Environmental Health Engineering and Management Journal 2016, 3(1), 47–53

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Environmental Health
Engineering and
Management Journal

Original Article

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Kinetic study of heavy metal ions removal from aqueous solutions using activated pumice stone

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Abstract

Background: The presence of heavy metals in aquatic and terrestrial ecosystems causes harmful effects to living organisms in the environment. This research aimed to determine the potential of activated pumice stone (APS) as a sorbent for the removal of Cd, Cu, and Zn from wastewater.

Methods: This research was conducted during 2013 on a laboratory scale. The study was performed using batch experiments with synthetic wastewater having Cd, Cu, and Zn concentrations of 10, 20, 40, and 80 mg/L. Various isotherm models, including Langmuir and Freundlich, were used to evaluate the sorption data. The influence of contact time and amount of sorbent on the removal of Cd, Cu, and Zn from wastewater was studied. All experiments were done at pH = 7 and at room temperature (20 ± 1°C). The solution pH was adjusted using 1N NaOH or 1N HCl solutions, and the pH value was determined by a pH meter.

Results: The results showed that the adsorption of Cd, Cu, and Zn approaches equilibrium after about 2 hours, while the rates of removal efficiency for Cd, Cu, and Zn at equilibrium were 91.6%, 91.8%, and 82.9%, respectively. Kinetic studies showed that the sorption of Cd, Cu, and Zn onto APS were fitted to the pseudo-second order kinetic models.

Conclusion: The results indicate that the APS is a good choice, because it is a low-cost and effective sorbent. The sorption capacity of APS as a sorbent was considerably affected by the initial concentration of metal ions in the solution and by contact time.

Keywords: Adsorption, Aqueous solution, Environment, Heavy metals, Pumice.

Citation: Babakhani N, Reyahi-Khoram M, Sobhanardakani S. Kinetic study of heavy metal ions removal from aqueous solutions using activated pumice stone. Environmental Health Engineering and Management Journal 2016; 3(1): 47–53.

Article History:
Received: 22 January 2016
Accepted: 5 March 2016
ePublished: 17 March 2016

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Introduction

Heavy metal ions such as zinc, lead, mercury, chromium, nickel, copper, and cadmium are dangerous materials, non-biodegradable, and can be toxic and carcinogenic, even at very low levels. Hence, they usually cause a serious threat to the environment (1). Heavy metals are generated from various industries, including but not exclusive to metal plastering, paint manufacturing, textile, steel fabrication, storage batteries alloy industries, fertilizers, dyeing, and other chemical industries (2). Cadmium is extensively used and highly toxic in relatively low doses. It is one of the most toxic heavy metals causing renal disorder, kidney damage, high blood pressure, destruction of red blood cells, and bone fracture. Human beings have reported nausea and vomiting at a cadmium level of 15 mg/L (3). Copper is known to be one of the heavy metals most toxic to living organisms, and it is one of the more widespread heavy metal contaminants of the environment. The main sources of copper in industry include plating baths, metal cleaning, pulp, paper board mills, wood pulp production, fertilizer industry, paints and pigment (4).

Zinc is one of the most commonly occurring elements in the earth's crust and an essential micronutrient for all living things. Zn has a multitude of industrial uses; the major industrial use of Zn is as a corrosion resistant coating for Fe and steel. It is used in dry cell batteries, mixed with other metals to form alloys, and used in the manufacture of products such as ceramics, paints, rubber, wood preservatives, dyes and fertilizers, dietary foods, drugs, and other medicinal preparations. Exposure to Zn in large concentrations is highly toxic to living organisms. In humans, it can cause a range of serious ailments including damage to the pancreas, decreased immune functions, growth retardation, anemia, metal fume fever, birth defects, and cancer (5).

