Water Air Soil Pollut (2015) 226:34
DOI 10.1007/s11270-015-2318-5

Adsorption Characteristics of Bisphenol A onto Low-Cost Modified Phyto-Waste Material in Aqueous Solution

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Received: 8 November 2014 / Accepted: 19 January 2015

Abstract The potential of agricultural waste materials for the removal bisphenol A (BPA) from aqueous solution was investigated. BPA is an endocrine-disrupting compound (EDC) used mainly in the plastic manufacturing industry. It may be hazardous to humans and animals because of its estrogenic activity. Agricultural wastes are sustainable adsorbents because of their low cost and availability. Hence, this study investigated the removal of BPA from water by adsorption onto treated coir pith, coconut shell and durian peel. The adsorption of BPA from water onto adsorbent was evaluated using field emission scanning electron microscopy (FESEM), Fourier transform infrared spectroscopy (FTIR), and Brunauer–Emmett–Teller (BET). The effects of morphology, functional groups, and surface area on adsorption before and after pretreatment with sulfuric acid and reaction were investigated, and it was found that the treated adsorbent were able to remove BPA. Carbonyl and hydroxyl groups had appear in large number in FTIR analysis. The present study indicates that coir pith had removed 72 % of BPA with adsorption capacity of 4.308 mg/g for 24 h, followed by durian peel (70 %, 4.178 mg/g) and coconut shell (69 %, 4.159 mg/g). The results proved that these modified phyto-waste were promising materials as alternative adsorbent for the removal of BPA from aqueous solution.

Keywords Bisphenol A · Brunauer–Emmett–Teller · Coconut shell · Coir pith · Durian peel · FESEM · FTIR

1 Introduction

The awareness issue of contaminants in groundwater, rivers, and lakes is raised because of the increasing use of emerging contaminant-based products that threaten the environment and human wellness. From other studies, the products that contribute to this unhealthy environment such as medicines, house whole product, disinfectants, detergents, pesticides, paints, food container, and personal care products are undeniably loosed by the chemical and pharmaceutical industries which had also impended the aquatic life and water quality. Therefore, for the protection of the environment and obtaining safe drinking water, the removal of emerging contaminants is significant and is most concerned (Kümmerer 2009).

One of the identified emerging contaminants is bisphenol A (BPA) which encloses estrogenic properties and known as possible endocrine disruptor (Crain et al. 2007). It is persistently being used in food can coating and the production of plastic (Rubin and Soto 2009; Vandenberg et al. 2007). At room temperature, BPA is a white solid with a molecular weight of 228.29 and 150 to 155 °C for a melting point (Nakanishi et al. 2007). A previous study explores that BPA has a low vapor

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pressure of 5×10−6 Pa at 20 °C which vaporizes slightly at room temperature (Suzuki et al. 2004). The effluent from wastewater treatment plants (WTPs) and landfill sites were the primary route of BPA contamination sources within the aquatic environment (Kang et al. 2007). In the investigation in the river water that found the concentrations of BPA in the range of 2 to 230 (8.8×10^{-12} to 1.0×10^{-9} M) and under the aerobic conditions, BPA will disappear within 8 days of incubation (Suzuki et al. 2004).

Since BPA is used in many things, it affects and contaminates the water sources and drinking water and exposes aquatic lives to the toxicity of BPA. Then, humans ingest these contaminated fish and mammals which can interfere with human hormonal function. A survey has been made toward people 6 years and older in the USA conducted by the Centers for Disease Control and Prevention (CDC), and the result shows that 93 % of 2517 urine samples had found detectable levels of BPA. Besides, some animal studies had also reported that fetuses and newborns will be affected once exposed to BPA (Vandenberg et al. 2007).

