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Adsorption of bisphenol A from aqueous solutions by *Pleurotus eryngii* immobilized on Amberlite XAD-4 using as a new adsorbent

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ABSTRACT

The adsorption of bisphenol A (BPA) in a fixed-bed column was evaluated in this work. Fungus *Pleurotus eryngii* isolated from Tunceli (Turkey) immobilized on XAD-4 resin was used as an adsorbent for decontamination of BPA from aqueous solutions. The extent of BPA removal capacity was evaluated as a function of parameters such as pH, volume of the sample, and column bed height. Adsorption isotherms of BPA on immobilized *P. eryngii* were determined and correlated with common isotherm equations such as Langmuir and Freundlich models. The mechanism of adsorption of BPA was analyzed preadsorption (XAD-4 + fungus) and postadsorption (XAD-4 + fungus + BPA) using fourier transform infrared spectroscopy. The results show that adsorption ability of BPA by immobilized *P. eryngii* is possible and this system yields the best degree of BPA removal (90%) at pH 11 for an initial load of 120 mg L⁻¹. Therefore, this material can be used as a very effective adsorbent for BPA decontamination in aqueous solutions.

Keywords: Bisphenol A; Pleurotus eryngii; XAD-4 resin; Removal; Fixed-bed column

1. Introduction

Among the registered xenobiotics that are currently in use, BPA (2,2-bis-(4-hydroxyphenyl)-propane) has generated the greatest amount of interest and controversy during the past decade. Recent findings have revealed that BPA is a selective estrogen receptor modulator [1]. Furthermore, there is an evidence that BPA may be an estrogen-mimicking chemical in both *in vivo* and *in vitro* experiments [2,3]. BPA is extensively used and it is one of the key raw materials used to manufacture polycarbonate plastic and epoxy resins [4].

While the source for human exposure to BPA is food and liquid storage containers, BPA is released into the environment either through sewage treatment effluents (human-ingested BPA being eliminated through sewage) [5] or landfill leachate (hydrolysis of BPA from plastics [6] or natural degradation of polycarbonate plastics). Even though sewage effluent and landfill leachate are point sources of BPA release in the environment, fragments of epoxy resins and

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polycarbonate plastic debris entering the watershed through run-off are nonpoint sources, which create additional challenges for remediation [7].

Pleurotus eryngii (oyster mushrooms, higher Basidiomycetes) comprises a group of edible mushrooms with important biotechnological and environmental applications such as degradation of toxic chemicals and hazardous wastes. These mushrooms are able to produce the relevant hydrolytic (cellulases and hemicellulases) and oxidative (ligninolytic) extracellular enzymes required to degrade the major components of lignocellulosic biomasses into low-molecular weight compounds that can be assimilated for fungi nutrition [8–10].

Solid-phase extraction (SPE) is a very popular technique for rapid and selective sample preparation. The SPE has been used for purification, trace enrichment, desalting, derivatization, and class fractionation among others. The usage of biological substances for the preconcentration and separation of some organic materials at trace levels is popular due to good adsorption properties such as high surface area and high adsorption capacities for biological substances [11].

The sorption of BPA from various synthetic or natural mediums has already been studied with an example being the removal of BPA via a *Trametes versicolor* membrane bioreactor. Mixed consortia of immobilized microorganisms [12], and batches of activated and deactivated sludge [13] have also been used.

In this study, the fungus P. eryngii (collected from Tunceli-Pulumur region) is here studied as a solidphase adsorbent for the removal of BPA. This novel study is the first report about the adsorption of BPA by immobilized fungi on Amberlite XAD-4 by fixedbed column system. In order to determine the optimum conditions for quantitative recovery of BPA, various analytical parameters such as pH, amount of adsorbent, and volume of sample solutions were investigated. This firstly reported BPA removal fungus-loaded column system may have some advantages such as cost-effective, reusability, short process time, without toxic limitation, highly recovery, etc., compared with other previously studied methods (membrane bioreactor or batch system).

