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Adsorption of bisphenol A on oil palm biomass activated carbon: characterization, isotherm, kinetic and thermodynamic studies

Risky Ayu Kristanti [1], Tony Hadibarata [2,*], Hamad M.S. Al Qahtani [3]

[1]Faculty of Engineering Technology, Universiti Malaysia Pahang, Lebuhraya Tun Razak, Gambang, 26300 Kuantan, Malaysia
[2]Department of Environmental Engineering, Faculty of Engineering and Science, Curtin University, Miri, Malaysia
[3]Department of Botany and Microbiology, College of Science, King Saud University, Riyadh 11451, Saudi Arabia

*corresponding author e-mail address: ***hadibarata@curtin.edu.my***

ABSTRACT

Bispheol A (BPA) is one of the new emerging pollutants that frequently utilized in many industries. BPA is a chemical that indisputable can mimic human estrogen and disrupt the endocrine system. Thus, two low-cost adsorbents (oil palm trunk and palm shell) with various physicochemical parameters were analyzed for the removal of BPA. The most favorable pH for adsorption was found to be pH 3 (75% and 77%), the adsorbent dosage was 0.1 g (76% and 78%), while temperature was 25^{o}C (76% and 78%) and agitation speed was 150 rpm (77% and 77%) for both oil palm trunk and palm shell. It was found that the best correlation with the experimental data of BPA adsorption was the Langmuir isotherm model, while the kinetic study could be well described with the pseudo-second-order model. The Fourier Transform Infrared Spectroscopy (FTIR), Field Emission Scanning Electron Microscopy (FESEM), Brunauer–Emmett–Teller (BET) were carried out to understand the functional groups, morphology, and surface area of oil palm trunk and palm shell on adsorption of BPA.

1. INTRODUCTION

BPA is a hazardous material which can mimic the human and animal estrogen and disturb the endocrine system. BPA is one of the new emerging pollutants that widely used in production of a variety of consumer products such as plasticizer and other chemical product [1, 2]. The removal of BPA had been concerned due to extensive environmental and human exposure. The chemical from BPA can cause abnormal reproductive organ when ingested in the body [3]. The fate of BPA is frequently into water body and contamination of the drinking water leading to bioaccumulation in various food chains until human consumption. Studies on animals showed that exposure to BPA affects infants and fetuses. Moreover, a previous study by Centers for Disease a Control and Prevention (CDC), United States had found the occurrence of BPA in the urine sample of human [2].

As a result, alternative treatment had been investigated for the removal of emerging pollutant including membrane, microbial degradation, and adsorption [4-7]. Among those methods, adsorption technique has some advantages to eliminate the BPA such as being efficient treatment, allows reaching a good removal percentage, insensitivity, low operating cost and easy application [8-11]. This method is a process that involved physical/chemical forces between adsorbent and dissolved molecule in adsorbate. Activated carbon (AC) are increasing used as adsorbent than raw biomass for removal of emerging pollutant due to higher adsorption capacity and adsorption rate [12-16]. However, currently, AC from agricultural waste are most preferred and used widely compared to commercial activated carbon. Instead of agricultural waste are causes severe discarding problems, they have been widely investigated as low-cost adsorbent due to basic components and a variety of functional groups that potentially be useful for the capacity of sorption for organic pollutants, other than environmental friendly and reliable resources [17-22]. The agricultural waste came from different kind of shape, color and sources thus, its need pretreatment before use as adsorbent. All pollutants and any soluble organic compounds of adsorbent ought to remove with diverse types of modifying agents, such as oxidizing agents, organic compounds, acid and base solutions, dyes, and mineral [23, 24]. With this background, the aim of this study was to apply a chemical and physical activation of oil palm trunk and palm shell as a potential adsorbent to adsorb BPA with various condition. The textural and chemical characteristic of AC and functional groups of AC were consider to know the adsorption mechanism. The isotherm and kinetic model was also developed.

2. MATERIALS AND METHODS

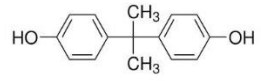
2.1. Chemicals and Materials.

