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ABSTRACT

Two lignocellulosic materials with different lignin contents (18 and 42 %wt) and pure lignin (PL) were evaluated for their effectiveness in binding cadmium from dilute solutions in various concentrations. Maximum sorption capacities (X_m), determined from equilibrium isotherms by applying the Langmuir model, indicated that PL ($X_m = 48.3\,\mathrm{mg/g}$) and the sample with the larger lignin content ($X_m = 22.2\,\mathrm{mg/g}$) showed a reasonable ability to uptake cadmium. An increasing relationship between X_m and the sample's lignin content was

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found, considering the tested materials together with others evaluated earlier under identical conditions. Pure lignin attained the highest value. Accordingly, the lignin content of lignocellulosic materials appears as an indicator of their ability to uptake cadmium. It could facilitate their screening for potential use as alternative cadmium sorbents from dilute wastewater. The effects of the sample's dose and the solution pH on cadmium uptake also were investigated.

Key Words: Alternative sorbents; Lignocellulosic wastes; Heavy metals removal; Wastewater treatment.

1. INTRODUCTION

Concern about the hazardous effects caused by the presence of heavy metals in aquatic environments in concentrations exceeding the admissible limits has induced the search for cost-effective solutions to mitigate water pollution. In the last few years, the feasibility of using low-cost, easily available materials for metals uptake from wastewater has been investigated to seek alternative sorbents that can be used more economically on a large scale than conventional ones.

Materials mostly examined include nonliving biomasses of yeast, bacteria, fungi, and algae,[1–4] as well as peat.[5,6] They are shown to be capable of concentrating metals from aqueous solutions, accumulating them within their structures.

Other cheap materials less explored as sorbents include lignocellulosic wastes generated from processing of agricultural products, food, and/or wood, either raw or chemically/thermally modified at low temperatures.[7–11] Many of them also have been demonstrated to be effective in binding several metals. Use of lignocellulosic wastes, as alternative sorbents, may constitute an attractive option for wastewater treatment because of their low cost, abundance, and renewable character. However, information on their effectiveness in removing trace metals from wastewater is still limited and, therefore, a thorough knowledge to predict the potential metal-binding ability of lignocellulosic materials in terms of their chemical features has not yet been attained.

Some results earlier obtained in our laboratory[12] suggested that the ability of lignocellulosic materials to uptake metals from dilute solutions could depend on their lignin content. Within this context, in the present work, two raw lignocellulosic materials with different lignin contents and pure lignin (PL) were purposefully selected and examined for their metal sorption ability, to gain insight into the incidence of the content of lignin composing this type of material on their performance as alternative metal sorbents from wastewater. An activated carbon (AC) with good metal-sequestering ability also was used for the sake of comparison. Dilute aqueous solutions of Cd(II) ion were used as models of low-metal concentration wastewater. Cadmium removal is of primary interest because of cadmium's acute toxicity and its increasing discharge into the environment.[13]

The effect of the samples' doses on the ability of the selected materials to sequester cadmium was first examined from uptake experiments conducted at fixed, preestablished equilibrium conditions. The influence of the solution pH on equilibrium cadmium uptake also was investigated for all the samples. Equilibrium isotherms were further determined and modeled to assess sorption capacities of Cd(II) ions for the selected samples.

2. MATERIALS AND METHODS

Lignocellulosic materials used were seed hulls (SDH) obtained from an easy-to-grow herbaceous plant (*Mirabilis jalapa*) and an agricultural waste, stems from yerba mate leaves (YLS). Yerba mate is a widely cultivated evergreen tree (*Ilex paraguariensis*), which belongs to the family Aquifoliaceae. The leaves of this tree are processed to prepare a traditional herbal tea beverage, very popular in Argentina, Paraguay, and southern Brazil. Large quantities of the leaves' stems are generated as waste from industrial processing. The YLS used were supplied by Mate Larangeira Co. (Province of Misiones, Argentina).

The SDH and YLS, without any further treatment, were ground, milled, and screen sieved. Fractions of particle diameter in the range 100–250 μm were used for samples' characterization and cadmium uptake experiments. Proximate analyses of the samples were performed according to ASTM (American Society of Testing and Materials) standards. An elemental analyzer (Carlo Erba model EA 1108, Carlo Erba Strumentazione, Milan, Italy) was used to assess their elemental compositions. In addition, contents of the major biopolymers constituents of the samples, i.e., holocellulose (cellulose + hemicellulose) and lignin, and solvent extractive components were determined by applying the TAPPI (Technical Association of the Pulp and Paper Industry) standard methods (T204 om-88, T222 om-88). Results are reported in Table 1.

