

## ADSORPTION OF BISPENOL A ONTO CATIONIC MODIFIED ZEOLITE

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### ABSTRACT

This paper concerns with the adsorption of bisphenol-A (BPA) by modified zeolites which were prepared using an orthogonal experimental design. The preparation process was optimized by the defined conditions, i.e., that 10% of cetyltrimethyl ammonium bromide was used as the optimal modifier and processing time and temperature were 36 h and 25°C, respectively. The adsorption equilibrium of BPA onto the modified zeolite fits the Freundlich isotherm with a high correlation coefficient 0.996 and was defined by a second-order rate constant ( $K_v$ ) of 1.07 g (mg h)<sup>-1</sup>. Furthermore, the effects of solution chemical properties on the adsorption of BPA by modified zeolites were examined. The result reveals that the best adsorption temperature was 25°C, and the optimal pH ranged from 5.0 to 10.0. In addition, the adsorption capacity of BPA increased with concentrations of HCO<sub>3</sub><sup>-</sup> and decreased with increasing in concentrations of NO<sub>3</sub><sup>-</sup>, OH<sup>-</sup>, and Al<sup>3+</sup> in the test solution.

**Keywords:** adsorption, bisphenol A, cationic modified zeolite, orthogonal experimental design, water treatment

### 1. Introduction

Bisphenol A (BPA) has been used as one of raw materials to make polycarbonate plastic and epoxy resins, along with other applications. It is one of endocrine disrupting chemicals presenting wastewaters which can mimic living animals own hormones and may lead to negative health effects (1). Regulatory organizations have provided guidelines of safety levels of BPA for humans, which are currently being under review as a result of new scientific discoveries (2). A recent published study (3) investigated the number of chemicals to which pregnant women are exposed and found that 99% of the investigated were detected BPA presenting in their bodies. Therefore the research for the removal of BPA from wastewater has great significance for the human beings, other living organisms and the environment.

Adsorption (4-7), ozonation, ultraviolet light irradiation<sup>8</sup>, catalyzed oxidation method (9, 10) and separation by membrane process (11) have been attempted to treat water and wastewater containing the BPA. Among them, chemical/physical adsorption is generally considered to be an effective method for quickly lowering the concentrations of organic molecules in an effluent because most adsorbents such as activated carbon generally possess large accessible internal and/or external surface areas or have specific affinity which can hold selected organic pollutants (12). Adsorbent materials for studies mainly include: zeolite (13), montmorillonite hybrid (14), activated carbon (15), and carbon nanotubes (16). Although activated carbon is the most commonly used adsorbent in the removal of organic compounds from liquid-phase media it presents some disadvantages (17), e.g., high capital and running costs and weak hydrophilic affinity (18) due to the presence of mineral impurities and oxygen-containing functional groups. On the other hand, important features of zeolite are that they are readily to be modified to adsorb both hydrophobic and hydrophilic pollutants and easily to be regenerated than activated carbons. Zeolite have thus been studied as an effective adsorbent for the removal of organic compounds from an aqueous solution, such as acetone and toluene vapors (19), ammonia and humic acid (20), nitrosamines (21), nicotine (22), atrazine (23) and tannic acid (24). However, limited studies

have been reported on the adsorption of BPA from aqueous solution by cationic modified zeolites and this is the objective of this study.

The specific objectives of this work were (a) to optimize modification conditions of zeolite using the orthogonal experimental design; (b) to compare the characteristics of selected raw and cationic modified zeolites; (c) to evaluate the applicability of common isotherm models (i.e., the Langmuir and Freundlich) and to determine the kinetics of BPA adsorption; (d) to extensively assess the effects of pH, reactions temperature and the co-ions presenting in the solution on the adsorption behavior of BPA.

## 2. Materials and methods

### 2.1. Materials

The natural zeolite powder used has the particle size 150-200  $\mu\text{m}$ . The target adsorbate, BPA [C<sub>15</sub>H<sub>16</sub>O<sub>2</sub>], has molecule size of 0.94 nm. The first deprotonation of BPA starts at around pH 8.0 with pKa<sub>1</sub> = 9.59 (Equation 1) and the second one starts at around pH 9.0 with pKa<sub>2</sub> = 11.30 (Equation 2) (25). In view of its high value of log K<sub>ow</sub>, 3.32 (26), the stock solution of BPA was prepared with 25 % methanol solution. Two different kinds of cationic surfactants were used as modifiers, namely, cetyltrimethyl ammonium bromide (CTAB) and cetylpyridinium bromide (CPB). All chemicals and solvents used in this study were of analytical grade.