All chemical reagents and BPA (CAS 80-0507; 98% purity) used in this study were high-purity analytical grade and obtained from Sigma-Aldrich (Milwaukee, USA). Using this standard, calibration curve of BPA were set up with ultrapure water obtained from Milli-Q equipment. BPA was dissolved with 10% ethanol, then distilled water has been added for the desired volume and concentration. For this study, 20 ppm of BPA has been prepared as a stock solution and for further experiment. The chemical properties of BPA was shown in Table 1. Two samples of agricultural waste (oil palm trunk and palm shell) were collected from the oil palm plantation in Miri, Malaysia.

2.2. Preparation of Adsorbents.

Oil palm trunk and palm shell were washed with tap water to remove any contaminations adhering to the surface and then repeated again with distilled water for the final wash. The samples then were cut into small pieces and dried at oven at 105 °C for 24h. The dried sample was ground to get the fine powder and 30 mesh sieve has been used to obtain a constant size of the adsorbent powder. The adsorbent powder was preserved in an airtight container for further experiments. Raw oil palm trunk and palm shell was washed by using distilled water to remove any impurities and dried in the oven at 105 °C overnight. The dried samples were impregnated with 8% Zinc chloride solution for 1 day. Horizontal furnace was used to activate the sample under nitrogen gas at 500 °C for 1 h. The activated carbon (AC) was adjusted to pH 7 using chloric acid or sodium hydroxide, and dried in the oven at 105 °C for 3 h. The samples were then has been cold down in room temperature and adjusted to neutral pH. Then, the adsorbents were oven dried again at the 105 °C for overnight. The AC was cooled down and stored in an airtight box for further analysis.

Table 1. Physical-chemical characteristics of bisphenol A.

Properties	Value
Structure	
Molecular formula	$C_{15}H_{16}O_2$
Appearance	Tough solid, white color
Molecular weight	228.3 g/mol
Melting point	150 - 157 °C
Boiling point	1.7kPa; 250-252°C
Solubility (g/100ml)	0.03 (poor)

2.3. Batch Studies.

To evaluate the potential of BPA sorption onto the natural and AC, experiment by the batch study has been conducted. The optimize data obtained by research variable study of parameters such as pH, adsorbent dosage, temperature, and agitation. For pH parameter study, the solution was manipulated from 3 to 9, and other variables were controlled and kept constant for 24 h. For the adsorbent dosage parameter, the weight was manipulated from 0.05g, 0.1g, 0.3g and 0.5g. While for the temperature parameter study, incubator shaker has been used to manipulate the temperature from room temperature, 30-60 °C. For the agitation parameter, the speed varied from 50rpm, 100rpm, 150rpm, 200rpm, and 250rpm. All the experiments were done in 30ml volume of 20 ppm BPA for a period time of 24h. The controlled variable value for the carried experiment was 0.1g for adsorbent dosage, pH 3 for the medium solution, and 150 rpm of agitation speed at room temperature. The solutions were then filtrated and stored in cold storage for further analysis.

2.4. Adsorption Kinetic.

The kinetic study of the adsorbent was carried for 38 h. Sample of the solution was collected for the first 2 h and followed with a determining interval of time as shown in the table for further analysis. The initial concentration of BPA was 20 ppm in 30ml of volume with an optimized parameter. The obtained data from the analytical method were then calculated for the removal rate (%) of BPA and the adsorption capacity (mg/g). Then, to identify the best adsorption kinetic for the adsorbents, a kinetic model was used as follow:

Pseudo-first-order model: $$\ln(q_e - q_t) = \ln q_e - K_1 t \quad (1)$$

Pseudo-second-order model: $$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (2)$$

Intraparticle diffusion: $$q_t = k_{diff} t^{1/2} + C \quad (3)$$

Where q_e and q_t (mg/g) respectively indicate the amount 1 of the BPA adsorbed at equilibrium and at time t (min); K_1 is the pseudo-first-order rate constant and K_2 is the pseudo-second order rate constant [7].

