



Single and binary adsorption of heavy metals on fly ash samples from aqueous solution

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ABSTRACT

Thousands of toxins are released every day into the environment through numerous industrial operations and this resulted in continuous search for newer adsorbents. Two types of adsorbents, 1:10 fly ash and 1:15 fly ash samples were prepared from coal fired thermal power plant fly ash by treating it with calculated amount of calcium carbonate and phosphoric acid. Adsorbents were characterized on the basis of FTIR, XRF, surface area, pH and particle size studies. Adsorption of Pb(II), Ni(II) and Cr(VI) from single metal solutions on 1:10 fly ash from aqueous solution was carried out as a function of initial metal ion concentration, adsorbent dose, contact time and pH of the solution. Adsorption data obtained was analyzed with Langmuir, Freundlich and Temkin adsorption isotherm equations. Binary adsorption of Pb(II) and Ni(II) was studied on 1:10 and 1:15 fly ash samples as a function of initial metal ion concentration to investigate their efficiencies in removing metal ions from aqueous solution. In the case of 1:10 and 1:15 fly ash samples, Pb(II) and Ni(II) removal was observed to be almost the same. The significant difference observed between single and binary metal adsorption is in the initial metal removal rate. Slightly lower initial metal removal rate observed in the case of binary adsorption is probably due to the presence of competing metal ions in the solution. Fly ash based adsorbents prepared in the laboratory exhibited comparatively more adsorption capacity than that of the parent material, fly ash. Further, used adsorbents can be disposed in the form of concrete blocks as leaching of toxic metals is almost nil both under mild acidic and basic conditions. As cost involved in developing adsorbents from fly ash is comparatively less than other commercial adsorbents, reactivation or regeneration of used adsorbents was not considered.

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1. Introduction

Fly ash is the by-product of thermal power stations and is a waste material available in large quantities free of cost. Chemical and physical properties of fly ash differ according to the nature of the coal used in thermal power stations. Heavy metals like Zn, Pb, Cd, Ni and Cu have been removed from municipal solid waste leachate by fly ash [1] of thermal power plant. About 2 g/L of fly ash removed 39%, 28%, 74%, 42% and 71% of Cu, Mn, Pb, Zn and Cd respectively. Adsorption process of Pb(II) and Cu(II) on thermal power plant fly ash sample is exothermic and metal uptake increased on increasing pH of solution [2]. Adsorption of Cu, Ni, Zn, Cd, and Cr from aqueous solutions on dry and wet fly ash samples at non constant and constant pH values was carried out by Ricou et al. [3]. They categorized metals in two groups, hydrolysable and non hydrolysable on the basis of efficiency of fly ash to remove metal ions at different pH values. Hydrolysable metals like Pb(II), Cu(II) showed increased adsorption as they were adsorbed in free and hydrated forms than non hydrolysable metal cations like Ni and Zn. Wet fly ash sample

removed fewer amounts of metal ions due to its lost alkalinity. Effect of humic acid on the adsorption of heavy metals like Pb(II) and Cu(II) was investigated by Wang and Tade [4]. Humic acid presence increased Pb(II) adsorption from 18 mg/g to 37 mg/g and Cu(II) adsorption from 18 mg/g to 28 mg/g as humic acid provided additional active site for the adsorption of heavy metals. Bayat [5] investigated adsorption of Zn(II), Cu(II) and Ni(II) on two Turkish fly ash samples and observed increase in Zn(II) and Ni(II) adsorption with increasing initial metal concentration, while, Cu(II) uptake decreased on decreasing initial metal concentration. It was concluded that increase in CaO content of fly ash sample increased adsorption of heavy metal ions. Fly ash pellets were used by Papandreou et al. [6] to adsorb Cu(II) and Cd(II) from aqueous solution and they proposed a safe disposal method of metals loaded fly ash pellets. Fly ash pellets were used to make concrete blocks. Metal ion charge is neutralized by alkaline fly ash [7] and this phenomenon was considered responsible for the adsorption of Cd and Cu from aqueous solution. Tailor made fly ash samples were used to investigate the role of carbon and minerals in Cu(II) adsorption from aqueous solution and results supported the role of carbon in the adsorption of Cu(II) from aqueous solution. Adsorption increased with increasing carbon fraction in the sample at pH 5. In the presence of less carbon in fly ash, Cu(II) removal

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was attributed to its precipitation in the solution. A mixture of fly ash and tungsten trioxide was utilized [8] for the treatment of waste water containing cadmium and methylene blue and a mechanism based on simultaneous photo catalysis and adsorption for the removal of heavy metals and dye from the waste water was proposed. Zho and Haynes [9] examined various solid waste materials like fly ash and saw dust, for the adsorption of heavy metal cations and anions from aqueous solution. According to them it is the specific adsorption and surface precipitation which is responsible for the adsorption of cations and anions on inorganic adsorbents while, functional groups present on the surface of organic adsorbents were responsible for the adsorption of heavy metals. They also suggested the need for the development of low cost adsorbents by combining more than one waste material. Cu^{2+} , Ni^{2+} , Zn^{2+} , Cd^{2+} and Pb^{2+} removal from aqueous solution was achieved to maximum extent by utilizing fly ash and lime mixture at pH 5 [10]. Minocha et al. [11] stressed the need for considering improved adsorption capacity, cost of developing adsorbents and disposal of spent adsorbents in an environmental friendly way, while utilizing low cost adsorbents for waste treatments. Many workers developed varieties of low cost adsorbents from several industrial waste materials like blast furnace slag from steel industries [12], carbon slurry of fertilizer industries [13], red mud [14] etc. It appears from the above perusal of the literature that increased calcium content and the presence of phosphate groups in the adsorbent sample increase the extent of adsorption of heavy metals. The present study is undertaken to investigate the efficiency of thermal power plant fly ash sample, an industrial waste material by modifying it with a mixture of calcium carbonate and phosphoric acid in calculated amounts. This treatment increases the amount of calcium content in the adsorbent sample and introduces phosphate groups on the fly ash particles. Two types of fly ash based adsorbents with varying content of calcium and phosphate groups were prepared. Single and binary adsorption of heavy metals on these fly ash samples from aqueous solution was studied.

