead and zinc solutions. An increase in adsorbent doses increases the percent removal of lead and zinc. A series of isotherm studies was undertaken and the data evaluated for compliance was found to match with the Langmuir and Freundlich isotherm models. To investigate the adsorption mechanism, the kinetic models were tested, and it follows second order kinetics. Kinetic studies reveals that blast furnace slag was not effective for lead and zinc removal. The bentonite and fly ash were effective for lead and zinc removal.

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### 1. Introduction

The presence of heavy metals in the environment is ubiquitous. As per the guidelines of World Health Organization (WHO), the drinking water containing heavy metals like aluminium, chromium, manganese, iron, cobalt, nickel, copper, zinc, cadmium, mercury and lead is major concern due to their toxicity. Among all, lead and zinc are most important because of their high toxicity. Lead leads the list of environmental threats because, even at extremely low concentration, lead has been shown to cause brain damage in children [1]. Zinc is essential in small quantity but when exceeds the prescribed limit it has also detrimental effect on human health. World Health Organization has set a provisional limit of 0.05 mg/L and 5 mg/L for lead and zinc, respectively.

Lead and zinc are mostly found in the inorganic form particularly in +1, +2 oxidation states for lead and +2 states for zinc. Lead is found in various industrial activities like manufacturing of alloys, electrical goods, chemical catalysis, metal surface finishing and batteries. Similarly zinc is used in the process of galvanization, pigment formation, stabilizers, thermoplastics, alloys and batteries. During the metallurgical processes, some amounts of metals are also released into the water bodies. The release of large quantities of heavy metals

into the natural environment e.g. irrigation of agricultural fields by using sewage has resulted in a number of environmental problems [2] and due to their non-biodegradability and persistence, can accumulate in the food chain, and thus may pose a significant danger to human health [3].

Constant efforts are being made to develop improved and innovative methods of waste water treatment. While developing new methods, economic feasibility and user friendly concepts are given much importance. Various processes for the removal of metals are available but the most commonly used process is precipitation. By the addition of suitable reagent of required quantity, under proper reaction condition makes the metal precipitated. But the sludge that is formed contains more amounts of heavy metals and the disposal of such sludge creates another problem. So other treatment processes like ion exchange, reverse osmosis or adsorption processes are required subsequently after the precipitation process and before discharging into water bodies. So the removal of metal requires two processes when the contents are very high. Because in first process it removes the high metal concentration and in the second process it removes the residual low metal concentration. So the cost for this two stage treatment is high which sometimes is not suitable for most of the industries. Keeping this in view, considerable attention has been given to develop low cost adsorbents for removal of the different heavy metals when the concentration is not very high.

In nature, there are so many materials which possess the properties of ion exchange and adsorption. Some materials are already

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**Table 1**  
Specific surface area of the adsorbents.

Materials	Specific surface area (m <sup>2</sup> /g)
Activated carbon	655.44
Kaolin	19.43
Bentonite	62.69
Blast furnace slag	0.70
Fly ash	0.75

in use even for commercial purposes. Some of the examples are zeolite, apatite, bentonite, etc. Similarly many waste materials like fly ash [4], red mud and blast furnace slag [5] are also used to remove the heavy metals from waste water. Activated carbon is the most widely used adsorbent [6] only because of its more surface area. The cost of activated carbon is high which leads to limited uses particularly in developing countries. Hence in search of materials having similar properties, the attention has been diverted towards the agricultural and forestry products such as saw dust, rice husk, used tea, straw, seeds etc. Many more common materials are also being used to reduce various pollutants [7–13].

Keeping in view the above, the present study was undertaken to investigate the adsorption of two heavy metals lead and zinc using different adsorbents like activated carbon as the reference and kaolin, bentonite, blast furnace slag, fly ash in single and binary solutions.

## 2. Materials and methods

All the chemicals used are of AR grade. Sample solutions were prepared by dissolving required amount of lead nitrate and zinc chloride salts obtained from Merck in deionized water. The concentration of the solutions prepared was in the range of 50–100 mg/L for lead nitrate and zinc chloride, respectively for single and binary systems.