There are many studies on BPA that occurred in the river around the world such as in Japan, USA, sewage treatment plant (STP) effluents in Germany, recreational bayou water, Netherlands surface water, Italy, and in the River Elbe and its tributaries (Suzuki et al. 2004; Kolpin et al. 2002; Staples et al. 2000; Fromme et al. 2002; Boyd et al. 2004; Belfroid et al. 2002; Patrolecco et al. 2006; Heemken et al. 2001; Stachel et al. 2003). The result indicated that the concentrations differ depending on the areas, mostly were lower than the limit of detection (<LOD 1 μg/L). Even though the concentrations were below LOD, it still gives effects in animals such as genital malformations, sexually dimorphic circuits in the hypothalamus, prostate weight and cancer, onset of estrus cyclicity and earlier puberty, mammary gland organization and cancer, low body weight, protein induction in the uterus, and many others (Richter et al. 2007). Recent results of molecular mechanisms of BPA action have revealed that at a very low concentration of BPA can still stimulate cellular response by a variety of pathways (Welshons et al. 2006). Other studies proved that BPA also has the efficacy of estradiol which are equally potent, changes in cell function at a dose of 1 pM (0.23 pg/ml culture medium), non-genomic, and involves membrane-associated forms of the estrogen receptors (Wetherill et al. 2007). Thus, the removal of BPA became precedence because these kinds of product are frequently used by human which can affect their health and disturb the ecosystem.

There are many studies on the removal of BPA especially by agricultural waste. The use of agricultural waste has been investigated widely because they represent available resources and always cause severe discarding problems (Ahmaruzzaman 2008; Bhatnagar and Jain 2005; Bhatnagar and Sillanpää 2010; Chun et al. 2004; Grassi et al. 2012; Khattri and Singh 2009; Rossner et al. 2009; Wan Ngah and Hanafiah 2008). Furthermore, agricultural wastes contain a variety of functional groups and basic components which have potential sorption capacities for several pollutants (Khattri and Singh 2009).

Various actions had been implemented for removing emerging contaminants from drinking and waste water, such as reverse osmosis (RO) membranes, filtration, coagulation/flocculation, advanced oxidation processes (AOPs), and microorganism (Rubin and Soto 2009; Vandenberg et al. 2007; Hadibarata et al. 2012a, b). Though these methods can remove the emerging pollutants, the cost demands high capital and maintenance besides multipart procedures involved in the treatment. Inversely, adsorption technique is applying simple design, insensitivity, and low cost of operation.

Activated carbons (AC) of agricultural waste are commonly used as adsorbent in adsorption process as for removal of emerging pollutant, for instance activated carbon from rice straw, activated carbon derived from oil palm empty fruit bunch, activated carbon coir pith, and so forth (Chang et al. 2012; Wirasnita et al. 2014; Kumar and Namasivayam 2014). Activated carbon has been suggested as a better adsorbent because it can afford to remove BPA rapidly and effectively due its high surface area and adsorptive capacity; however, preparation of activated carbon was costly. Therefore, many studies come out with natural materials as alternative adsorbent, which are low-cost, reliable resources and environment-friendly (Bolong et al. 2009; Chang et al. 2012).

Instead of activated carbon sources, agricultural waste materials are used either directly as adsorbent or applied in physical or chemical modification as pretreatment (Chun et al. 2004). Pretreatment of the adsorbents is for the purpose of removing all colors, metals, and any soluble organic compounds in aqueous solutions and in order to enhance its ability of adsorption (Elizalde-González et al. 2008). Different adsorbents are treated with different kinds of modifying agents, such as organic compounds, mineral, dyes, acid and base solutions, and oxidizing agents, depending on the kind of adsorbate or pollutant to remove (Bhatnagar and Sillanpää 2010).

The objective of this study was the application of sulfuric acid as modification reactions to coir pith, coconut shell, and durian peel in order to enhance the ability for BPA adsorption in aqueous solution. Various parameter conditions on the adsorption and removal of BPA have also been studied.

2 Materials and Methods

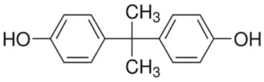
2.1 Preparation of BPA Solutions

The highest available grade of BPA (purity 95 %, chemical formula $C_{15}H_{16}O_2$, MW=228.29, λ_{max}=278 nm) was purchased from Sigma-Aldrich (Milwaukee, USA). The characterization of BPA is presented in Table 1. The stock solution of BPA was prepared by dissolving 0.02 g of BPA in 10 % of ethanol (95 %) and 90 % of distilled water. Distilled water was used to dilute the stock solution of BPA which depends to the desired concentrations.

2.2 Preparation of the Adsorbents

Coir pith, coconut shell, and durian peel were collected from an agricultural waste dumpsite in Johor Bahru respectively. The samples were washed with tap water and repeated with distilled water to remove any impurities adhering to the surface, and then the samples were cut into pieces and oven-dried at 105 °C overnight. The dried samples were grinded and sieved through 30 mesh to get a consistent size of adsorbent powder. The adsorbent was then treated with sulfuric acid according to the former method (Dhayabaran et al. 2012). The dried raw adsorbents were soaked in concentrated 98 % H_2SO_4 (1:1 *w*/*v*), then were kept in a hot air oven at 105 °C for 12 h. These samples were then washed with distilled water then were dried again in a hot air oven at 105 °C for hours. The treated adsorbents were stored in an airtight container for further experiments.