Common methods for the removal of heavy metals from water environments include chemical precipitation coagulation, oxidation, reduction, solvent extraction, electrolytic processes, membrane separation, ion exchange, reverse osmosis, membrane filtration, ultra-filtration, biological systems, and adsorption. Among these methods, adsorption is most highly recommended because it is effective and economical (1,2,4). The adsorption process has been identified as an efficient, cheap, and promising technology in wastewater treatment for the removal of metal ions (6). Several authors have reported on various adsorbents. A number of adsorbent materials, such as zeolites, activated carbon, tea factory waste, amorphous iron oxide, kaolinite, phenolated wood resin, modified cellulose, mesoporous silica, egg shell, hydrogle, protein granules produced, cationic resin Purolite, nano-materials, bentonite, and modified jute, have been used to remove heavy metals from wastewaters (7-13).

Pumice is a porous igneous rock; it is formed during massive volcanic eruptions when fluid lava is emitted into the air as a set containing mass of gas bubbles (14). Pumice has been used in ceramic, cement, brick, and concrete industries as an additive and coating material (15).

In the last two decades, many researchers have used pumice for the removal of fluoride (16), nitrates and nitrites (17), disinfection by-products (18), phosphorous ions (19), organic matter (20), pathogens (21), arsenic (22), azo dye (22), and p-chloronitrobenzene (23). In Iran, many studies have focused on the use of pumice for the adsorption of metal ions and other solutes (14,16,19,20), but not much research related to activated pumice stone (APS) absorption has been done. Access to sources of pumice, its lower costs, its availability in Iran, and innovation are the motivations behind this research, which aimed to determine the adsorption capacity of APS for removing (Zn), (Cu), and (Zn)ions from aqueous solutions through a batch process.

Methods

This study was carried out in the laboratory of Kermanshah Provincial Directorate of Environmental Protection during 2013. First, a pre-test was performed to identify possible issues and problems for this study and to determine optimum pH. Based on the pre-test results, the optimum pH was found to be pH 7.

Preparation of sorbent

The sorbent used in this study was made in the lab from natural sources. Pumice stone was obtained from Ghorveh township of Kurdistan province in the western part of Iran. To remove contaminants, the pumice was washed thoroughly with double-distilled water until the wash water ran free from color and turbidity. Subsequently, the prepared sorbent was kept in an oven at 110 ± 1°C for 6 hours and was then transferred to a furnace and held at a temperature of 550°C for one hour. In the next stage, the activated sample was crushed with laboratory-scale crushers and granulated using standard ASTM sieves with a mesh size of 20 (0.85 mm) and an effective size of 2 mm. Finally, the sorbent was rinsed with distilled water and kept at 110 ± 1°C for 14 hours until it was completely dried.

Preparation of chemical agents

All employed chemicals were of the purest grade. Cd, Cu, and Zn in granular form were provided by Merck Co. (Germany).The stock solutions (1000 mg/L) of each element were prepared by dissolving an adequate quantity of Cd, Cu, and Zn powder in double-distilled water. Then, different working concentrations (10, 20, 40, and 80 mg/L) for each metal were prepared by sufficiently diluting stock solutions.

Sorption experiments

This research was performed on a laboratory scale. Erlenmeyer flasks with 250 mL were used as a simple batch reactor; initial concentrations of Cd, Cu, and Zn solutions were selected at four different values (10, 20, 40, and 80 mg/L), and 2, 4, 6, and 8 g of APS sorbent were added to the batch reactor.

Erlenmeyer flasks were filled with 200 mL of solution. All experiments were done at pH = 7 and at room temperature (20 ± 1°C). The solution pH was adjusted using 1N NaOH or 1N HCl solutions; pH values were determined by a pH meter. The mixture was agitated on a shaker at a speed of 200 rpm. In order to ensure reliability and reproducibility, all experiments were performed in duplicate, and the results are reported as averages. Sorption studies were also carried out at different contact times (10, 20, 40, 80, 120, and 180 minutes) to determine the effect of contact time on the removal of heavy metals (Cd, Zn, and Cu) from aqueous solutions.

