Removal of Bisphenol-A from Aqueous Solution Using Polymeric Resin Impregnated with Phosphorous Based Extractant

Sakshi Batra and Dipaloy Datta

***Abstract*—A study was conducted for the adsorption of Bisphenol A (BPA) from aqueous solution using solvent impregnated resin (Amberlite XAD-7 was impregnated with trioctylphosphine oxide). FTIR, FE-SEM and EDX analysis characterized this prepared resin. Various parameters like contact time, the mass of adsorbent, BPA initial concentration, and the temperature was studied. For impregnated resin percentage removal was obtained 88.09% within 120 min. For BPA adsorption, optimum adsorbent dosage was found to be 8 $g \cdot L^{-1}$. Adsorption of BPA decreased with increase in temperature. Different models like Langmuir, Freundlich and Temkin models were used to predict the equilibrium points. Kinetics of BPA adsorption was predicted using pseudo-first order, pseudo-second order, and intraparticle diffusion models.**

***Index Terms*—Amberlite XAD-7, bisphenol–A, solvent impregnated resin, trioctylphosphine oxide.**

I Introduction

Bisphenol A (BPA), found to be an endocrine disrupting chemical (EDCs) that is used as an essential chemical intermediate [1].The release of this compound into the environment has become a significant concern of public health. Usually used BPA discharged into an aquatic environment through industrial wastewater effluent [2]. It was reported that it mainly affects the reproductive and endocrine system of females. Hence it is necessary to remove BPA from water using highly efficient technology. Till now many adsorptive and oxidative methods like Ozonation [3], adsorption using different adsorbents [4]-[5], photo catalysis [6], membrane separation [7] and solvent extraction [8] were applied for the removal of BPA.

Although these techniques have some disadvantages like leaching of extractant into the aqueous phase, the problem of entraining, reusability of adsorbents, expensive and synthesis complexity [9]. A Solvent impregnated resin is one of the prominent techniques that combine the process of solvent extraction and ion exchange resin [10]. Solvent impregnated resins are macroporous resins in which extractant is impregnated into the pores of resin. The Essential advantage of this method is the usage of prepared adsorbent up to several numbers of times without loss of extractant [11], [12]. In this study, Bisphenol A (BPA) was removed using a prepared impregnated resin, i.e. Amberlite XAD-7 impregnated with extractant TOPO (Trioctylphosphine oxide). Various experimental parameters such as contact time, mass of adsorbent, initial BPA concentration and temperature were studied and their effect on adsorption of BPA was analyzed.

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II Methods and Materials

A. Materials

Amberlite XAD-7 (AX7), Trioctylphosphine oxide (TOPO) and Bisphenol A (BPA) were procured from Sigma-Aldrich, Ethanol (> 99% of purity, Merck Germany) was used for purification of AX7. For filtration Whatman quantitative filter paper was used. Deionized water was used for our work which was produced by Millipore Milli-Q water system.

B. Preparation of SIRs

For the preparation of SIR wet impregnation method was used [7]. It includes following steps like washing, drying, impregnation, and filtration. Firstly, washing was done using ethanol and water to remove present impurities and to maintain the neutral pH using magnetic stirrer at room temperature. After that, filtration was done and this washed resin (WAX7) was kept for drying in an oven. Then extractant TOPO (1 g TOPO per g of XAD-7) was used for impregnation of WAX7. Impregnation was done in a temperature bath shaker maintained at room temperature for 24 h. These impregnated beads (I-WAX7) was filtered and kept for overnight drying and later in an oven. Properties of Amberlite XAD-7 are given below [13].

TABLE I: Physical Properties of Amberlite XAD-7 [13]

Physical Properties of Resin	
Type (Matrix)	Aliphatic ester
Surface Area	450 m^2/g
Pore Size	450 Å
Particle Size	20-60 mesh

C. Characterization

Characterizations of WAX7 and I-AX7 done using FE-SEM (Scanning Electron Microscopy), EDX (Energy Dispersive X-Ray Spectroscopy), FTIR (Fourier Transform Infrared Spectroscopy). FTIR was performed to study the functional groups present in the resin in the range of 4000 to