Furthermore, total polar or acidic surface oxygen functional groups (TOFG) of the lignocellulosic samples, reportedly influencing metal uptake by various biosorbents,[4] were quantified. These groups may include carbonyls, phenols, lactones, and/or carboxyl acids. They were determined by titration with sodium ethoxide. This base is the same one used in the Boehm's method,

Table 1. Chemical characteristics of the lignocellulosic samples.

Sample	YLS	SDH	ADS[12]	BNS[12]	PRS[12]	SCB[12]
Proximate analysis[a] (wt%)						
Volatile matter	72.0	56.8	71.3	76.1	75.4	84.9
Fixed carbon	24.9	39.5	24.1	22.2	22.1	12.3
Ash	3.1	3.7	4.6	1.7	2.4	2.8
Ultimate analysis[b] (wt%)						
Carbon	46.4	48.3	49.3	50.0	47.1	46.9
Hydrogen	5.9	6.1	6.0	5.8	6.2	5.6
Nitrogen	1.2	1.4	0.3	0.7	0.4	1.2
Oxygen[c]	46.5	44.2	44.4	43.5	46.3	46.3
Lignocellulosic composition[d] (wt%)						
Holocellulose	82	58	77	43	72	72
Lignin	18	42	23	57	28	28
TOFG (meq/g)	1.0	2.5	1.1	2.7	1.5	1.8

[a] Dry basis.
[b] Dry and ash free basis.
[c] Estimated by difference.
[d] Dry and extractive-free basis.

which is widely applied to ACs and other carbonaceous materials for the same purpose. The amount of base uptake by each sample is considered as an approximate comparative indication of the TOFG of the samples. Details of the experimental procedure have been reported earlier.[14] Mean values of the TOFG, expressed as milliequivalents per gram of sample, also are listed in Table 1.

Analytical grade lignin (PL) from Aldrich Chemical Co., and a prepared AC also were used in cadmium uptake assays. The AC was obtained by phosphoric acid activation of the stems from a fast-growing grass (*Arundo donax* L.) and characterized following the procedures reported in previous works.[14,15] Main features of the AC were BET (Brunauer, Emmett and Teller) surface area of 1194 m^2/g, total pore volume of 1.03 cm^3/g, and TOFG of 3.3 meq/g.

For the sorption experiments, a stock cadmium solution (1000 mg/L) was prepared by using analytical grade $Cd(NO_3)_2 \cdot 4H_2O$ (Carlo Erba) and distilled water. Standard solutions of cadmium concentrations ranging between 5 and 100 mg/L were obtained by dilution of the stock solution.

The influence of the sample's dose on cadmium ion uptake was first investigated under fixed preestablished equilibrium conditions. For these experiments, different preweighed amounts of each sample (0.02–1 g) were contacted with 100 mL of 20 mg/L cadmium solutions in capped glass flasks. The pH of the suspensions was 5.8. A pH-meter (Orion model 9107WP, Orion Research Inc., Beverly, Massachussetts, USA) was used for pH measurements. No buffer was added, based on results published in previous works that have reported optimum cadmium uptake for $pH > 4$.[16,17]

The capped glass flasks containing the suspensions were kept in a shaker at constant temperature ($T = 28^{\circ}C \pm 1^{\circ}C$) for 7 hr. From preliminary experiments conducted for different prolonged periods, it was verified that this contact time was long enough to ensure equilibrium attainment for all the investigated systems. To check for any significant drift during the sorption tests, the pH was controlled at the start and at the end of each experiment. Almost no variation was detected, except for the experiments using the larger sample doses. Nevertheless, even in these cases, pH values were maintained within ± 0.5 units. Afterward, the suspensions were filtered through 0.45-μm membranes. Equilibrium cadmium concentrations were determined in the filtrates by using a selective ion electrode (Cole-Parmer 27502-07, Cole-Parmer Instrument Co., Vernon-Hills, Illinois, USA), with a reproducibility of $\pm 2\%$ and a lower detection limit of 0.05 ppm.

The effect of the solution pH on the equilibrium cadmium uptake for each sample was further examined by using 20 mg/L cadmium solutions and sample doses of 0.3 g/100 mL, under otherwise constant conditions. The pH of the suspensions was adjusted to values in the range of 1–7 by adding drops of nitric acid or sodium hydroxide solutions. Sorption experiments were then performed following the procedure described above.

