Batch Studies of Heavy Metal Sorption Using Nano-Metal Oxides

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Abstract

Safe drinking water is paramount to human survival. Current treatments do not adequately remove all heavy metals from solution, are expensive, and use many resources. Metal oxide nanoparticles may be ideal sorbents for heavy metals due to their smaller size and increased surface area in comparison to bulk media. Heavy metal adsorption (Pb, Zn, Cd) to hematite (Fe_2O_3) and aluminum oxide (Al_2O_3) nanoparticles was examined as a function of pH for use as a contaminant removal substrate in water treatment technology. Batch sorption experiments were conducted with 0.1 and 0.5 g/L nanoparticles. Adsorption results showed metal pH-dependency with a Freundlich isotherm fit. Two-step desorption experiments using Pb and Cd indicated that Pb and Cd appeared to be irreversibly sorbed to the surface of hematite nanoparticles in the same solution conditions. Other experiments were conducted to determine the removal efficiency of multiple metals in solution by the nanoparticles. 0.5 g/L hematite nanoparticles simultaneously removed greater than 70% of Pb, Zn, Ni and Cd.

Introduction

Much of the world's population does not have adequate access to clean drinking water for basic survival needs. Demands are increasing for this resource as fresh water availability is decreasing due to extended drought, population growth, more stringent health-based regulations, and competitive user demands[1]. The existing water treatment infrastructures are in need of updating, are not one hundred percent efficient, and one treatment method does not work for all situations. Therein indicates the need for diversification of engineering technologies to treat drinking water issues. One of the biggest challenges is to create a simple, low-cost, environmentally-friendly and efficient method to remove contaminants, especially heavy metals, from potential potable waters. Levels of heavy metals found in rocks and soils are usually not cause for concern, but anthropogenic activities often disperse heavy metal pollution throughout the environment. Furthermore, problems associated with metal retrieval and purification from the environment may pose human health risks when these metal concentrations reach high levels[2]. Although the United States Environmental Protection Agency monitors water quality for some metal contaminants, acceptable levels may need to be reduced and more metals monitored as more studies on negative health effects arise[3,4].

Various treatments exist for removing some heavy metals from water: chemical precipitation, ultrafiltration, adsorption and ion exchange, reverse osmosis, electrodialysis, and alternative biological treatments[5-9]. The most commonly practiced methods include coagulationflocculation, membrane processes, and adsorption by bulk materials, though current methods require large amounts of bulk media and increasingly more intricate treatment configurations to produce potable water of acceptable quality. The main disadvantages of these methods include high cost, high volume of generated waste material, and little recovery of metals for reuse, so much research now focuses on use of low cost adsorbent materials or exchangers for water purification[10]. Much work exists demonstrating the ability of industry standard bulk crystal media (e.g., iron oxides, activated carbon, and aluminum oxides) to remove isolated contaminants from water, but they carry significant disadvantages: expensive reactivation, slow kinetics, lack of flexibility in design, and less than 100% removal capacity[11]. As the world's population continues to increase, more stringent demands will be placed on water treatment plants to supply safe drinking water to adequately protect this resource[12].

One possible solution for removing heavy metals from drinking water is the use of metal oxide nanoparticles as heavy metal adsorbents. Lab-based characterization studies for trace metal adsorption, such as lead onto iron oxide surfaces, are well studied[13,14]. Aluminum hydroxides are sometimes used as coagulants in drinking water treatment for removing organic matter and suspended particles[15]. Metal oxide nanoparticles (e.g., Al_2O_3 and Fe_2O_3) have roughly the same physical dimensions within each type and are among the most used nanoparticle type for research[16]. These nanomaterials fall within 1-100 nanometers in size and some of their physical properties (e.g., surface area) are enhanced as their volume is decreased in comparison to their bulk media counterparts[17], thus representing great potential as water-purification catalysts and redox active media[18]. Greater surface area may translate into much greater sorption capacities while using significantly less adsorbant amounts. Additional possible applications exist of being more cost effective in treatment processes with much less waste for disposal.

