Production of a new industrially viable green-activated carbon from *Artocarpus integer* fruit processing waste and evaluation of its chemical, morphological and adsorption properties

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**Abstract**

*Artocarpus integer* is widely cultivated in Malaysia for its edible fruit which is used as an ingredient in the production of various confectioneries. The large amount of thick solid peel waste formed from processing of this fruit is a burden to local *Artocarpus integer* fruit processing industries. In this work, the feasibility of converting *Artocarpus integer* fruit processing waste into an industrially viable and low-cost green-activated carbon was evaluated. The novel activated carbon was prepared by physical activation with steam and characterized by elemental analysis, Boehm titration method, Fourier Transform Infrared Spectroscope (FTIR), Scanning Electron Microscope (SEM) and X-ray powder Diffraction (XRD). In addition, the potential use of the prepared activated carbon as an adsorbent and the effects of activation temperature and duration on iodine adsorption were examined. Experimental results revealed the suitability of *Artocarpus integer* fruit processing waste to be used as a precursor for activated carbon. The raw waste material was found to be a lignocellulosic biomass which is rich in cellulose (53.6 wt%) and carbon (60.8 wt%). Steam activation temperature, 750 °C and a short activation time of 60 min were appropriate to attain a high quality activated carbon with a large iodine adsorption capacity (1411 mg/g), highly micro-porous and amorphous structure with predominantly basic surface functional groups which are all advantageous properties for adsorption applications. The newly developed activated carbon is a promising cost-effective (~1.67 US$ per kg) alternative to commercial carbon which may be used as a cleaner and ecologically compatible adsorbent for improving water and air quality, consequently, benefiting human health and wellbeing.

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**1. Introduction**

The demand for activated carbon (AC) is increasing day by day ever since its discovery by Raphael Von Ostrejko, who is widely regarded as the “Father of Activated Carbon” (Menéndez-Díaz and Martin-Gullón, 2006). A market research firm reported that the global market for AC has reached US$2.1 billion in 2014 (Ceskaa, 2015). This figure is expected to rise as there are ever expanding applications for carbon materials. AC is widely used on the industrial scale as an adsorbent for the purification, separation and recovery processes, as a catalyst or a catalyst support in the catalytic processes and as electrode materials in electrochemical devices and processes (Ozdemir et al., 2014). In spite of its prolific use, AC remains as a pricey industrial material since the higher the quality of AC, the greater is its cost. This situation makes the existing commercial AC no longer attractive to be widely used in small-scale industries, especially in underdeveloped and developing countries.

Over the past few decades, commercially available AC is manufactured from non-renewable resources such as coal or petroleum based residues. However, in recent years, the increasing cost and scarcity of raw material has encouraged researchers to find other low cost alternatives from renewable resources. Utilization of natural sources as precursors for production of AC will reduce the use of coal and minimize the environmental impacts of coal mining. With innovations in AC industry, wood is the natural renewable material most often used globally to produce AC in a large scale. As a result, the pace of global deforestation accelerates. This motivates many researchers across the globe to investigate the use of agricultural waste materials as carbon source, instead of trees. Previous studies proved that lignocellulosic agro-wastes such as papaya peel (Abbaszadeh...
et al., 2016), orange peel (Fernandez et al., 2014), *Jatropha curcas* seeds (Hsu et al., 2014), longan seed (Yang et al., 2015), langsat empty fruit bunch (Njoku et al., 2015) corn cob (Song et al., 2013), almond shell (de Yuso et al., 2014), palm kernel shell (Kundu et al., 2015), cocoa shell (Saucier et al., 2015), grape stalk (Ozdemir et al., 2014), rice husk (Liu et al., 2016), mung bean husk (Mondal et al., 2015), grape bagasse (Demiral and Gungor, 2016), tomato waste (Saygili and Güzel, 2016), pigeon peas hulls (Venkata Ramana and Min, 2016), soybean hull (Chandane and Singh, 2016) and Fox nutshell (Kumar and Jena, 2016) being inexpensive, renewable and available in abundance with distinctive chemical compositions are workable option for production of AC (Yahya et al., 2015).