Equilibrium isotherms for all the samples were determined by using doses of 0.3 g/100 mL, as assessed from the experimental dosage curves and solutions of cadmium concentrations varying from 5 to 100 mg/L. The suspensions at pH 5.8 were shaken at constant temperature ($T = 28^{\circ}C \pm 1^{\circ}C$) up to equilibrium, filtrated, and further analyzed for Cd(II) ions concentration as already detailed.

Duplicate experiments were carried out for all the investigated systems, differences between replicate experiments being less than 3% in all the cases. Average values were used. Metal and sorbent free blanks were also used for control in all the experiments.

3. RESULTS AND DISCUSSION

3.1. Effect of Sorbent Dose

Figure 1 shows the effect of varying samples' doses on the equilibrium metal uptake for the tested materials when using cadmium solutions of

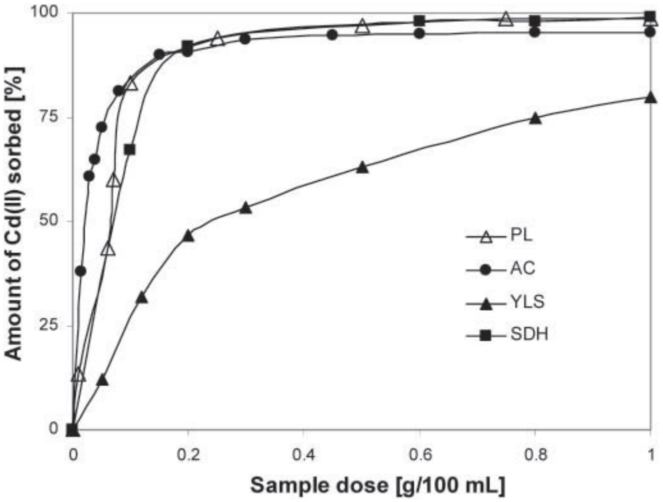

Figure 1. Effect of the sample dose on the equilibrium cadmium uptake by the SDH, YLS, PL, and the prepared AC. $C_0 = 20\,\text{mg/L}$; pH = 5.8; $T = 28°\text{C}$; $t = 7\,\text{hr}$. Solid lines only to guide the eyes.

20 mg/L. Percentages of Cd(II) ions sorbed at equilibrium are represented as a function of the samples' doses in the figure.

As expected, for a fixed metal initial concentration, increasing the sample's dose enhanced the percentages of cadmium uptake. Although both lignocellulosic samples showed appreciable cadmium uptakes, noticeable differences can be seen in Fig. 1. The SDH were more effective in capturing cadmium than the YLS over the whole dose range. The maximum cadmium uptake for the YLS did not exceed 80% even for the largest dose used.

The SDH exhibited a quite similar behavior to PL and the AC for doses larger than 0.2 g/100 mL, whereas differences in their sorptive behavior became more evident at low doses.

3.2. Influence of the Solution pH

The effect of the solution pH on the equilibrium cadmium uptake by the samples is illustrated in Fig. 2, for a cadmium concentration of 20 mg/L. As seen in the figure, cadmium sorption was strongly dependent on the solution pH. Cadmium uptake by PL and AC showed a sharp increase from negligible or very low to maximum values in the narrow pH range of 2.5–4. For the

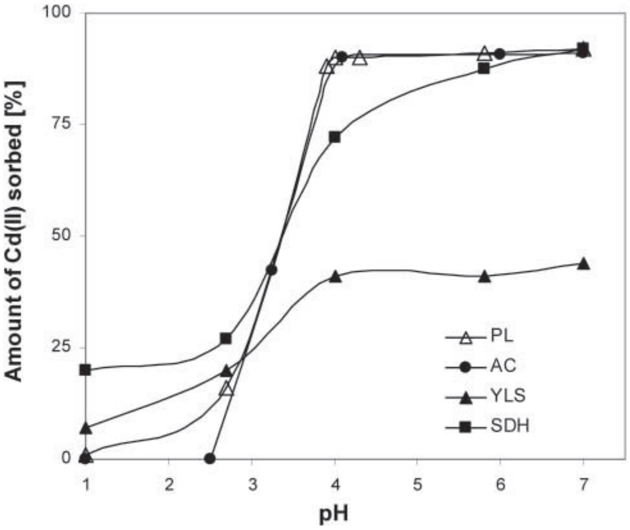

Figure 2. Influence of the solution pH on the equilibrium uptake of cadmium by the SDH, YLS, PL, and the prepared AC. $C_0 = 20\,\mathrm{mg/L}$; sample dose $= 0.3\,\mathrm{g/100\,mL}$; $T = 28^\circ\mathrm{C}$; $t = 7\,\mathrm{hr}$. Solid lines only to guide the eyes.

lignocellulosic samples, the uptake of cadmium ions increased more gradually with pH variation from 1 up to 7.

