

Article

A New Activated Carbon Prepared from Sago Palm Bark through Physicochemical Activated Process with Zinc Chloride

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Abstract. This study aimed to use sago palm bark to formulate a new adsorbent activated carbon (AC) contains highly surface area through physicochemical method via $ZnCl_2$ activation. Conduction of the activation process was performed at varying impregnation ratios (0.5-2.0). Thermal decomposition was determined using thermogravimetric analysis (TGA). Porosity characterizations of AC were conducted by using N_2 adsorption-desorption in order to characterise properties like pore volume, surface area, and micropore volume. To detect the presence of functional groups which were found on the surface of AC, Fourier Transform Infrared Spectroscopy (FTIR) analysis was utilised. Morphology of AC was determined using scanning electron microscopy (SEM) and X-ray spectroscopy (EDX). Experimental results showed that maximum AC surface area was $1737 \text{ m}^2/\text{g}$. Activation temperature was revealed to be 700°C , with chemical impregnation ratio of zinc chloride to a precursor equal to 1.5/1.

Keywords: Activated carbon, sago palm bark, physicochemical activation, zinc chloride, surface area.

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1. Introduction

When it comes to advanced treatment of water and wastewater, adsorption theory is thought to be a successful process. The term adsorbent refers to solid materials that offer a surface for the adsorption process to occur while the species adsorbed on the solid surface is referred to as the adsorbate [1]. Activated carbon (AC) is widely utilised in adsorptive of pollutants that may be present in water and wastewater because its structure is highly microporous and it has high porosity and internal surface area [2]. Several studies in recent years have aimed to produce more efficient materials that have lower costs and widely available [3, 4]. Because of their availability and abundance, agricultural products are considered an excellent source to produce AC [5]. The most commonly used raw materials among these agricultural by-products were palm of date [6, 7], palm oil waste [8, 9, 10], woods [11, 12, 13, 14], coconut shell [15, 16, 17], bagasse [18]. A waste material converted to AC is a great potential form of waste recycling [19, 20, 21]. A few researchers investigated on the characterization of AC [22, 23, 24] and on utilization of it [25, 26].

Generally, physically or chemically method is used to prepare AC. Physical process involves two steps: first step involves carbonisation [27] and the second step is activating the resulting char under high temperatures and in the presence of a gaseous activating agent like air or steam, The carbonisation process is vital in the transformation of cellulose structures that have several oxygen- and hydrogen- containing functional groups into a carbonaceous material. Dehydrating chemical agents can be used to remove such cellulose structures. Dehydration is normally performed at elevated temperatures under pyrolytic conditions. This results into the destruction of the cellulose structures. Tar formation is inhibited and carbon yield is increased by the chemical activation of the agents [28, 29]. Several researchers have made use of $ZnCl_2$ for the chemical activation step of the AC preparation. Ozdemir et al. (2014) [30] used $ZnCl_2$ activating agents in CO_2 atmosphere for the chemical activation process for their AC preparation from grape stalk. They obtained a $1411\text{ m}^2/\text{g}$ BET surface area value. Azevedo et al. (2007) [31] used $ZnCl_2$ under nitrogen and vacuum conditions to prepare AC from coconut shell. The resulting preparation had a surface area of $2527\text{ m}^2/\text{g}$ at temperature of 700°C , and 2-hours holding time.

Researchers used new method to prepare AC using a physicochemical activation process, which is a combined physical with chemical method. Pore development step of this method is carried out using both of the previously described activation procedures. Physicochemically prepared AC obtained a higher surface area compared with physically or chemically prepared ones (Aber et al., 2009; El Qada et al., 2008) [32, 33]. Salman et al. (2010) [34] manufactured AC using palm fronds as a raw material. Their physicochemical activation was carried out using CO_2 and KOH and as physical and chemical activation agents, respectively. The procedure involves a 2-hour carbonisation step at 700°C in presence of N_2 flow, before being soaked in KOH solutions with different impregnation ratios. The preparation's last step involves activation under CO_2 atmosphere at varying times and temperatures. It was revealed that optimum value of temperature was 850°C with impregnated ratio of 3.75, and 1 hour activation time. Under optimum conditions, prepared carbon has BET surface area $1237.13\text{ m}^2/\text{g}$ and carbon yield 21.6%, respectively.