By focusing on surface area and its attributes to surface reactions (e.g., adsorption, desorption), natural and engineered nanoparticles may increase the overall environmental and cost effectiveness of water treatment strategies to remove heavy metals[19] by developing proper affinity, capacity, and selectivity for contaminants. Furthermore, although these studies use commercially available for convenience and uniformity of variables, comparable natural nanoparticles of these materials exist. However, reports of the use of natural nanoparticles in literature are extremely sparse in comparison to engineering nanoparticles and their potential uses for environmental applications.

In this work, heavy metal adsorption (Pb, Zn, Cd) to hematite (Fe_2O_3) and aluminum oxide (Al_2O_3) nanoparticles was examined as a function of pH for use as a contaminant removal substrate in water treatment technology. Batch sorption experiments were conducted with 0.1 and 0.5 g/L nanoparticles under pH conditions at 4.0, 6.0, and 8.0 as well as varying initial metal concentrations to determine pH-dependency and isotherm fit. Two-step desorption experiments were performed using Pb and Cd to determine possible irreversible desorption to the surface of hematite nanoparticles in the same solution conditions. Other experiments were conducted to determine the removal efficiency of multiple metals in solution by the nanoparticles.

Materials and Methods

Materials and mineral sorbents

Commercially prepared 33 nm hematite (Fe_2O_3) and < 50 nm aluminum oxide (Al_2O_3) nanoparticles from Sigma Aldrich (St. Louis, MO) were used. Sigma Aldrich reports the SSA of the hematite to be 35 m^2/g and the aluminum oxide to be 35-43 m^2/g. Future characterization studies will be completed to confirm size and SSA provided by Sigma Aldrich.

50 mg/L Pb (II), Cd (II), Ni (II), Zn (II), and Cu (II) stock solutions were prepared from $Pb(NO_3)_2$, $CdCl_2$, $NiCl_2$, $ZnSO_4$, and $Cu(NO_3)_2$ (Fisher Scientific, Houston, TX). Sample solutions were prepared by diluting the stock solutions to 10, 100, 500, 1000, in the buffered solutions. The electrolyte solution used in desorption experiments was 0.01 M $NaNO_3$. The pH adsorption experiments were conducted at 25°C and pH 4, 6, and 8 and desorption experiments at pH 8. All pH values were ±0.05. 0.01M $NaNO_3$ sodium acetate buffer solutions were used for pH 4 and 6. 0.01M $NaNO_3$ THAM buffer solutions (0.01 M tris(hydroxymethyl)aminomethane) were used for pH 8. Adjustments of pH solutions were accomplished using the following: 0.1M CH_3COOH/1N NaOH (pH 4 and 6), 6N HNO_3/1N NaOH (pH 8). Sterile plastic centrifuge containers were rinsed several times with nano-water before use. Surfactant-Free Cellulose Agent (SFCA) 0.45 µg syringe filters were used.

Water was prepared by treating reverse osmosis water with sand and carbon nanofiltration and UV, using a NANOpure Diamond Barnstead D3750 with Hollow Fibre Filter, Gamma Irradiated pore size rating 0.2 um and maximum operating pressure of 50 psi (3.4 kg/cm^2).

A Labnet MinilabRoller was used to rotate the samples at 24 rpm for one 24 hour period during the sorption experiments. The HACH sensION pH meter was used to measure the pH of buffer solutions and samples.

Heavy metal concentrations were measured on a Perkin Elmer DRC-e ICP-MS having a quadrupole mass spectrometer. The plasma is Argon gas with optimized parameter operating conditions for nebulizer gas flow of 0.88 L/min, a lens voltage of 6.5V, and RF power of 1000 watts. The heavy metal standards were prepared by making 1 g/L standard stock solutions. Then each stock solution was diluted to the desired calibration standard concentrations of 1, 10, and 100µg/L. The calibration standards were acidified with 1% HNO_3 by volume. The correlation coefficients were generally 0.999 or better, and the relative standard deviation of three replicate analyses was always below 5%. The intensity of the background was taken into account, with estimated values being significantly lower than the intensity of the 1 µg/L standard (about 100 times greater than the background intensity).