Artocarpus integer, also called Chempedak (English) is a member of *Moraceae* family which is widely cultivated in Malaysia for its edible aggregate fruit (Nakasone and Paull, 1998). Apart from being eaten raw, *Artocarpus integer* fruit (AF) is used as an ingredient in the production of various confectionaries. AF weigh from 600 to 3500 g, with the total edible portion (perianths + seeds) amounting to between 25 and 50% of a fresh fruit weight (Nakasone and Paull, 1998). According to agro-food statistics reported by the Ministry of Agricultural and Agro-Based Industry Malaysia (MOA), the annual production of AF in 2014 was projected at 35,563 MT, translating to approximately 22,237 MT of peel waste (MOA, 2015). The large amount of AF solid peel waste formed from processing of this fruit is a burden to local AF processing industries. This waste is disposed of by burning or throwing in municipal dumps. Most agro-wastes contain phenolic compounds and other compounds of toxic potential which may cause environmental degradation when discharged to the nature (Mussatto et al., 2012). Hence, the innocuous disposal of AF agro-industrial residue is fundamentally important from the ecological point of view.

The main objective of this work is to investigate the possibility of converting AF processing waste into an industrially viable and low-cost AC by steam pyrolysis. This conversion will minimize the landfill space occupied by AF waste disposal, overcome pollution and odor problems arising with poor management of AF solid waste in Malaysia and provide a cost-effective alternative to the existing disposal of AF agro-industrial residue. This waste is disposed of by burning or throwing in municipal dumps. Most agro-wastes contain phenolic compounds and other compounds of toxic potential which may cause environmental degradation when discharged to the nature (Mussatto et al., 2012). Hence, the innocuous disposal of AF agro-industrial residue is fundamentally important from the ecological point of view.

The cleaned and chopped pieces of peels (from AFP AC preparation step mentioned above) were air-dried for 24 h. The dried peels were weighed ($M_0$, g) and leached with a mixture of benzene and ethanol (2:1 v/v) at room temperature for 3 h. The residue is then dried in an oven at 105 °C to a constant weight, cooled to room temperature in a desiccator and reweighed ($M_l$, g). The weight difference of the peel before and after leaching is the amount of the extractives.

The extractives content in the raw peel (dry basis wt%) were calculated as

$$W_1 = \frac{M_0 - M_1}{M_0} \times 100\%$$  \hspace{1cm} (1)

**b) Analysis of hemicellulose**

1 g of the extractives-free dried peel (from part (a)) was placed in a conical flask and a 150 ml of NaOH solution (20 g/l) was then added to the sample. The mixture was boiled for 3.5 h, filtered and washed with distilled water to remove the NaOH solution (indicated by the pH value of the filtrate approaching 7). The residue is then dried in an oven at 105 °C to a constant weight, cooled to room temperature in a desiccator and reweighed ($M_2$, g). The difference between the sample weight before and after this treatment is the hemicellulose content. The hemicellulose content (dry basis wt%) in the raw peel was calculated as

$$W_2 = \frac{1 - M_2}{M_1} \times 100\%$$  \hspace{1cm} (2)

**c) Analysis of lignin**

1 g of the extractives-free dried peel (from part (a)) was placed in a conical flask and a 30 ml of sulphuric acid (72%) was added to the sample. The mixture was kept at 15 °C for 24 h. A 300 ml of distilled water was then added to the sample and the mixture was...
boiled for 1 h. After cooling, the filtered residue was washed until the sulfate ion in the filtrate was undetectable (through titration of a 10% barium chloride solution). The residue is then dried to a constant weight, cooled to room temperature in a desiccator and reweighed ($M_3$, g). The weight of the residue is recorded as the lignin content. The lignin content in the raw peel (dry basis wt%) was calculated as

$$W_3(\text{dry basis wt\%}) = \frac{M_3}{T} \times 100\% \quad (3)$$

d) Analysis of cellulose

Finally, the content of cellulose is calculated by the difference, assuming that extractives, hemicellulose, lignin, and cellulose are the only components of the entire peel waste. The cellulose (dry basis wt%) was calculated as

$$W_4(\text{dry basis wt\%}) = 100 - (W_1 + W_2 + W_3) \quad (4)$$

2.2.2. Proximate analysis

The proximate analysis of raw AF peel was conducted according to ASTM D 3172–3175 test standards and the results were given as moisture, volatile matter, ash and fixed carbon content. 1 g of cleaned and air-dried raw AF peel was placed in a crucible with cover and heated in an oven at 108°C for 3 h. The crucible was then placed in a desiccator, allowed to cool at room temperature and the sample was reweighed. The difference between the sample weight before and after heat treatment is the moisture content. To determine the volatile matter, 1 g of the sample was placed in covered crucible and heated in the muffle furnace from room temperature to 850°C for 7 min. The crucible was then placed in a desiccator, allowed to cool at room temperature and the sample was reweighed. The volatile matter content was then obtained by subtracting moisture content value from the weight of residue obtained. To determine the ash content, 1 g of sample was placed in covered crucible and heated in the muffle furnace from room temperature to 750°C for 3 h. The crucible was then placed in a desiccator, allowed to cool at room temperature and the sample was reweighed. The weight of the residue was recorded as the ash content. The fixed carbon content was then obtained by subtracting moisture, volatile matter and ash content from 100%.