### 2.2. The preparation of modified zeolite

The natural zeolite were firstly washed with distilled water for three times in order to remove the soluble material on its surface, and then dried in a muffle at 500 oC for 2 h, in order to remove trace organic matter in zeolite. The dried zeolite was then grinded to 150  $\mu\text{m}$  particle size. The natural zeolite after pretreatment was simply named PZ. Next 10g of the PZ was mixed with a specified amount of modifier solution. Then the mixtures were placed in a 250 mL conical flask and shaken at 150 rpm for given temperature and given time. The resulting modified zeolite then was washed with distilled water until free of Cl<sup>-</sup> (as judged by AgNO<sub>3</sub>), and collected by centrifugation, dried at 110 oC for 3h and grinded to 150  $\mu\text{m}$ . The properties of the zeolite were characterized using X-ray photoelectron spectrometer (XPS), X-ray fluorescence (XRF) and Brunauer-Emmett-Teller (BET) surface area measurement.

### 2.3. Orthogonal experimental design

An orthogonal experimental design L<sub>9</sub>(3<sup>3</sup>) was used to evaluate the effects of modification of zeolite such as processing time, the time of zeolite mixing with surfactants, solution temperature and the mass percentage of modifiers used. These factors were treated as the variable factors in orthogonal experimental design method. Each factor had three levels as shown in Table 1.

**Table 1.** The levels of each factor

factors	Percentage of modifier (%)	Processing time (h)	Processing temperature (°C)
	A	B	C
Level 1	3.0	12	25
Level 2	6.5	24	35
Level 3	10.0	36	45

### 2.4. Batch sorption experiments

Batch adsorption experiments were carried out to obtain the adsorption isotherms. 50 mg modified zeolite was weighted into 250 mL conical flasks and mixed with 50 mL of test solution at given BPA concentrations and this made the zeolite dose to be 1 g L<sup>-1</sup>. The suspension was under shaking for given time at 150 rpm and given temperature , and allowed to age for 1 h. The solution

pH was manually adjusted (before mixing with zeolites) to the given values by HCl (0.1 mol L<sup>-1</sup>) or NaOH (0.1 mol L<sup>-1</sup>) solution.

## 2.5. Analytical testing

The resulting suspension solutions were centrifugated at 10000 rpm for 10 min, and were filtered by 0.45 µm membrane filtration. BPA concentrations were measured by ultraviolet spectrophotomete at a 276nm wavelength and followed up the procedures recommended by the Standard Methods (27).

## 3. Results and discussion

### 3.1. Orthogonal experimental design

The results of orthogonal experimental designs for the zeolite modification by CTAB and CPB are showed in Tables 2 and 3. As shown in Tables 2 and 3, the adsorption of BPA by CTAB modified zeolite (CTABZ) was stronger than CPB modified zeolite (CPBZ), and therefore, further studies will be focus to CTABZ only. The range analysis of orthogonal experimental designs of CTABZ was to evaluate the effects of modified conditions. The means of grade and range difference of each level are listed in Table 4. The absolute of the range difference of factor A was the largest, indicating that A was the strongest influence factor. The second stronger factor was B. From Table 4, the best level was A3B3C3. Since the range difference of C was little, considering energy conservation, level A3B3C1 was determined as the defined best modification conditions for preparing CTABZ.

**Table 2.** L9(33) orthogonal array and BPA removal onto CTAB modified zeolite

Number	A	B	C	BPA removal (%)
1	1	1	1	41.31
2	1	2	2	33.78
3	1	3	3	44.09
4	2	1	2	77.84
5	2	2	3	82.53
6	2	3	1	80.77
7	3	1	3	89.13
8	3	2	1	92.96
9	3	3	2	96.00

**Table 3.** L9(33) orthogonal array and BPA removal onto CPB modified zeolite.

Number	A	B	C	BPA removal (%)
1	1	1	1	32.55
2	1	2	2	19.58
3	1	3	3	27.05
4	2	1	2	65.73
5	2	2	3	71.91
6	2	3	1	71.75
7	3	1	3	87.79
8	3	2	1	87.65
9	3	3	2	86.74

**Table 4.** Range difference analysis on each level

Factors	Means to each level			Range difference
	1	2	3	
A	39.73	80.38	92.70	52.97
B	69.42	69.76	73.62	4.20
C	71.68	69.21	71.92	2.71