Hu et al. (2007) [35] prepared AC using pistachio shells through chemical and physicochemical activation methods by utilising KOH and KOH- CO_2 , respectively. The results revealed that using chemical activation, the prepared AC surface area $1013\text{ m}^2/\text{g}$ when prepared at 780°C for one hour. Chemical activation with CO_2 increased the surface area of AC up to $2145\text{ m}^2/\text{g}$, which shows that AC prepared with physicochemical activation method has a surface area that is twice as high as that of AC prepared only with chemical activation.

This study was able to utilise a new raw material sago palm bark (SPB) to prepare AC. SPB is considered a renewable source that contains lignocellulosic material [36]. In Malaysia, typically use sago to produce sago starch. The sago starch industry discards more than 20,000 ton/yr of SPB. Sago thrives in humid tropical lowlands. The plant has maximum height of 25, and 40 cm diameter [37]. SPB is one of Malaysia's main carbohydrate sources, as it contains about 60 to 70% cellulose and hemicellulose [36]. This alone makes it an important factor for AC preparation. The main objective of this study was to prepare AC from SPB with high porosity and a large specific surface area, considered a high potential adsorbent. Many studies used different agricultural wastes as precursor to prepare AC, however, based on our literature survey, no extensive study was conducted on AC preparation from SPB.

2. Experimental Work

2.1. Preparation Method of AC

Trunks of plant were bought from a local plantation located in Melaka, Malaysia. The trunks of the sago palm were debarked and the piths were removed (the inner portion) to obtain the bark fraction (the outer layer). After collection, drying of the bark was conducted for 24 hours using a drying oven set at 105°C. Afterwards, the bark was crushed into smaller sizes ranging from 0.3 to 0.6 mm. AC preparation involves the two steps described below:

a) Carbonisation

From the prepared sample, about 10 g was transferred to a cylindrical stainless steel reactor that had a sealed end. Cylindrical reactor was then inserted into an electric horizontal tubular furnace given a continuous nitrogen flow of 100 mL/min. The dimensions of the furnace tube are 50 mm in diameter and 800 mm length. Carbonisation was carried out at 300°C and an average heating rate of 10°C/min. The reaction lasted for a total of 4 hours.

b) Activation

Chemical activation of the carbonised sample was done using $ZnCl_2$ under varying impregnation ratios (0.5, 1.0, 1.5 and 2.0). The impregnation process was done in a cylindrical flask that is simultaneously agitated with a heating magnetic stirrer. This process was conducted at room temperature. The chemical agent and precursor were thoroughly mixed for 24 hours. Afterwards, drying of the resulting sample at 100°C for 24 hours. Once sample was dried, insertion of the impregnated material into regulated furnace at different activation temperatures (700, 800 and 900°C) and under continuous nitrogen flow (100 ml/min) for one hour. After cooling, hot water was used to wash sample then with cold distilled water till neutral pH. Last step is drying sample overnight at 100°C before being stored in a desiccator for future uses. Yield of AC is calculated using following formula:

$$Yield (\%) = \frac{\text{weight of AC after Activation}}{\text{weight of the raw material}} \times 100$$

2.2. Sample Characterization

The oven-drying test method (ASTM D2867-09) was used to calculate the moisture content [38]. First, an AC sample is transferred to a dry and closed capsule with a known weight before being subsequently weighted accurately. Then, drying capsule in oven with temperature ranging from 145-155°C. Once the sample is dried, the capsule is closed and removed from the oven before being cooled. Accurate weighing of capsule is then performed again. The moisture content is represented by percentage difference of weight.

The chemical components of raw material of SPB are mainly consisting of hemicelluloses, cellulose, lignin and ash [39]. The ash content of sample was determined by weighed the dried sample to the nearest 0.1 mg and taken it into the crucible of known weight. The crucible was placed in the muffle furnace at 650°C and ashing was considered to be completed when content weight is achieved. The crucible is cooled to room temperature in a desiccator and the ash content was considered as the percentage weight of the sample remained [40].