2.2.3. Iodine number analysis

Iodine number of AFP AC was obtained using Na$_2$S$_2$O$_3$ volumetric method ([Tounsiadi et al., 2016]). Standard iodine solution was added over 0.50 g of AFP AC produced and after an equilibration time of 30 s, the residual iodine concentration was determined by titration with standard sodium thiosulfate using starch as an indicator.

2.2.4. Boehm titration procedure

Surface functional groups of samples were determined using Boehm titration technique ([Hajati et al., 2015]). 1.0 g of AFP AC was added to a series of flasks containing 30 cm$^3$ of 0.05 M: NaOH, Na$_2$CO$_3$, NaHCO$_3$ and HCl solutions. These flasks were then sealed and shaken for 48 h at room temperature. After 48 h, the solutions were filtered and 10 ml of each solution was pipetted to a conical flask and was titrated with 0.05 M HCl or NaOH, depending on the original solution used. The amount of acidic groups was determined based on the assumptions that NaOH neutralizes carboxylic, lactonic, and phenolic groups; Na$_2$CO$_3$ neutralizes carboxylic and lactonic groups; and NaHCO$_3$ neutralizes only carboxylic groups. The number of basic sites was calculated from the amount of HCl that reacted with AFP AC.

2.2.5. Instrumental analysis

Fourier transform infrared (FT-IR) spectra were recorded using a Perkin–Elmer RX1 FT-IR with potassium bromide (KBr) pellets. The morphology of the samples was examined from the scanning electron microscope (SEM) Leica S440. Samples were coated with gold in E-1010 Ion sputter prior to SEM observations. X-ray diffraction analysis was performed using XRD Siemens D5000 and elemental analysis of the samples was carried out using EDX-OXFORD elemental analyzer.

3. Results and discussion

3.1. Proximate, ultimate and biochemical component analyses

Agro-waste is a high-grade raw material for preparing AC due to its high carbon content (usually more than 45%) ([Ahmedna et al., 2000]). Proximate and ultimate analyses carried out in this study (Table 1) revealed that AF peel contains a high amount of carbon. These results suggested the suitability of the raw material to be used as carbon precursor. Cellulose, hemicellulose and lignin are the three main components of biomass. Lignocellulosic biomass typically has cellulose content ranging from 40 to 60%, hemicellulose from 20 to 40% and lignin range of 10–25% ([Yang et al., 2007]). The biochemical component analysis carried out in this work showed that the percentage compositions of cellulose, hemicellulose and lignin in AF peel (Table 1) fall within the standard ranges of lignocellulosic biomass. This result is also in conformity with the FTIR signal intensities obtained for the functional groups of cellulose, hemicellulose and lignin in the raw material (Fig. 1), giving affirmation of the lignocellulosic nature of AF peel. In general, cellulose is a homogenous polymer of glucose while hemicellulose macromolecules are polymers of pentoses, hexoses and a number of sugar acids. Lignin contains three aromatic alcohols (coniferyl, sinapyl and p-coumaryl) ([Collard and Blin, 2014]). About 15.3 wt% of extractives were found in the raw material used in this study (Table 1), revealing the presence of small amounts of water, ash, cyclic hydrocarbons and inorganic materials which are common constituents of lignocellulosic materials. AF peel, being a lignocellulosic bio-waste, is a good choice of material to be evaluated as precursor for production of AC because it is not dense and therefore the activation process is expected to occur much faster than with other more common carbon precursors such as coal and coconut shells.