### 3.2. Characterization of zeolite

Chemical compositions and BET surface areas of natural zeolite (PZ) and CTAB modified zeolite (CTABZ) are summarized in Table 5. The inorganic elemental composition of PZ and CTABZ are listed in Table 6. The content of carbon and nitrogen containing groups on the surfaces of CTABZ was higher than that of PZ, reflecting that the modifier, CTAB, which consists of C, H, N and Br, was attached onto the surface of zeolite. Therefore, after modifying, the content of Br element was increase from 0 to 0.94 % (Table 6), and the surface area was reduced from 16.39 to 9.78 m<sup>2</sup> g<sup>-1</sup> as shown in Table 5, since CTAB occupies a part of zeolite's surface

**Table 5.** Surface organic elemental composition and surface areas of PZ and CTABZ

Adsorbents	Surface elemental composition			Surface area (m <sup>2</sup> g <sup>-1</sup> )
	C (%)	O (%)	N (%)	
PZ	10.49	89.51	0.00	16.39
CTABZ	46.06	51.67	2.27	9.78

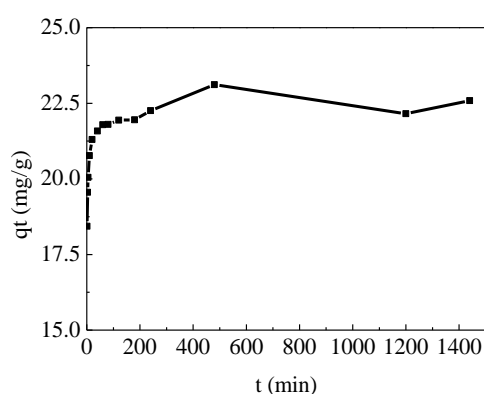
**Table 6.** Inorganic elemental composition of PZ and CTABZ

Adsorbents	Elemental composition (%)							
	Si	Al	Na	Ca	K	Br	Fe	Mg
PZ	35.54	7.58	2.46	1.77	1.61	0	0.58	0.41
CTABZ	35.23	7.75	2.00	1.72	1.58	0.94	0.58	0.49

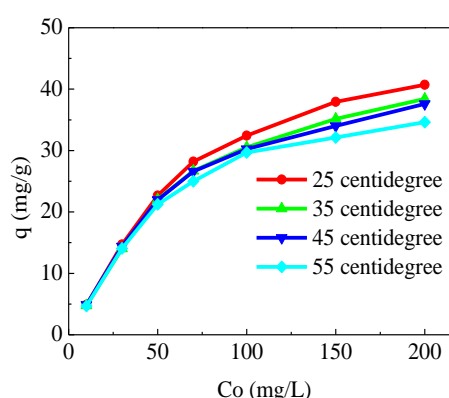
### 3.3. Adsorption kinetics and isotherms

The process of adsorption of BPA onto CTABZ could be divided into three stages. As shown in Figure 1, the first stage was identified by a fast intake of BPA in the initial 30 minutes of the reaction (Table 7), which was followed by a second stage of a sluggish approach to equilibrium lasting approximately 210 min, and a final stage characterized by a slight inversion approximately after 240 minutes of contact. Sorption data were fitted well to the pseudo second-order kinetic model described. A suitable fitness between calculated and experimental data indicated that the sorption of BPA by CTABZ follows the pseudo-second-order reaction kinetics (R<sup>2</sup> = 0.996) with a rate constant of (K<sub>v</sub>) of 1.07 g mg<sup>-1</sup> h<sup>-1</sup> (Table 8). The pseudo second-order kinetic model can thus be described by Equation 3, where q<sub>t</sub> is the amount adsorbed at given time,

$$dq_t = 1.07(22.46 - q_t)^2 \quad [3]$$



**Figure 1.** Kinetics of adsorption. 50 mL of 50 mg L<sup>-1</sup> BPA solution, 100 mg CTABZ, T=25 oC, 150 rpm. q<sub>t</sub>, adsorption capacity, t, adsorption equilibrium time (min).



**Figure 2.** The effect of BPA initial concentration and temperature on the adsorption capacity. 50 mL of 50 mg L<sup>-1</sup> BPA solution, 100 mg CTABZ, 150 rpm. q, adsorption capacity, C<sub>0</sub>: initial concentration.