Sorption Isotherms

Adsorption experiments for the metal of interest (Pb, Cd, Zn) on nanoparticles (Fe_2O_3, Al_2O_3) were performed as batch experiments for pH 4.0, 6.0, and 8.0. Metal concentrations ranged from 10 to 1000 µg/L in a background 0.01M $NaNO_3$ electrolyte. Concentrations of nanoparticles were 0.1 g/L and 0.5g/L as Fe_2O_3 Al_2O_3. The concentrations for the metal of interest were added to plastic centrifuge containers and filled to 50 mL with appropriate buffer solution. To each series of concentrations in their respective containers, 0.1 or 0.5 g/L metal oxide nanoparticles was added. The containers were sealed, placed in a tumbler, and rotated for 24 hours. Samples were taken, filtered using a 0.45 um Nalgene SCFA filter, and acidified with 1% HNO_3 for ICP-

MS analysis. The pH of each sample was monitored at the end of each experiment to verify any negligible changes.

Multi-Element Adsorption

Batch adsorption experiments for multiple elements (Pb, Cu, Ni, Cd, Zn) on nanoparticles (Fe_2O_3, Al_2O_3) were performed as batch experiments at pH 8.0 using 500 µg/L for each metal concentration in a background 0.01M $NaNO_3$ electrolyte. Concentrations of nanoparticles were 0.1 g/L and 0.5g/L as Fe_2O_3 Al_2O_3. Metal concentrations were added to plastic centrifuge containers and filled to 50 mL with appropriate buffer solution. To each multi-element solution, 0.1 or 0.5 g/L metal oxide nanoparticles were added. The containers were sealed, placed in a tumbler, and rotated for 24 hours. The pH was verified, and samples were taken, filtered using a 0.45 um Nalgene SCFA filter, and acidified with 1% HNO_3 for ICP-MS analysis.

Desorption

Twenty-four hour batch adsorption experiments at pH 8.0 were repeated for 500 µg/L initial concentrations of Pb(II) and Cd(II) using the 33 nm hematite (Fe_2O_3) at 0.5g/L and the previously described procedure in order to prepare samples for desorption experiments. Desorption was performed by adding metal-free 0.01M $NaNO_3$ electrolyte to the aliquot, following a 24-hour adsorption. Samples were allowed to react for 24 hours at 24 rpm on a Labnet MiniLabRoller. The sample was then centrifuged at 5000 rpm for 15 minutes and the supernatant decanted into another container to determine the amount of metal desorbed. The pH for each sample was evaluated immediately to verify any negligible changes. A portion of each sample was acidified with 1% HNO_3 for ICP-MS analysis. For each adsorption sample, two steps of desorption were performed.

Results and Discussion

Adsorption isotherms

All adsorption data were fitted with the Fruendlich isotherm which provided the best fit for the data overall for heterogeneous adsorption to the surface of the nanoparticles solids [Eq. (1), solid lines in Fig. 1, 2]:

$$q = K_F C^{1/n} \tag{1}$$

where q is the adsorbate mass per adsorbent unit mass at equilibrium; K_F is the adsorbent capacity measure; C is the aqueous adsorbate concentration; and n is a measure of how adsorbate affinity changes with adsorption density changes[20]. Tables 1 and 2 list the isotherm parameters for experimental adsorption data at 4.0, 6.0, and 8.0 pH for Fe_2O_3 and Al_2O_3, respectively. Overall the correlation coefficients for the isotherm fit are good (r= >0.96) except for Pb at pH 6 suggesting adsorption is not heterogeneous to the surface and that other isotherms might describe the data better.

Figure 1a, b, and c shows the adsorption to hematite nanoparticles at pH 4.0, 6.0, and 8.0 for Pb(II), Zn(II) and Cd(II) respectively and the respective isotherm fit. The adsorption of Pb(II), Cd(II), and Zn(II) to hematite nanoparticles was pH dependent (Fig. 1). Higher adsorption capacities were observed at pH 8. According to the literature[21,22], the surface of α-Fe_2O_3 is negatively charged above pH 8 and thereby attracts the metal cations. Conversely, below this pH value the surface becomes positively charged, thus repelling the metal cations of interest which means less adsorption occurs. Zn has a larger adsorption capacity than Pb and Cd at pH 8; however, Pb has a higher affinity for the hematite since more is adsorbed from solution (~100%) at pH 8.

