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Influence of pH on cadmium, copper, and lead removal from wastewater by steel slag

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ABSTRACT

Steel slag is a byproduct of steel manufacturing which raises an important environmental concern due to the high volume of generation and, specially, its basicity. The aim of this work was to determine its recycling potential and use for the removal of Cd, Cu, and Pb given its ability to increase the pH of a solution to values close to 13.5. Remediation of cadmium-, copper-, and lead-contaminated water was studied by means of induced precipitation/sorption reactions. Precipitation of these metals was evaluated by kinetic and batch tests that were analyzed by commonly used sorption models. The results confirmed that Cd, Cu, and Pb can be removed from solutions by the addition of steel slag particles, while the removal efficiency depends on metal type and on the ion interaction, concentration, and pH of precipitation of each metal.

Keywords: Steel slag; Recycling; Heavy metals removal; Wastewater; Precipitation; Sorption

1. Introduction

Most pollutants appear as the result of human activities and cause risk to the surrounding environment [1]. These disturbances may be chemical (metals or organic compounds), physical (particulate matter, sound, or thermal energy), biological (bacteria, fungi, or virus), or from ionizing radiation. In particular, heavy metal contamination is mainly related to anthropogenic sources and causes major impacts on human health due to its persistency, toxicity, and cumulative effects. Given their importance, uses, and impact on the environment, cadmium (Cd), copper (Cu), and lead (Pb) are among the most usual heavy metal contaminants [2]. Spills of these metals are found individually, related to particular industrial activities, or combined as in the case of landfill leachate.

Remediation technologies for heavy metal-contaminated water depend on several factors, including whether the water is contained in a facility or dispersed in the environment. The different techniques to be applied for each metal depend on wastewater components, initial metal concentration, cost of treatment, further use of treated water, and associated environmental impacts [3]. Frequently used remediation strategies involve either metal removal by their adsorption on filter materials or isolation/containment of contaminated water by means of slurry walls or barriers [4–6]. Alternatively, permeable reactive barriers (PRB) can be used to adsorb, precipitate, or change the speciation of inorganic contaminants [2]. The use

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of PRBs has been increasing in the last two decades due to their capacity to remove metallic ions from groundwater at low cost and with high efficiency [7]. Alternative low-cost reactive materials for this purpose are thus continuously being developed or discovered [8,9].

Steel slag is a byproduct of steel manufacturing mainly used as flux in steel production, and recent applications are related with concrete and asphalt production and the construction of road grades and sub-grades [10]. Its physical properties and chemical composition promote that new applications for steel slag are frequently found. Successful results have been obtained in wastewater treatment, iron recovery, CO_2 sequestration, and as fertilizer [9,11,12]. It can also be used for the treatment of metalloids and heavy metal-contaminated wastewater due to its high alkalinity (equilibrium pH near 14) and capacity to produce metal precipitation [13–15].

Precipitation is widely used for heavy metal separation: Chemical compounds are added to water, reacting with the metallic compounds and producing insoluble species [16]. The process is controlled by chemical equilibrium and depends on each species formed, with precipitation results being influenced by the solution's pH and Eh conditions. In general, anions such as hydroxides, sulfites, sulfides, carbonates, and phosphates are used to form insoluble compounds with non-alkali metals [17–19]. Lead also precipitates as sulfate, and this metal as well as copper precipitates as chloride or bromide species. The reactive media in PRB are usually a natural compound: limestone ($CaCO_3$) is used for carbonate barriers, hydroxyapatite for phosphate precipitation, and slaked lime ($Ca(OH)_2$) for hydroxide precipitation and pH regulation in acid mine drainage [20–22].

The chemical equilibrium (Eq. (1)) in precipitation/dissolution reactions is characterized by an equilibrium constant named solubility product constant (K_{sp}):

$$M_bA_n(s) \rightleftharpoons bM^{+n}(\mathrm{aq}) + nA^{-b}(\mathrm{aq}) \tag{1}$$

$$K_{\mathrm{sp}} = \left[M^{+n}\right]^b \left[A^{-b}\right]^n \tag{2}$$

where M is a metal, A is an anion, n and b are the chemical charges of metal and anion, respectively, (aq) is aqueous (dissolved) species, and (s) is solid species. Brackets represent the molar concentration (mol/L) of each compound or species. The higher the K_{sp}, the higher the dissociation of the metal, and the smaller the K_{sp}, the easier would be the metal precipitation [23]. It should be noted that species with K_{sp} values lower than hydroxide-based compounds sometimes pose greater environmental concerns than the metals, as in the case of lead arsenate.