Table 1 Physical-chemical characteristics of bisphenol A

Properties	Value
Structure	
Molecular formula	$C_{15}H_{16}O_2$
Appearance	White solid
Molecular weight	228.29 g/mol
Melting point	158 to 159 °C
Boiling point	220 °C, 4 mmHg
Aqueous solubility (15 °C)	120–300 ppm (21.5 °C)

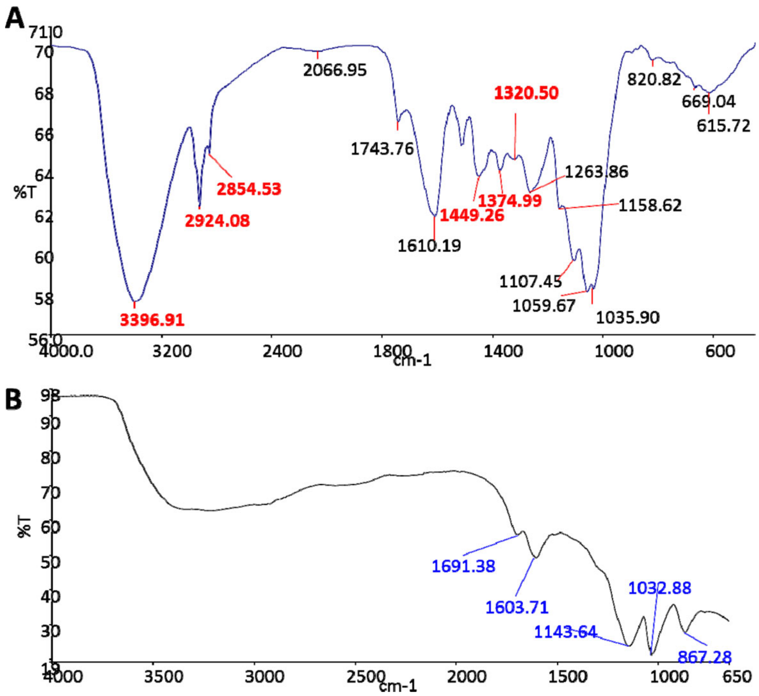
2.3 Characterization of the Adsorbent Materials

Surface texture and morphology before and after of coir pith, coconut shell, and durian peel were analyzed by the field emission scanning electron microscopy (FESEM, JEOL 6335F-SEM, Japan), and elementary analyses were performed simultaneously using an EDX spectrometer. The Brunauer–Emmett–Teller (BET) surface areas and monolayer pore volumes of the adsorbents were determined by using the provided software, surface analyzer (Quantachrome Instrument, USA). Fourier transform infrared spectroscopy (FTIR) (Spectrum one, Perkin Elmer, USA) was used to analyze the functional groups on the surface before and after of coir pith, coconut shell, and durian peel with the spectral range varied from 4000 to 400 cm^{-1}.

2.4 Adsorption Experiments

In other to obtain a concentration of 20 mg/L BPA, 0.02 g of BPA powder was dissolved in 10 % of ethanol and 90 % of distilled water, and then the solution was diluted to various concentrations depending on the experiment. Adsorption experiments were performed by adding 30 ml of 20 mg/L BPA solution into 100 ml conical flask containing 0.1 g of treated adsorbent. The pH value has been adjusted by NaOH or HCl solution. The mixtures were sealed and shaken at a rate of 150 rpm at room temperature for 24 h. The solutions were then filtrated by using Whatman no. 1 filter paper and determined the BPA concentration by using a UV/Vis spectrophotometer (*NANOCOLOR*® VIS, Macherey-Nagel, Germany) at 277 nm. All experiments were performed in triplicate. Ultimately, the solids obtained were oven-dried at 105 °C for one daytime and analyzed by FESEM, FTIR, and BET after treatment.

Fig. 1 FTIR spectrum of raw coir pith (**a**) and coir pith after pretreatment (**b**)



The adsorption kinetics of the agricultural residues was examined by performing equilibrium experiments. The samples were shaken for periods running from 2 to 24 h, with the initial BPA concentrations of 20 mg/L.

