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ORIGINAL PAPER

Adsorption of bisphenol A on lignin: effects of solution chemistry

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Abstract Adsorption of bisphenol A on a lignin isolated from black liquor, a waste product of the paper industry, was investigated to assess the possibility of using the lignin to remove bisphenol A from waters. Effects of pH, ionic strength, heavy metals, and dissolved organic matter (DOM) on adsorption were examined. Adsorption equilibrium was approached within 5 h. The adsorption capacity of bisphenol A on lignin was as high as 237.07 mg/g. Ionic strength had no influence on the adsorption, while higher pH above 7.5 inhibited bisphenol A adsorption due to the repulsive electrostatic interaction between bisphenolate anion and the negatively charged lignin surface. The presence of heavy metals of copper and lead increased the adsorption by 11.90 and 26.80 %, respectively, possibly through modifying the physiochemical configuration characteristics of labile fraction of the lignin and reducing the polarity of it. No obvious impact of DOM on the adsorption was observed. The results of this study suggest that lignin is a promising adsorbent material to remove bisphenol A in wastewater containing complex components such as heavy metals and DOM, particularly at acid and neutral conditions.

Keywords Adsorption · Bisphenol A · Dissolved organic matter · Heavy metals · Lignin

Introduction

Endocrine disrupting chemicals (EDCs) can cause adverse health effects on wildlife and humans; therefore, their contamination has drawn increasing scientific and public concerns in recent years (Ifelebuegu 2011; Shareef et al. 2006; Sun et al. 2010; Vandenberg et al. 2010). Bisphenol A (BPA) with structure shown in Fig. 1 is one of the highest volume EDCs produced worldwide with annual production of over 8 billion pounds (Gong et al. 2009; Vandenberg et al. 2010). BPA has been detected in wastewater, surface water, groundwater and even drinking water due to its wide applications in plastic products (Dong et al. 2010; Bautista-Toledo et al. 2005). As a stable compound, BPA is highly resistant to chemical and biological degradation in waters (Bautista-Toledo et al. 2005; Mohapatra et al. 2010). It is, therefore, imperative to explore ways to reduce BPA in the aquatic environment and to minimize its potential exposure to aquatic organisms.

Low cost sorbents obtained from plant and wood wastes have shown ability to efficiently remove organic pollutants and heavy metals from waters (Ngah and Hanafiah 2008a, b; Rodriguez-Cruz et al. 2009). Lignin is a natural polymer and has an aromatic three-dimensional polymer structure with apparent infinite molecular weight and high surface area (Wu et al. 2008). It is present in large quantities in the cell walls of terrestrial plants and is the main binding agent for fibrous plant components, comprising about 16–30 % of plant biomass (Guo et al. 2008). More than 50 × 10^4 metric tons/year of lignin are produced worldwide by pulping (Gosselink et al. 2004). Previous studies have demonstrated that lignin has high adsorption capacities for heavy metals including Pb, Cu, Cr, Cd, Sr, Zn, Ni, and Hg through binding by various organic functional groups (e. g.,

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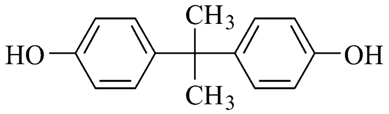

Fig. 1 The molecular structure of bisphenol A

carboxylic and phenolic) on its surface (Guo et al. 2008; Suhas et al. 2007; Wu et al. 2008; Ngah and Hanafiah 2008a, b). Lignin has also been used to efficiently remove organic contaminants such as dyes, bile acid, surfactants, pesticides, phenols, and polycyclic aromatic hydrocarbons (PAHs) from waters (Suhas et al. 2007; Wang et al. 2007). It is, therefore, anticipatable that lignin exhibits high adsorption capacity for BPA and thus can be employed as an adsorbent to remove BPA from the aquatic environment. To the best of knowledge, the removal of BPA by lignin has not been investigated previously. Researches have indicated that solution chemistry parameters such as pH, ionic strength, and co-occurrence of heavy metals strongly affect adsorption of BPA on sediments (Li et al. 2007). Dissolved organic matter (DOM) exists universally, particularly at high concentrations in wastewater.

Effects of DOM on the adsorption of some organic contaminants have been reported (Hollrigl-Rosta et al. 2003). It is unknown whether the presence of DOM affects the adsorption of BPA on lignin. Consequently, investigating these effects on the adsorption is necessary to comprehensively understand BPA adsorption on lignin.