The contents of cellulose, hemicellulose and lignin determine to some extent, the porosity development of the produced AC. Our

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Proximate, ultimate and biochemical component analyses of AF peel waste.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Proximate analysis (as received, wt%)</td>
<td></td>
</tr>
<tr>
<td>Moisture</td>
<td>11.4</td>
</tr>
<tr>
<td>Volatile matter</td>
<td>33.1</td>
</tr>
<tr>
<td>Fixed carbon</td>
<td>51.7</td>
</tr>
<tr>
<td>Ash</td>
<td>3.8</td>
</tr>
<tr>
<td>Ultimate Analysis (dry basis, wt%)</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>60.8</td>
</tr>
<tr>
<td>O</td>
<td>37.1</td>
</tr>
<tr>
<td>S</td>
<td>0.2</td>
</tr>
<tr>
<td>Si</td>
<td>0.3</td>
</tr>
<tr>
<td>Component Analysis (dry basis, wt%)</td>
<td></td>
</tr>
<tr>
<td>Cellulose</td>
<td>53.6</td>
</tr>
<tr>
<td>Hemicellulose</td>
<td>22.5</td>
</tr>
<tr>
<td>Lignin</td>
<td>2.6</td>
</tr>
<tr>
<td>Extractives a</td>
<td>15.3</td>
</tr>
</tbody>
</table>

*a* Benzene/ethanol (2/1) (v/v).

studies showed that the contents of cellulose and hemicellulose are more than lignin in raw AF peel (Table 1), indicating the possibility of easier activation of the bio-char and greater development of pores in AC, as observed in previous works done by Muley et al. (2016). Cellulose and hemicellulose are the volatile fractions removed during pyrolysis and activation processes, causing the development of pores, whereas lignin which imparts hardness to the biomass matrix, is the main constituent for char formation.

Elemental analysis of AFP AC prepared under activation conditions of 750 °C and 60 min revealed that the steam activated sample has 71.2% C, 24.2% O and 0.9% Si. As can be seen in Fig. 2, the carbon content of AFP AC substantially increased compared to the raw lignocellulosic AF peel after activation process. In the pyrolysis and activation processes, AF peel decomposes. As a result of decomposition, volatile compounds containing mainly oxygen and sulphide leave the biochar which becomes rich in carbon. A similar phenomenon had been previously observed by Saygil et al. (2015) and Coromina et al. (2016) for AC prepared from other lignocellulosic materials.

3.2. Surface chemistry

3.2.1. FTIR

FTIR spectra can provide beneficial information about the functional group distributions in the raw material and prepared carbon. The FTIR spectra of raw AF peel and AFP AC were demonstrated in Fig. 1. AF peel precursor contained more signals than the prepared carbon. Referring to the spectrum registered for raw AF peel, an intense and broad band located around 3400 cm⁻¹ is reflected to hydroxyl stretching vibration, suggesting the participation of the OH groups in hydrogen bonds because, for non-bonded OH groups, the band is usually located above 3500 cm⁻¹. For AF peel, this band also can be caused by silanol groups, because elemental analysis result obtained in this study revealed the presence of Si element in raw AF peel. Peaks occurring at 2920 and 2854 cm⁻¹ can be assigned to C–H stretching vibrations in aliphatic, alkyl and aromatic compounds. The band at 2311 cm⁻¹ is identically shaped to a band reported previously at 2325 cm⁻¹, which was ascribed to C≡C stretching vibration in alkyne group (Saka, 2012). The signals at 2190 and 2011 cm⁻¹ may also be attributed to the C≡C stretch of alkynes. The band located around 1700 cm⁻¹ is the characteristic of C=O stretching vibration in ketones, aldehydes, lactones and carboxylic groups. The 1642 cm⁻¹ band denotes the existence of carbonyl groups such as quinones, which are highly conjugated with the polyaromatic matrix. The 1600–1500 cm⁻¹ band can be assigned to not only the stretching vibration of C=C bonds in olefin structures but also in-plane skeletal C=C vibrations of aromatics. Peaks occurring at 1440 and 1375 cm⁻¹ are ascribed to oxygen functionalities such as highly conjugated C=O stretching, C–O stretching in carboxylic groups, and carboxylate moieties (Tongpoothorn et al., 2011). The signal at 1440 cm⁻¹ also indicates the presence of compounds containing methoxy–O–CH₃. The peak at 1232 cm⁻¹ indicates the presence of C=O–C stretching vibration in aryl-alkyl ether linkage. The intense band positioned around 1052 cm⁻¹ can be associated with C–O stretching vibrations in acids, alcohols, phenols, ethers and esters. The band centered at 617 cm⁻¹ is likely due to in-plane ring deformation. Finally, the presence of weak bands in the 731–864 cm⁻¹ wavenumber ranges and at 550 cm⁻¹ could be due to C–H out-of-plane bonding in benzene derivatives.