Fig. 2 FTIR spectrum of raw coconut shell (**a**) and coconut shell after pretreatment (**b**)

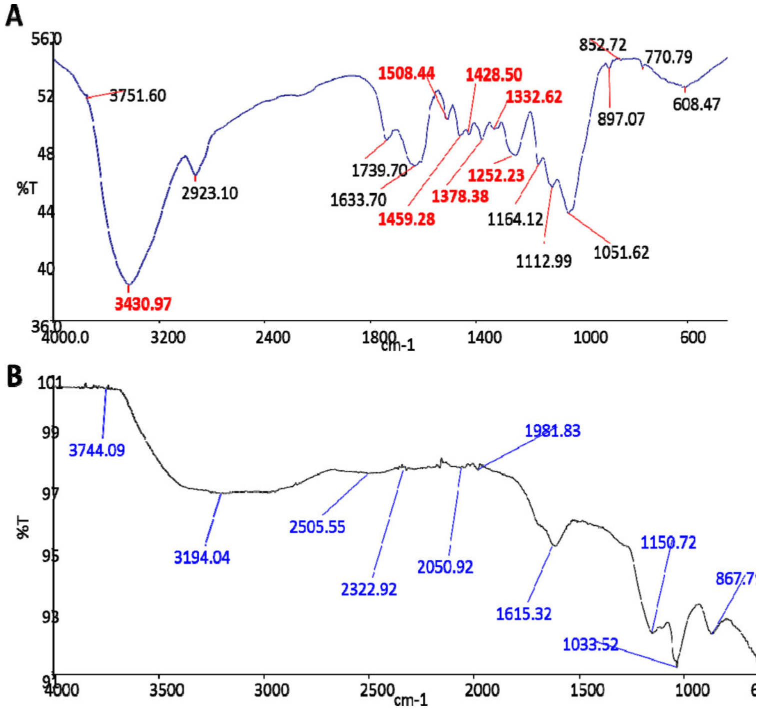
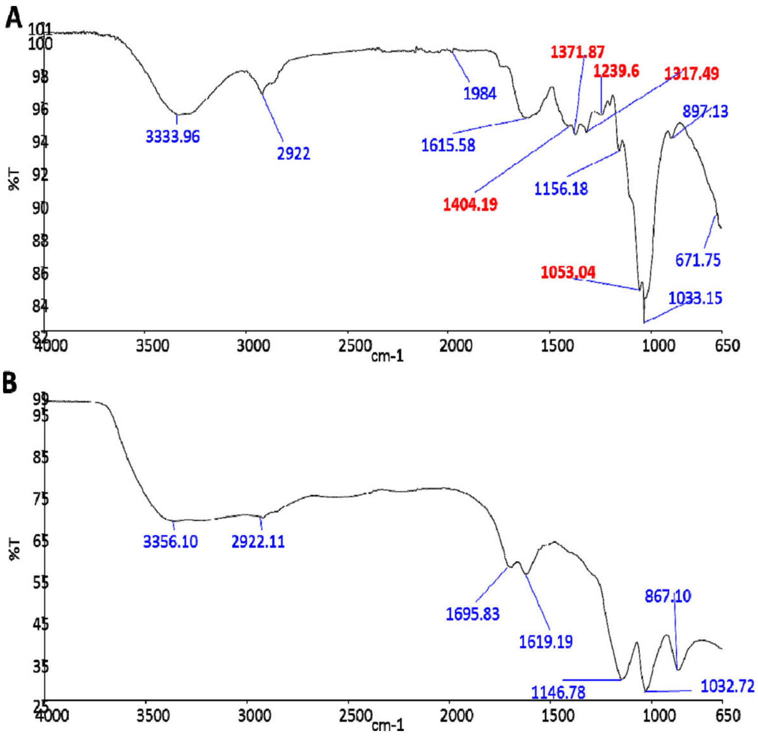


Fig. 3 FTIR spectrum of raw durian peels (**a**) and after durian peels pretreatment (**b**)



The BPA rate (%) and adsorption capacity (mg/g) will be calculated by:

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

$$\text{Adsorption Capacity} = A = \frac{(C_0 - C_x)V}{M}$$

where A (mg/g) is the BPA adsorption capacity, C_0 (mg/L) and C_x (mg/L) are, respectively, the initial and equilibrium BPA concentrations in the solution, V (L) is the solution volume, and M (g) is the mass of adsorbent (Wirasnita et al. 2014).