Granular materials can also remove heavy metals by means of sorption, depending on mineralogy and surface charges (e.g. negative surface charges in clay particles and in oxides and hydroxides forming the steel slag) [24,25]. Sorption processes can be divided into three different mechanisms, which are difficult to differentiate at macroscale: (a) *adsorption* or *physisorption*, involving a physical, electrostatic interaction, which is fast and usually reversible; (b) *absorption* or *chemisorption*, where the contaminant enters in the

Table 1
Isotherm models analyzed

Model	Equation
Linear isotherm model	$q_{\mathrm{e}} = k_{\mathrm{d}}C_{\mathrm{e}}$
Freundlich isotherm model	$\log q_{\mathrm{e}} = \log k_{\mathrm{F}} + \frac{1}{n}\log C_{\mathrm{e}}$
Langmuir isotherm model	$\frac{q_{\mathrm{e}}}{C_{\mathrm{e}}} = \alpha\beta - \beta q_{\mathrm{e}}$
Dubinin–Radushkevich isotherm model	$\ln q_{\mathrm{e}} = \ln sq_{\mathrm{s}} - k_{\mathrm{ad}}\varepsilon^2$
Temkin isotherm model	$q_{\mathrm{e}} = \frac{RT}{b_{\mathrm{T}}}\ln A_{\mathrm{T}} + \frac{RT}{b_{\mathrm{T}}}\ln C_{\mathrm{e}}$

Notes: q_{e} is the amount of sorbate sorbed per unit mass of sorbent (mg/g); C_{e} is the equilibrium concentration of the metal in solution (mg/L); k_{d} is the linear model constant (L/g); k_{F} (mg/g) and n (adim) are Freundlich model's fitting parameters related with the adsorption capacity and the adsorption intensity, respectively; α is the maximum monolayer coverage (mg/g), and β is a Langmuir fitting parameter; q_{s} is the theoretical saturation capacity of the D–R model (mg/g); $\varepsilon = RT\ln(1 + 1/C_{\mathrm{e}})$ is Polanyi's potential; k_{ad} is a fitting parameter for the D–R model, ($\mathrm{mol}^2/\mathrm{kJ}^2$); R is the universal constant for ideal gases (8.314 J/mol/K); T is the absolute temperature (K); A_{T} is Temkin's equilibrium bonding constant (L/g), and b_{T} is a fitting parameter for Temkin's model.

solid phase and forms a new solution, which usually takes longer and in many cases is irreversible; and (c) *ionic exchange*, by which ions from the solid structure are exchanged for those in solution with higher affinity [19]. Different models have been proposed to relate the concentration of sorbate in solution and the amount sorbed, but those with one and two fitting parameters are the most simple and widely accepted [11,26]. Table 1 summarizes the sorption isotherm models commonly used to evaluate the removal capacity of different substrates. Sorption barriers usually apply physical mechanisms (adsorption) for metal removal, which depend highly on subsurface conditions (pH, O_2, redox potential) [18].

The purpose of this work was to assess the potential use of recycled steel slag for the removal of Cd, Cu, and Pb from contaminated water. The effects of pH and ionic strength are studied for individual species and multi-species tests. Removal capacities are evaluated by means of adsorption models, while remediation kinetics is considered by means of reaction-controlled and diffusion-controlled mechanisms. The results obtained show that steel slag has significant potential for the treatment of contaminated water.

2. Materials and methods

2.1. Metallic solutions

Cd, Cu, and Pb solutions were prepared in a metal concentration of 1,000 mg/L. These solutions were prepared by dissolving cadmium nitrate tetrahydrate ($Cd(NO_3)_2{\cdot}4H_2O$, Anedra), copper (II) nitrate trihydrate ($Cu(NO_3)_2{\cdot}3H_2O$, Taurus), and lead nitrate ($Pb(NO_3)_2$, Anedra), respectively. All reagents were of analytical grade and were used without further purification. Depending on the tests, different dilutions between 1 and 500 mg/L were prepared with distilled water (electrical conductivity < 3 μS/cm). These concentrations fall within recorded concentrations in wastewater from the chemical industry that may be accidentally released into the environment.