2.5. Adsorption Isotherm.

The adsorption isotherm study of the samples was conducted by manipulating the concentration of BPA in experiments. Other parameters were controlled as optimized studied and were run for 24 h. To describe the best adsorption isotherms of BPA onto the adsorbents, the following isotherm models were used:

Langmuir equation: $$\frac{C_e}{q_e} = \frac{1}{k_L q_m} + \frac{C_e}{q_m} \quad (4)$$

Freundlich equation: $$\ln q_e = \ln k_f + \left(\frac{1}{n}\right) \ln C_e \quad (5)$$

Temkin equation: $$q_e = B \ln A + B \ln C_e \quad (6)$$

Where C_e (mg/L) is the concentration of BPA solution at equilibrium, q_e (mg/g) is the amount of sorbed BPA at equilibrium, q_m is the maximum sorption capacity, K_L is the Langmuir constant, K_F and n is the Freundlich constant, A is the Temkin equilibrium binding constant (L/g) and B is the Temkin constant related to the heat of sorption (J mol^{-18}).

2.6. Thermodynamic Study.

The thermodynamic parameters involved in the adsorption process, ΔG^o (kJ mol^{-1}), ΔS^o (J K^{-1} mol^{-1}) and ΔH^o (J mol^{-1}) could be determined using the following equation.

$$\Delta G° = -RT.\ln K_c \quad (7)$$

$$\Delta G° = \Delta H° - T\Delta S° \quad (8)$$

Equation (10) and (11) can be simplified as follows:

$$\ln K_c = \frac{\Delta S°}{R} - \frac{\Delta H°}{RT} \quad (9)$$

Where, K_c is the distribution coefficient of adsorbate at absolute temperature T(K) and is calculated by equation. R represents the universal gas constant (8.314 J K^{-1} mol^{-1}). The plot between ln K_c and *1/T* provides the values of ΔS^o and ΔH^o. All these parameters are considered valid when the change of enthalpy remains constant over the temperature range studied.

2.7. Analytical Methods.

The filtrated solutions from the batch study were analyzed by using a UV-Visible spectrophotometer (CECIL CE7200 double-beam spectrophotometer) at 278 and 258 nm. The removal of BPA (%) and the adsorption capacity (mg/g) was calculated by using formulas as follows:

Removal rate (%) = $$\frac{C_0 - C_x}{C_0} \times 100 \quad (11)$$

Adsorption Capacity (mg/g) = $A = \frac{(C_0 - C_x)V}{M}$ (12)

Where A (mg/g) is the BPA adsorption capacity, C_0 (mg/L) and C_x (mg/L) is initial and equilibrium of BPA concentrations in the solution, V (L) is the solution volume, and M (g) is the mass of adsorbent.

The adsorbents were then characterized by FESEM, FTIR, and BET for before and after chemical treatment with sulphuric acid.

3. RESULTS

3.1 .SEM Images and BET.

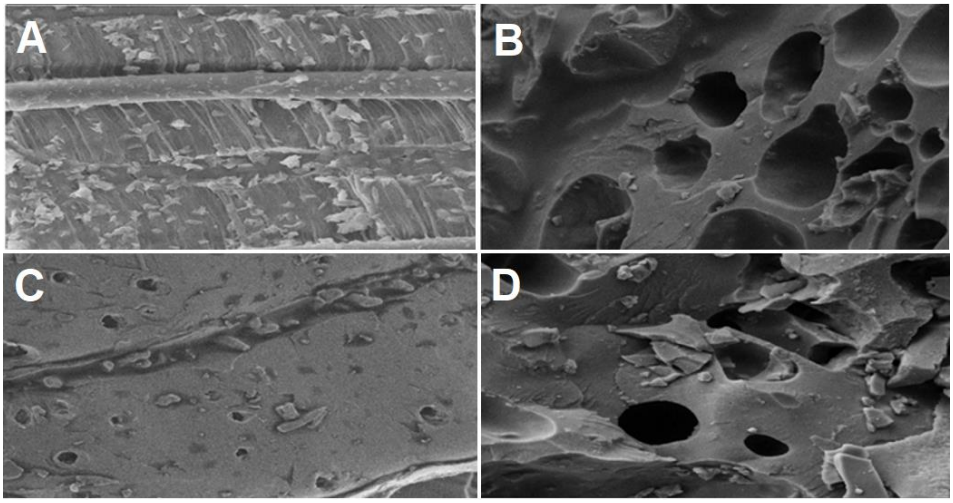


Figure 1. Field Emission Scanning Electron Microscope (FESEM) image of raw oil palm trunk (A), oil palm trunk after activation (B), raw palm shell (C), and palm shell after activation (D).