Equilibrium adsorptions of BPA by CTABZ were analyzed by the Langmuir and Freundlich models (Table 8). The Langmuir isotherm showed a deviation from linearity ( $R^2 = 0.9626$ ). The Freundlich isotherm gave a good fit ( $R^2 = 0.9956$ ). The Freundlich isotherm can thus be described by Equation 4 where  $C_{eq}$  is the concentration of adsorbate at equilibrium in the liquid phase,  $Q_m$  is the corresponding amount of adsorbate in the solid phase, and two coefficients,  $K_f$  and  $n$ , have been established.

$$Q_m = 16.47C_{eq}^{0.19} \quad [4]$$

**Table 7.** Kinetics of adsorption between 0-30 minutes

t(min)	1.00	3.00	5.00	10.00	20.00	30.00
q(mg g <sup>-1</sup> )	17.81	19.13	19.62	19.88	20.73	21.01

**Table 8.** Adsorption models parameters of BPA by CTABZ

Pseudo-Second-order kinetic			Langmuir isotherm			Freundlich isotherm		
$K_v$ (g mg <sup>-1</sup> h <sup>-1</sup> )	$q_e$	$R^2$	$Q_m$ (mg g <sup>-1</sup> )	$b$ (L g <sup>-1</sup> )	$R^2$	$K_f$	1/n	$R^2$
1.07	22.46	0.999	37.85	0.65	0.963	16.47	0.19	0.996

### 3.4. The effect of solution chemical properties on BPA adsorption by modified zeolite

The effects of solution chemical properties on the adsorption of BPA onto CTABZ were examined and discussed below. Batch adsorption experiments were carried 4h at 150 rpm and given temperatures (e.g., 25 oC).

#### Initial BPA concentration

The sorption of BPA onto CTABZ was investigated for the initial concentrations from 50 to 200 mg /L at various temperatures as shown in Figure 2. The adsorption capacity of BPA increased from 4.88 to 40.72 mg L<sup>-1</sup> with increasing the initial concentration from 50 to 200 mg L<sup>-1</sup>. Higher BPA initial concentration provided the power to overcome the resistance to mass transfer from water phase to solid surface (28), and also had effects on the efficiency and ability of sorption. Generally, increase in the initial concentrations would elevate the adsorption capacity until a critical initial concentration reaches when adsorbate is quickly saturated in the surface and inter layer of the zeolite.

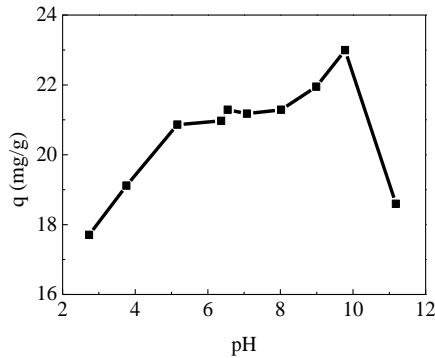
#### Temperature

The adsorption of BPA onto CTABZ was investigated at temperature of 25, 35, 45 and 55 oC. The results of sorption isotherms were illustrated in Figure 2. The results showed that adsorption capacity of BPA at 25 oC was the largest, and increasing in temperature slightly decreased the adsorption capacity. This can be attributed to the exothermic sorption mechanism; that the adsorption capacity of BPA by surfactant-modified zeolite and activated carbon was diminished with increasing in solution temperatures (26, 29). Thus, further studies were conducted at temperature of 25 oC.

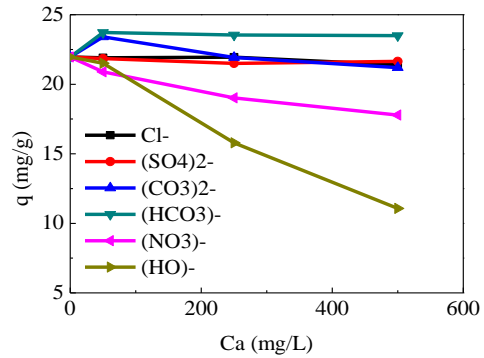
#### Solution pH

The effect of solution pH on the adsorption of BPA onto CTABZ was investigated at the pH range from 2.0 to 11.0. Results shown in Figure 3 indicated that adsorption efficiency of BPA onto the CTABZ was good, and the adsorption capacity increased with increasing in solution pH ranging from 3-10. However, when solution pH was greater than 10.0, the adsorption capacity decreased. The phenomena can be interpreted based on the concept of first and second deprotonation equilibriums of BPA, which were occurred at pH 9.6 and pH 11.3, respectively. When solution pH was less than 10, the BPA was dissociated to form H<sup>+</sup> and BPA anions which will be easily bonded by the cationic modified CTABZ via the charge neutralisation and thus the adsorption efficiency of BPA on CTABZ increased in raising solution pH. While pH was greater than 10, major compound of BPA was to be anions and this increased the solubility of BPA and thus leading to the decrease in adsorption efficiency of BPA by CTABZ. It is observed that the relative low

adsorption capacity of BPA was observed when solution pH was less than 5 (Figure 3). Lower pH solutions (higher H<sup>+</sup> concentration) intend to exchange cationic ions of the modifiers combined with CTABZ and this changes CTABZ to be less hydrophobic on its surface and leads to the decrease in the reactions between BPA and CTABZ and thus adsorption efficiency. pH 10 was determined to be the optimal pH to adsorb BPA by the CTABZ.