2. Material and method

2.1. Chemicals and apparatus

The Amberlite XAD-4 resins were purchased from Alfa Aesar (LOT: 10170146, 76185 Karlsruhe, Germany). BPA, with purity of 99%, was purchased from Sigma Aldrich (LOT: MKBH6262V, Germany). Acetonitrile was obtained from Carlo Erba Reagents (LOT: 41237200, Italy). All other chemicals and solvents used in the study were of analytical and HPLC grade purchased from standard manufacturers. For SPE, a 1.0 cm \times 10.0 cm glass column was used (Isolab, Germany). A Thermo Orion 5 Star model (Germany) digital pH meter was used to adjust the pH of BPA solutions. A Shimadzu Prominence LC-20A model (Japan) equipped with a diode array detector (HPLC-DAD) was used for all chromatographic measurements.

Fourier transform infrared (FTIR) spectra of the pretreated *P. eryngii*-loaded Amberlite XAD-4 were compared to the FTIR of the *P. eryngii*-loaded Amberlite XAD-4 after BPA sorption. The spectra were recorded in the frequency range of 500–4,000 cm⁻¹ using an FTIR spectrophotometer (Nicolet, Protégé 460, Madison, WI, USA). The samples were analyzed with Attenuated Total Reflectance.

2.2. Pretreatment of Amberlite XAD-4

Amberlite XAD-4 is a polymeric and a nonionic hydrophobic polyaromatic resin. It was used as the adsorbent for the adsorption behavior of BPA in aqueous solutions. Therefore, this adsorbent resin is one of the best polymeric adsorbents to remove of the phenolic compounds in contaminated water sources. But, Amberlite XAD-4 has some extreme hydrophobic surface. So, it requires pretreatment with solvents such as methanol and acetonitrile; this process enhances its surface contact with the aqueous solutions [14–16]. Through the following steps, before usage, XAD-4 was purified from contaminants. The resin was washed with deionized water several times (until neutral pH). Then, it used to remove inorganic impurities such as 1 M NaOH and 3 M HCI and washed with ethanol several times and rinsed for 24 h to remove organic impurities [17]. The resin was again washed with deionized water several times. When all processes were done, no yellow color was observed and no precipitate was detected by 0.1 M AgNO₃ solution in the eluent. As a result, Amberlite XAD-4 was considered purified [18]. Finally, the resin was dried at 40°C in an oven (UNB 400 Memmert, Germany) for 10 h prior to use and then put into contact with fungus (P. eryngii).

2.3. Preparation of fungal biomass as an adsorbent

Indigenous isolate of *P. eryngii* var. *ferulae* was collected from the province of Tunceli-Pulumur in Turkey and used for all described procedures. The fungal sample was washed twice with distilled water to remove contaminants and then dried at room temperature. The dried fungal biomass was ground in a porcelain mortar to obtain a fine powder. It was then

dried at 80°C in an oven for 24 h to confirm the complete death of the dried cells. For viability testing, the cells were inoculated to a Sabouraud dextrose agar medium at 27°C for 24 h [19]. The absence of mycelian *P. eryngii* indicated the complete death of the fungus. The dead cells were stored at -5°C in a deep freeze until further use. Dry biomass powder (0.2 mg) was mixed with 2 g of Amberlite XAD-4 resin.

2.4. Preparation and immobilization of fungus (P. eryngii) on Amberlite XAD-4

The immobilization of fungus (*P. eryngii*) on the substrate was performed as follows: 0.1 g of fungus powder was mixed with 1 g of Amberlite XAD-4. The mixture was wetted with 2 mL of ultra-pure water several times, thereby improving the immobilization efficiency and ensuring it was thoroughly mixed. After these processes, the mixture was heated in an oven at 50°C for 24 h to dry the mixture, thereby improving the immobilization efficiency. The product obtained was then ground to get original size of less than 60 mesh and used as an adsorbent for BPA. The turbidity analyses were made to determine the immobilization efficiency of XAD-4 with fungus. Turbidity analyses show that all amount of fungus (0.1 g) immobilized on XAD-4.