The SEM image of the raw oil palm trunk taken at magnitude 2.5 K x and AC oil palm trunk taken at magnitude 1.0 K x is showed in Fig. 1 (A & B). The surface morphology of the raw oil palm trunk is significantly different from that of the ACoil palm trunk[25]. This a photograph of raw oil palm trunkes has no pores which indicated that the adsorbent has a low surface area [26]. The analytical method of BET has been done to confirm the surface area of the adsorbent. However, results from single point BET showed that the surface area was not so much different before and after treatment, which obtained 4.80 m^2/g and 5.83 m^2/g respectively. The ACoil palm trunk showed more pore appear resulting in the adsorbent has more surface area and active site to react with BPA. Thus, this study showed that the sulphuric acid treatment responsible for the growth in the porosity of the oil palm trunkes adsorbent, which increased the number of available binding sites and its ion-adsorption capacity [26-28]. While the palm shell morphology showed the structure appears like it has some cavities and has no pores on the surface (Fig. 1C and Fig. 1D). Palm shell adsorbents have a surface area of 3.75 m^2/g. However, result from BET analysis showed that the surface area of adsorbent after treatment are seem occupied with the sulfonic functional group which gave 2.15 m^2/g of area, the pore is clearly seen which this improved in BPA adsorption. The morphology of the palm shell exhibits a caves-like, uneven and rough surface morphology. The effectiveness of adsorbent to remove the contaminant is based on the role of the physical properties such as pore volume, pore size distribution, functional groups of the contaminants, surface area, surface chemistry of adsorbent, and the solution characteristics [29].

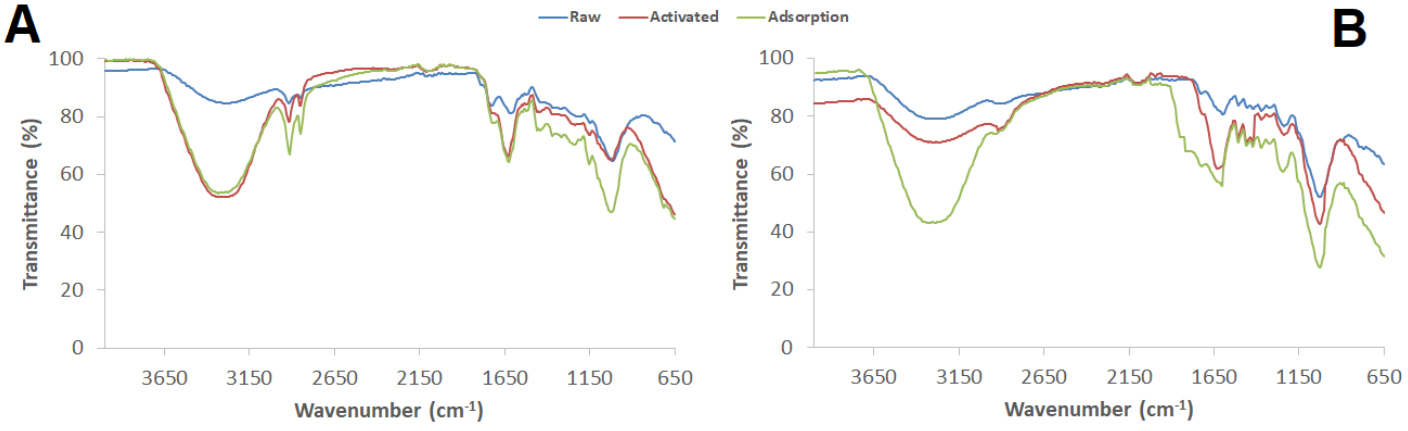


Figure 2. FTIR spectrum of oil palm trunk (A) and palm shell (B)