According to Yang et al. (2007), the highest IR absorbance of O=H and C–O was found with commercial cellulose while commercial hemicellulose contained higher C=O compounds. For AF peel used in this study, the highest IR absorbance is observed for C–O. This complies with biochemical component analysis result which also proved that AF peel is rich in cellulose (53.6%). All the

![Fig. 1. FTIR spectra of raw AF peel and AFP AC produced under activation conditions of 750 °C and 30 min.](image1)

![Fig. 2. EDX spectra of raw AF peel (a) and AFP AC produced under activation conditions of 750 °C and 30 min (b).](image2)
functional groups suggested for raw AF peel are in good agreement with those reported by Yang et al. (2007) for commercial cellulose, hemicellulose and lignin. This is an indisputable evidence of the lignocellulosic structure of AF peel. It can be concluded that lignocellulosic AF peel waste consists of alkene, esters, aromatics, ketone and alcohol, with different oxygen-containing functional groups. Similar results were also reported for other lignocellulosic bio-wastes such as pine sawdust (Du et al., 2016) and barley grain (Kodama et al., 2015).

The main features of the FT-IR spectrum registered for AFP AC show a resemblance to that of raw AF peel but with different band intensities. This situation may be attributed to the decomposition of cellulose, hemicellulose and lignin in the AF peel during carbonization and activation processes. A considerable decrease in the intensities of the bands around 3400 and 1052 cm⁻¹ most likely indicate the decrease of OH and C–O in the prepared AFP AC. This is in accordance with Boehm titration result obtained in this study (Table 2) which also showed the presence of a small amount of acidic surface functional groups in the prepared carbon. The disappearance of bands at 2920 and 2854 cm⁻¹ can be associated with the breaking of aliphatic C–H groups in AFP AC during carbonization and activation processes. The high carbon content of AFP AC, which was determined through elemental analysis (Fig. 2), confirms this result.

3.3. Adsorption capacity

Iodine number analysis is an experiment that determines the adsorption capacity and porosity of AC. It is defined as the milligrams of iodine adsorbed by 1 g of AC. This adsorption is a measure of the micro-pore content of AC (0–20 Å, or up to 2 nm) which is equivalent to surface area of carbon between 900 and 1100 m²/g. A high iodine adsorption capacity generally indicates the existence of high surface area and large amount of micropores in AC (Tounsiadi et al., 2016). AC with an iodine number value greater than or equals to 900 mg/g is classified as high quality AC (Ozdemir et al., 2014).

In this work, the effect of activation temperature on iodine adsorption of AFP AC was investigated in the range of 550–750 °C. This temperature range was chosen because previous work done by Demiral et al. (2011) showed that very high activation temperatures (800, 850 and 900 °C) resulted in higher percentage of carbon burn-off and decreased yield of AC prepared from olive bagasse by physical activation. The weight loss may be attributed to the continual evaporation of the bio-char and carbon burn-off due to C–H₂O reaction. During steam activation, carbon in the bio-char reacts with steam (H₂O) to form CO, CO₂ and H₂ according the following equations (Chowdhury et al., 2013):

\[
\text{C} + \text{H}_2\text{O} \rightarrow \text{CO} + \text{H}_2
\]

(5)

\[
\text{Char to CO and H}_2:
\]

\[
\text{C} + 2\text{H}_2\text{O} \rightarrow \text{CO}_2 + 2\text{H}_2
\]

(6)

These reactions result in the removal of carbon atoms from the char and are widely known as chief mechanisms contributing to the development of pores in steam-gasified carbon.

Referring to Fig. 3, it is irrefutable that activation time and temperature have significant effect on iodine number of AFP AC. Our results indicated that an increase in activation time from 40 to 60 min, caused iodine sorption capacity to increase at temperatures 750, 650 and 550 °C. This is due to longer time allows heat to pervade deeper inside particles and aids in the vaporization of