After each sorption experiment, the mixture was centrifuged at 200 rpm for 5 minutes and the supernatant fluid was decanted. Then, the final concentrations of metal ions in the samples were determined by ICP (Varian ES-710) in ppb.

All analyses were performed according to the procedures outlined in standard methods (24). Excel software was used to input, analyze, and graphically display the results. The effects of contact time, initial metal ion concentration, and sorbent dosage on sorption efficiency were investigated. Pseudo-first order and pseudo-second order kinetic models were used to represent the kinetics or rate of the process and obtain the main kinetic parameters. Common linear isotherms, such as the Langmuir and Freundlich isotherms, are usually used by researchers to study the adsorption mechanism (7-10,22); here, they were assayed for their ability to fit the sorption data. The quantity of metal ions sorbed per gram of sorbent was fulfilled according to the equation below (25):

$$q_t = \frac{(c_1 - c_t)}{m}.v \qquad (1)$$

where, q_t is the mass of metal ion sorbed as mg metal/g of the sorbent at time t; c_1 is the initial concentration of metal in the solution in mg/L; c_t is the equilibrium concentration of the metal in the solution; m is the amount of the sorbent sample on a dry basis in g, and v, the liquid volume in L. The proportion of metal ions decreased from the aqueous solution was determined using the following equation (25):

$$R = \frac{(c_1 - c_t)}{C_1}.100 \qquad (2)$$

Results

Effect of contact time

Contact time is an important factor in evaluating sorption efficiency. The removal rate of Cd, Cu, and Zn from solution is shown in Figure 1, from which it can be seen that the adsorption of Cd, Cu, and Zn approached equilibrium after about two hours, while removal efficiency rates for Cd, Cu, and Zn at equilibrium were 91.6%, 91.8%, and 82.9%, respectively. Initially, the uptake rate was higher, because all sites on the APS were vacant and the concentration was high; however, the decrease in sorption sites reduced the uptake rate. As shown in Figure 1, considering technical and economic aspects, a contact time of 120 minutes was chosen for Cd, Cu, and Zn removal from aqueous solutions by sorption onto APS.

Effect of the sorbent dosage

Adsorption capacity is highly dependent on the amount or concentration of the sorbent. The effect of sorbent dosage on the removal of Cd, Cu, and Zn from aqueous solutions was studied using batch sorption experiments. Experiments were conducted by contacting known amounts of the adsorbent with sorbent concentrations varying from 10 to 40 g/L. The results are presented in Figure 2. It is apparent that the amount of metal ions sorbed increased with increasing concentrations of sorbent. This is an expected result, because as the amount of sorbent increased, the available surface area increased, thereby exposing more active sites for the binding of metal ions. A similar trend was observed for the effect of sorbent concentration and is discussed in the literature (26).

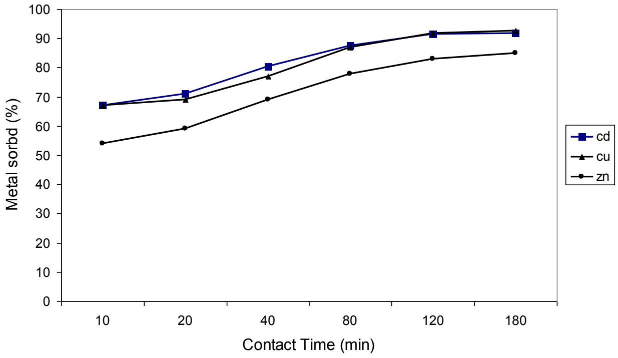

Figure 1. Effect of contact time on the sorption of Cd, Cu, and Zn onto APS. Conditions: 10 g/L APS, pH 7, 200 mL 10 mg/L metal ions solution, temperature = 20 ± 1°C, agitation speed = 200 rpm.