Thus, the objective of this study was to thoroughly investigate the adsorption of BPA on lignin which included the adsorption kinetics and isotherms, and factors that were expected to influence the adsorption such as pH and ionic strength. In addition, influences of coexisting heavy metals and DOM on the adsorption were also examined to explore the adsorption efficiency of lignin for BPA in the actual environment. Copper and lead (Pb and Cu) were taken as the representatives of heavy metals since they are typical divalent metals and commonly found in polluted waters. The experiments were conducted in the laboratory from September 2010 to February 2011.

Materials and methods

Materials

Lignin was obtained from black liquor of a paper mill located in Yunnan, south China. The pretreatment and purification procedures were presented in previous studies (Guo et al. 2008; Wu et al. 2008). BPA (analytical standard, for drug analysis) was purchased from Sigma-Aldrich Co. and was dissolved in methanol (HPLC grade) with the concentration of 2 g/L as the stock solution. Chemicals of $Cu(NO_3)_2$ and $Pb(NO_3)_2$ of analytical grade were purchased from Beijing Chemical Company (Beijing, China).

Two different types of DOM purified from a peat and a commercial humic acid (HA) were applied to examine the influence of DOM on the adsorption of BPA on lignin. They were obtained with the following methods. Peat suspension obtained by mixing 10 g of peat with 300 mL of 0.01 mol/L $NaNO_3$ solution was shaken for 24 h, centrifuged at 4,000 rpm for 20 min, and then filtered through a 0.45 μm membrane. The filtrate called peat dissolved organic matter (PDOM) was stored at 4 °C in the dark before use. About 1.0 g of commercial HA (Tianjin Chemical Factory, China) was dissolved in 1 L 0.01 mol/L $NaNO_3$ solution and the pH was adjusted to 6 with diluted HNO_3 and NaOH. The suspension was filtered through a 0.45 μm membrane and dissolved HA (DHA) was obtained and stored before use. The dissolved organic carbon (DOC) contents in the lignin (at the solid-to-liquid ratio of 1:2) and the DHA and PDOM solutions were determined using a Phoenix 8000 TOC analyzer (Tekmar-Dohrmann, USA).

Total contents of carbon (C), hydrogen (H), nitrogen (N) and sulfur (S) were determined by dry combustion in an auto-analyzer (Vario EL, Elementar Analysensyteme, Hanau, Germany). The oxygen (O) content was calculated by mass difference. The pH value of lignin suspension was measured at a solid-to-water ratio of 1:1 (w/v). Cation exchange capacity was determined by the method of Guo et al. (2008). Zeta potentials in the presence of heavy metals were determined by micro-electrophoresis using a zeta potential meter (Zeta-Meter 3.0 system, Nicolet, USA).

Adsorption experiments

A mixture of lignin and water (3 g in 1L) was vigorously stirred magnetically for 24 h, and then a portion of the suspension containing 15 mg of lignin was transferred into a 40-ml glass centrifuge vial with 30 mL of background solution inside. The background solution contained 0.01 mol/L of $NaNO_3$ to maintain a constant ionic strength and 100 mg/L of NaN_3 to inhibit biological activity. Batch adsorption experiments were conducted at pH 6.0 ± 0.2 except for special notes. Then BPA was added with a 50 μL micro-injector and methanol content in the test solution was less than 0.16 % in volume to avoid co-solvent effect.

The vials sealed with polytetrafluoroethylene (PTFE)-line caps were shaken on a rotary shaker (115 rpm at 23 ± 1 °C) for a given time, centrifuged at 3,200 rpm for 20 min and then about 1.0 mL of supernatant was removed into an injection vial for immediate detection. Each sample and blank control without BPA was conducted in triplicate. The preliminary experiment showed the loss of BPA due to biodegradation, photo-degradation and adsorption on the vials was negligible (<2 %). The amount of BPA adsorbed by lignin was, therefore, calculated by mass balance between solid and liquid phases.

The initial concentration of BPA in kinetic experiments was 2 mg/L, and varying concentration from 0.33 to 300 mg/L was used for the adsorption isotherm experiments. Lignin of 33–10,000 mg/L was used to investigate the effect of dosage on BPA (2 mg/L) adsorption. To determine the effect of ionic strength on adsorption, two different background electrolyte concentrations, i.e., 0.01 and 0.1 mol/L $NaNO_3$, were applied. At the same time, effect of pH (2–9) on adsorption was investigated by adjusting the solution pH with diluted HNO_3 and NaOH.