2.2. Steel slag

The slag used in this work is from a basic oxygen furnace in Argentina. Its chemical composition can be found in Table 2. Due to process variations and the rejects in steel casting, the greatest variations in composition are found for iron compounds (Fe°, FeO, and Fe_2O_3). The main characteristic of the slag is its basicity, increasing the pH of an aqueous suspension to 13.5.

Table 2
Chemical composition of the steel slag

Compound	Percentage
Iron compounds	8.5–27.2
CaO	37.6–44.8
SiO_2	35–37.3
Al_2O_3	9.3–14.7
MgO	5.6–9.6
K_2O	0.41
Na_2O	0.2–0.3
S	0.4
MnO	0.4–0.7
TiO_2	0.4–0.6
Basicity (%CaO/%SiO_2)	1.1–1.2

All tests were performed using the slag fraction passing sieve #100, and retained in sieve #200 (150 μm > particle size > 74 μm).

2.3. Precipitation test

Solutions with initial metal concentrations of 50 mg/L were prepared in 50 mL Falcon tubes and regulated at different pH values, between 1 and 14, by the addition of 1 M HCl or NaOH. The solutions were mixed in an orbital shaker for 24 h, and the pH of the tubes was measured and readjusted at 4, 8, and 24 h. After equilibrium was reached, an aliquot was collected in 1.5 mL Eppendorf tubes and centrifuged at 10,000 rpm for 10 min. Initial and final metal concentrations were determined to evaluate the influence of pH on precipitation.

2.4. Remediation kinetic tests

Removal kinetic testing of Cd, Cu, and Pb by steel slag was carried out in magnetically stirred beakers. Table 3 summarizes the most important kinetics test conditions. Individual ions were tested at a 50 mg/L concentration, while combined ions were tested at individual concentrations of 100 mg/L (300 mg/L in total). Steel slag concentrations ranged from 0.25 to 0.5 g/L.

The steel slag was added to the Cd, Cu, and Pb solutions, and these mixtures were stirred for 48 h. A representative sample of the solution was taken at different times to measure the final metal concentration. The results obtained were analyzed by reaction kinetic models (pseudo-first-order, pseudo-second-order and Elovich's equation) and diffusion kinetic models (liquid film diffusion, intraparticle diffusion, and mass transfer).

Table 3
Test conditions

Test	Slag concentration (g/L)	Metal	Test duration (h)
Kinetics	0.25	Cadmium	48
	0.50	Copper	48
	0.50	Lead	48
	0.25	Cd/Cu/Pb	48
Batch removal	0.25	Cadmium	24
	0.25	Copper	24
	0.50	Lead	24
	0.25	Cd/Cu/Pb	24

Removal efficiency (RE) was computed from initial concentrations (C_0) and instant concentrations (C_t) as follows:

$$\mathrm{RE} = \frac{C_0 - C_t}{C_0} \tag{3}$$

2.5. Batch removal tests

Table 3 summarizes solid and metal concentrations, as well as test durations. Batch removal tests were carried out in acrylic 50 mL Falcon tubes placed in an orbital shaker. Tests were performed isothermally at 20˚C for 24 h. After this period, samples collected were centrifuged at 3,500 rpm for 30 min in 50 mL Falcon tubes. The supernatant was then centrifuged for 10 min at 10,000 rpm in 1.5 mL Eppendorf tubes, and the new supernatant was tested to determine the final metal concentration.

For each metal and slag concentration, two different tests were performed at different pH. Solution pH values were either 4 or 10.5, which were selected to analyze the behavior of the metals at pH values higher and lower than the precipitation pH ($\mathrm{pH_p}$). The results obtained were analyzed by the sorption models described in Table 1, which were implemented by means of nonlinear least square fitting.

2.6. Chemical analyses

Spectrometric determination was performed for dissolved Cu, Cd, and Pb, using a visible range ThermoFisher Scientific Aquamate® spectrophotometer. Commercial test kits were used for cadmium (Merck), copper (Hach), and lead (Merck), following the standard procedures. Table 4 presents significant test conditions used for the detection of each metal. When needed, solutions were diluted in distilled water in order to obtain Cd, Cu, and Pb concentrations within the quantification range for each metal.