### 2.1. Adsorbents

Five adsorbent materials activated carbon, kaolin, bentonite, blast furnace slag and fly ash were used to study the removal efficiency of lead and zinc. Kaolin and Bentonite were obtained from Merck. Bentonite contained over 80% montmorillonite. Blast furnace slag and fly ash were collected from the dumping site and the ash bond of Rourkela Steel Plant of Steel Authority of India Limited respectively. The surface area was measured for all the materials at liquid nitrogen temperature using BET surface area analyzer and the data is represented in Table 1.

Activated carbon, kaolin and bentonite were used without any pretreatment. Blast furnace slag and fly ash were oven dried at 105 °C for 24 h, which was manually ground in a mortar after cooling in a desiccator and then screened to eliminate the coarser sizes and to get homogeneous particle size ranging between 0.05 mm and 1.0 mm.

### 2.2. Adsorbent dosage and contact time studies

Adsorption of 50 mg/L of lead nitrate and 100 mg/L zinc chloride solution by different adsorbent doses (5–20 g/L) for each adsorbent was carried out at the optimum pH at different time intervals.

### 2.3. pH studies

In order to investigate the effects of pH on metal ion adsorption, the pH of the metal ion solution was adjusted to different values (2,

5, 7 and 10) by adding required amount of dilute NaOH or HCl solution. pH was measured using a pH meter. pH adjusted solution and 0.5 gm adsorbent were used in batch experiments conducted at the determined equilibrium time. The pH value at which the maximum lead and zinc removed was determined.

### 2.4. Adsorption studies

Adsorption of lead and zinc ion onto different adsorbents was studied by batch experiments. A fixed amount of each dry adsorbent 0.5 gm and 100 mL of lead nitrate and zinc chloride solution was placed in a capped volumetric flask and shaken at 140 rpm using a temperature controlled water bath with shaker at ambient temperature  $25 \pm 2$  °C. Then, the adsorption samples were centrifuged and the lead and zinc concentration in supernatant liquid was determined.

Metal concentration was determined by lead and zinc ion selective electrode. Methanol–formaldehyde reagent was prepared by adding 3 drops 37% formaldehyde to 1 L methanol to preserve the electrode used for the determination of lead. Ionic strength adjuster (ISA) was prepared by adding 80.25 g of sodium perchlorate to 100 mL distilled water in a volumetric flask for lead and 100 mL of  $10^{-3}$  M NaNO<sub>3</sub> solution was prepared for zinc.

The ion analyzer was calibrated using the standard solution supplied with instruments. Ionic strength adjuster solution was added to all solutions to ensure that the samples and standard have similar ionic strength and proper pH and to reduce the effect of interfering ions.

### 2.5. Kinetic studies

In kinetic studies, 50 mL of lead nitrate and zinc chloride solution each and 0.5 gm adsorbent were used. Batch experiments were repeated at different periods of all adsorbents, lead and zinc ion concentration in supernatant have been constant after a certain time period which was accepted as equilibrium time for relevant adsorbent.

### 2.6. Desorption and regeneration studies

The reusability of the adsorbent mainly depends on the ease with which Pb (II) and Zn (II) ions desorbed from loaded adsorbent sample. For this 100 mL of 10 mg/L, 50 mg/L and 100 mg/L lead nitrate and zinc chloride solution was tested with each of adsorbent (0.5 g) and was kept under stirring for 24 h. The content of the flask was filtered and separated. The filtered adsorbent was retreated with 100 mL neutral distilled water and distilled water adjusted to different pH with the help of 1 M HCl. It was stirred for 24 h. The residual lead and zinc concentration was measured. The study was carried at ambient temperature  $25 \pm 2$  °C.