Using EDX (Thermo scientific, USA), the chemical concentration of elements for SPB was performed. The composition characteristics analysis of SPB is mentioned in Table 1.

Table 1. Proximate and composition of SPB.

Analysis	Dry weight (%)
Proximate	
Moisture content	9
Ash content	1.53
Chemical composition	
Cellulose	44.13
Hemicellulose	21.09
Lignin	23.3

2.3. Porosity Characterisation

To characterise the porosity, N₂ adsorption-desorption isotherms at (-195.6°C) need to be determined using an automatic adsorption instrument (Micromeritics ASAP 2020 V3.04). Evaluation of surface area for sample was done used BET method [41].

2.4. Analysis of Chemical Nature

Surface functional groups were estimated using FTIR spectroscopy (Thermo Scientific, Nicolet 6700). The different samples' FTIR were recorded to be between 400 and 4000 cm⁻¹.

2.5. Scanning Electronic Microscopy (SEM) and EDX Analysis

SEM (model S-3400 N, Hitachi, Japan) coupled with EDX were used to identify the morphology of the AC and conduct elemental analysis of the adsorbent.

3. Experimental Results and Discussions

3.1. Thermal Characterisation of SPB

This study used SPB as a raw material to prepare AC. As shown in Table 1, SPB was found to have ash low content (1.53%), this make SPB is the best choice for AC preparation and production [29]. To calculate the production yield of the precursor, the mass of AC is divided by raw material initial mass that was utilised for activation [42]. The thermal degradation of the raw material of SPB was examined using thermogravimetric analysis (TGA). The TGA of SPB was conducted in N₂ atmosphere with the temperature range of 25-900°C as shown in Fig. 1. There are three stages involved in the thermal decomposition of SPB: (1) the first stage at 25 to 125°C, resulting in the loss of weight due to remove of bound and unbound water; (2) most loss in weight taking place in the second stage due to thermal degradation of the main component of biomass cellulose and hemicellulose at temperatures ranging from 150 to 400°C [43, 44]; and (3) slow weight loss in the third stage taking place at temperatures above 400°C because of the sample's higher lignin content. Jin et al. (2013) [44] reported that the thermal degradation of lignin occurred at a low rate in the range of 100 to 700°C. This thermal degradation can be accompanied with a tiny peak at 340°C.

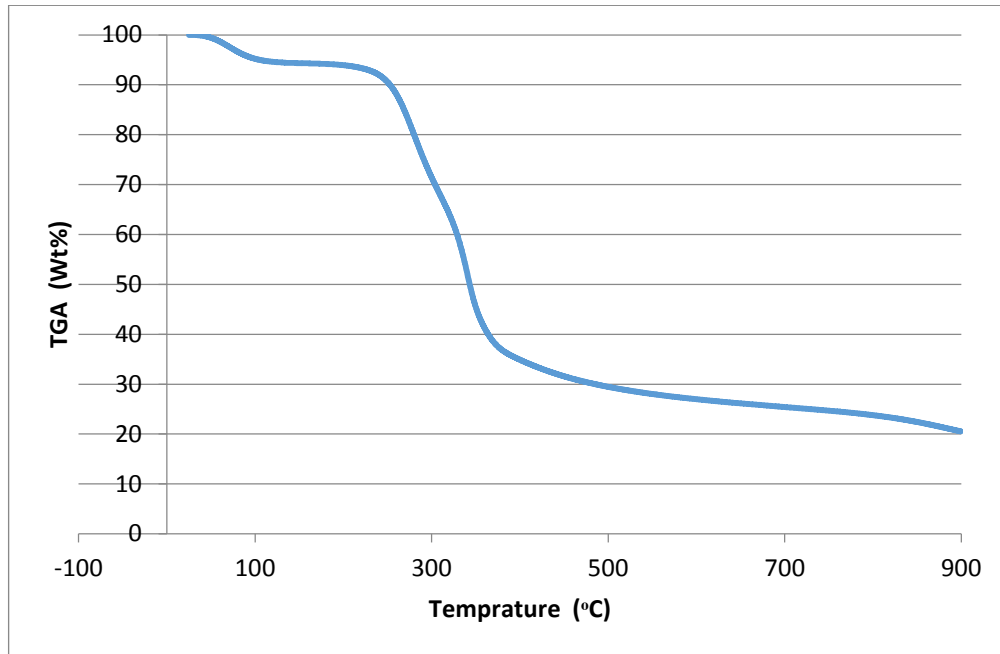


Fig. 1. Thermogravimetric analysis (TGA) of the raw material of SPB.