The Zeta potential of the slag was analyzed in a DelsaNano HC apparatus (Beckman Coulter) by means of photon correlation spectroscopy. These tests consisted in measuring the fluctuations on dispersed light intensity caused by solid particles moving in the liquid due to an applied electric field. The equipment actually measures the velocity of a particle in a unit electric field, which is referred to as its electrophoretic mobility. Zeta potential is related to the electrophoretic mobility by the Henry equation:

$$U_{\mathrm{E}} = \frac{2\varepsilon\zeta f(\kappa a)}{3\eta} \tag{4}$$

where U_{E} = electrophoretic mobility, ζ = zeta potential, ε = dielectric constant, η = viscosity, and $f(\kappa a)$ = Henry's function (equals to 1.5 according to Smoluchowski approximation).

Prior to testing, the steel slag samples were washed with distilled water to eliminate impurities. The pH of the solutions was modified from 2.5 to 11.5 by adding 1 M HCl or NaOH. Zeta potential tests

Table 4
Commercial test kits used for metal quantification

Metal	Cadmium	Copper	Lead
Commercial brand	Merck	Hach	Merck
Reaction	Red complex formation between cadmium and a cadion derivate in alkaline solution	Purple complex formation between copper and bicinchoninate in neutral solution	Red complex formation between lead and 4-(2´-pyridylazo)-resorcinol in alkaline solution
Wavelength (nm)	525	420	525
Quantification range (mg/L)	0.010–0.500	0.01–5.00	0.10–5.00
Sample pH	3–11	4–6	3–6

were performed at each pH with three different ionic strengths (obtained with NaCl concentrations of 0.001, 0.01, and 0.1 M). The pH of the solution was measured before and after the zeta potential tests, and the change observed in pH was lower than 0.5 UpH.

3. Results

3.1. Zeta potential

The Zeta potential of steel slag indicates the net surface charge of solid particles, which is a pH-dependent property (Fig. 1). The slag isoelectric point (pIE) is at pH 7.8, with negligible influence of the ionic strength. Fig. 1 also shows that Z potential decreases at pH < 4, probably indicating the dissolution of mineral phases or the complexation of anions at the slip plane.

Positive surface charges, associated with positive zeta potentials, determined at pH < 7.8, indicate the slag could be used to adsorb anionic compounds (e.g. arsenate, chromate), while it would act as a cation sorbent at higher pH values. However, it is important to highlight that the solubility of cationic metals decreases with the increase in pH, promoting a dual adsorption/precipitation mechanism at high pH values.

3.2. Precipitation behavior

Fig. 2 presents the removal results of Cd^{2+}, Cu^{2+}, and Pb^{2+}, as individual ions or in a multispecies test, by means of pH adjustment with HCl and NaOH. The simultaneous presence of different metals in the solution influenced the pH_p for Cd and Pb, while no significant changes were observed in the case of Cu. Cd was in its soluble form for pH < 7, with this pH value increasing by one unit when in the presence of Pb and Cu. Cu showed a rapid decrease in its solubility for pH > 6 due to the formation of highly insoluble hydroxides [27,28]. Finally, Pb was in solution for pH < 7, and the simultaneous presence of Cu and Cd decreased the pH_p by one unit. However, Pb compounds were redissolved at pH > 11, probably due to the formation of $Pb(OH)^{3-}$ [29].

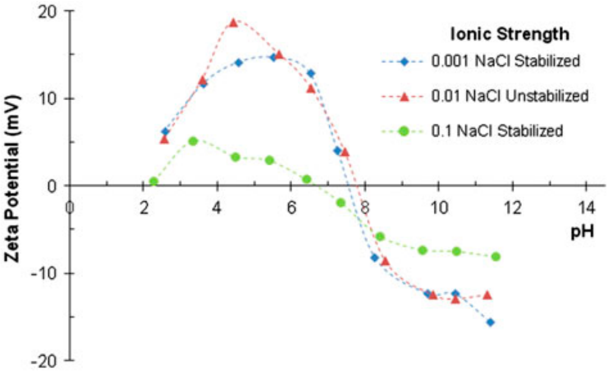

Fig. 1. Zeta potential of steel slag at different ionic strengths.