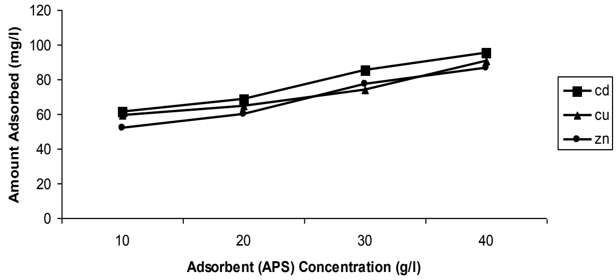

Figure 2. Effect of sorbent concentration on the sorption of Cd, Cu, and Zn onto APS. Conditions: pH = 7, 200 mL 40 mg/L metal ions solution, contact time = 120 min, temperature = 20 ± 1°C, agitation speed = 200 rpm.

Sorption isotherm studies

Several mathematical relationships have been developed to describe the equilibrium distribution of solute between the solid and liquid phases and thus aid in the interpretation of adsorption data. These relationships apply when sorption tests are conducted at a constant temperature and are referred to as sorption isotherms (27). Analytical isotherm equations, such as Langmuir and Freundlich isotherms, are widely used for modeling sorption data (28). In this research, sorption isotherms were obtained from the previous data using four different concentrations of APS and under constant pH conditions for the solution (pH = 7).

Langmuir equation

The Langmuir equation is generally written as (27):

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{b.q_m}\left(\frac{1}{c_e}\right) \qquad (3)$$

where, C_e is the equilibrium concentration of sorbent (mg/L), q_m is the maximum sorption capacity of the adsorbent (mg/g), b is the Langmuir constant, and q_e is the equilibrium sorption capacity of the sorbent in mg metal/g sorbent. The plots of specific sorption ($1/q_e$) against equilibrium concentration ($1/C_e$) gave the linear isotherm parameters of q_m and b. These coefficients are presented in Table 1. On this basis, the monolayer (maximum) adsorption capacities (q_m) were found to be 3.01, 2.62, and 2.53 mg/g for Cd, Cu, and Zn, respectively. The basic factor of the Langmuir adsorption isotherm can be obtained from the analysis of a distinctive feature commonly known as R_L and described as (22):

$$R_L = \frac{1}{1 + b.c_0} \qquad (4)$$

where, C_0 is the initial concentration and R_L indicates the confirmation of the Freundlich adsorption isotherm ($R_L>1$, unfavorable, $R_L = 1$ linear, $0<R_L<1$, favorable, $R_L<0$

Table 1. Langmuir isotherm parameters for adsorption of Cd, Cu, and Zn from aqueous solution on APS adsorbent

Element	q_m (mg/g)	b (L/mg)	R^2
Cu	2.616	0.739	0.987
Zn	2.526	0.347	0.993
Cd	3.008	1.141	0.996

irreversible). The Langmuir isotherm model is based on the assumption of homogenous surface energy distribution (22).

For the present study, the values of R_L are shown in Table 2 and indicate that sorption intensity for Cd, Cu, and Zn is most favorable.

It can be observed from Figure 3 that the isotherm data fits the Langmuir equation well as it is a straight line. The values of q_m and *b* were determined from the slope and the intercept of the plot of $1/q_e$ versus $1/C_e$ (Figure 3) and are reported in Table 1.

The Langmuir isotherm yielded a better fit to the experimental data for sorbent, probably because of the homogenous natures of their surface sites involved in the uptake of cadmium, copper, and zinc.

Freundlich equation

Freundlich developed an empirical equation that is used mainly to describe the results of adsorption isotherm experiments. One form of the equation is:

$$q_e = K_f C^{1/n} \qquad (5)$$

In this equation, *qe* is the mass of solute adsorbed per mass of absorbent, mg/g; C is the equilibrium concentration of solute concentration after sorption is complete (mg/L); *Kf* and *n* are Freundlich adsorption parameters that must be determined for each temperature and solute (27).