Concentration of BPA was determined by HPLC (Agilent 1200 series) equipped with a fluorescence detector and the excitation wavelength and emission wavelength were set at 228 and 316 nm, respectively. An Agilent eclipse XDB-C_{18} column (4.6 × 150 mm, 5 μm particle size) was used and the injection volume was 10 μL. The flow phase was acetonitrile:ultra pure water (containing 1 % oxalic acid) 50:50. The retention time of BPA was 3.5 min.

Data analysis

The kinetic data of BPA adsorption were treated with pseudo-first-order and pseudo-second-order kinetic models (Gong et al. 2009; Wu et al. 2008). The pseudo-first-order kinetic equation is $ln\ (q_e - q_t) = lnq_e - k_1 t$, where k_1 is the rate constant for pseudo-first-order kinetic equation and q_e and q_t are the amounts of BPA adsorbed per unit adsorbent at equilibrium and at time t, respectively. The pseudo-second-order kinetic equation is $t/q_t = 1/k_2 q_e^2 + t/q_e$, where k_2 is the rate constant for the pseudo-second-order kinetic equation and q_e and q_t are the amounts of BPA adsorbed per unit adsorbent at equilibrium and at time t, respectively.

Langmuir and Freundlich equations (Wu et al. 2008; Wang et al. 2007) were used to describe the adsorption of BPA on lignin. The Langmuir equation is $q_e = KC_e M/(1 + KC_e)$, where q_e is the adsorbed BPA (mg/g); C_e is the equilibrium concentration of BPA (mg/L); M is the maximum capacity of BPA adsorption (mg/g); K is the adsorption equilibrium constant related to bonding energy (L/mg). The Freundlich equation is $q_e = K_f C_e^n$, where q_e is the adsorbed BPA (mg/g); C_e is the equilibrium concentration of BPA (mg/L); K_f is the sorption coefficient (L/g) and n (dimensionless) is a site energy heterogeneity factor.

The data fit of adsorption model was performed by Origin 7.5. Statistical analysis was completed in SPSS (version 13.0) using ANOVA (Tukey test, $p < 0.05$).

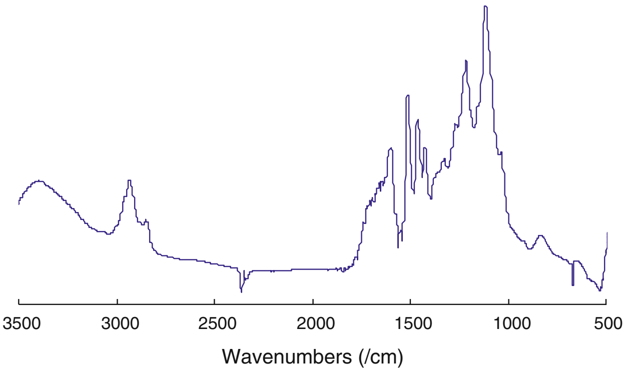

Fig. 2 FTIR spectra of lignin

Results and discussion

Characterization of lignin

The element analysis showed that the lignin had the following elemental percentage composition (%): C, 60.8; H, 5.8; N, 1.3; S, 2.1; and ash, 5.5. The pH value of the lignin suspension was 5.5 at a solid-to-water ratio of 1:1 (w/v), and the cation exchange capacity was 23.9 cmol/kg. The point of zero charge (pH_{PZC}) of lignin was at about pH 2.3. The DOC content of lignin suspension at the solid-to-liquid ratio of 1:2 (g/L) was 5 mg/L.

The functional group composition of the lignin was obtained from FTIR analysis. As shown in Fig. 2, the broad and strong band at about 3,400 cm^{-1} was assigned to aromatic and aliphatic OH groups. Peaks at 1,600, 1,513 and 1,425 cm^{-1} were related to aromatic ring vibrations of the phenyl propane skeleton. The peak at 1,216 cm^{-1} showed C–O stretch vibration of phenolic OH and ether. The 1,034 cm^{-1} band arose from C–O vibration of aliphatic OH together with ether. These observations are consistent with the previous reports about the general chemical composition of lignin (Guo et al. 2008).