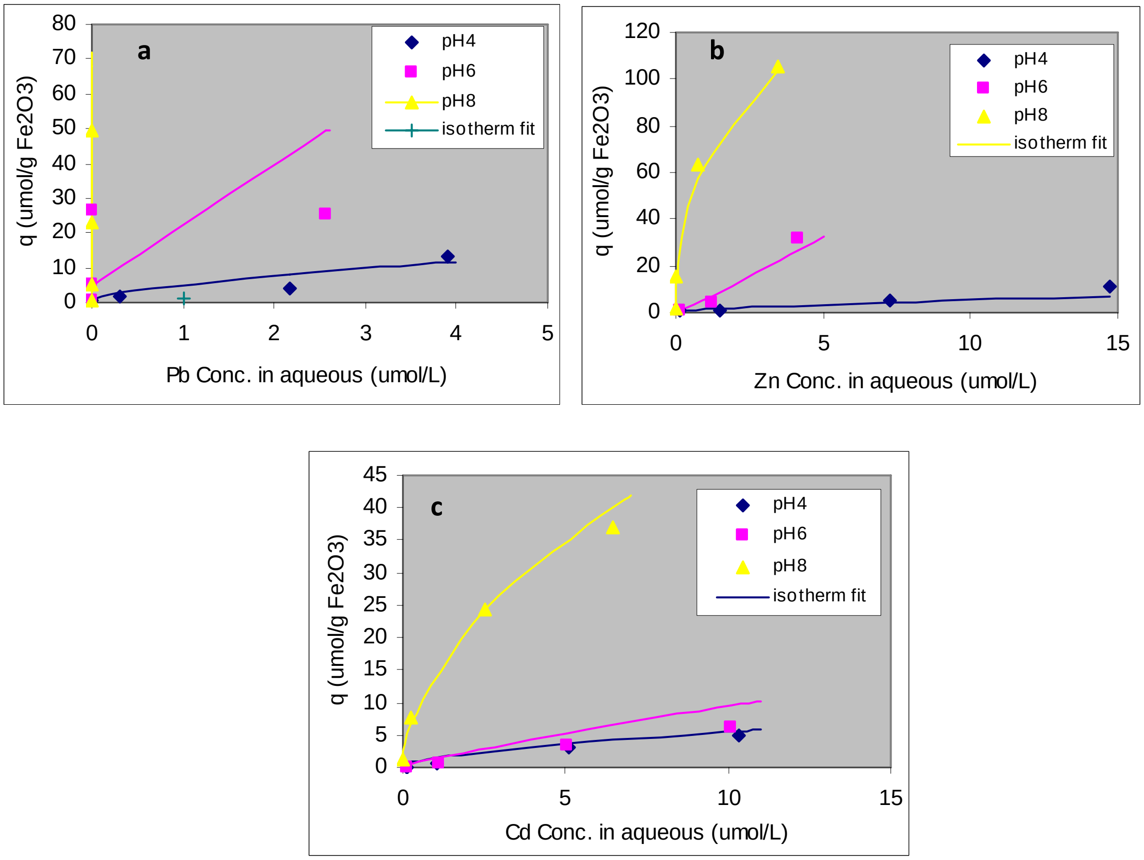

FIG. 1. Adsorption to 33 nm hematite at pH 4.0, 6.0, and 8.0 for Pb(II) (a), Zn(II) (b), and Cd(II) (c). The solid lines are drawn using the curve-fitted isotherm data in Table 1.

TABLE 1. Fruendlich adsorption isotherm parameters for Pb(II), Cd(II), and Zn(II) onto 33 nm hematite at pH 4.0, 6.0, and 8.0.

Metal	pH	K_F (L/g)	n	r
Pb	4.0	5.43	1.87	0.98
	6.0	46.20	14.92	0.84
	8.0	1.49e26	0.82	0.98
Zn	4.0	1.51	1.76	0.96
	6.0	64.79	2.63	0.99
	8.0	1.48	1.74	0.99
Cd	4.0	1.48	1.74	0.96
	6.0	1.31	1.16	0.99
	8.0	14.76	1.87	0.99

Figure 2 [a, b, and c] shows the adsorption to aluminum oxide nanoparticles at pH 4.0, 6.0, and 8.0 for Pb(II), Zn(II) and Cd(II) respectively and associated isotherm fit. The adsorption of Pb(II), Cd(II), and Zn(II) to aluminum oxide nanoparticles was also pH dependent (Fig. 2) as seen with hematite. Higher adsorption capacities were observed at pH 8 for Pb and Cd while for Zn similar adsorption was observed at pH 6 and pH 8. According to the literature[23,24] for compositionally pure Al_2O_3, the surface of aluminum oxide becomes negatively charged between pH 8 and 10 which might be why similar adsorption was seen for Zn at pH 6 and pH 8 since the surface still has some positive charges. At pH 8 both Zn and Pb had similar adsorption capacities to the aluminum oxide nanoparticles but Pb had the higher affinity with about 70% removed from solution as the concentration increased. Zn results showed an inverse relationship between increasing concentration and amount of metal removed from solution.