## 3. Results and discussion

### 3.1. Effect of adsorbent dose

The effect of adsorbent dose on the removal of lead and zinc was studied at ambient temperature  $25 \pm 2$  °C and contact time of 3 h for initial lead and zinc concentrations of 50 mg/L and 100 mg/L for both single and binary systems. The maximum removal of lead and zinc was obtained for the adsorbent dose of 20 g/L (Figs. 1 and 2). However, it is observed that after dosage of 20 g/L, there was no significant change in percentage removal of both lead and zinc in single and binary systems. It may be due to the overlapping of active sites at higher dosage. There is decrease in the effective surface area resulting in the conglomeration of exchanger particles [14]. So

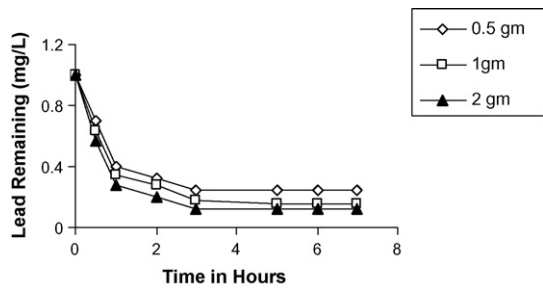


Fig. 1. Kinetic curves for lead removal using bentonite at different adsorbent doses.

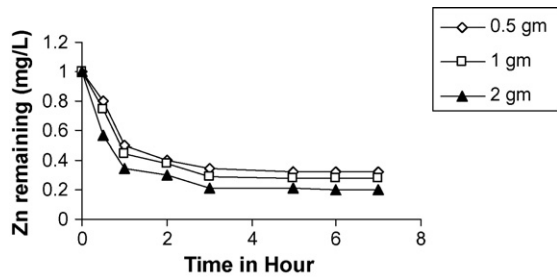


Fig. 2. Kinetic curves for zinc removal using bentonite at different adsorbent doses.

20 g/L was considered as optimum dose and was used for further study for both single and binary systems.

### 3.2. Effect of contact time

Adsorption of lead and zinc at different contact time was studied for initial lead and zinc concentration of 50 mg/L and 100 mg/L keeping all other parameters constant. There is increase in adsorption with increase in contact time and maximum adsorption takes place at 3 h (Figs. 3 and 4) and again after 3 h contact time there was no further adsorption. This may be due to the fact that ini-

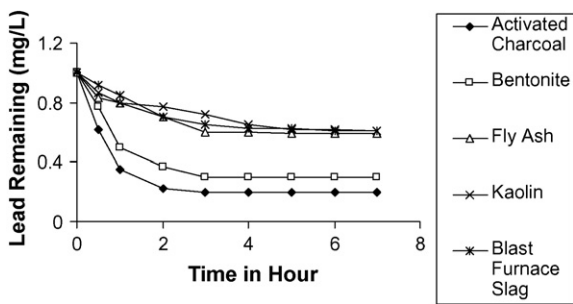


Fig. 3. Time decay curves for different adsorbents used for removal of lead from water.

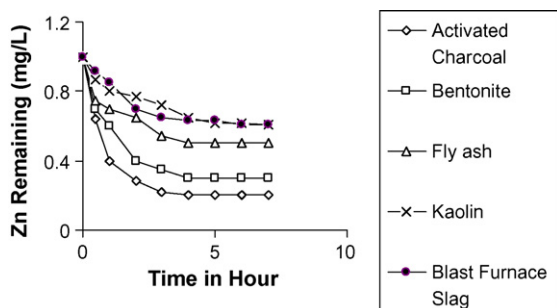


Fig. 4. Time decay curves for different adsorbents used for removal of zinc from water.

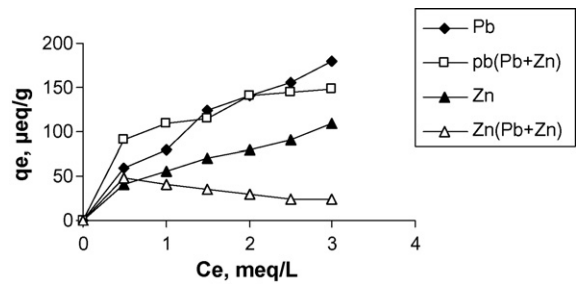


Fig. 5. Experimental isotherms for the adsorption of Pb and Zn in single and binary solutions onto activated carbon.

tially all adsorbent sites were vacant and the solute concentration gradient was high. Later, the lead and zinc uptake rate by adsorbent was decreased significantly, due to the decrease in number of adsorption sites as well as lead and zinc concentration. Decrease in removal rate, towards the end of the experiments, indicates the possible monolayer formation of lead and zinc ion on the outer surface.