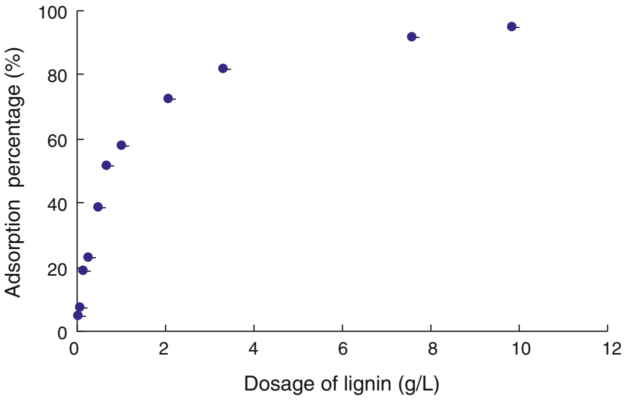

Fig. 3 Effect of lignin concentration on BPA adsorption

Effect of lignin dosage on BPA adsorption

Effect of lignin dosage on BPA adsorption was investigated to optimize the sorbent-to-solution ratio. Figure 3 shows the adsorption efficiency of BPA (2.0 mg/L) as a function of lignin content in suspension. The adsorption percentage increased rapidly from 5 to 95 % with the increase of lignin dosage from 33 to 10,000 mg/L due to a large number of available adsorption sites, and then became relatively stable.

Adsorption kinetics and isotherms

The adsorption kinetics of BPA on lignin consists of an initial rapid adsorption followed by a much slower but continuous adsorption (Fig. 4). The adsorption capacity increased sharply in the first 5 h with more than 98 % of the amount being adsorbed. The adsorption equilibrium was reached with the contact time of less 24 h, and thus 24 h was adopted as the contact time in the following experiments.

The pseudo-first-order and pseudo-second-order models were tested to fit the kinetic adsorption data. The correlation coefficient (R^2) of pseudo-first-order equation was only 0.52 and the equilibrium adsorption capacity, q_e, derived from the equation was 0.23 mg/g which was far lower than the experimental value of 1.56 mg/g. A better fitting was obtained with pseudo-second-order model with the correlation coefficient R^2 of 0.99. Moreover, the calculated equilibrium adsorption capacity (1.43 mg/g) was close to the actual amount of adsorption (1.56 mg/g). The pseudo-second-order model is based on the assumption that the rate-limiting step is a chemical adsorption involving valance force through sharing or exchange of electrons between adsorbent and adsorbate (Mohan et al. 2006). Successful fitting of this model suggests that chemosorption is the rate-controlling step (Wu et al. 2008). The fast adsorption process showed the high adsorption efficiency of lignin for BPA.

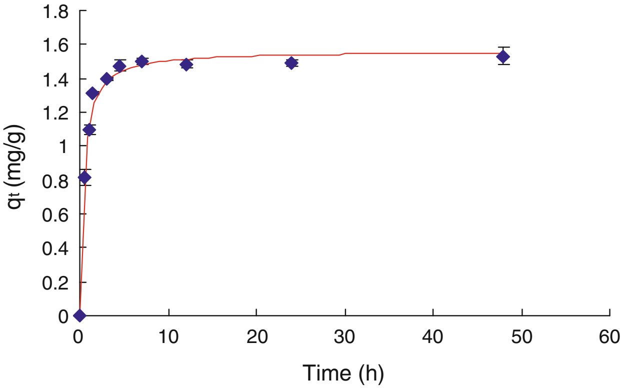

Fig. 4 Adsorption kinetics of BPA on lignin fitted with the pseudo-second-order equation

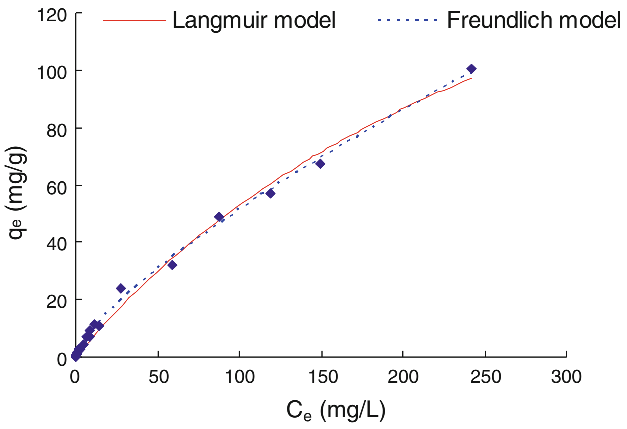

Fig. 5 Adsorption isotherm of BPA on lignin fitted with the Langmuir and Freundlich equations