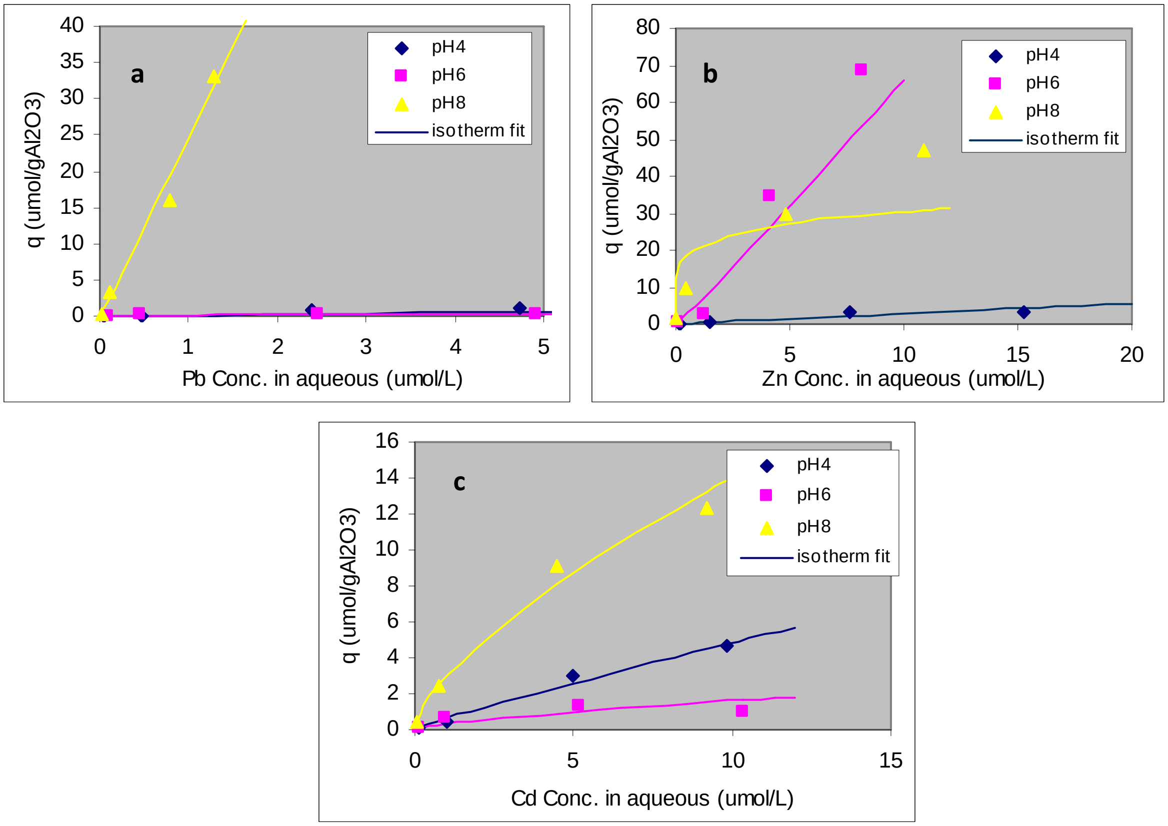

FIG. 2. Adsorption to <50 nm aluminum oxide at pH 4.0, 6.0, and 8.0 for Pb(II) (a), Zn(II) (b), and Cd(II) (c). The solid curves are drawn using the curve-fitted isotherm data in Table 2.

TABLE 2. Fruendlich adsorption isotherm parameters for Pb(II), Zn(II), and Cd(II) onto <50 nm aluminum oxide at pH 4.0, 6.0, and 8.0.