### 3.3. Effect of pH

Adsorption of lead and zinc was studied under similar condition at different pH. There is practically no removal at pH lower than 3 may be due to high  $H^+$  ion concentration, which reverses the process of adsorption. There is a gradual increase in adsorption with increase in pH from 3 to 6 and the maximum adsorption is at pH 6. Again the percentage of adsorption increases gradually with increase in pH from 6 to 10 may be due to the formation of the precipitate of  $Pb(OH)_2$  and  $Zn(OH)_2$ . So pH 6 was considered as optimum condition and was used for further study for both single and binary systems.

### 3.4. Adsorption isotherm

The adsorption isotherms obtained with single and binary solutions with the different adsorbent materials are shown in Figs. 5–10. The best results are obtained with bentonite, since its metal uptake

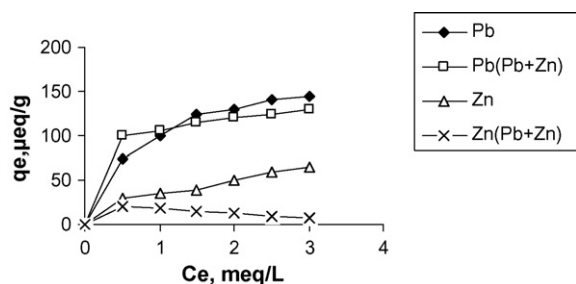


Fig. 6. Experimental isotherms for the adsorption of Pb and Zn in single and binary solutions onto kaolin.

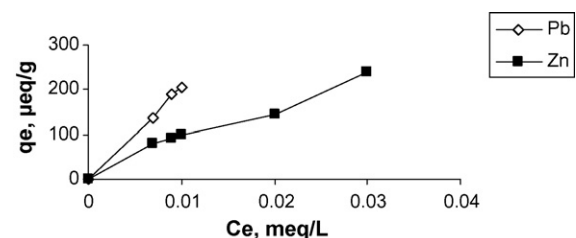


Fig. 7. Experimental isotherms for the adsorption of Pb and Zn in single solution onto bentonite.

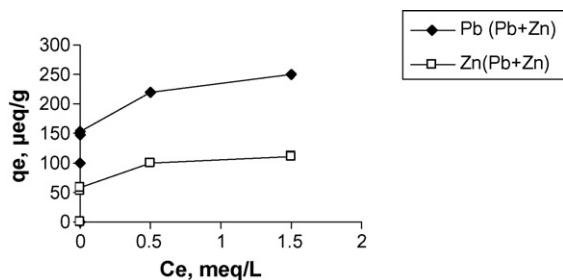


Fig. 8. Experimental isotherms for the adsorption of Pb and Zn in binary solution onto bentonite.

capacities are the highest and hence the residual metal concentrations in the solutions are the lowest. It is observed that adsorption onto this material is practically linear in solutions containing one metal, whereas in binary solution the linearity can only be observed for the lower concentrations. This may be due to the saturation of bentonite at higher concentration, as can be observed in Fig. 5.

For activated carbon and kaolin, the presence of Zn in the solution adversely affects the adsorption of lead; on increasing the initial concentration of both metals, the adsorption capacity of the different materials for this later metal decreases. This indicates that these materials have a greater selectivity for lead, in accordance with the experimental results found in the literature for similar materials, in which this behavior is justified on the basis of the ionic size of the hydrated metal ion [15] or the different electromagnetivity of the two ions [16].

With respect to fly ash, it was found in both cases that the presence of one of the metals in solution decreases the uptake of the other and this decrease is more pronounced for zinc, indicating the greater affinity of these materials for lead.

The equilibrium data obtained in the different adsorbent–metal systems fitted the Langmuir and Freundlich models. The Langmuir isotherm fits the following equation:

$$q_e = \frac{Qk_c c_e}{(1 + k_c c_e)} \quad (1)$$

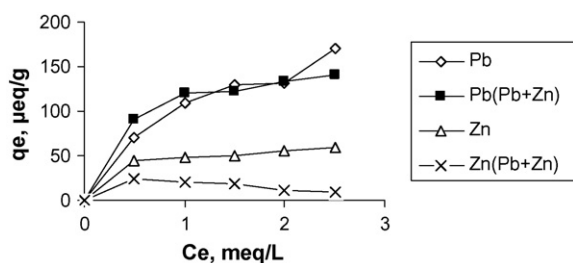


Fig. 9. Experimental isotherms for the adsorption of Pb and Zn in single and binary solutions onto blast furnace slag.