The adsorption isotherms were well fitted with the Langmuir model ($K = 0.0029$ L/mg and $M = 237.07$ mg/g, $R^2 = 0.99$) and the Freundlich model ($K_f = 1.67$ L/g, $n = 0.74$, $R^2 = 0.99$) (Fig. 5). No significant differences ($p > 0.05$) were observed in the adsorption capacities at the two different background electrolyte concentrations (data not shown), indicating that adsorption of BPA on lignin was ionic strength-independent. Although salting-out effect may influence BPA adsorption (Bautista-Toledo et al. 2005), Shareef et al. (2006) observed that ionic strength (from 0 to 0.1 mol/L KNO_3) exerted no obvious effect on BPA solubility.

Hydrophobic partition and π–π interaction may play an important role in the adsorption of BPA on lignin. Particularly, the π–π interaction between lignin and BPA contributes importantly to the adsorption of BPA on lignin due to the abundant aromatic moieties in the lignin (evidenced by the FTIR spectra). The aromatic ring with hydroxyl group of BPA is a π donor and lignin acts as a π acceptor similar to the binding between BPA and black carbon composed of stacked aromatic sheets (Koelmans et al. 2006; Sun et al. 2010).

Effects of pH, heavy metals and DOC on the adsorption

Solution pH is an important parameter governing the adsorbent–adsorbate electrostatic interactions. Figure 6 shows the effect of pH from 2 to 9 on BPA adsorption on lignin. The adsorption of BPA on lignin showed a slight decrease when the pH increased from 2 to 7, and then decreased significantly when the pH was above 7.5. Change in the adsorption with pH can be explained by the following two reasons. Firstly, raising pH will increase the solubility of lignin. Some adsorbed BPA were consequently released into the liquid phase, which mitigated

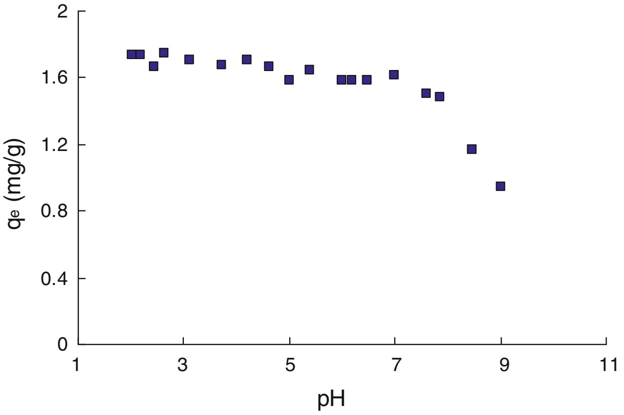

Fig. 6 Effect of pH on BPA adsorption on lignin

BPA adsorption on lignin (Kauper 2004). Secondly, BPA exists in different species at different pH values. It exists in molecular form in acid solution, while $HBPA^-$ and BPA^{2-} are the main species when pH is above 8 (Bautista-Toledo et al. 2005). It is known that zeta potential decreases with the increase of pH. Lignin surface would be covered with negative charges when the pH was higher than pH_{pzc} of lignin, and more negatively charged with the increase of the pH. Therefore, the repulsive electrostatic interaction between bisphenolate anion and the negatively charged lignin surfaces was together responsible for the decreased adsorption.

The influence of metal cations of Cu^{2+} and Pb^{2+} on BPA adsorption by lignin was investigated. The pH was controlled at 5.0 to avoid precipitation of the metal cations. At the concentration of 0.4 mmol/L, the presence of Cu^{2+} and Pb^{2+} increased the BPA adsorption by 11.90 and 26.80 % on average, respectively (Fig. 7). The presence of metal cations may modify the surface physiochemical characteristics of lignin. The lignin has a strong affinity for heavy metal cations (Guo et al. 2008) and adsorption of Cu^{2+} and Pb^{2+} increased the zeta potential of lignin by 23.20 and 49.42 %, respectively. Less negatively charged surface of lignin was more favorable for the adsorption of anion species of BPA. At the same concentration, adsorption of Pb^{2+} increased the zeta potential of lignin more obviously than did Cu^{2+} due to the higher affinity of the lignin for Pb^{2+} than Cu^{2+} (Guo et al. 2008; Suhas et al. 2007), therefore, resulting in a more remarkable enhancement of BPA adsorption on lignin than Cu^{2+}.