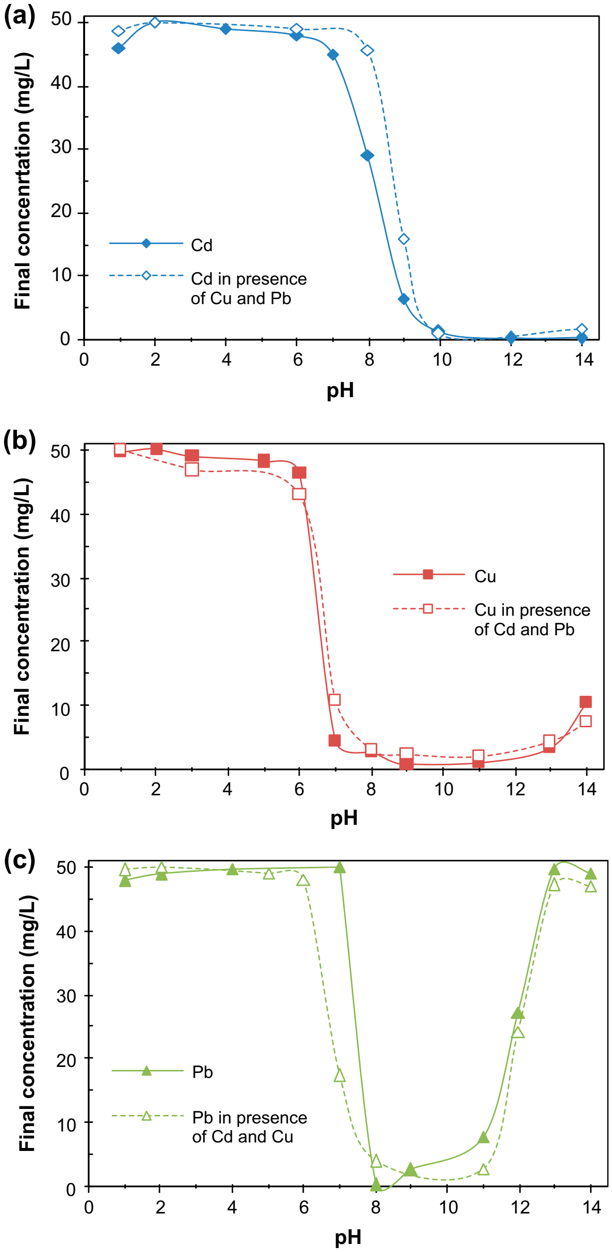

Fig. 2. Precipitation of cadmium (a), copper (b), and lead (c) as function of the pH.

3.3. Sorption/precipitation kinetics

The addition of steel slag raised the suspension's pH to reach values between 9.5 and 11 UpH, depending on the slag concentration. The high acid neutralization capacity of the slag promoted the pH increase and is thus partially responsible for precipitating Cd, Cu, and Pb.

Concentrations of soluble Cd, Cu, or Pb in the presence of slag decreased with time, as shown in Fig. 3(a). Addition of the metallic solutions reduced the water pH, while the inclusion of the slag raised the solution's pH, with the latter effect being more important than the former. Cu^{2+} showed a higher decrease in concentration than Cd^{2+} and Pb^{2+}, although Cd^{2+} reached equilibrium faster. No significant influence of slag concentration was observed in the amount of metallic ions removed from the solutions. Fig. 3(b) shows the results for the multispecies test when the solution with slag particles simultaneously contained Cd, Cu, and Pb ions. In this case, the higher concentration of metals reduced the pH after 48 h to 6.3 UpH. Decreases in concentrations resulted in the removal efficiencies (Eq. (3)) of 6, 81, and 2% for Cd, Cu, and Pb, respectively. These results are in agreement with the precipitation trends at pH 6.3 shown in Fig. 2.

The remediation process kinetics in all cases was fast and followed a pseudo-second-order kinetic model. Optimal removals were reached between 5 min and 4 h of testing and were highly related to the effective mixing of the solution, due to the constant stirring. The best fit for a reaction kinetic model was at pH 10.5 with strong mixing.

3.4. Remediation isotherms

Fig. 4 shows the RE (Eq. (3)) of individual heavy metals due to their interaction with the steel slag in the bath tests at pH 4 and 10.5, while Fig. 5 presents results in the case of the simultaneous presence of Cd, Cu, and Pb at the same pH values. The most significant differences were found between samples treated at pH 4 and pH 10.5, while the behavior for ions treated individually or combined showed similar patterns in most cases.