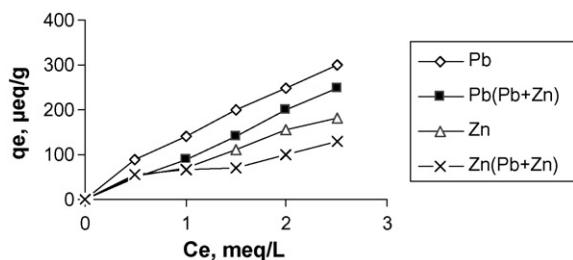


Fig. 10. Experimental isotherms for the adsorption of Pb and Zn in single and binary solutions onto fly ash.

Table 2

Langmuir parameters for the adsorption of Pb and Zn in single solution.

Adsorbents	Langmuir constants					
	Lead (Pb)			Zinc (Zn)		
	Q (mg/g)	K (L/mg)	r	Q (mg/g)	K (L/mg)	r
Active carbon	6.68	0.05	0.90	11.24	0.02	0.96
Kaolin	4.50	0.14	0.90	3.05	0.07	0.84
Bentonite	7.56	3.69	0.98	9.12	22.08	0.94
BFS	5.52	0.08	0.84	3.25	0.35	0.71
Fly ash	4.98	0.20	0.99	5.82	0.05	0.99

where,  $q_e$  = the specie concentration in the adsorbent;  $k$  = the constant of Langmuir's equation, related to the enthalpy of the process;  $Q$  = the adsorption capacity to form the single layer;  $c_e$  = the concentration in the solution.

This isotherm is an example of a favorable isotherm and is applicable under the following hypothesis: a uniform surface of the solid, the absence of interaction between the different adsorbed molecules and adsorption in a single layer.

Freundlich's isotherm fits the following equation:

$$q_e = K_F c_e^{1/n} \quad (2)$$

where,  $q_e$  = the amount adsorbed per specified amount of adsorbent (mg/g);  $K_F$  = a constant related to the adsorption capacity;  $c_e$  = the concentration in solution;  $n$  = an empirical parameter related to the intensity of adsorption, which varies with the heterogeneity of the adsorbent. For values in the range  $0.1 < 1/n < 1$ , adsorption is favorable [17,18].

This model is valid for heterogeneous surfaces and predicts an increase in the concentration of the ionic specie adsorbed onto the surface of the solid when increasing the concentration of said specie in the liquid phase.

Fittings were carried out separately for the single and binary systems. The results are shown in Tables 2–5. With regards to the single systems, it can be seen that the Langmuir model fits the results better, in spite of the heterogeneity of many of the materials employed as adsorbents. The best fittings obtained with both models for bentonite and the fly ash, though the correlation coefficients are somewhat higher in Langmuir model. With respect to the adsorption of lead, the capacities obtained varied between 7.56 mg/g for the lead–bentonite system and 4.98 mg/g for lead–fly ash system. Similarly, with respect to the adsorption on Zn, the capacities

Table 3

Freundlich parameters for the adsorption of Pb and Zn in single solution.

Adsorbents	Freundlich constants					
	Lead (Pb)			Zinc (Zn)		
	$K_F$	1/n	r	$K_F$	1/n	r
Active carbon	0.85	0.44	0.87	0.64	0.52	0.94
Kaolin	1.42	0.21	0.77	0.74	0.24	0.94
Bentonite	17.98	0.84	0.95	1.84	0.45	0.96
BFS	1.01	0.34	0.75	1.72	0.14	0.85
Fly ash	1.92	0.23	0.97	0.90	0.37	0.98

Table 4

Langmuir parameters for the adsorption of Pb and Zn in binary solution.