3 Results and Discussions

3.1 Characteristics of Adsorbents

Adsorbents were run in FTIR spectroscopy to determine the functional groups that are present on the surface area. Commonly, agricultural residues composed almost the same functional groups that can bind pollutants to some extent, such as phenol, aldehydes, alcohols, ether, ketones, and carboxyl groups (Pagnanelli et al. 2003). Figure 1 shows the FTIR spectra of the coir pith before

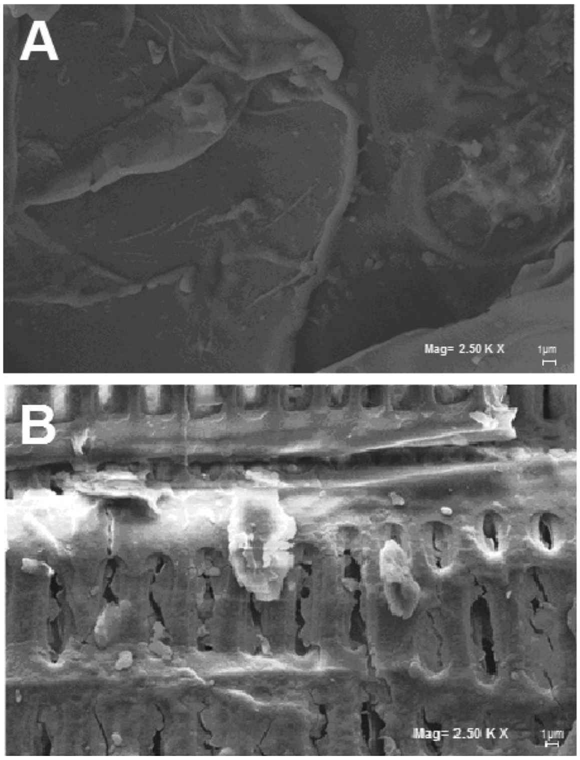


Fig. 4 Field emission scanning electron microscope (FESEM) image of coir pith (**a**) and coconut shell (**b**) after adsorption of bisphenol A

and after the pretreatment. Band shifting around the broad peak at 3396.91 cm^{-1} has shown the possible involvement of hydroxyl groups. The peaks at 2924.08 and 2854.53 cm^{-1} are due to the CH stretching causes vibrations of CH, CH_2, and CH_3 groups. The absorption bands are at around 2066.95 and 1515.10 cm^{-1} that show the characteristic of C═C bonds in aromatic rings. The major changes can be seen are at the increments in the C–O carboxyl bands at 1449.26 and 1035.90 cm^{-1}. Usually, the activity of carboxyl oxygen atoms causes the alterations in these bands area. The major deviations are in the regions from 3396.91 and 2924.08 1 cm^{-1} to 1691.38 and 1603.71 cm^{-1}, which are assigned to the oscillations of the N–H and O–H functional groups. The region at 2923.06 and 2924.75 cm^{-1} shows the weak bands that cause the C–H stretching mode. It reflects the aliphatic nature of the coir pith, while the peaks at 1143.64 and 1032.88 cm^{-1} are recognized as Si–O stretching and bending, which indicate the presence of silica.

FTIR spectrum of the raw coconut shell and after pretreatment is shown in Fig. 2. From the gained result, show band shifting around the broad peak at 3430.97 cm^{-1} indicates the possible involvement of hydroxyl group. The peak at 2923.10 cm^{-1} is due to the CH stretching that causes vibrations of CH, CH_2,