At low pH, the unfavorable high concentration of protons in solution and electrostatic repulsion forces between cadmium cations and the positively charged surface of the samples appear to predominate, affecting, detrimentally, the uptake of cadmium by all the samples. Differences observed at $\mathrm{pH} < 2.5-3$ may be attributed to electrostatic interactions of Cd(II) ion and sample's surface of different strength, which could arise from different kinds and/or amounts of polar or acidic functional groups present on the sample's surface. The results suggest stronger repulsion interactions for PL and AC. They could explain the almost negligible cadmium uptake by these samples at the lower pH values, compared with those determined for the SDH and YLS. As the solution pH increases, repulsion interactions seem to be reduced and the extent of cadmium uptake increases for all the samples, depending on their specific features, likely due to a ion-exchange mechanism between the surfaces' protons and cadmium cations. Similar trends also have been reported for the pH effect on cadmium ion sorption by ACs,[16,17] peat,[18] and certain biomasses.[19]

3.3. Equilibrium Sorption Isotherms

The Langmuir model (LM) for adsorption equilibrium was applied to the experimental data obtained for the uptake of Cd(II) ions by all the samples:

$$C_e/q_e = \frac{1}{(X_m K)} + \frac{C_e}{X_m} \tag{1}$$

C_e and q_e in Eq. (1) denote the equilibrium metal ions concentration and the amount of metal ions adsorbed at equilibrium per sample mass unit, respectively. X_m and K are parametric constants related to the maximum adsorption capacity and the affinity of the metal to the sorbent, respectively. The ability of the LM to describe the experimental data is illustrated in Fig. 3. As observed, the LM succeeded in representing properly all the examined systems with high correlation coefficients ($r^2 > 0.995$). The LM parameters estimated by regression analysis are listed in Table 2. They showed significant differences depending on the material used.

Differences in the K values indicate that Cd(II) ions interact with each material differently. These values were used to calculate the dimensionless separation factor R_L, defined as $R_L = 1/(1 + K\,C_0)$.[20] R_L between 0 and <1 was obtained for all the systems, pointing to a favorable uptake of cadmium ion in all the cases.

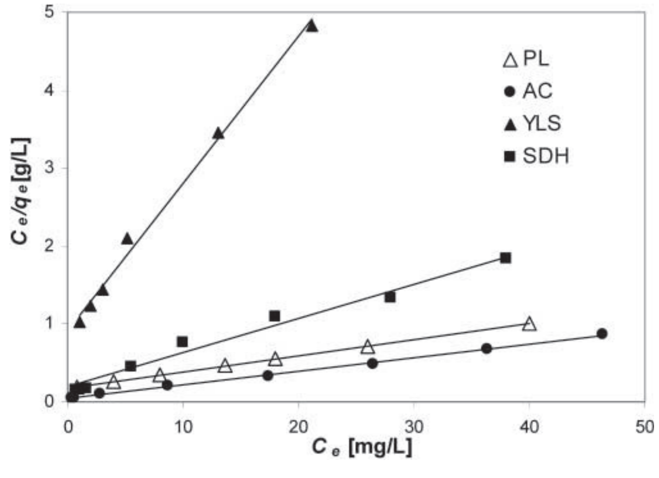

Figure 3. Langmuir plots for equilibrium cadmium uptake by the SDH, YLS, PL, and the AC: comparison between the experimental data (points) and model predictions (solid lines). $C_0 = 5–100$ mg/L; sample dose = 0.3 g/100 mL; pH = 5.8; $T = 28°C$; $t = 7$ hr.

Table 2. Langmuir model parameters estimated for the investigated systems.

Sample	X_m (mg/g)	K (L/mg)
Pure lignin	48.3	0.12
Seed hulls	22.2	0.34
Yerba leaves stems	4.8	0.25
Arundo donax stems[12]	5.7	0.70
Sugarcane bagasse[12]	10.7	0.25
Prosopis ruscifolia sawdust[12]	7.4	1.01
Brazil nutshells[12]	19.4	1.32
Activated carbon	57.3	0.36

As seen in Table 2, the X_m value estimated for PL was higher than those obtained for the SDH and YLS. Nevertheless, it was rather lower than the X_m for the AC, possibly because of its highly developed pore structure and large content of TOFG, already reported in Section 2, the latter being recognized as playing a key role on metal adsorption on ACs.[14,21]

Table 1 summarizes chemical characteristics of the lignocellulosic samples tested earlier[12] as potential biosorbents of cadmium ions, by using the same methods depicted before. The tested samples were sawdust from *A. donax* L. (ADS) *and Prosopis ruscifolia* (PRS), *Sugarcane bagasse* (SCB), and Brazil nutshells (BNS). Table 2 lists the LM parameters for these samples, obtained from cadmium isotherms, applying identical experimental conditions and methodology as the ones used in this work. From the data reported in Tables 1 and 2 for all the lignocellulosic samples tested, it appears that the cadmium uptake, as judged by the calculated X_m values, increases, in general, with the TOFG content. This trend and the results for the incidence of the solution pH on Cd(II) ion uptake suggest that among the different possible mechanisms responsible for metal uptake,[2] ion exchange or chelation apparently prevails.