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400 cm^{-1} (FT-IR Spectrum 2, Perkin Elmer, USA). FE-SEM was done (Nova NanoSEM 450, Perkin Elmer, USA) to observe the surface structure of prepared adsorbent imaged at a magnification of 100000×. EDX was performed to analyze the present elements in the SIRs

D. Adsorption Experiments

A kinetic study was conducted using I-AX7 (8 $g \cdot L^{-1}$) with 10 $mg \cdot L^{-1}$ of initial BPA concentration up to 120 min. Water bath shaker was used to conduct adsorption experiments. Effect of the mass of adsorbent was performed in the range of 1 $g \cdot L^{-1}$ to 10 $g \cdot L^{-1}$ at 298 K for 60 min. Equilibrium study was conducted by varying a concentration of BPA from 10 $mg \cdot L^{-1}$ to 50 $mg \cdot L^{-1}$ .To observe the outcome of temperature on the adsorption capacity experiments was performed at 298 K, 313 K, 323 K, and 333 K for 60 min at 10 $mg \cdot L^{-1}$ BPA concentration. The final concentration of BPA was analyzed using UV-VIS spectrophotometer (UV-1800, Shimadzu) at 276 nm. The adsorption performance of resin was evaluated by calculating the percent removal (% *R*), and the adsorption capacity (q_e or q_t) of BPA was calculated using the following equations (1) and (2), respectively

$$\% \text{ Removal} = \left(\frac{C_o - C_e}{C_e}\right) \times 100 \quad (1)$$

$$q_e \text{ or } q_t = \left(\frac{C_o - C_e}{m}\right) V \text{ or } \left(\frac{C_o - C_t}{m}\right) V \quad (2)$$

Here,

C_o, C_e and C_t represents the BPA initial, equilibrium and at time t concentrations, respectively in $mg \cdot L^{-1}$. *V* and *m* represent the volume of aqueous solution of BPA and adsorbent mass L and g, respectively.

III Results and Discussion

A. Characterization: FTIR

FTIR, FE-SEM and EDX analysis was performed for WAX7 and I-WAX7. From the FTIR analysis it was observed in Fig. 1 , in the WAX7, a sharp peak at 2956.00 cm^{-1} indicates the moisture present in the resin [2]. A peak was observed at 1728 cm^{-1} assigned to the ester group in the resin. After impregnation of TOPO, two new peaks were observed at 2926 cm^{-1} and 2850 cm^{-1} which represent the adsorption of extractant into the pores of resin. A new peak at 1143.21 cm^{-1} was observed. This peak denotes the phosphoryl group that indicates the proper impregnation of extractant into the pores of resin.

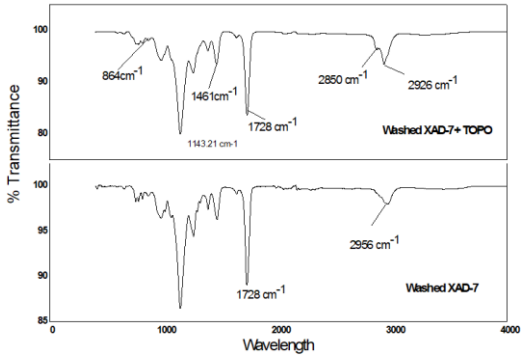
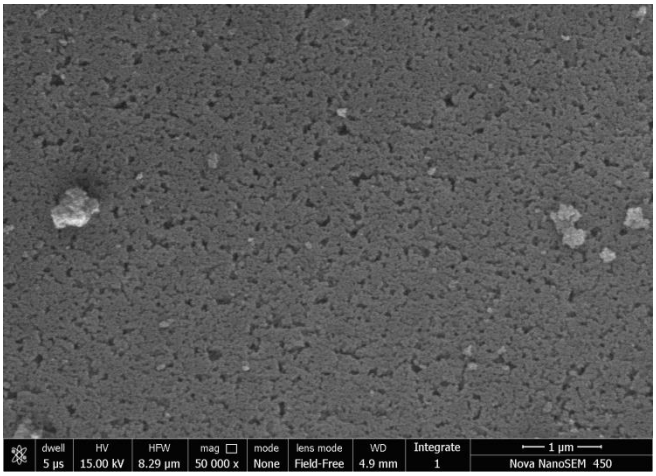