Cd presented the greatest differences in RE with pH, showing no removal when pH < pHp and RE

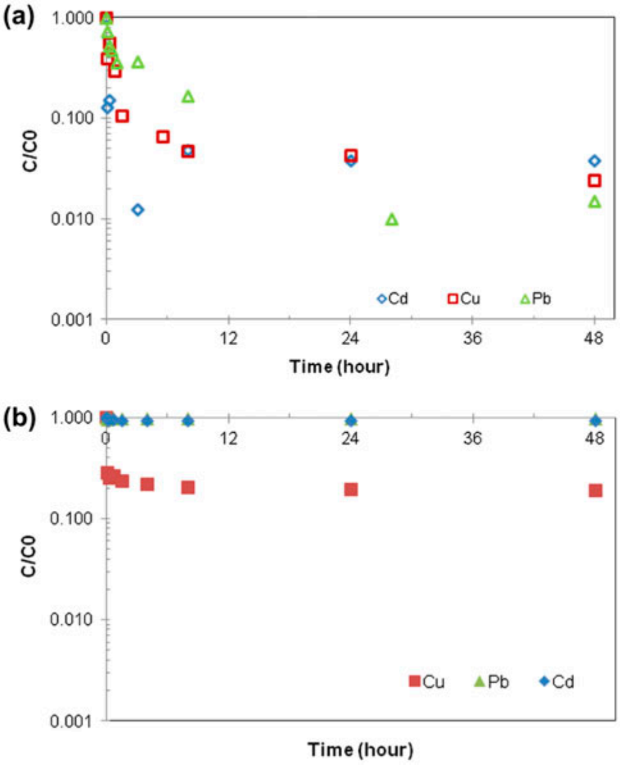

Fig. 3. Remediation kinetics for individual heavy metals (a) and combined heavy metals (b).

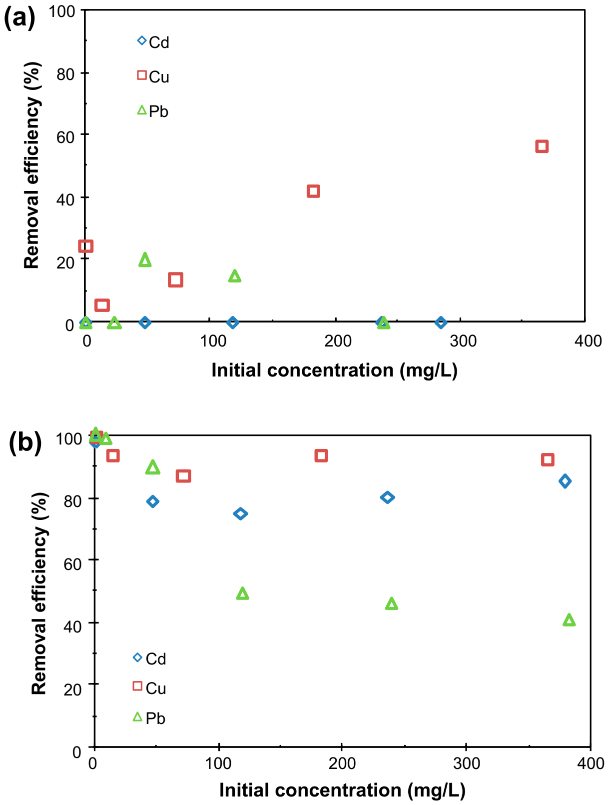

Fig. 4. Removal isotherms for individual ions at (a) pH 4 and (b) pH 10.5.