### Table 2

<table>
<thead>
<tr>
<th>Activated carbon</th>
<th>Activation agent/Impregnation ratio</th>
<th>Activation temperature (°C)</th>
<th>Surface functional groups (mmol/g)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Olive bagasse</td>
<td>steam/...</td>
<td>900</td>
<td>1.59</td>
<td>0.17</td>
</tr>
<tr>
<td>Commercial coal (F-400)</td>
<td>air/-</td>
<td>800</td>
<td>0</td>
<td>0.01</td>
</tr>
<tr>
<td>Artocarpus integer fruit peel</td>
<td>steam/...</td>
<td>750</td>
<td>1.34</td>
<td>0.21</td>
</tr>
<tr>
<td>Flamboyant pod</td>
<td>sodium hydroxide/3:1</td>
<td>700</td>
<td>0.96</td>
<td>0</td>
</tr>
<tr>
<td>Bamboo</td>
<td>phosphoric acid/1:1</td>
<td>600</td>
<td>0.64</td>
<td>0.62</td>
</tr>
<tr>
<td>Jackfruit peel</td>
<td>phosphoric acid/1:1</td>
<td>550</td>
<td>0.78</td>
<td>0.74</td>
</tr>
<tr>
<td>Pine cone</td>
<td>phosphoric acid/3:1</td>
<td>500</td>
<td>1.74</td>
<td>0.72</td>
</tr>
<tr>
<td>Tomato waste</td>
<td>zinc chloride/6:1</td>
<td>400</td>
<td>0.52</td>
<td>0.52</td>
</tr>
<tr>
<td>Jackfruit peel</td>
<td>phosphoric acid/4:1</td>
<td>350</td>
<td>1.3</td>
<td>0.65</td>
</tr>
</tbody>
</table>
vocates from the inner part of particles. However, the increase of activation time from 60 to 100 min resulted in lower iodine values at temperatures 750, 650 and 550 °C. Prolonged activation most probably had caused acceleration of surface erosion and destruction of micro-porosity of AFP AC resulting in collapsing of some walls between pores and widening of micro- and meso-pores to macro-pores, thus decreasing iodine sorption capacity.

Iodine numbers of the prepared carbons increased with increasing activation temperature from 550 to 750 °C. This trend is expected because the volatiles resulting from the decomposition of major components of AF peel continue to evolve with increasing activation temperature, creating new porosities, thus, resulting in greater surface area. A shorter activation time for the preparation of AFP AC with a high iodine number is favorable for large scale production of the carbon as shorter activation time results in reduced production cost.

Based on our results, a maximum iodine adsorption of 1411 mg/g was achieved under activation conditions of 750 °C and 60 min for AFP AC. It can be said that, AFP AC prepared under these conditions is of high quality with a large amount of micropores. Therefore, AFP AC prepared under activation conditions of 750 °C and 60 min was used for all subsequent analyses. The maximum iodine number of AFP AC is comparable with those reported by other researchers for activated carbons prepared using various agro-waste precursors under different conditions (Table 3). It should be pointed out here that the optimum iodine adsorption capacity of AFP AC is slightly higher than that of a commercial coal-based first grade AC, which has an iodine number value of 1000 mg/g (Song et al., 2013).

### 3.4. Microstructure

In order to investigate the surface topography of the samples, raw AF peel, AF peel char (collected prior to activation process) and AFP AC with the highest iodine number value were subjected to scanning electron microscopy (SEM). The surface of the raw material (Fig. 4a) is quite dense, moderately smooth and homogenous without any cracks, voids and pores. Similar surface topography has been previously observed for other lignocellulosic carbon precursors such as grape stalk (Ozdemir et al., 2014) and raw jute (Du et al., 2016). On the contrary, it can be seen from the microstructures of the AF peel char (Fig. 4b and c) that a major surface damage appeared, which means carbonization is developed. The wrecked surface of the char is quite dense with some occasional rudimentary pores and cracks. Similar surface destruction has been observed by Vargas et al. (2011) for the char obtained from lignocellulosic flamboyant pod. As can be seen in Fig. 3c, the surface of the char is not highly porous. Pores were not completely formed and the pores formed were not widely opened. Porosity of carbon can be closed by deposits of non-condensable liquids (tars) which are residuals of the carbonization process. Apart from deposition of tar, agglomeration and migration of inorganic matter from the bottom all the way up to the outer surface of bio-char during pyrolysis, may also give rise to pore blockage.

In this study, steam is used as the activating gas to open the clogged pores and develop new porosity in the bio-char. From the micrographs of AFP AC (Fig. 5a–c), it is obvious that the morphological characteristics differed strongly between the prepared carbon, AF peel char (Fig. 4b–c) and raw AF peel (Fig. 4a). AFP AC possesses a rough texture with heterogeneous surface. The external surface of the prepared carbon showed more crevices and full irregular cavities compared to that of bio-char, indicating that activation process happened and the surface was aggressively attacked and eroded by steam. The thick wall of raw AF peel opened and pores of different sizes were created by steam activation. A highly porous structure was formed because most of the volatiles were evolved, leaving behind the ruptured surface of AFP AC with

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### Table 3

A comparison of iodine adsorption capacity of agro-waste based activated carbons prepared under different conditions.