2.5. Preparation of the fixed-bed column

2.2 g of Amberlite XAD-4 loaded with *P. eryngii* was wetted with 3 mL of methanol. The mixture was transferred to a $1.0 \text{ cm} \times 10.0 \text{ cm}$ glass column bed





Notes: (1) supernatant sample, (2) Amberlite XAD-4 + fungus fixed-bed column system, (3) peristaltic pumps, and (4) magnetic stirrer. heights (BH₁, BH₂, BH₃, and BH₄) of 2.6, 5.2, 8.0, and 11.4 cm, respectively (Fig. 1). Ultra-pure distilled water was passed through the column in order to condition and clean it before use. For preparation of BPA solution as 120 mg L^{-1} , initial concentration was solved in methanol–water mixture (1:9, v:v). A flow rate of 0.81 mL min⁻¹ was employed throughout the experiments. The effluents were collected fractionally and analyzed by HPLC-DAD.

Besides, to calculate the influence of pH on the BPA adsorption, an amount of 1.0 g Amberlite XAD-4 loaded with 0.1 g (10:1, w:w) *P. eryngii* was fixed in a 10-mm i.d. glass column and 10 mL BPA solution containing 0.12 g L^{-1} was passed at an optimum 0.81 mL min⁻¹ flow rate at various pH values (pH 2–11) through BH₃ column bed height.

2.6. Desorption procedure of BPA

After all adsorption process, 10 mL of various solutions (acetonitrile, hydrochloric acid, methanol) were passed through the column for desorption step. The result of this process was given in Table 3. Desorption recovery was calculated in eluent.

2.7. Analysis methods

Each effluent sample was pretreated by filtering it through a membrane filter of 0.22 μ m pore size. HPLC analysis was performed using a liquid chromatography, equipped with a diode array detector (DAD), a binary pump, a vacuum degasser, an autosampler, and a temperature-controlled column oven. The column used was Kromasil C-18 (4.6 mm × 150 mm, 5 μ m). An isocratic mode was used and the mobile phase was acetonitrile/water (50:50). The flow rate was set to 1 mL min⁻¹. The injection volume was set to 10 μ L. Column temperature was set at 40 °C and the excitation wavelength of 226 nm was used. The retention time of BPA was 5.0 min (Fig. 2). The limit of detection and limit of quantification for BPA were calculated to be 5.0 and 16.7 μ g L⁻¹, respectively.

3. Results and discussion

3.1. Effect of pH

The adsorption of phenolic compounds including their cationic or anionic form by microorganisms is associated to the attraction exerted on organic wastes through functional groups found of this kind of biomass [20,21]. Therefore, the effect of the pH on the adsorption is important in this system. The pH value of the sample solutions was adjusted to a range of 2–11



Fig. 2. Chromatogram of BPA (a) the diagram of before BPA adsorption and (b) the diagram of after BPA adsorption.



Fig. 3. Effect of pH on adsorption of BPA (initial concentration: 120 mg L^{-1} , flow rate: 0.81 mL min⁻¹, solution volume: 10 mL).

with NaOH or HCL. The dependence of BPA adsorption on pH is shown in Fig. 3. The maximum adsorption was carried out at pH 11. Obviously, the adsorption capacity exhibits an increasing extent when the pH was increased from 7 to 11, while the BPA adsorption shows a decreasing trend at a higher pH ranging from 2 to 7. These results are consistent with the studies by Dogru et al. [17].



Fig. 4a. Effect of sample volume on adsorption of BPA by Amberlite XAD-4 resin loaded with *P. eryngii* (initial concentration: 120 mg L^{-1} , flow rate: 0.81 mL min⁻¹, pH 11, BH₃: 8.0 cm).



Fig. 4b. The effect of bed height on adsorption of BPA (Initial concentration: 120 mg L^{-1} , flow rate: 0.81 mL min^{-1} , solution volume: 10 mL).



Fig. 4c. Adsorption equilibrium isotherm of BPA in aqueous solution onto the *P. eryngii* immobilized on Amberlite XAD-4.