Adsorbents	Langmuir constants					
	Lead (Pb + Zn)			Zinc (Pb + Zn)		
	Q (mg/g)	K (L/mg)	r	Q (mg/g)	K (L/mg)	r
Active carbon	4.77	0.20	0.98	–	–	–
Kaolin	4.36	0.17	0.99	–	–	–
Bentonite	7.56	3.73	0.99	5.80	1.04	0.98
BFS	4.27	0.33	0.91	–	–	–
Fly ash	4.55	0.13	0.99	1.82	0.39	0.72

**Table 5**  
Freundlich parameters for the adsorption of Pb and Zn in binary solution.

Adsorbents	Freundlich constants					
	Lead (Pb + Zn)			Zinc (Pb + Zn)		
	$K_F$	$1/n$	$r$	$K_F$	$1/n$	$r$
Active carbon	1.99	0.19	0.99	–	–	–
Kaolin	2.74	0.09	0.96	–	–	–
Bentonite	4.92	0.14	0.96	3.57	0.13	0.97
BFS	2.16	0.15	0.77	–	–	–
Fly ash	1.55	0.23	0.95	1.38	0.06	0.71

obtained varied between 11.24 mg/g for the zinc–activated carbon system and 5.82 mg/g for zinc–fly ash system.

Worth noting with regards to adsorption in binary systems is the fact that in the system of zinc–activated carbon, zinc–kaolin and zinc–blast furnace slag, the equilibrium data obtained does not fit the Langmuir and Freundlich models, although this fitting was possible in single systems. This may be because on increasing the amount of zinc in the solution, the expected increase in adsorption capacity is not achieved but rather it decreases (Figs. 5–10), since as the amount of lead also increases, a competitive phenomenon arises between both species, lead being preferentially adsorbed.

In order to understand the adsorption type, equilibrium data was tested with Dubinin–Radushkevich isotherm [19].

The linearized D.R. equation can be written as

$$\ln q_e = \ln q_m - K\varepsilon^2 \quad (3)$$

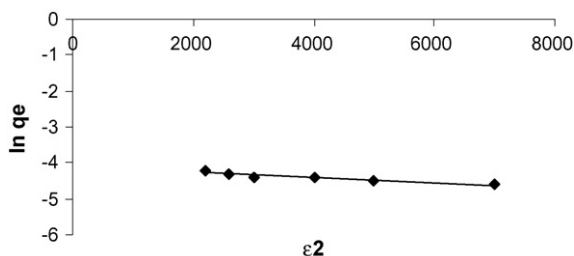
where  $\varepsilon$  is Polanyi potential and is equal to  $RT \ln(1 + 1/C_e)$ ,  $q_e$  is the amount of lead adsorbed per unit mass of adsorbent,  $q_m$  is the theoretical adsorption capacity,  $C_e$  is the equilibrium concentration of lead,  $K$  is the constant related to adsorption energy,  $R$  is the universal gas constant and  $T$  is the temperature in Kelvin. Fig. 11 shows the plot of  $\ln q_e$  against  $\varepsilon^2$ , which was almost linear with correlation coefficient,  $R^2 = 0.9631$ . D.R. isotherm constants  $K$  and  $q_m$  were calculated from the slope and intercept of the plot, respectively. The value of  $K$  was found to be  $2.56 \times 10^{-4} \text{ mol}^2 \text{ kJ}^{-2}$  and that of  $q_m$  was 0.0069 g/g.

The mean free energy of adsorption ( $E$ ) was calculated from the constant  $K$  using the relation [20].

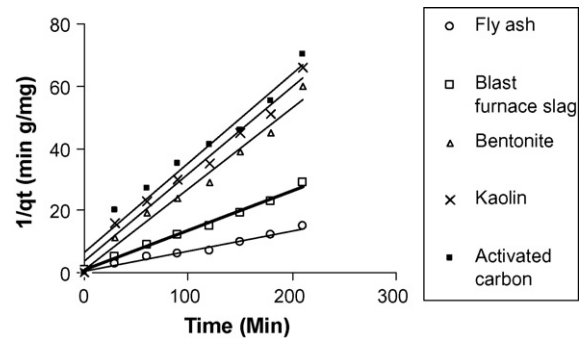
$$E = (2K)^{-1/2} \quad (4)$$

It is defined as the free energy change when 1 mol of ion is transferred to the surface of the solid from infinity in solution. The value of  $E$  was found to be 43.01 kJ/mol. The value of  $E$  is very useful in predicting the type of adsorption and if the value is less than 8 kJ/mol, then the adsorption is physical in nature and if it is in between 8 kJ/mol and 16 kJ/mol, then the adsorption is due to exchange of ions [21]. The value in the present study was found to be little greater than 16 kJ/mol. This is due to different chemical processes accompanying the ion exchange process.