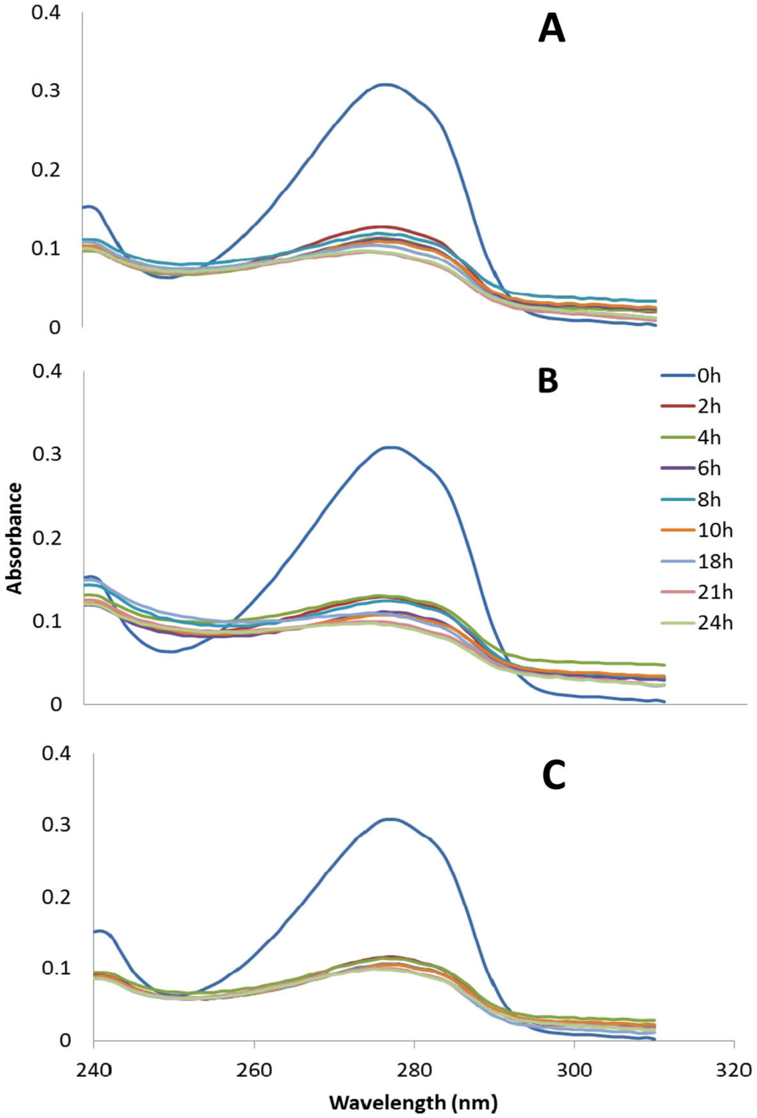


Fig. 5 Visible absorbance spectra of bisphenol A removal by coir pith (**a**), coconut shell (**b**), and durian peel (**c**)

Table 2 Removal rate and adsorption capacity of BPA in liquid solution by coir pith, coconut shell, and durian peel

Time (h)	Removal rate (%)			Absorption capacity (mg/g)		
	Coir pitch	Coconut shell	Durian peel	Coir pitch	Coconut shell	Durian peel
2	59±2.5	58±0.8	62±1.6	3.525±0.15	3.507±0.05	3.712±0.10
4	62±1.1	58±3.0	63±1.4	3.693±0.07	3.488±0.18	3.768±0.08
6	63±0.8	60±2.8	65±0.2	3.824±0.05	3.581±0.17	3.898±0.01
8	64±1.6	64±0.5	65±0.4	3.786±0.09	3.842±0.03	3.917±0.02
10	65±2.0	65±1.0	65±0.9	3.880±0.12	3.898±0.06	3.917±0.06
18	66±2.8	67±2.3	66±1.9	3.973±0.17	3.880±0.14	3.954±0.11
21	69±1.6	67±1.5	67±1.3	4.122±0.09	3.991±0.09	4.029±0.08
24	72±1.2	69±0.5	70±1.1	4.308±0.07	4.159±0.03	4.178±0.07
30	71±0.7	69±0.5	68±0.5	4.233±0.04	4.122±0.03	4.084±0.03
36	69±0.7	68±0.5	67±0.5	4.159±0.04	4.103±0.03	4.047±0.03
38	69±0.7	68±0.8	67±1.3	4.159±0.04	4.066±0.05	4.029±0.08

and CH_3 groups. The absorption bands at about 1739.70 and 1252.23 cm^{-1} show the characteristic of $C{=}C$ bonds in aromatic rings. The major changes that can be seen are at the increments in the C–O carboxyl bands at 1633.70 and 1252.23 cm^{-1}. Usually, the activity of carboxyl oxygen atoms causes the alterations in these band areas. The major deviations are in the regions from 3430.97 and 2923.10 cm^{-1} to 3194.04 and 2505.55 cm^{-1}, which are assigned to the vibrations of N–H and O–H functional groups. The peaks at 1150.72 and 1033.52 cm^{-1} are attributed to Si–O stretching and bending, indicating the presence of silica.