The log form of the Freundlich equation is:

$$\log q_e = \log K_f + \frac{1}{n}\log C \qquad (6)$$

The Freundlich model is an empirical and suitable isotherm model for nonhomogenous systems (22). A plot of log q_e against log C_e yielding a straight line was made to confirm the model. The values of K_f and *1/n* were obtained from the intercept and slope of the straight line.

In this research, the experimental data from the sorption study of the three metal ions on APS were plotted logarithmically (Figure 4) using the linear Freundlich isotherm equation.

The linear Freundlich isotherm constants obtained for the sorption of Cd, Cu, and Zn onto APS are presented in Table 3. The Freundlich isotherm parameter *1/n* measures the adsorption intensity of metal ions on sorbent. In this research, the values of the constant *1/n* were found to be 0.291, 0.259, and 0.258 mg/g for Cd, Cu, and Zn, respectively. The value of *1/n* is less than 1, indicating the favorable adsorption of Cd, Cu, and Zn, probably due to the ability of the APS to remove these three metal ions from aqueous solutions (3).

Table 2. Dimensionless Langmuir parameter R_L for Cd, Cu, and Zn sorption onto APS

Concentration mg/L	Cd	Cu	Zn
10	0.08	0.12	0.22
20	0.04	0.06	0.12
40	0.02	0.03	0.07
80	0.01	0.02	0.03

Table 3. Freundlich isotherm parameters

Element	K_f	N	1/n	R^2
Cu	1.108	3.862	0.259	0.984
Zn	0.912	3.866	0.258	0.855
Cd	1.101	3.433	0.291	0.941

Sorption kinetic studies

Kinetic studies are important in determining the sorption capacity of the sorbent. Most sorption (or desorption) processes of the solid phases are time dependent (29). To study the control and operation of sorption processes such as chemical reaction and mass transfer, various kinetic models were applied to the experimental data obtained from sorption processes.

Pseudo-first order kinetic model:

The pseudo-first order is one of the most commonly used kinetic models for describing the adsorption mechanism (22). It is expressed as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \qquad (7)$$

In this equation, q_e and q_t represent the sorption capacity of sorbent material (mg/g) at equilibrium and at time (min), respectively, and k_1 is the rate constant of the model (1/min).

The straight line plot of log ($q_e - q_t$) against time (t) should give a linear relationship, and in this situation, k_1 and q_e

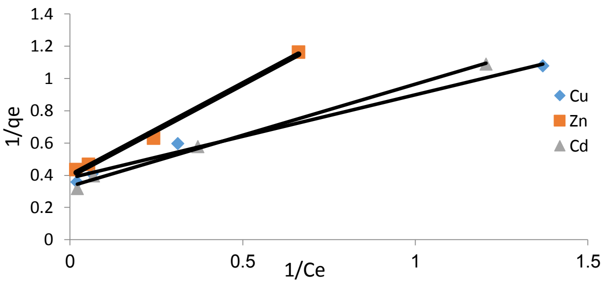

Figure 3. Fit of experimental data to Langmuir isotherm model.

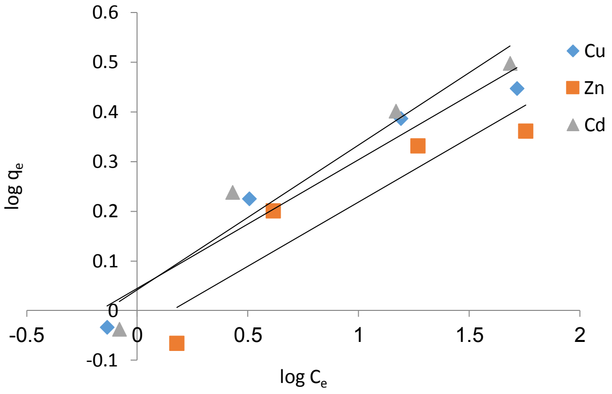

Figure 4. Fit of experimental data to Freundlich isotherm model.