The adsorptive capacities obtained with the Langmuir model, considering the highest correlation coefficients varied between



**Fig. 11.** D–R adsorption isotherm,  $\ln q_e$  versus  $\varepsilon^2$ .



**Fig. 12.** Plots of second order model for nitrate adsorption by different adsorbents.

7.56 mg/g [22] obtained for the [Pb (Pb+Zn)]–bentonite system and 4.36 mg/g for [Pb (Pb+Zn)]–kaolin system. In the case of the adsorption of zinc, and in the cases where the fitting was possible, the capacities varied between 5.80 mg/g for the [Zn (Pb+Zn)]–bentonite system and 5.36 mg/g for the [Zn (Pb+Zn)]–blast furnace slag system.

### 3.5. Adsorption kinetics

In order to examine the controlling mechanisms of adsorption processes such as mass transfer and chemical reaction, several kinetic models are used to test the experimental data.

The rate constant of adsorption is determined from the following first order rate expression given by Lagergren.

$$\log(q_e - q_t) = \log q_e - \left( \frac{K_1 t}{2.303} \right) \quad (5)$$

where  $q_e$  and  $q_t$  are the amounts of lead and zinc adsorbed (mg/g) at equilibrium and at time  $t$  (min), respectively, and  $k_1$  (1/min) is the rate constant of first order adsorption.

A second order equation based on adsorption equilibrium capacity may be expressed in the form.

$$\frac{t}{q_t} = \left( \frac{1}{k_2 q_e^2} \right) + \left( \frac{t}{q_e} \right) \quad (6)$$

where  $k_2$  g/(min mg) is the rate constant of second order adsorption. If the above two equations cannot give definite mechanisms, the intraparticle diffusion model is tested. The fractional approach to equilibrium changes according to a function of  $(Dt/r^2)^{1/2}$ , where  $r$  is the particle radius and  $D$  is diffusivity of solute within the particle. The initial rates of intraparticle diffusion can be obtained by linearization of the curve  $q_t = f(t^{1/2})$ .

The fit of these models was checked by each linear plot of  $\log(q_e - q_t)$  versus  $t$ ,  $(t/q_t)$  versus  $t$  and  $q_t$  versus  $t^{1/2}$ , respectively and by comparing to the regression coefficients for each expression. First order rate expression and intraparticle diffusion model is not fully valid for the present systems due to low correlation coefficients. Hence for the first order rate expression, the experimental  $q_e$  values do not agree with the calculated ones obtained from the linear plots. A good agreement of the experimental data with the second order kinetic model was observed for different adsorbent which is presented in Fig. 12. The correlation coefficients for the second order kinetic models are greater than 0.996 and the calculated  $q_e$  values agree very well with the experimental data.

### 3.6. Desorption and regeneration studies

In order to know the nature of adsorption i.e. physical, chemical or both, desorption study was carried out; the regeneration study at varying pH was studied in a range of pH 3–10. Dilute HCl and NaOH were used to adjust the pH of the solution. Change of