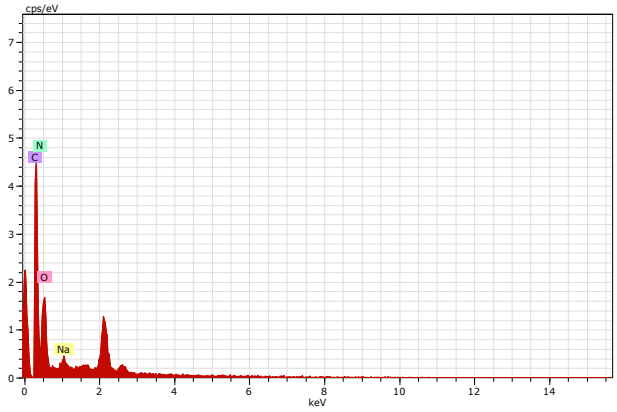
Fig. 1. FTIR analysis of WAX7 and I-WAX7.

B. Characterization: FE-SEM and EDS Analysis

SEM analysis was represented in Fig. 2 and Fig. 3 at magnification of 50000 x. From the SEM analysis, it was observed that in the micrographs of WAX7 pores are present. After impregnation of extractant, these pores are filled that confirm the impregnation of the extractant in I-WAX7 Further from the EDS analysis also it was seen that initially, only C and O elements are observed but after impregnation P element was also observed that prove the conformity of presence of extractant into the pores of resin.



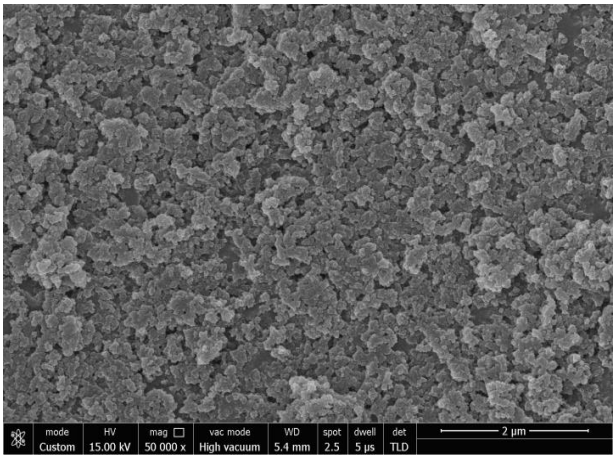
(a)



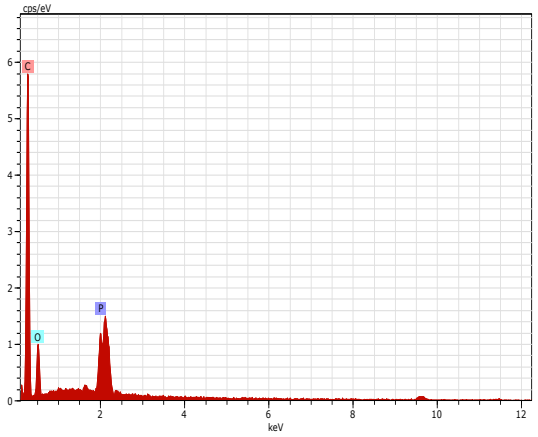
Element	Percentage
O	31.52
C	25.41

(b)

Fig. 2. (a) FE-SEM image of WAX7 at 50000 × magnification (b) EDS image of WAX7.



(a)



Element	Percentage
O	15.35
C	80.07
P	4.58

(b)

Fig. 3. (a) FE-SEM image of I-WAX7 at 50000 × magnification (b) EDS image of I-WAX7.

C. Effect of Contact Time

Contact time study was done to observe the effect on the BPA adsorption using I-WAX7 shown in Fig.4. From the figure, it was observed that initially, as adsorption time increases from 2 to 60 min adsorption of BPA increased from 32% to 88.09% and it becomes constant after 60 min 88.09 % Equilibrium time was found to be 60 min for the further experiments. Different kinetics models (Pseudo first order (PFO), Pseudo second order (PSO) and intraparticle diffusion model (IPD) were used to determine the adsorption kinetics of BPA. It was observed that PSO model give best fit with the experimental data. Fitting of PSO model represented in Fig. 5.