2. Materials and methods

2.1. Fly ash based adsorbents preparation

Two portions of 50 g of accurately weighed analar grade CaCO_3 were taken in two 2 L flasks separately and were dissolved in 100 mL of phosphoric acid. 500 g of fly ash sample was added to the first flask containing clear solution obtained by dissolving 50 g of analar grade calcium carbonate. Contents of the flask were stirred well and further heated in a microwave oven for 15 min at 220 °C. Solid material obtained was cooled, powdered and marked as 1:10 fly ash sample. 750 g of fly ash sample was added to the second flask and the same procedure mentioned in the case of 1:10 fly ash sample preparation was followed. Solid material obtained was labeled as 1:15 fly ash sample. These two fly ash sample based adsorbents were stored in air tight containers for further use.

2.2. Preparation of stock solution of heavy metals

All chemicals used in this study are of analytical grade and procured from Ranbaxy India Limited. Distilled water was used for all purposes unless specifically stated.

Nickel(II): The stock solution of 1000 mg of nickel was prepared by dissolving 4.478 g of nickel sulfate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$) in 1000 mL of demineralized water.

Lead(II): The stock solution of 1000 mg of lead was prepared by dissolving 1.598 g of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) in 1000 mL of demineralized water.

Chromium(VI): The stock solution of 1000 mg of chromium(VI) was prepared by dissolving 5.658 g of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in 1000 mL of demineralized water.

2.3. Analysis of adsorbates

Pb(II), Ni(II) and Cr(VI) were analyzed using an Atomic Absorption Spectrophotometer (ECIL, India) using standard procedures [15]. FTIR (Perkin Elmer) and XRF studies were carried out at Panjab University, Chandigarh.

2.4. Equilibrium adsorption studies

Equilibrium adsorption experiments were performed in duplicate by adding 0.1 g of adsorbent to labeled 250 mL reagent bottles. 50 mL aliquots of Cu(II), Ni(II), Pb(II), Cr(VI), Xylenol orange solutions of various initial concentrations (Co mg/L), and pH 5 ± 0.1 , were equilibrated at 25 ± 0.5 °C by shaking the capped bottles for 24 h on a horizontal bottle shaker at 100 rpm. Preliminary experiments had indicated that equilibrium could be reached within 24 h. At the end of this period, equilibrium concentration C_e of the metal ion in the supernatant liquid was determined by atomic absorption spectroscopy.

2.5. Initial metal concentration studies

Batch adsorption studies were carried out for different initial metal concentrations. Accurately weighed adsorbent samples were added to 50 mL of metal solutions taken in 250 mL stoppered conical flasks. The suspensions were stirred well for an hour. After an hour, the solutions were centrifuged and the clear solutions were analyzed for metals using AAS.

2.6. Contact time studies

50 mL of metal solution (20 mg/L) was taken in 250 mL reagent bottle and the solution was adjusted to pH 5 and 100 mg of fly ash was added to it. This adsorbent adsorbate slurry was stirred well and allowed to equilibrate in a thermo stated water bath for varying period of time interval. The temperature of the water bath was maintained at 25 ± 2 °C and the clear liquid was drawn after the stipulated time period and analyzed for Pb(II), Ni(II), Cr(VI) dye concentration with AAS. Experiment was carried out simultaneously for other time periods.

2.7. Adsorption dose studies

50, 100, 150 and 200 mg of adsorbent samples were added to 50 mL of pH adjusted metal solutions taken in 250 mL reagent bottles. Adsorbent adsorbate suspensions were kept in a thermo stated water bath cum shaker well beyond the equilibrium. Thereafter, clear supernatant solutions were drawn and analyzed for metal concentration with AAS.

2.8. pH studies

5 g of the adsorbent sample was taken in a 250 mL stoppered conical flask and 100 mL of CO_2 free demineralized water was added. The suspensions were shaken occasionally for 24 h. The pH of supernatants obtained by centrifugation was assayed using microprocessor based pH meter (Century model 931, India).

3. Results and discussion

FTIR peak was observed at 1087.9 cm^{-1} in the thermal power plant fly ash. Splitting and widening of this peak take place in 1:10 and 1:15 fly ash samples. This may be probably due to the introduction of phosphate groups on fly ash particle surface and the presence of phosphate compounds in the adsorbent samples. Three peaks observed in 1:10 fly ash sample in the wavelength range of 1235.6 – 969.5 cm^{-1} disappeared and a single peak appeared in Pb(II), Ni(II) and Cr(VI) loaded fly ash sample. This may be due to the formation of heavy metal phosphate oxygen bond. Fly ash samples were sieved

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