The X_m estimated for the PL, SDH, and YLS also was found to be in accordance with their lignin contents (Tables 1 and 2). Figure 4 illustrates the X_m values obtained for the SDH and YLS, together with those published earlier involving other lignocellulosic materials—Cd(II) ion systems[12] as a function of the samples' lignin content. An increasing relationship between X_m and the lignin content of the samples can be noticed in Fig. 4. It indicates that the lignocellulosic materials with larger lignin content have a greater ability to uptake cadmium. Lignin, constituting the cells walls of lignocellulosic materials together with cellulose and hemicellulose, is the

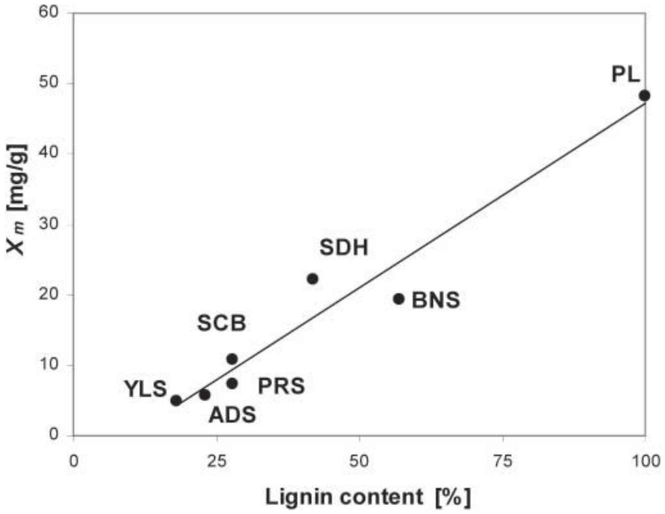

Figure 4. Influence of lignin content on the estimated maximum sorption capacity (X_m) of cadmium for the lignocellulosic samples: ADS, BNS, PRS, SCB, SDH from *M. jalapa*, and YLS., PL. $C_0 = 5\text{–}100\,mg/L$; sample dose $= 0.3\,g/100\,mL$; pH $= 5.8$; $T = 28°C$; $t = 7\,hr$. Solid line only to guide the eyes.

major noncarbohydrate component. It is a complex, cross-linked three-dimensional oxygenated polymer based primarily on three different phenyl-propane units in various proportions, i.e., guaicyl, syringyl, and *p*-hydroxyphenyl monomeric units.[22] Hence, it appears that phenyl-propane units composing lignin provide dominantly surface functional groups for cadmium uptake by the lignocellulosic materials, regardless of other specific features of their cell walls. Surface "free" functionalities on lignin comprise hydroxyl groups, either alcoholic –OHs on the linear chains or the phenolic –OHs on the aromatic groups of the building units, methoxyls ($-OCH_3$) attached to the aromatic rings, and aldehydes and ketones on the linear chains. Thus, although additional complementary techniques should be required to identify specific functional groups responsible for metal binding,[2,23] the increasing trend found for X_m with the TOFG and lignin contents seems to indicate that phenolic groups, which distinguish lignin structure from that of cellulose and hemicellulose, could be the functionalities mainly involved in cadmium uptake. In addition, the apparent easier access of the metal species to lignin in the cell wall of lignocellulosic materials compared with cellulose and hemicellulose could also favor, preferentially, the uptake of cadmium.

4. CONCLUSIONS

The content of lignin composing lignocellulosic materials appeared to have a predominant incidence on their performance in the uptake of cadmium ion from dilute aqueous solutions. Though more data should be needed to attain a completely definite correlation, the present results point to the lignin content of lignocellulosic materials as a proper, indicative tool of their potential cadmium-sorption capability, at least as a first approximation. Accordingly, it might provide useful information to facilitate the screening of lignocellulosic materials for their use as alternative cadmium sorbents from dilute wastewater, contributing to reduce partially tedious experimental work involved in screening tests.

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