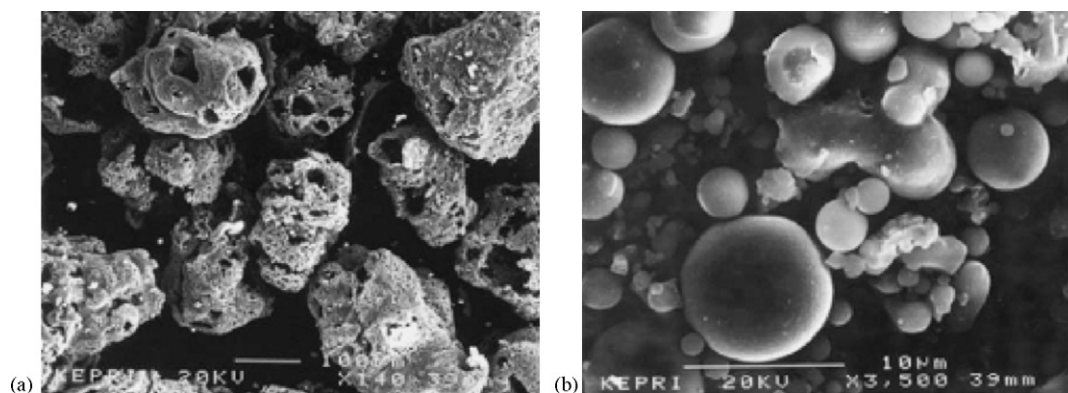


Fig. 13. SEM pictures: (a) +200 mesh particles of the fly ash and (b) –200 mesh particles of the fly ash.

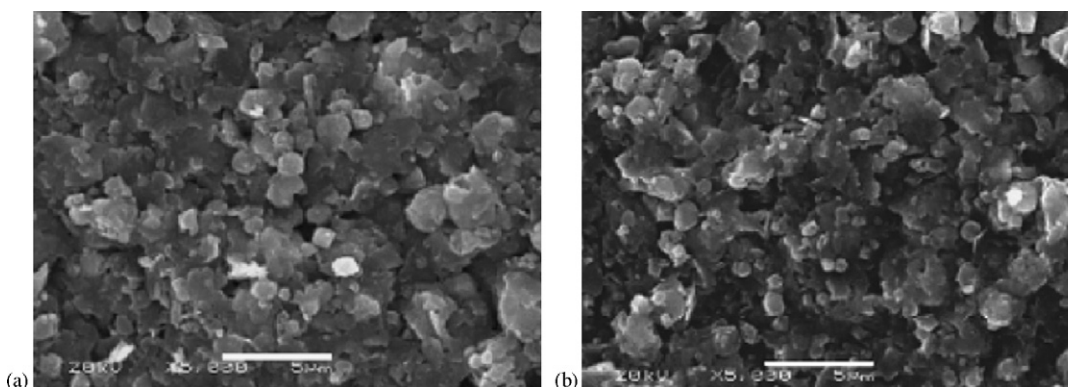


Fig. 14. (a)+200 mesh particles of bentonite and (b) –200 mesh particles of bentonite.

percentage sorption in acidic environment was insignificant. At pH of 6, maximum sorption of lead and zinc was observed. The desorption of adsorbed lead and zinc on different adsorbents resulted about 5.2% thereby indicating the process of adsorption chemical in nature [22].

Amount adsorbed–80.4 mg/L.

Amount desorbed–4.2 mg/L

Percentage desorption–5.2%.

### 3.7. Characterization of fly ash and bentonite

Fly ash is a heterogeneous material consisting largely of small spheres, formed by the condensation of aluminous and siliceous glass droplets in the air. Also found in fly ash samples are irregular, porous, coke-like particles of unburned carbon material, which are often concentrated in the larger size fractions. The SEM (Fig. 13) image clearly shows that finer fly ash particles (smaller than 200 mesh) are primarily spherical, whereas the coarser particles (larger than 200 mesh) are mainly composed of irregular and porous particles. The bentonite shows loose aggregates with porous structure (Fig. 14).

## 4. Conclusion

Of the materials studied, bentonite and fly ash exhibited the highest adsorption capacities, the lowest residual concentration and thus the greatest removal capacities of lead and zinc. The presence of the two metals in solution creates competition for the linking sites on the adsorbent material. With the exception of blast furnace slag, all the materials studied exhibited greater selectivity for lead.

In general, both the Langmuir and the Freundlich models achieve good fittings in single solutions. In binary systems, it was observed that within the range of concentrations studied, the presence of lead in the solution provoked a decrease in the adsorption capacity for zinc of activated carbon, kaolin and blast furnace slag. In these cases, it was not possible to carry out the fitting with said models.

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