FTIR spectra of the durian peel adsorbent before and after the pretreatment are depicted in Fig. 3. The spectrum indicates a band shifting and possible involvement of hydroxyl groups around the broad points at 3333.96 and 2922 cm^{-1}. The absorption bands at about 1984 and 1317.49 cm^{-1} are the characteristic of $C{=}C$ bonds in aromatic rings. Alterations in these bands usually result from the activity of carboxyl oxygen atoms. Furthermore, peaks at 1615.58 and 1317.49 cm^{-1} are recognized as Si–O stretching and bending, which indicate the presence of silica. The changes in the FTIR spectra for coir pith, coconut shell, and durian peel after pretreatment indicate that those functional groups on the surface of the adsorbents were involved in the adsorption process (Chang et al. 2012). In this study, raw coir pith, coconut shell, and durian peel were tested for BPA

Table 3 Removal of bisphenol A by different value of pH

pH	Removal rate (%)	Adsorption capacity (mg/g)
3	70.55	4.233
5	69.31	4.159
7	68.38	4.121
9	67.14	4.028

Table 4 Comparison of the maximum monolayer adsorption of BPA onto various adsorbents

Adsorbents	Adsorption capacity (mg/g)	References
Coir pith	4.308	This study
Coconut shell	4.159	This study
Durian peel	4.178	This study
Banana bunches	4.532	This study
Coconut bunches	4.662	This study
Almond shell	188.9	Bautista-Toledo et al. (2005)
Activated rice straw	181.19	Chang et al. (2012)
Commercial activated carbon	129.6	Bautista-Toledo et al. (2005)
Commercial activated carbon	78.13	Tsai et al. (2006)
Carbon nanotube	61	Kuo (2009)
Bamboo	58	Goodson et al.(2004)
Carbon nanomaterial	45	
Minerals	0.33–0.86	Tsai et al. (2006)

removal by batch studies method; however, the result by UV–Vis spectrometer showed that the absorbance of coir pith and coconut shell was in negative value; this means that the adsorbents cannot afford to remove BPA. While for durian peel, it can only remove BPA from aqueous solution four about 10 %. Thus, these adsorbents were treated by sulfuric acid to improve the adsorption capability.

The field emission scanning electron microscopy (FESEM) photographs of the prepared raw coir pith and coconut shell are indicated in Fig. 4. These photographs coir pith have no pores. The lack of porosity indicated a low surface area of the adsorbent (Wan Ngah and Hanafiah 2008). To support the expected relationship between the open area and porosity, the open areas of the coir pith were determined by BET (5.79 m^2/g). Nevertheless, later on the pretreatment, the resulting adsorbent was found to be quite efficient in getting rid of BPA. The sulfuric acid treatment might be responsible for the growth in the porosity of the coir pith adsorbent, which increased its ion adsorption capacity, probably because the number of available binding sites increased (Ho et al. 2005; Hussain et al. 2007; Wan Ngah and Hanafiah 2008). The coconut shell has no pores, although the structure appears like it has some cavities on the airfoil. The BET analysis of the coconut shell indicated a surface area of 4.75 m^2/g.

3.2 Batch Studies

Visible absorbance spectra of BPA removal versus contact time by coir pith are shown in Fig. 5a. BPA control was at a wavelength 277 nm, and the absorption value was 0.319. The BPA concentration was reduced by the coir pith adsorbent after being shaken for 38 h. The peak at 277 nm indicated an absorption value of 0.085. The BPA removal rate after 24 h was calculated to be 72 %, while the adsorption capacity was 4.308 mg/g. Figure 5b shows the reduction in the BPA concentration due to coconut shell adsorbent after being shaken for 38 h. The peak at 277 nm indicates an absorption value of 0.093. The BPA removal rate was estimated to be 69 %, while the adsorption capacity was 4.159 mg/g. Figure 5c shows the reduction in BPA concentration due to the durian peel adsorbent after being stirred for 38 h. The peak at 277 nm indicates an absorption value of 0.092. The BPA removal rate was calculated to be 70 %, while the adsorption capacity was 4.178 mg/g. This result proved that coir pith, coconut shell, and durian peel, as promising materials, are able to remove BPA in aqueous solution. Other studies showed that varying kinds of pollutants can be removed by agricultural waste such as use modified bagasse to remove methylene blue (MB) dye; coconut (*Cocos nucifera* L.) shell for Pb^{2+}, Cu^{2+}, Cd^{2+}, and As^{3+} adsorption, treated rice husks for adsorption of malachite green (MG), coconut husk-based activated carbon for adsorption of 2,4,6-trichlorophenol (TCP), bamboo charcoal for adsorption of Cd (II), olive bagasse activated carbon for adsorption of chromium(VI); banana and orange peel for adsorption of Cu^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}, and Pb^{2+}; treated sunflower for the adsorption of Cr(VI); and treated maize corn cobs for removal of methyl orange (Elizalde-González et al. 2008; Jain et al. 2010; Annadurai et al. 2003; Demiral et al. 2008; Hameed et al. 2008; Wang et al. 2010; Okafor et al. 2012; Rahman et al. 2005; Low et al. 2011).