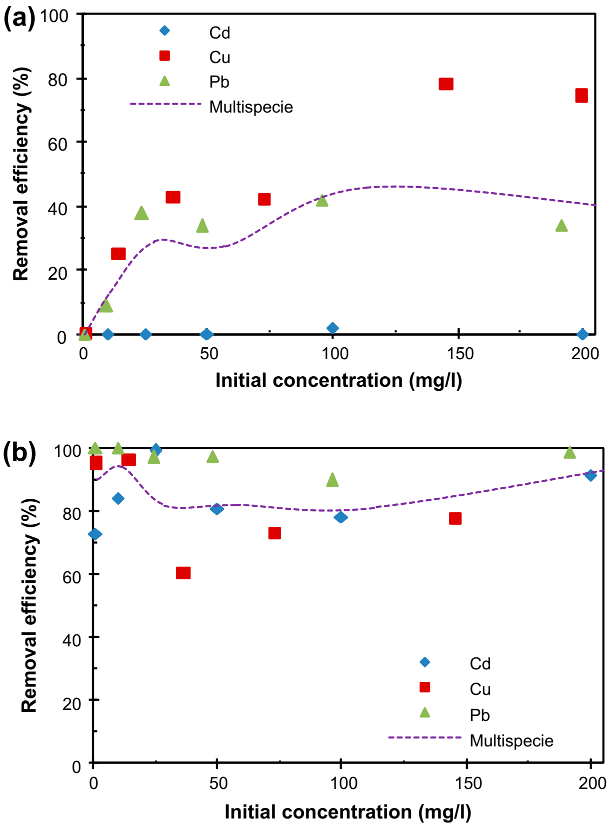

Fig. 5. Removal isotherms for combined ions at (a) pH 4 and (b) pH 10.5.

between 75 and 98% when $pH > pH_p$. Cd removal levels at $pH > pH_p$ changed with the initial concentration, but show no direct trend. Removal levels were similar for the pure Cd solution and in the presence of Cu and Pb, as shown in Figs. 4 and 5, respectively. Cu removal efficiencies also depended on pH values and initial concentration, changing from 10% ($pH < pH_p$) to 90% ($pH > pH_p$) at an initial concentration of 10 mg/L, or from 60% ($pH < pH_p$) to 90% ($pH > pH_p$) when the initial concentration was 200 mg/L. Finally, pH also had a great impact on Pb removal, promoting no removals at low pH for almost every initial concentration, while showing RE between 40 and 100% when pH was 10.5. These results confirmed the impact of precipitation on the remediation of Cd, Cu, and Pb produced by the addition of steel slag particles in the solutions.

4. Discussion

The physical and chemical reactions involved in the contribution of slag to removing heavy metals from contaminated water are numerous, including acid neutralization and carbonate dissolution from slag components, formation of complex phases, and the surface adsorption or chemical diffusion of metals in the slag pores. Additionally, oxidation–reduction reactions may occur due to zerovalent iron in the slag, which promotes the reduction of metals and their precipitation as native elements. These interactions depend on fluid and flow conditions and can take place at the same time, making it difficult to determine the individual contribution of each process. In these conditions, kinetic and remediation models constitute a first approximation to the observed behavior, given that several mechanisms may simultaneously be present. This is important when considering the possible use of steel slag as a reactive material in PRBs.

These remediation results were achieved at $pH > pHp$ and $pH > pIE$, and thus, they involve simultaneous precipitation and adsorption phenomena. However, it was possible to simulate the observed decrease in metal concentration in the solution with the adsorption models shown in Table 1. Fig. 6 presents the removal results of Cd, Cu, and Pb, with steel slag at pH 10.5 and implementing adsorption models. Different error functions were considered when fitting the models. Given that adsorption and precipitation operate together, the best fit of these equations represents a global remediation model rather than an adsorption trend.

The error functions considered were linear regression coefficient (R^2), sum of the squared errors, sum of the absolute errors, average relative error, hybrid fractional error function, Marquardt's percent standard deviation, Spearman's correlation coefficient, standard deviation of relative errors, and chi-squared nonlinear test. The nonlinear error functions avoid biases for linearized sorption models, and confer different weights to different concentration ranges, enabling deeper understanding of the removal process. The linear analysis using the linear regression coefficient (R^2) shows that removals of Cu and the multispecies test (simultaneous presence of Cd, Cu, and Pb) follow a linear model, while Cd and Pb removals can be better represented with the Freundlich and Langmuir equations, respectively. The nonlinear analysis shows that R^2 accurately represents the removal of Cu, Pb, and combined metals. In addition, most error functions show a better match between Cd removal results and the linear model, rather than the Freundlich model as indicated by the R^2 coefficient ($R^2 = 0.954$).