3.2. Effect of the volume of sample solutions

Actual samples such as wastewater, industrial wastes consist of various organic compounds, have different concentrations. Thus, the amount of BPA in the water solution is important in order to define the effect of sample volume on maximum removal capacity of the adsorbent. In order to calculate this capacity, 5, 10, 20, 50, and 100 mL solutions of BPA

(120 mg L⁻¹, in BH₃: 8.0 cm) were passed through the column under optimum conditions. It was discovered that BPA could be adsorbed quantitatively (107.7 mg g⁻¹) up to 10 mL (1.2 mg BPA) of the sample solution (Fig. 4a). As shown in Fig. 4a, the amount of adsorbed BPA increased from 5 to 10 mL sample volume, while other sample volumes (20, 50, and 100 mL) exhibit a constant extend. This revealed that the adsorbent is fully loaded by BPA.

3.3. Effect of adsorbent amount (column bed height)

The effect of the amount and column bed height of adsorbent (Amberlite XAD-4 loaded with *P. eryngii*) on the retention and the recovery was studied for BPA (Fig. 4b). The mass and height of adsorbent varied from BH₁ to BH₄, as shown in Table 1. The maximum adsorption capacity of BPA was achieved on BH₃. Therefore, BH₃ was used for BPA in subsequent experiments. The adsorption capacity of BPA was increased up to BH₃ and then this amount of adsorbed BPA was reduced at BH₄. The reason for this decreasing derived from increasing in adsorbent mass, while initial concentration is constant. Experimental results comply with the literature data [22,23].

3.4. Adsorption isotherms

The adsorption isotherms are the data at the equilibrium point. Some models were matured to describe adsorption system [24,25]. Due to the shape of the isotherms, the sorption data were calculated according to Freundlich and Langmuir equations as given below Eqs. ((1) and (2)):

$$q_{\rm e} = K_{\rm F} C_{\rm e}^{1/n} \tag{1}$$

$$\frac{C_{\rm e}}{q_{\rm e}} = \frac{1}{K_{\rm L}C_{\rm e}} + \frac{C_{\rm e}}{Q_{\rm m}} \tag{2}$$

where q_e is the amount of BPA per unit weight of the fungus (mg g⁻¹), C_e , the equilibrium concentration of BPA (mg L⁻¹), while K_F , K_L , and n are constants that

Table 1

The mass and bed height of *P. eryngii* immobilized on Amberlite XAD-4 in fixed-bed column system^a

Adsorbent	BH_1	BH ₂	BH ₃	BH_4
Height (cm)	2.6	5.2	8.0	11.4
Mass (g)	0.38	0.76	1.14	1.52

^aP. eryngii and Amberlite XAD-4 ratio: (0.1–1.0 g) (w/w).

give estimates of the adsorption capacity and intensity, respectively. $K_{\rm L}$ is a direct measure of the intensity of adsorption process (L mg⁻¹), and $q_{\rm m}$ is a constant relating to the surface area occupied by a monolayer of BPA, reflecting the adsorption capacity (mg g⁻¹).

Based on the data of q_e from the fittings of the pseudo-second-order adsorption rate model, q_m and K_L can be determined from its slope and intercept from a typical plot of $1/q_e$ vs. $1/C_e$. In Eq. (1), the slope 1/n, ranging between 0 and 1, is a measure of adsorption intensity or surface heterogeneity. K_F is a constant for the system, related to the bonding energy.

The amount of BPA increases with the equilibrium concentrations of BPA on solutions as seen in Fig. 4c. The results of the Langmuir and Freundlich isotherms fitted using the data of adsorption capacity from the regression of Eq. (2) at 25 °C are presented in Table 2. Obviously, it can be seen in Table 2 that the Langmuir model yields a somewhat better fit than the Freundlich model as reflected by correlation coefficients (R^2) of 0.99 and 0.97, respectively. At the same time, the values of K_L and K_F define a measure of adsorption capacity (Table 2) [24–26].