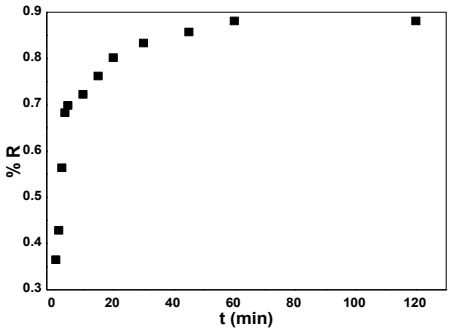


Fig. 4. Effect of contact time on adsorption of BPA using I-WAX7 (m = 8g·L^{-1}; C_o = 10 mg·L^{-1}; T = 298 K; and speed of shaker = 100 rpm).

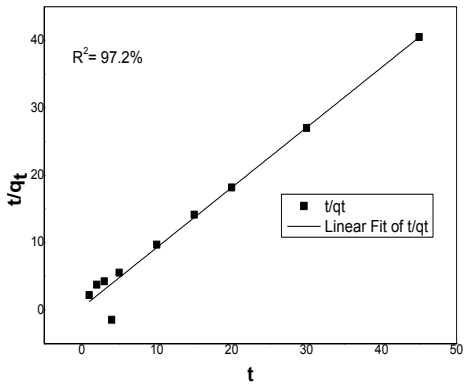


Fig. 5. Fitting of PSO with experimental data (m = 8g·L^{-1}; C_o = 10 mg·L^{-1}; T = 298 K; and speed of shaker = 100 rpm).

D. Effect of Mass of Adsorbent

Experiments were conducted at 10 mg·L^{-1} initial BPA concentration at 298 K and experiments was conducted to observe the effect of the mass of adsorbent on BPA adsorption. A Dosage of adsorbent (I-WAX7) was varied from 1 g·L^{-1} to 10 g·L^{-1} and data points are shown in Fig. 6. It was concluded from the figure that with increasing the amount of adsorbent from 1 to 8 g·L^{-1} percentage removal of BPA increased (~ 75% to ~ 89%). Initially, at lower dosage of adsorbent less number of adsorption sites are present for the removal of BPA. With increased in the adsorbent dosage the removal of BPA enhanced. Therefore, 8 g·L^{-1} found to be optimum dosage for BPA adsorption.

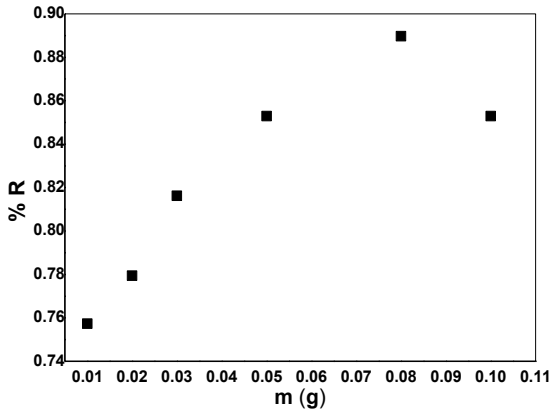


Fig. 6. Effect of mass of adsorbent on adsorption of BPA using I-WAX7 (C_o = 10 mg·L^{-1}; T = 298 K; and speed of shaker = 100 rpm).

E. Effect of Initial BPA Concentration

Concentration study was conducted by changing a BPA concentration from 10 mg·L^{-1} to 50 mg·L^{-1} and results are shown in Fig. 7. 8 g·L^{-1} of I-AX7 was mixed in 100 ml of flask with different concentration of BPA and put this solution into shaker for 120 min and at 298 K. it was observed from the figure that if concentration increases from 10 mg·L^{-1} percentage removal for BPA decreased. Three different models (Langmuir, Freundlich, and Temkin) were applied to predict the experimental values. It was found that the Langmuir model gives the best fit (R^2 = 0.996) with the experimental data. It can be concluded that adsorption of BPA is found to be monolayer adsorption on the surface of adsorbent. Fitting of Langmuir isotherm model was shown in Fig. 8