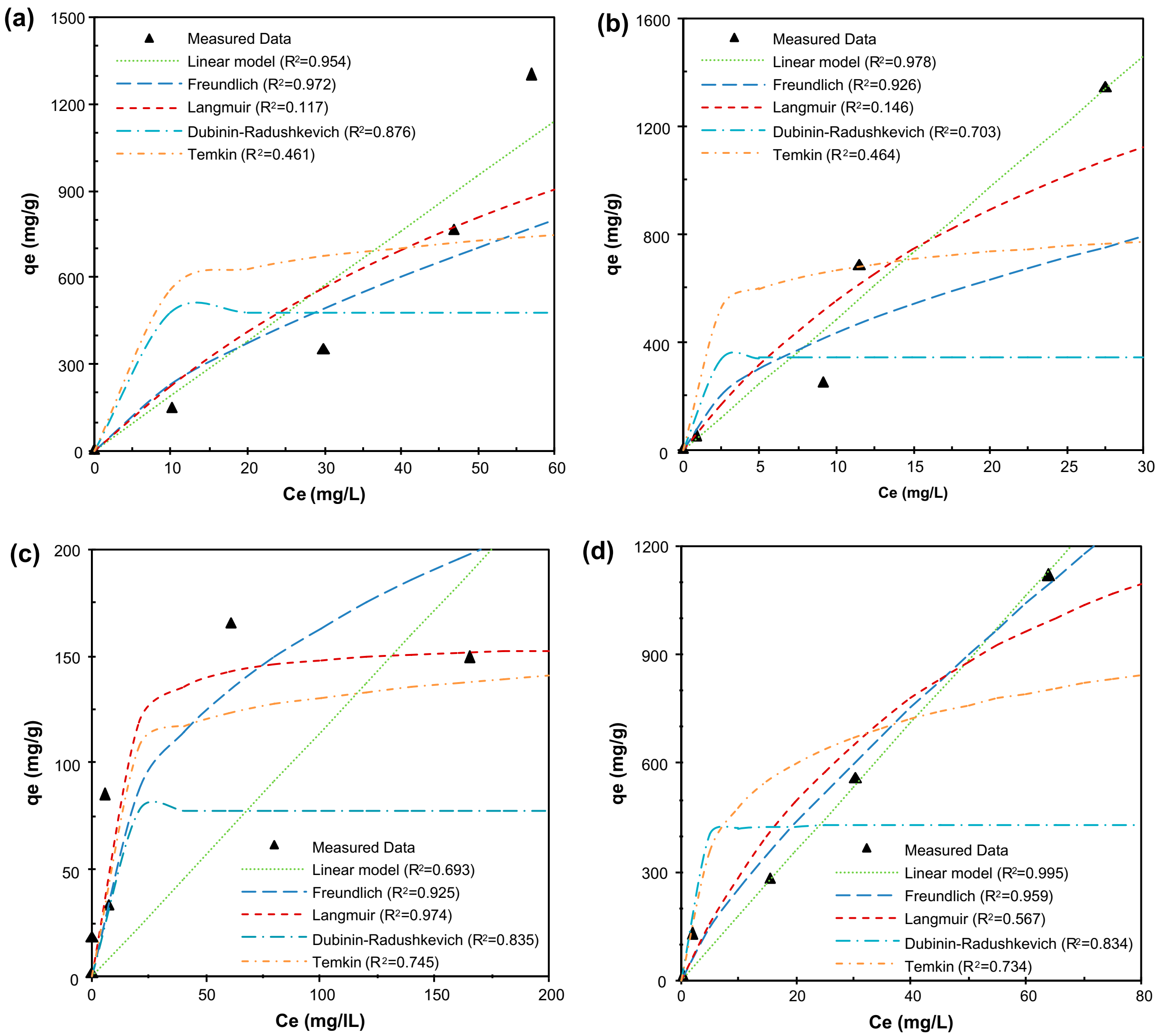

Fig. 6. Sorption models for removal at pH 10.5 of (a) cadmium, (b) copper, (c) lead, and (d) multispecies.

5. Conclusions

This work presents results and analysis of Cd, Cu, and Pb removal with steel slag. The influence of metal type, concentration, pH, and ion competition were evaluated. The results show that the solution pH is fundamental for the metal removal process, not only because of the charge that pH confers to the solid particles, but also because of the formation of insoluble species of Cd, Cu, and Pb at pH values higher than 7, 6, and 7, respectively. The kinetics of the reactions is very fast and follows a pseudo-second-order kinetics model. The simultaneous presence of the metals affects the removal efficiencies obtained in the case of Pb, while Cd and Cu removals with steel slag are independent of whether these elements are treated individually or combined. The remediation process can be related with different sorption models. Cadmium and copper follow a linear behavior, while lead removal is best described by the Langmuir model. Copper and cadmium contributions, increased by the higher removals of these metals in the combined removal test, confer a linear behavior on the mixed metals. We can confirm that steel slag can successfully remove Cd, Cu, and Pb from water due to the capacity of slag to increase the pH of the solution, inducing precipitation/sorption reactions.

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