

Short Communication

Lead removal in fixed-bed columns by zeolite and sepiolite

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Abstract

The removal efficiency of zeolite (clinoptilolite) and sepiolite from lead containing aqueous solutions was investigated. A series of experiments were conducted in batch-wise and fixed-bed columns. Synthetic wastewaters containing lead (50 mg l^{-1}) and acetic acid (0.001 N) along with untreated and regenerated clinoptilolites and sepiolites were used in the adsorption studies. Batch tests were mainly conducted to isolate the magnitude of lead precipitation from real adsorption. Adsorption isotherms for both abstraction and adsorption were constructed. The removal of lead is found to be a sum of adsorption induced by ion exchange and precipitation of lead hydroxide. The breakthrough curves were obtained under different conditions by plotting the normalized effluent lead concentration (C/C_0) versus bed volume (BV). The ion exchange capacity of sepiolite and clinoptilolite for lead removal showed good performance up to approximately 100 and 120 BV where the C/C_0 remained below 0.1, respectively. The lead removal capacity of clinoptilolite bed from wastewater containing only lead yielded 45% higher performance compared to that of acetic acid partly due to a decrease in the effluent pH and consequently in precipitation. Also, the presence of acetic acid in the sepiolite column decreased the bed volumes treated by about 40%. Removal efficiency of lead–acetic system both in untreated clinoptilolite and sepiolite columns was found higher than that in regenerated columns.

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1. Introduction

Heavy metal ions accumulated in the receiving environment by natural and synthetic means are toxic. Industrial wastewaters are considered as the most important source of heavy metal pollution. Metal plating, automobile, and oil industries produce more heavy metal ions than other industries (Reed and Arunachalam, 1994).

Lead is often encountered in industrial wastewaters and one of the heavy metals that have been classified as priority pollutants by the US Environmental Protection Agency (USEPA). The national interim primary drinking water regulations gives the enforceable maximum contaminant level (MCL) of lead as 0.05 mg l^{-1} . Also the USEPA describes its proposed MCL as related to the optimal corrosion control. Although chemical precipitation is most economic for treatment of wastes with high lead concentrations, ion exchange and adsorption are widely used in the removal of lead at low concentrations (Reed and Arunachalam, 1994).

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Numerous investigators have studied lead removal from wastewater by adsorption using activated carbon in batch mode (Schindler et al., 1976; Wang, 1981; Corapcioglu, 1984; Netzer and Huges, 1984) and column reactor (Arulanantham et al., 1989; Reed and Arunachalam, 1994) using iron-containing industrial wastes as a low-cost adsorbent namely blast furnace sludge (Lopez et al., 1995), waste iron (III)/chromium (III) hydroxide (Namasivayam and Raganthan, 1995), and recycled iron material (Smith and Amini, 2000). Clinoptilolite is the most abundant natural zeolite. Removal by ion exchange particularly with zeolite is more effective when the metal species are cationic. Therefore, ion exchange methods using zeolite and sepiolite exhibit a significant potential for the removal of lead from wastewaters (Semmens and Martin, 1988; Bowman et al., 1995; Vaca-Mier et al., 2001). Mesoporous minerals, clinoptilolite and sepiolite, were also applied for ammonia removal (Sirkecioglu and Erdem-Senatalar, 1995; Celik et al., 2000, 2001; Turan et al., 2000) and color removal (Armagan et al., 2003a,b) from wastewaters.

This paper presents lead removal from wastewaters by clinoptilolite and sepiolite as a low-cost adsorbent. A series of adsorption experiments was conducted both in batch-wise and in ion-exchange columns. Adsorption isotherms are obtained to isolate the contribution of precipitation to adsorption. The breakthrough curves were constructed by plotting the effluent lead concentration normalized with respect to the influent lead concentration (C/C_0) versus bed volumes (BV).

2. Materials and methods

Clinoptilolite and sepiolite samples used in the experiments were received from Incal zeolite (Gordes, Turkey) and Mayas sepiolite (Sivrihisar, Turkey) companies, respectively. The sample was classified into different size groups: 4 mm, 2.8–4 mm, 2–2.8 mm, 1–2 mm. The chemical analyses of the clinoptilolite and sepiolite samples are given in Table 1. Since the sample is rich in Ca^{2+} and K^+ it was identified as Ca-clinoptilolite. The clinoptilolite sample contains 90.5–92.0% clinoptilolite, 4.2–5.0% smectite, 2.0–3.5% cristobalite and 1.0–1.3% mica. Gordes clinoptilolite has the following properties: cation exchange capacity 1.9–2.2 meq g^{-1} , pore diameter 4 Å, purity 92%, bed porosity 40%, density 2.15 g cm^{-3} , apparent density 1.30 g cm^{-3} , and suspension pH of 7.5–7.8 at 5% solids content. The X-ray diffraction and chemical analysis of sepiolite indicate that calcite and dolomite are the major impurities accompanying sepiolite.

2.1. Batch experiments

Adsorption experiments were conducted in 40-ml vials using particles in the size range of 1–2 mm. One

Table 1
Chemical analyses of Gordes clinoptilolite and Sivrihisar sepiolite

Constituent	Clinoptilolite (wt%)	Sepiolite (wt%)
SiO_2	70.00	51.93
CaO	2.50	0.12
K_2O	2.30	0.33
SO_3	0.01	–
Al_2O_3	14.00	1.52
MgO	1.15	24.20
TiO_2	0.05	0.08
P_2O_5	0.02	–
Fe_2O_3	0.75	0.70
Na_2O	0.20	0.12
LOI	9.02	21.00

gram of clinoptilolite (or sepiolite) sample was mixed with 20 ml lead solution of desired concentration at a solid-to-liquid ratio of 0.05 g ml^{-1} . The vials were shaken for 2 h on a shaker and centrifuged for 15 min. The supernatant was analyzed for the lead by atomic absorption spectrophotometer TJA Solutions model Solar 969. All analyses were made at ambient temperature (22.5 ± 1 °C).

Precipitation tests were conducted in the absence of mineral bed by introducing $\text{Pb}(\text{NO}_3)_2$ at a particular constant pH value. Lead nitrate undergoes above a certain pH value depending on the initial lead concentration. The precipitated lead at each pH value is collected as a separate phase by centrifugation followed by supernatant analysis for lead using AAS. The depleted amount of lead is indicative of the lead ion removal through precipitation.

2.2. Column reactor experiments

The laboratory scale experimental setup consists of a set of fixed ion exchange columns, wastewater and regeneration solution tanks, feed pumps, valves and treated water tank. The cylindrical Plexiglas column has a diameter of 3 cm and height of 100 cm. The particle size of clay mineral was 1–2 mm with a bed height of 50 cm and filling weight of 370 g. Aqueous solution made of $\text{Pb}(\text{NO}_3)_2$ was used. During the adsorption process, the samples were taken in 2 h periods and analyzed using AAS. Experiments were conducted with the synthetic wastewater containing only lead (50 mg l^{-1}) and lead (50 mg l^{-1})–acetic acid (0.001 N) to simulate an acidic wastewater, respectively. A peristaltic pump (Master flex 100) was used to feed the column at a filter rate of 6 m h^{-1} .

When the normalized effluent concentration (C/C_0) that is the ratio of the lead concentration in treated water to the influent lead concentration approached 1.0, the process was terminated. The breakthrough

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