3.2. FTIR Analysis.

FTIR analysis of the oil palm trunk and palm shell is shown in Figure 2. From Figure 2, clearly seen the broad peak at 3205 cm^{-1} which indicated the involvement of hydroxyl groups which possibly present in lignin, hemicellulose and cellulose of oil palm trunk. Structure group of CH, CH_2, and CH_3 contain in oil palm trunk also causes stretching of C−H which proved by a peak appeared at a wavelength of 2922 cm^{-1}. While a peak that showed at a wavelength of 1650 cm^{-1} indicated as C=C bonds in aromatic rings which also known as absorption bands. This band is attributed to the bending mode of the absorbed water. Besides, the figure showed CH_2 bends, OH bends, and C–O skeletal vibrations at the bands of 1489.98, 1384.54, and 1313.70 cm^{-1} respectively. While, C–O is stretching in hemicellulose at the band of 1244.82 cm^{-1}. This result also showed the presence of silica as bending peaks at 1140 cm^{-1} indicated the Si−O stretching. The result of FTIR for the AC oil palm trunk showed an overlapped band of sulphonic acid hydroxyl and free phenolic and alcoholic hydroxyl groups of the raw oil palm trunk. This can be seen by a broad peak centered at 3400 cm^{-1}. Besides, the presence of SO_3H groups was definite by the peaks shifting at 1143 cm^{-1} (SO_2) and 1032 cm^{-1} (SO_2). The presence of carboxylic acid groups also was confirmed by peak shifting at bands of 1695 cm^{-1} (C−O) and 1620 cm^{-1} (C−O) [25]. FTIR spectrum of the palm shell showed the possible involvement of the hydroxyl group as band shifting around the broad peak at 3300 cm^{-1}.

The peaks at 1730 cm^{-1} and 1248 cm^{-1}indicated as C=C bonds in aromatic rings which also known as absorption bands. The peak showing at 1603 cm^{-1} and 1050 cm^{-1} known as the C−O carboxyl bands that indicated the activity of carboxyl oxygen atoms occurred. This result also showed the presence of silica as bending peaks at 1140 cm^{-1} indicated the Si−O stretching band. The efficiency of the oil palm trunk and palm shell treatment is shown on the dominance of wave numbers in inorganic groups. The increased binding of adsorbents is characterized by Si-O groups. After the binding proses are complete, there is a change of Si-O wave number. This proves the involvement of Si-O groups in binding Biphenyl. The presence of a number of functional groups and the absorption peak in the adsorbent indicated the complex nature of the adsorbent from oil palm trunk and palm shell. All changes in peaks and bands were detected to identify the significance different especially the functional group for all adsorbents. These changes were assumed the contribution of those functional groups in the adsorption process. The most dominance element in the adsorbents was carbon, then, followed by oxygen that derived from carboxylic acid of functional groups. While the presence of Sulphur element in the adsorbents was derived from sulfuric acid that used as a modified agent for pre-treatment process [29].