Metal	pH	K_F (L/g)	n	r
Pb	4.0	0.10	0.87	0.98
	6.0	0.15	1.56	0.89
	8.0	24.54	0.99	0.99
Zn	4.0	0.39	1.15	0.99
	6.0	5.83	0.95	0.98

	8.0	20.89	6.23	0.97
Cd	4.0	0.63	1.13	0.99
	6.0	0.36	1.53	0.96
	8.0	2.98	1.49	0.99

Figure 3 compares the surface area normalized adsorption capacities for hematite and aluminum oxide nanoparticles for Pb, Zn, and Cd. The adsorption capacities for all the metals are larger for the hematite nanoparticles than the aluminum oxide nanoparticles. For example, the adsorption capacity with Zn for hematite nanoparticles (3.0 μmol/m^2) is about two times larger than for the aluminum oxide nanoparticles (1.3 μmol/m^2). The adsorption capacities of the hematite and aluminum oxide nanoparticles are largest with Zn, followed by Pb and then Cd. These results suggest that hematite nanoparticles have more potential to remove larger amounts of metals from solution than aluminum oxide nanoparticles due to their larger adsorption capacities.

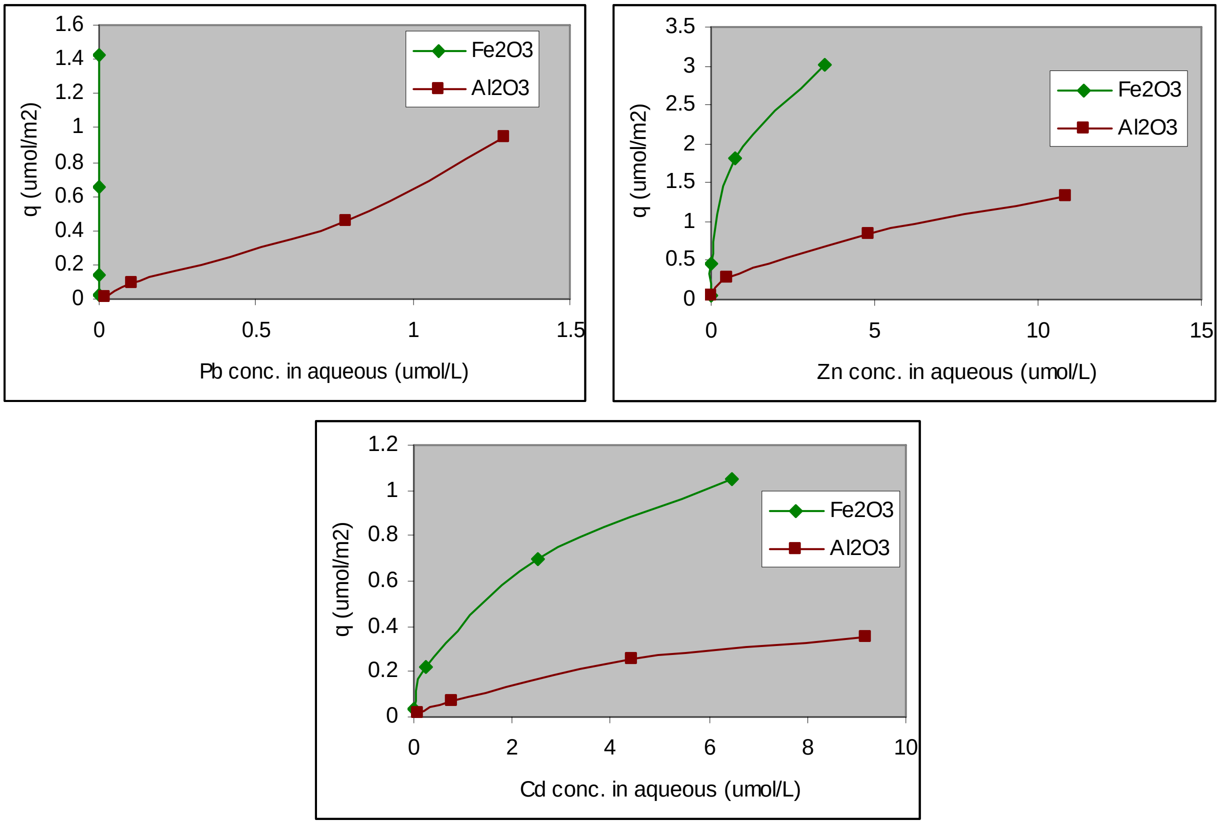

FIG. 3. Comparison of adsorption capacities (μmol/m^2) of hematite (Fe_2O_3) and aluminum oxide (Al_2O_3) nanoparticles versus the amount of Pb, Zn, or Cd in aqueous (μmol/L) at pH 8.