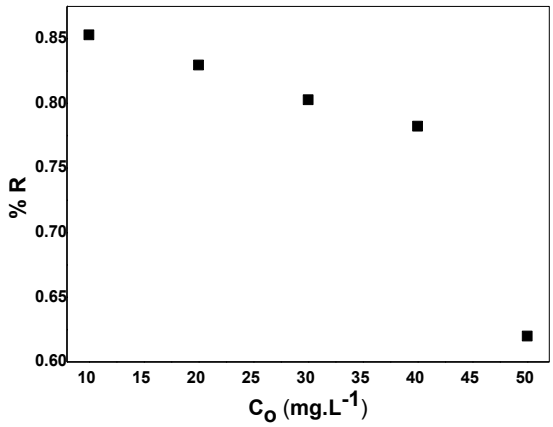


Fig. 7. Effect of concentration on BPA adsorption using I-WAX7 (m = 8g·L^{-1}; T = 298 K; and speed of shaker = 100 rpm).

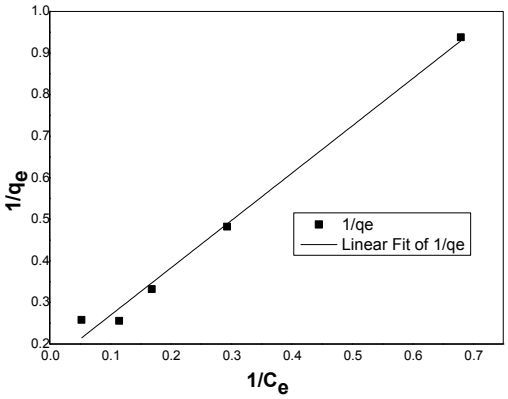


Fig.8. Fitting of Langmuir isotherm with the Experimental data (m = 8g·L^{-1}; C_o = 10 mg·L^{-1}; and speed of shaker = 100 rpm).

F. Effect of Temperature on Removal of BPA

Temperature effect on the removal of BPA using I-WAX7 was shown in Fig. 9. A study was conducted by mixing 8 mg·L^{-1} of the adsorbent into BPA solution (10 mg·L^{-1}) for 120 min in a water bath shaker. From the figure, a decrease trend was observed between the temperature and percentage removal because of increase in the kinetic energy of solute molecules. This increased in the kinetic energy reduce the interaction between the BPA molecules and adsorbent [14]. With increasing in temperature from 298 K to 333 K, % R of BPA decreased. It was found that maximum percentage removal (84.5%) was obtained at 298 K.

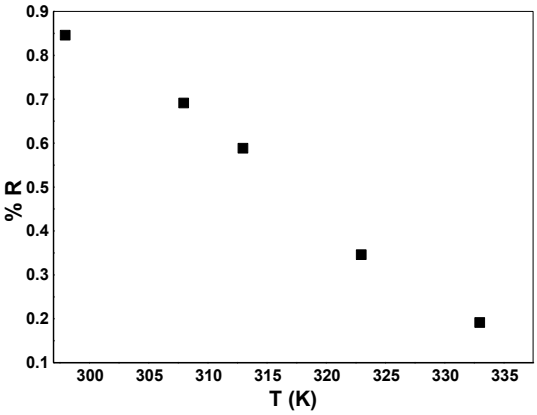


Fig. 9. Effect of temperature on BPA adsorption using I-WAX7 (m = 8g·L^{-1}; C_o = 10 mg·L^{-1}; and speed of shaker = 100 rpm).

IV Conclusion

In this study, phosphorous based extractant (Trioctylphosphine oxide) was used to impregnate the Amberlite XAD-7 for the adsorption of BPA. Impregnation was confirmed using FTIR, FE-SEM and EDX analysis. Various parameters like contact time, the mass of adsorbent, an initial concentration of BPA and temperature was performed to observe the effect on BPA removal. It was concluded that equilibrium time for BPA adsorption was obtained in 120 min. Pseudo second order model gives best fitted isotherm that shows the chemisorption phenomena of BPA. With increasing in dosage percentage removal of BPA increased and at 8g·L^{-1} the removal of BPA found to be optimum. Percentage removal decreased at higher temperature. Concentration studies concluded that at lower concentration removal of BPA enhanced due to more adsorption sites are present. Langmuir model gives the best fit that indicates the monolayer adsorption of BPA on the surface of the impregnated resin.

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