can be calculated from the slope and intercept, respectively. The pseudo-first order kinetic constant k_1 and the correlation coefficients for Cd, Cu, and Zn adsorption were calculated from the linear plots of log (q_e-q_t) versus t (Figure 5) and are listed in Table 4. The correlation coefficients for the pseudo-first order kinetic model are at below 0.9. As observed in Figure 5, the pseudo-first order model gives a good description of the sorption. The experimental kinetic data for Cd, Cu, and Zn onto APS were fitted to the pseudo-first order kinetic model, and its expressions are shown in Table 4.

Pseudo-second order kinetic model

The pseudo-second order equation has many advantages and is one of the best models to describe adsorption behavior (30). The pseudo-second order equation is represented as shown in Equation 8:

$$\frac{t}{q_t}=\frac{1}{k_2q_e^2}+\frac{1}{q_e}t \qquad (8)$$

where k_2 is the constant rate of pseudo-second order kinetic (g/(mg.min)), and q_e and q_t are defined in the preceding paragraph.

Also:

$$h=k_2q^2{}_e \qquad (9)$$

where h can be regarded as the initial sorption rate since q_t/t, when t approaches 0 (30).

In the present study, the pseudo-second order model showed the best fit to the experimental data related to the adsorption of Cd, Cu, and Zn onto APS with the highest squared correlation coefficient (Figure 6).

The pseudo-second order kinetic rate constant (k_2) and the theoretical q_e can be calculated from the plots of t/qt versus t as shown in Figure 6.

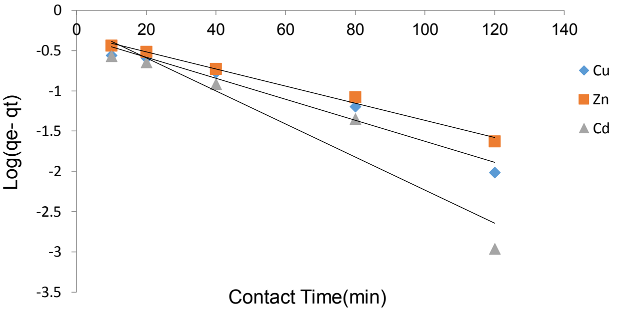

Figure 5. Pseudo-first order model for Cd, Cu, and Zn sorbtion onto APS.

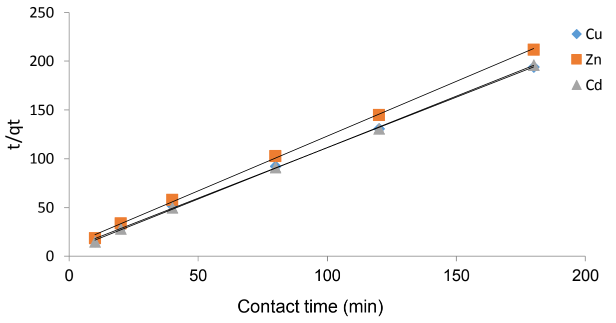

Figure 6. Pseudo-second order model for Cd, Cu, and Zn sorption onto APS.

The ways to obtain the kinetic rate constant k_2 (g/mg min), initial sorption rate h (mg/g min) and the amount of metals adsorbed at equilibrium q_e (mg/g) are presented in Table 5. The correlation coefficients for the pseudo-second order kinetic model are all extremely high (greater than 0.99), indicating that the adsorption system belongs to the pseudo-second order kinetic model (31).

This study has developed a new medium, pumice stone, for the removal of Cd, Cu, and Zn from aqueous solutions. Based on the results and on the correlation coefficients calculated, the sorption kinetics were found to follow a pseudo-second order kinetic model. In general, the isotherm gives the R^2 value closest to unity, but the Langmuir isotherm is better fitted for describing the equilibrium adsorption data for Cd, Zn, and Cu than the Freundlich isotherm.