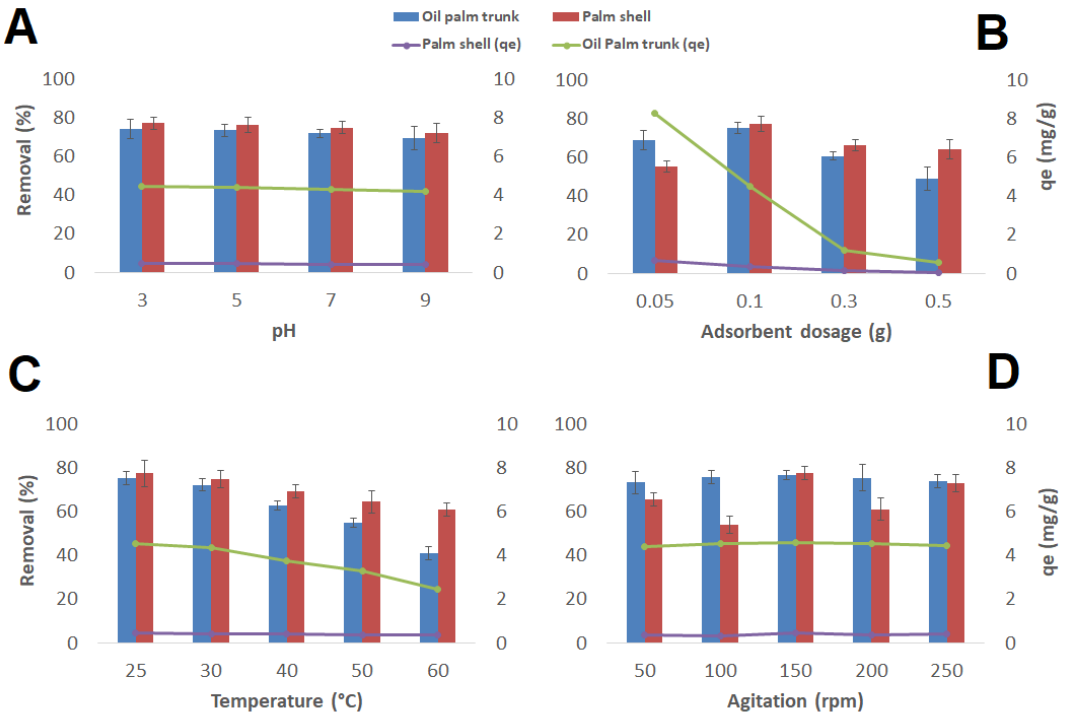


Figure 3. Effect of some parameters on the adsorption of BPA: pH (A), adsorbent dosage (B), Temperature (C), and Agitation (D).

3.3. Batch studies.

The effect of some parameters on the adsorption of BPA was shown in Figure 3. The percentage removal of BPA for oil palm trunk and palm shell decreased from 75.53% to 41.06% and 77.70% to 61.24%, when the temperature increased. The adsorption capacities also decreased with the increasing of temperature. Thus, temperature gives a significant effect on BPA removal, which the result indicated that the temperature of 25°C was the best condition for BPA removal for these adsorbents. In the adsorption process, the parameter of pH contributes to the important role as pH value influencing the ionic state of the solution [30].

This current study showed that oil palm trunk and palm shell adsorbents were pH-dependent which indicated that the functional group on the surface of adsorbent were influenced by ionic state and the component of the solution. In alkaline condition, as pH

value increased in solution, the electrostatic repulsion between BPA and the adsorbent also increased which causes the surface area of adsorbent becomes negative and inclined the binding affinity. Thus, this led to the low in percentage removal and adsorption capacity of BPA onto the adsorbents.

The parameter of the dosage on the adsorption of BPA showed that the percentage removal increased when adsorbent added from 0.05 to 0.1 g, then regularly reduced when adsorbent dosage added from 0.1 to 0.3 and 0.5 g respectively. However, the adsorption capacities of all adsorbents decreased by increasing the adsorbent dosage. This result showed that the adsorption ability was dependent on the adsorbent dosage and volume of solution added. Thus, the optimum adsorbent dosage for this experiment was 0.01 g in 30 ml that can achieve a reasonably good removal performance. This was considerately advantaged for economic purposes [31]. While the removals of BPA and adsorption capacity for adsorbents were not constant by the increase of agitation. The results showed that the different rpm gave the little bit differ of percentage removal and adsorption capacity. However, in moderate agitation which is 150 rpm, the results showed the highest in the removal and adsorption capacity of BPA. Thus, agitation at 150 rpm was the most suitable speed in this study.