Competitive Adsorption

Table 3 shows results of multi–metal adsorption to 0.1 g/L and 0.5g/L hematite and aluminum oxide nanoparticles at pH 8.0 and 500 μg/L of each metal of interest. Overall, 0.5g/L of both nanoparticles showed higher percentage of metals adsorbed due to more available surface area. Hematite appears to be more effective at adsorbing the metals than aluminum oxide, since the

0.1g/L Fe_2O_3 adsorption values are greater than the 0.5g/L Al_2O_3 values, with the exception of zinc (70.2% to 83.0%, respectively). This may suggest that Zn has a stronger affinity to aluminum oxide nanoparticles but due to their smaller adsorption capacities, less Zn is adsorbed than with the hematite nanoparticles. Both nanoparticles have similar adsorption affinities (Pb > Zn > Ni ≥ Cu=Cd) when multiple metals are in solution. These results show the potential of hematite nanoparticles to be used to simultaneously remove multiple metals from solution.

TABLE 3.Multi-element metal adsorption to 0.1g/L and 0.5g/L Fe_2O_3 and Al_2O_3 nanoparticles at pH 8.0 and 500 µg/L for each contaminant.

Metal Contaminant	Percent Metal Adsorbed			
	Fe_2O_3		Al_2O_3	
	0.1 g/L	0.5 g/L	0.1 g/L	0.5 g/L
Pb (II)	98.7	100.0	59.7	94.0
Zn (II)	70.2	100.0	35.8	83.0
Ni (II)	37.4	100.0	21.2	32.4
Cd (II)	4.9	71.9	0	2.0
Cu (II)	8.3	65.5	0	0.1

Desorption

Desorption experiments were performed on hematite nanoparticles to evaluate the reversibility of the adsorption processes for Pb (II) and Cd (II). The remaining metals and aluminum oxide nanoparticles were not tested for desorption due to time limitations. The two-step desorption experiments concluded these metals were irreversibly sorbed to hematite surfaces at pH 8 due to their negligible release of Pb and Cd to solution after each 24 hour desorption step (Table 4). The hematite nanoparticles offer unique advantages because of their strong sorption due to increased surface area and negligible bleed-off at common environmental pH conditions due to desorption hysteresis. Both strong adsorption and resistant desorption and bleed-off are a significant advantage in water treatment and solid waste disposal.

TABLE 4. Two-step 24-hour desorption results for 500 µg/L Pb(II) and Cd(II) using 0.5 g/L 33 nm hematite nanoparticles at pH 8.0.

Metal Contaminant	Percent Adsorbed	Desorption 1: Percent Desorbed	Desorption 2: Percent Desorbed
Pb (II)	100	0.03	0.01
Cd (II)	98.17	1.31	1.35

Conclusion

Metal adsorption to hematite and aluminum oxide nanoparticles is strongly dependent on pH with more adsorption occurring at pH 8. When comparing the two nanoparticles, hematite has larger adsorption capacities than aluminum oxide nanoparticles suggesting hematite may be the better sorbent. The competitive adsorption results showed hematite adsorbs higher percentages of metals than aluminum oxide with a general adsorption trend of Pb > Zn > Ni ≥ Cu=Cd. Desorption results suggest Pb and Cd may be irreversibly sorbed to hematite nanoparticles at normal environmental conditions, but more work is needed to confirm this conclusion as well as exploration using other metals. These results demonstrate the ability of the hematite and aluminum oxide nanoparticles to remove Pb, Zn, and Cd from solution. The increased surface area of these nanoparticles, coupled with the strong adsorption at pH 6 and 8 (which is representative of many natural waters), offers a potential alternative method of removal for metals from water intended for drinking. This method may be comparable or even a more viable option than bulk materials, especially when other factors (e.g., volume of adsorbent needed, waste generated, and cost) are considered.

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