Discussion

Comparable results were obtained by Nasseri and Heidari (14). Their study investigated and compared arsenic removal from water resources using aluminum-coated pumice (ACP) and aluminum-coated zeolite (ACZ) as new adsorbents. The effects of parameters including adsorbent doses, pH, contact time, and initial arsenic concentration were studied. They concluded that more than 96% of arsenic with an initial concentration of 250 μg/L was removed by 10 g/L aluminum-coated zeolite at pH = 7 and in 60 minutes to achieve an arsenic concentration of 10 μg/L, while only 71% of arsenic could be removed by aluminum-coated pumice. In a recent study done in Iran, the removal of acid red 14 and acid red 18 azo dyes by acid-treated pumice stone as an efficient adsorbent was investigated under various experimental conditions. It was revealed that maximum sorption capacities were 3.1 and 29.7 mg/g for acid red 14 and acid red 18, respectively. These results were significantly higher than those reported in the literature, even for activated carbon (22). Another study performed by Kaplan Bekaroglu et al (18) investigated the effectiveness of iron oxide-coated pumice and volcanic slag particles in removing disinfection by-product precursors from a raw drinking water source with high specific UV absorbance. It was found the iron oxide-coated pumice/slag particles are effective adsorbents for removing natural organic matter and

Table 4. Pseudo-first order rate equations; parameters related to the kinetic models for APS sorbent

R^2	q_e (mg/g)	k_1 (1/min)	Element	Initial concentration (mg/L)
0.958	0.725	0.013	Cu	10
0.990	0.738	0.010	Zn	10
0.904	0.835	0.020	Cd	10

Table 5. Pseudo-second order rate equations

Initial concentration (mg/L)	Element	k_2 (g/mg-min)	qe (mg/g)	R2	h (mg/g. min)
10	Cu	0.139	0.964	0.999	0.129
10	Zn	0.116	0.891	0.999	0.092
10	Cd	0.178	0.949	0.999	0.161

control disinfection by-product formation in waters; but, they may not be effective for waters with alkalinity levels above 250 mg $CaCO_3$/L. Also, Asgari et al (16) reported that surfactant-modified pumice was shown to be an affordable and promising option for the removal of fluoride from drinking water.

Conclusion

The removal of cadmium, copper, and zinc from aqueous solutions using APS as a sorbent is possible. The results of the current study indicate that APS is a good choice for a low-cost and effective sorbent. The sorption capacity of APS was considerably affected by the initial concentration of metal ions in the solution and by contact time. The Langmuir isotherm provided the best correlation for the sorption of cadmium, copper, and zinc onto APS. At a constant pH of 7, the sorption capacities as calculated from the Langmuir isotherm were 3.01, 2.62, and 2.53 mg/g for Cd, Cu, and Zn, respectively. The kinetic data was well fitted by a pseudo-second order model. The authors believe that the sorbent APS can be recommended as a suitable alternative for the control and treatment of industrial wastewater where heavy metals or toxic chemicals threaten the quality of ground or surface waters. Because of the excellent qualitative and quantitative capabilities of new instruments, it is recommended that future researchers apply the latest technology such as x-ray diffraction (XRD), x-ray fluorescence (XRF), and scanning electron microscopy (SEM) to evaluate the capability of APS to absorb heavy metals and characterize their mechanisms of action.

Acknowledgements

This research was carried out as a Master's Thesis at the Department of Environment, financially supported by Hamedan Branch, Islamic Azad University, and technically supported by Kermanshah Provincial Directorate of Environmental Protection, to which the authors' thanks are due.

Ethical issue

The authors certify that all data collected during the study is presented in this manuscript, and no data from the study has been or will be published separately.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

All authors participated in the study design, literature search, writing of the manuscript, and data acquisition, analysis, and interpretation. All authors critically reviewed, refined, and approved the manuscript.

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