3.4. Adsorption Isotherm.

Both oil palm trunk and palm shell showed that the adsorption equilibrium was reached at the time of 24th h with the percentage removal and adsorption capacity of BPA were 75.53%, 4.53 mg/g and 77.7%, 4.66 mg/g respectively. Oil palm trunk and palm shell suggested following the Langmuir model which obtained the R^2 equal to 0.9566 and 0.9056 respectively (Fig. 4). For this study, the Langmuir model provides a better fit for explaining the adsorption of BPA onto both of the adsorbents by comparing the correlation factors of the Langmuir, Freundlich, and Temkin isotherm models. This Langmuir model indicated that the adsorption mechanism applies monolayer adsorption with no transmigration of the adsorbate in the smoothness of the surface and assumes a surface containing a limited number of adsorption sites [32]. Langmuir model also assumes that the surface of adsorbent was homogenous indicated the homogeneous distribution of active sites onto the surface of the adsorbents [31]. The value of R^2 obtained indicated the type of the isotherm were favorable at the concentration studied since the R^2 value of more than 0 and less than 1 ($0< R^2<1$). The correlation coefficient (R^2) of the experimental data fits quite well with the Langmuir isotherm since the value was more than 0.90 (>0.90), whereas, the correlation coefficients for Freundlich and Temkin model were less than 0.90 (<0.90) indicated that the experimental data have not fit with the models. In the Langmuir, q_m value was the most important parameter to compare by measure the adsorption capacity of the adsorbent by using isotherms based equilibrium modelling. This current study showed that the monolayer adsorption capacity of oil palm trunk and palm shell was 26.88 and 28.65 mg/g respectively.

3.5. Adsorption Kinetic.

The outcomes indicated that BPA removal and adsorption capacity increase gradually over time. The most effective for BPA adsorption was at the first 2 h of experiment. This was because of the availability of readily accessible sites and the morphology effect of the adsorbents. At the initial stage, the capacity of adsorption was highly increased due to the number of vacant sites that available were greater. Thus, this increased concentration gradient between adsorbate in the adsorbent and adsorbate in solution [25]. The stationary states for all the adsorbents were reached at the time of 24 h, which showed the highest removal rate, adsorption capacity and indicated that the adsorbent was fully saturated [33]. The observation after 24 h, showed that the adsorption rate tends to decrease.

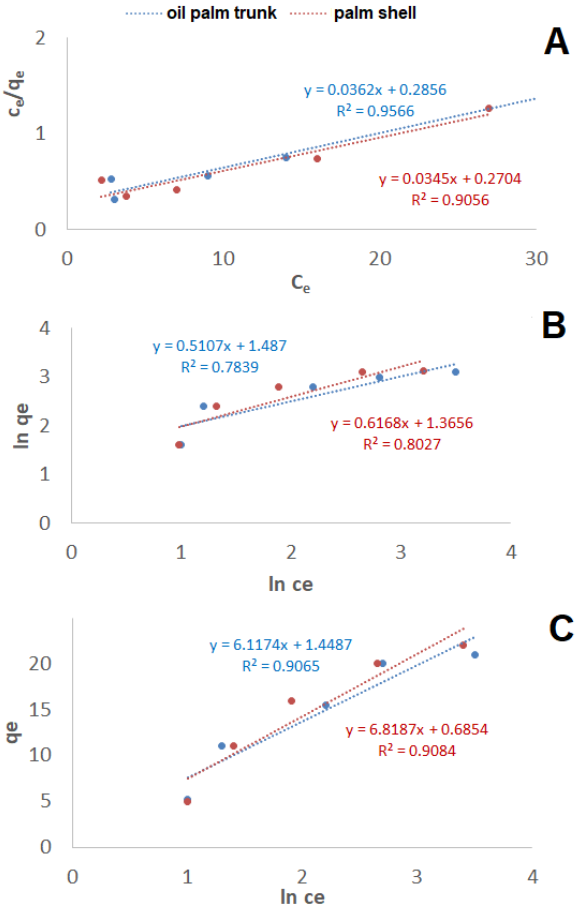


Figure 4. Adsorption isotherm: Langmuir (A), Freundlich (B), and Temkin (C).