**Figure 3.** The effect of pH on the adsorption capacity. 50 mL of 50 mg L<sup>-1</sup> BPA solution, 100 mg CTABZ, T=25 oC, 150 rpm. qt, adsorption capacity.



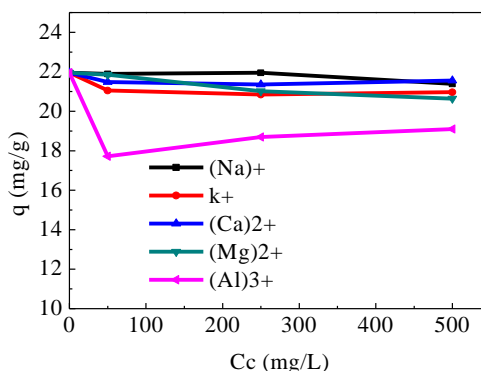
**Figure 4.** The effect of anions on the adsorption capacity. 50 mL of 50 mg L<sup>-1</sup> BPA solution, 100 mg CTABZ, T=25 oC, 150 rpm. qt, adsorption capacity, Ca, the concentration of anions.

The presence of anions in the test solution

The influence of representative inorganic anions on the BPA sorption by CTABZ was investigated and the results are shown in Figure 4. It can be seen that the adsorption capacity did not change in the presence of CO<sub>3</sub><sup>2-</sup>, SO<sub>4</sub><sup>2-</sup>, Cl<sup>-</sup>. However, it increased from 21.95 to 23.72 mg L<sup>-1</sup> with the presence of HCO<sub>3</sub><sup>-</sup>, and decreased from 21.95 to 17.78 mg L<sup>-1</sup> and 21.95 to 11.07 mg L<sup>-1</sup> respectively, in the presence of NO<sub>3</sub><sup>-</sup> and OH<sup>-</sup> with their concentrations up to 500 mg L<sup>-1</sup>.

The presence of cation in solution

The influence of several inorganic cations in solution on BPA sorption by CTABZ was studied and the result is shown in Figure 5. Obviously, the presence of the monovalent (K<sup>+</sup>, Na<sup>+</sup>) and bivalent cations (Ca<sup>2+</sup>, Mg<sup>2+</sup>) made no difference to the adsorption capacity. However, it decreased from 21.95 to 17.72 mg L<sup>-1</sup> with increasing in the concentration of Al<sup>3+</sup> up to 500 mg L<sup>-1</sup>. Possible reasons of this would be that solution pH decreased to below 10 resulting from hydrolyzing reactions of Al<sup>3+</sup> in aqueous solutions and then adsorption capacity decreases as demonstrated in Figure 3.



**Figure 5.** The effect of cations on the adsorption capacity. 50 mL of 50 mg L<sup>-1</sup> BPA solution, 100 mg CTABZ, T=25 oC, 150 rpm. qt, adsorption capacity, Cc, the concentration of cations.

#### 4. Conclusions

Specific modifications of zeolite were optimized using an orthogonal experimental design and a range of analysis. The best performed modified zeolite for the removal of bisphenol-A (BPA) was prepared by 10% cetyltrimethyl ammonium bromide (CTAB) with pre-treated zeolite under operating temperature 25 oC and processing time 36 h. By comparing the property characteristics of raw zeolite (PZ) and CTAB modified zeolite (CTABZ), it is evident that the modifier, CTAB, was binded with zeolite after modification.

The results of adsorption characteristics of BPA onto CTABZ from aqueous solutions show that the sorption kinetics can be well described by a pseudo-second order reaction model with a rate constant ( $K_v$ ) of 1.07 g mg<sup>-1</sup> h<sup>-1</sup>. The BPA sorption onto the CTABZ fitted better onto the Freundlich isotherm ( $R^2 = 0.996$ ) than the Langmuir model. Also, 25 oC was the best adsorption temperature for BPA to be adsorbed by CTABZ. Furthermore, the adsorption capacity of CTABZ increased with increasing in solution pH, ranging from 5-10, and the optimal adsorption pH was pH 10. Finally, the adsorption capacity increased when the test solution consisted of HCO<sub>3</sub><sup>-</sup> but decreased when the test solution was composed of NO<sub>3</sub><sup>-</sup>, OH<sup>-</sup>, Al<sup>3+</sup> ions with their concentrations up to 500 mg L<sup>-1</sup>.

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