<table>
<thead>
<tr>
<th>Activated carbon type</th>
<th>Activation agent/Impregnation ratio</th>
<th>Activation temperature (°C)</th>
<th>Activation time (min)</th>
<th>Iodine number (mg/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Artocarpus integer fruit peel</td>
<td>Physical steam</td>
<td>750</td>
<td>60</td>
<td>1411</td>
</tr>
<tr>
<td>Olive waste-cake</td>
<td>Physical steam</td>
<td>850</td>
<td>60</td>
<td>1495</td>
</tr>
<tr>
<td>Rubber-seed shell</td>
<td>Physical steam</td>
<td>880</td>
<td>60</td>
<td>1326</td>
</tr>
<tr>
<td>Corn</td>
<td>Physical steam</td>
<td>800</td>
<td>120</td>
<td>665.23</td>
</tr>
<tr>
<td>Moringa oleifera seed husk</td>
<td>Physical steam</td>
<td>800</td>
<td>30</td>
<td>703</td>
</tr>
<tr>
<td>Olive bagasse</td>
<td>Physical steam</td>
<td>900</td>
<td>45</td>
<td>500</td>
</tr>
<tr>
<td>Rubberwood sawdust</td>
<td>Physical steam</td>
<td>750</td>
<td>60</td>
<td>765</td>
</tr>
<tr>
<td>Apricot stones</td>
<td>Physical steam</td>
<td>900</td>
<td>na</td>
<td>1200</td>
</tr>
<tr>
<td>Grape stalk</td>
<td>Chemical zinc chloride/2:1</td>
<td>700</td>
<td>120</td>
<td>1760</td>
</tr>
<tr>
<td>Acorn shell</td>
<td>Chemical zinc chloride/1:1</td>
<td>600</td>
<td>30</td>
<td>1209</td>
</tr>
<tr>
<td>Date Pit</td>
<td>Chemical Ferric chloride/1:5:1</td>
<td>700</td>
<td>60</td>
<td>761.4</td>
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<tr>
<td>Jatropha curcas fruit shell</td>
<td>Chemical sodium hydroxide/4:1</td>
<td>800</td>
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<td>1138.13</td>
</tr>
<tr>
<td>Waste ramulus mori</td>
<td>Chemical dianmonium hydrogen phosphate/2:1</td>
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<td>120</td>
<td>847.21</td>
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<td>Corn cob</td>
<td>Chemical potassium hydroxide/3:1</td>
<td>800</td>
<td>120</td>
<td>1262.25</td>
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</table>

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![Fig. 3](image_url)  
**Fig. 3.** Effect of activation temperature and time on iodine adsorption capacity of AFP AC.
Fig. 4. Surface views of raw AF peel (a) and AF peel bio-char collected prior to steam activation (b)–(c) obtained by SEM.
inhomogeneous pores. As is widely known, lignocellulosic materials are not rigid. The fragmentation of cellulose and hemicellulose during carbonization and activation causes non-carbonic elements such as oxygen and hydrogen to change to volatiles. The accumulation of these volatiles leads to development of pressure inside the particles. When these trapped volatiles diffuse out through the particles, the pores are evolved.

The presence of small, large and transitional pores with cylindrical and conical shapes in AFP AC could be clearly identified from the micrographs shown in Fig. 5a and b. It is also obvious that AFP AC prepared in this study is rich in open pore, which was convincingly proved by SEM (Fig. 5b). These open pores which connect to the surface of the prepared bio-carbon suggest the suitability of the newly prepared AC for functional applications.
such as adsorption, catalysis and sensing.

It is known that lignocellulosic materials with a greater content of lignin develop AC with macroporous structure, while those with a higher content of cellulose yield activated carbons with a predominantly microporous structure (Ioannidou and Zabaniotou, 2007). It is also generally accepted that carbon dioxide activating gas develops mainly microporosity, and that steam produces a wider pore size distribution, with larger development of wide micropores and mesopores in carbons prepared from lignocellulosic precursors (Sun and Chun Jiang, 2010). Therefore, it can be said that AFP AC prepared in this study using cellulose rich AF peel with steam activation method may contain a large number of both micro- and mesopores. Hence, AFP AC is also expected to have a high micro- and mesopore adsorptive capacity.

It is commonly admitted that activated carbons contain variable amounts of mineral matter (ash content) depending on the nature of the raw material used as precursor (Bandosz, 2006). As can be seen from Fig. 5b, the surface of AFP AC presents small amounts of white particles, probably ash content, which may fill or block some portions of the pores.

3.5. Amorphous structure

Activated carbon is usually in the form of amorphous structure which is composed of microcrystalline carbon fragments (graphite-like microcrystalline, single reticular-plane carbon and non-organized carbon) randomly (Tang et al., 2012). X-ray diffraction analysis is crucial to determine the amorphous nature of AFP AC prepared in this study.