Table 2 shows the BPA removal rate and adsorption capacity of the coir pitch, coconut shell, and durian peel adsorbent with contact of time of 38 h. The removal rate and adsorption capacity of all the adsorbents were increased gradually over time. All three adsorbents had almost similar BPA removal rates and absorption capacity at optimum time, ranging from 69 to 72 % and 4.159 to 4.308 mg/g, respectively. The adsorption test was observed over 38 h of adsorption time, and the optimum adsorption equilibrium was reached at time 24th hour for all adsorbents. At the first 2 h, durian peel was the most effective BPA adsorbent compared to coir pith and coconut shell. However, at equilibrium time, the coir pith adsorbent showed the highest removal, followed by durian peel and then coconut shell (coir pith > durian peel > coconut shell). This might be because of morphology effect of the adsorbents and due to the availability of readily accessible sites. The stationary states for all adsorbents were reached after 24 h, indicating that the adsorbents were saturated (Demiral et al. 2008). After the optimal adsorptions were achieved, at time 30th onward, the percentage removals of BPA were slightly decreased. This might be due to the release of some BPA molecules from adsorbent surface. The absorption capacity of treated coir pith, coconut shell, and durian peel was not as high as other activated adsorbent, as shown in Table 4; however, the removal of BPA by percentage is over 69 % in only 24 h of contact time to reach equilibrium state compared to the removal of BPA by EFB-activated carbon and sugi chip and sawdust that need 48 h to reach equilibrium (Wirasnita et al. 2014;

Nakanishi et al. 2002). The greater removal of BPA in river water will be less risky of BPA toxicity exposure toward human and aquatic live.

The pH values of the solution play the important role in the adsorption process (Budinova et al. 2009). Fluctuations in pH can affect the states of the ionic species in solution. An experiment was conducted at an initial range of 3 to 9 pH with a contact time of 24 h to determine the adsorption behavior of BPA. The adsorption capacities of adsorbents were found to be pH-dependent, as indicated in Table 3. Adsorption capacities decreased from 3.708 to 3.429 mg/g when the pH was increased from 3 to 9. This pH dependence might be largely related to the types and ionic states of the functional groups in the adsorbents and to the compound speciation in the solution (Jain et al. 2010). When the pH value in the solution was increased, the percentage removal decreased slightly. This is due the electrostatic repulsion between partially ionized BPA and negatively charged adsorbent surface at alkaline condition. Thus, this lessened the overall consumption of BPA. Comparability of the maximum monolayer adsorption of BPA by various adsorbents is shown in Table 4.

4 Conclusions

This research investigated agricultural waste as potential adsorbents for removal of BPA from aqueous solution. Adsorbent of coir pith, coconut shell, and durian peel had achieved almost similar percentage removal rate and absorption capacity with equilibrium time of 24 h respectively. Selection of an appropriate adsorbent is one of the key issues regarding the maximum removal of a given type of pollutant, depending upon the characteristic of the adsorbent and adsorbate. This work proved that treated durian peel, coconut shell, and coir pith could replace the used of activated carbon, which is known expensive in preparation. These adsorbents are beneficial since they are used as low-cost adsorbents, economical and easily available adsorbent, and simple design of adsorption technique, leading to environmental benefits such as cutting the bulk of waste material and monetary values of water and effluent handling.

Acknowledgments The authors would like to acknowledge the Universiti Teknologi Malaysia and Ministry of Education Malaysia for providing LRGS Grant on Water Security entitled Protection of Drinking Water: Source Abstraction and Treatment (203/PKT/6720006; R.J130000.7809.4L810).

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