3.5. Desorption studies

For desorption studies, methanol, hydrochloric acid, and acetonitrile were used as eluting solutions to obtain the desorption rate of BPA. The intent of elutions is to remove the reloading BPA as complete as possible. The conditions of adsorption were used to determine the desorption of BPA. The effect of various solutions on the percentage of desorption was illustrated in Table 3. The similar results reported in another studies [27]. Nevertheless, the acetonitrile was better than the methanol solutions in removing BPA from fixed-bed columns as opposed to the hydrochloric acid which did not manage to effectively remove any of the BPA from fixed-bed columns.

3.6. FTIR studies

The fungi have a thick cell wall, rigid structure composed of complex layers which have polysaccharides, proteins, lipids, and polyphosphates. The most

Table 2

Parameters in the Langmuir and Freundlich adsorption isotherm models of BPA onto adsorbent at 25 $^\circ\!C$

Langmuir			Freundlich		
$\overline{q_{\rm m}} \ ({\rm mg} \ {\rm g}^{-1})$	$K_{\rm L} ({\rm Lm \ g}^{-1})$	R^2	$\overline{K_{\rm F}} \ ({\rm mg \ g}^{-1})$	п	R^2
238	0.000021	0.99	101.7	1.2	0.97

Effect of the type and volume of elution solutions on the recovery of BPA				
The kind of elutions	Concentration (mg L^{-1})	Volume (mL)	Recovery (%)	
Acetonitrile	500	10	26	
Hydrochloric acids	500	10	10	
Methanol	500	10	0	



Fig. 5. FT-IR spectra of BPA (a) the diagram of after BPA adsorption and (b) the diagram of before BPA adsorption.

common constituent of the wall is chitin, consisting of N-acetyl glucosamine residues. Amberlite XAD-4 is a polymeric resin, supplied as white insoluble beads. It is a nonionic cross-linked polymer which drives its adsorptive properties from its patented macro-reticular structure (containing both a continuous polymer phase and continuous pore phase), high surface area, and the aromatic nature of its surface. This structure gives Amberlite XAD-4 polymeric adsorbent an excellent physical, chemical, and thermal stability. On account of these reasons, fungi both physically and chemically interact with polymeric resin surface. The mechanism of fungi binding to XAD-4 might be explained by which it involves surface adsorption to functional groups. These interactions between fungus and XAD-4 have contributed to adsorption of BPA in this column system.

Table 3

The FT-IR spectra before and after the adsorption of BPA were compared in Fig. 5. In this figure, the peaks observed around 2,900 cm⁻¹ can be attributed to the stretching vibrations of the C-H alkyl groups for

BPA. The peak around $1,600 \text{ cm}^{-1}$ is due to the C=C aromatic or may be asymmetric and symmetric stretching of C=O vibration for BPA [28]. The band around 3,407 cm⁻¹ can be attributed to the presence of O-H and N-H groups for BPA. The shifts and intensity changes of the FTIR bands observed were rather weak, which can be an indication of the dominance of ion exchange over the precipitation/coprecipitation occurring during BPA sorption on sorbent.

This study is the first report about adsorption of BPA using column system. Previously, batch system has been investigated using different adsorbents such as, grapheme [29], carbon nanomaterial [30], zeolite [20], and silica [31] for BPA adsorption.

4. Conclusion

The adsorption of BPA from aqueous solutions by P. eryngii immobilized on Amberlite XAD-4 in a fixed-bed column system was investigated and the parameters (such as pH, bed height) were optimized to achieve maximum adsorption. Both Freundlich and Langmuir isotherms show good fit to the experimental sorption equilibrium data. From the isotherm data obtained from the fittings of the models, the Langmuir model yields a comparatively better fit than the Freundlich model for the adsorption of BPA by *P. eryngii*. It should be noted that about 90% of the total BPA load (120 mg L⁻¹) was removed during the adsorption. Thus, this material can not only be used as a novel effective adsorbent for removal of BPA but also other phenolic components from aqueous solutions. In addition, researchers can use this column system again and again, and this system can be applied to the field scales and also laboratory scales.

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Nomenclature

BPA	_	bisphenol A
BH_1	—	bed heights 1
BH ₂	—	bed heights 2
BH ₃	_	bed heights 3
BH ₄	_	bed heights 4

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