The X-ray diffractogram of AFP AC (Fig. 6) exhibits two broad peaks at approximately $2\theta = 23^\circ$ and $2\theta = 43^\circ$ which are assigned to the reflection from (002) and (100) planes respectively. The occurrence of these two broad peaks signifies an increasing regularity of crystal structure, resulting in better layer alignment. The (002) and (100) planes can be attributed to microcrystalline and turbostratic graphite structures respectively. In contrast, the absence of broad peak and presence of sharp peaks in X-ray diffraction profile of the AF peel char (Fig. 6), gives no indication of any amorphous structure. Similar findings have been previously reported for AC prepared from rice-straw (Adinaveen et al., 2015).

The strong (100) surface diffraction peak of AFP AC is comparable to that of commercial activated carbon obtained by Tang et al. (2012). This indicates that microcrystalline size of the prepared carbon is very close to that of commercial AC. The XRD pattern proved the amorphous state of AFP AC, which is a beneficial property for well-defined adsorbents. Yang and Lua (2006) surmised that a very high activation temperature leads to the burn-off of the less ordered components, resulting in less amorphous structure. This shows that 750 °C, the activation temperature used in this study, is appropriate to obtain AFP AC with highly amorphous nature.

3.6. Cost estimation for production of AFP AC

One of the main circumstances that slightly influence whether an agro-industrial residue is practical or not is the processing cost. The prime components that need to be considered in the production of AFP AC are the raw material, transportation, consumables (chemicals and gases) and power consumption. Referring to Table 4, the estimated cost involved in the production of AFP AC is 1.67 US$/kg, which is indeed a very reasonable expense. The cost of manpower utilization for industrial-scale production of AFP AC is not approximated due to its huge variability. It should be pointed out that steam gasification is an endothermic process. At industrial scale, the external heating process which is necessary for maintaining the activation temperatures will add to the cost of final product. Direct fire-heated systems with both steam and air fed into the kiln are the most economic (Menendez-Diaz and Martin-Gullon, 2006). According to our search from various sources through the internet, the cost of commercial AC in the world market varies between US$ 2.2–5 per kg based on the quality of AC. From the financial point of view, conversion of AF processing waste into AC would increase economic value of the agro-industrial residue and most importantly provide a promising cost-effective alternative to the existing commercial activated carbons.

4. Conclusions

The origin of carbon may play a vital role in the quality of AC produced, but in order to reduce ecological impact, local AF processing waste can be used as an alternative in AC manufacturing process. AF peel was found to be lignocellulosic with high cellulose (53.6 wt%) and carbon (60.8 wt%) content making it a suitable precursor for production of AC. AF processing solid waste is successfully converted into a novel AC by steam pyrolysis. Steam activation temperature, 750 °C and activation time of 60 min were the optimum conditions for preparing high quality AFP AC with a large iodine adsorption capacity (1411 mg/g), highly micro-porous and amorphous structure and primarily basic surface functional groups. It is evident from the microstructure images that the raw material is non-porous, pyrolysis only resulted in the formation of rudimentary pores and steam activation process is the main contributing factor to the development of porosity and

<table>
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<tr>
<th>Component</th>
<th>US$/kg</th>
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<tbody>
<tr>
<td>Transportation</td>
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</tr>
<tr>
<td>Hydrochloric acid</td>
<td>0.1</td>
</tr>
<tr>
<td>Distilled water</td>
<td>0.5</td>
</tr>
<tr>
<td>Nitrogen gas</td>
<td>0.2</td>
</tr>
<tr>
<td>Water (for steam)</td>
<td>0.02</td>
</tr>
<tr>
<td>Power consumption</td>
<td>0.8</td>
</tr>
<tr>
<td>Total</td>
<td>1.67</td>
</tr>
</tbody>
</table>

Table 4 Estimated cost production of AFP AC (US$/kg).
intensification of micro-pores in the produced AC. The newly developed AFPC AC showed structural characteristics that are very appropriate to adsorption processes. The production of AFPC AC could be of great interest and the findings of this study can be a helpful reference to expand the profit of AFPC processing industry. A short activation time (60 min) for the preparation of AFPC AC is favorable for large scale industrial production as it resulted in reduced production cost. AFPC AC can be produced at an affordable price of ~1.67 US$/kg which may be used by both large- and small-scale industries for a cleaner and greener adsorption processes, consequently, improving environmental quality.

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