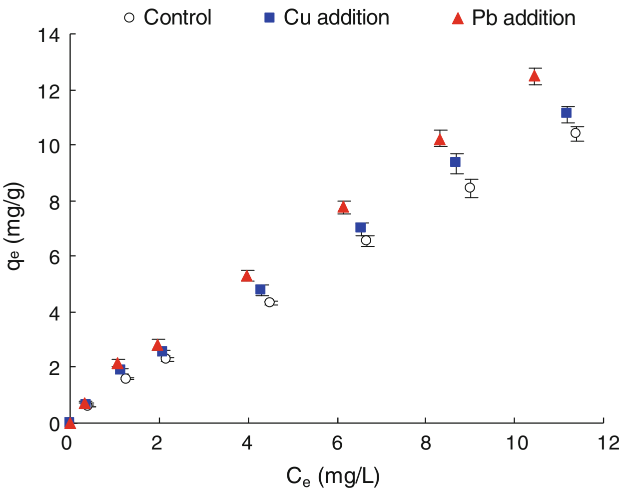

Fig. 7 Influences of Cu^{2+} and Pb^{2+} (0.4 mmol/L) on BPA adsorption on lignin

Moreover, heavy metal cations can also modify the composition and conformation of soft or labile fraction of organic carbon including DOC in the lignin (Luo et al. 2010). Condensation of labile organic carbon in the suspension of lignin can enhance the BPA adsorption. Li et al. (2007) found that the presence of metal cations increased the adsorption of BPA on sediments and proposed that aggregation and flocculation of humus in sediments accounted for the adsorption promotion. Furthermore, Cu^{2+} can complex with O-containing groups (abundant indicated by IR spectra) in the interfaces and reduce the polarity of lignin (Wang et al. 2007). Therefore, more hydrophobic domains were responsible for hydrophobic interactions and thus the adsorption was enhanced.

Influence of DOM on the adsorption of BPA by lignin was investigated using the DHA and PDOM with the DOM concentration in the range of 0–290 mg/L, close to their contents in wastewater (Katsoyiannis and Samara 2007). Results showed that both PDOM and DHA had negligible influences on BPA adsorption on lignin. Possible interaction between lignin and DOM was further determined. The DOC concentrations showed no significant change ($p > 0.05$) after equilibrium of lignin with DOM (DHA and PDOM) for 24 h (data not shown), indicating lignin did not adsorb DOM and adsorption sites on lignin surfaces were not significantly affected by the presence of DOM. This feature favors lignin to be used to remove BPA from wastewaters which normally contain high concentrations of DOM.

Comparison adsorption capacity of BPA on lignin and other sorbents

The maximum adsorption capacity of BPA on lignin derived from the Langmuir equation is 237.07 mg/g, which is higher than that of mineral sorbents (e.g., andesite, diatomaceous earth, titanium dioxide, activate bleaching earth, Fe/Cr hydroxide, and montmorillonite) with the adsorption capacity less than 32.0 mg/g (Namasivayam and Sumithra 2007; Dong et al. 2010). Although the adsorption efficiency of BPA on lignin is a little weaker than that of some reported activated carbon sorbents (263.2–378.3 mg/g) (Bautista-Toledo et al. 2005; Dong et al. 2010), it is still much higher than that of some other activated carbons (23.5–188.9 mg/g), porous carbon and carbonaceous materials prepared by combustion (2.1–41.8 mg/g) (Dong et al. 2010; Bautista-Toledo et al. 2005; Nakanishi et al. 2002). Moreover, the low cost of lignin obtained as by-product, especially compared with activated carbon which is often currently used in wastewater treatment, provides it the advantage to be used as an economical and efficient adsorbent for the removal of BPA from waters.

Conclusion

The lignin shows a strong adsorption affinity for BPA with a maximum capacity of 237.07 mg/g and the adsorption was inhibited at higher pH (>7.5). The adsorption of BPA on lignin is independent of ionic strength. The presence of Cu^{2+} and Pb^{2+} promotes the adsorption probably by modifying the surface characteristics of lignin. DOM has no influence on the adsorption of BPA on the lignin. The adsorption capacity of BPA on the lignin is much higher than many other carbonaceous adsorbents. The low cost and high adsorption capacity provide lignin a great potential as an excellent adsorbent for BPA and perhaps other organic contaminant removal from wastewaters.

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