Previous study described that initially, the solute concentration gradient was relatively high and the active sites on the surface of the adsorbent were vacant, however, with the increased of the contact time, the adsorption capacity of BPA was significantly decreased; due to the decreased in the number of vacant sites on the surface of the adsorbent [25]. The adsorption data obtained were calculated with the pseudo-first-order and pseudo-second-order models. The adsorption of BPA at equilibrium (q_e) for oil palm trunk was 4.53 mg/g, which is almost the same as the calculated value for pseudo-second-order model which is 4.503 mg/g. While the R^2 in pseudo-second-order model gives a value of 0.999 that indicated a better fit, which is a little bit higher than the pseudo-first-order with R^2 value, was 0.967. Thus, the adsorbents proposed that the overall rate of the BPA adsorption mechanism was chemisorption [34].

3.6. Adsorption Thermodynamics.

The values of the thermodynamic parameters calculated at temperatures 298, 303, 313, 323 and 333K are presented in Table 3. The negative output of $\Delta H°$ for both BB and CB demonstrates the exothermic nature of adsorption interaction. Moreover, the gradual decrease of adsorption uptake with the increase of temperature is due to the involvement of the desorption step within the sorption mechanism. Another reason might be due to the reduction of strong sorptive forces among the active sites present on the BPA molecules and adsorbents or between BPA molecules onto the sorbed phase. ΔS^o values for BPA-adsorbent interactions were found -19.84 J K^{-1} mol^{-1} on BB and 7.053 J K^{-1} mol^{-1} on CB respectively. This positive value of ΔS^o proves the affinity of the palm shell for BPA and randomness at the solution interface during adsorption. On the other hand, the negative value of $\Delta S^o$for BB suggests the decrease of randomness at the solid-solution interface at the time of adsorption of BPA from the water body. A higher value of ΔG^o with the increase of temperature (K) for both oil palm trunk and (from 5.9 to 6.6 kj mol^{-1}) and palm shell (from -7.8 to -8.1 kj mol^{-1}) indicates a fall of spontaneity and feasibility of the adsorption process at higher temperature [35].

Table 2. The kinetic parameter of BPA adsorption onto oil palm trunk and palm shell.

Kinetic Parameter	Oil palm trunkes (q_e = 4.53 mg/g)	Palm shelles (q_e = 4.66 mg/g)
Pseudo-first-order		
q_e (calculated)	1.004	0.334
K_1	0.076	0.070
R^2	0.967	0.677
Pseudo-second-order		
q_e (calculated)	4.440	4.606
K_2	1.178	2.362
R^2	0.999	1
Intraparticle Diffusion		
C	3.362	4.167
K_{diff}	0.216	0.099
R^2	0.941	0.704

Table 3. Thermodynamic parameters for the adsorption of BPA onto oil palm trunk and palm shell

Adsorbent	T (K)	*Kc*	*ΔG* (kJ mol−1)	*ΔH* (J mol−1)	*ΔS* (J mol−1 K−1)	R^2
Oil palm trunkes	298	22.7	5.9			0.95
	303	21.7	5.9			
	313	18.8	6.2	-13.8	-19.8	
	323	16.6	6.4			
	333	12.3	6.6			
Palm shelles	298	23.3	-7.8			0.99
	303	22.5	-7.8			
	313	20.8	-7.9	-5693.7	7.1	
	323	19.4	-8			
	333	18.4	-8.1			

4. CONCLUSIONS

Two low-cost adsorbents (oil palm trunk and palm shell) showed the ability to remove BPA with various physicochemical parameters. The most favorable condition for BPA removal is as follows: pH 3, the adsorbent dosage at 0.1 g, temperature at 25°C, and agitation speed at 150 rpm. Treated sulphuric acid adsorbents showed increasing in the removal rate and adsorption capacity compared to the raw adsorbent. The adsorption isotherm process was well fitted with the Langmuir model which indicated that the monolayer adsorption of BPA takes place on the homogeneous surface of the adsorbent. While, adsorption kinetic was fitted well with and pseudo-second order model, which suggests that the adsorption mechanism was chemisorption.

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6. ACKNOWLEDGEMENTS

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