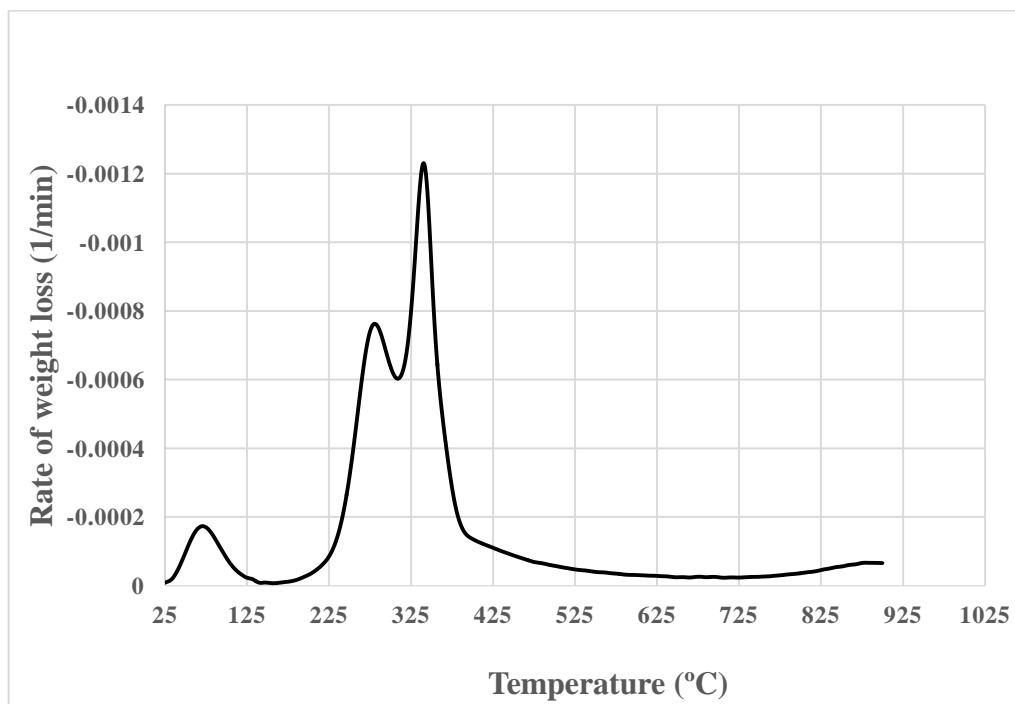


Fig. 2. Differential thermogravimetric analysis (DTG) of SPB.

Figure 2 shows the differential thermogravimetric analysis (DTG) of the SPB. The weight loss observed at the first peak at 38°C in DTG can be attributed to the moisture, while the two peaks observed at 260 and 350°C are attributed to hemicellulose and cellulose thermal degradation [43]. For lignin, no clear peaks were detected, though it might have been located at the same position as the cellulose peak, since lignin has tiny peak at 340°C [44].

3.2. Effect of Activation Temperature on the Yield of AC

The yield of AC can be determined from the weight of resultant AC divided by the weight of dried sago palm bark. The yield of AC was calculated at different activation temperatures (600, 700, 800 and 900°C). Figure 3 shows the effect of activation temperature on the yield of AC for sago palm bark. The yield decreases from 65 to 30% with increasing of activation temperature from 700 to 900°C. This is due to the promotion of carbon burn-off and tar volatilization at higher temperature [45]. So, the activation temperature of AC would be equal to 700°C, which resulted in higher percentage of AC yield.

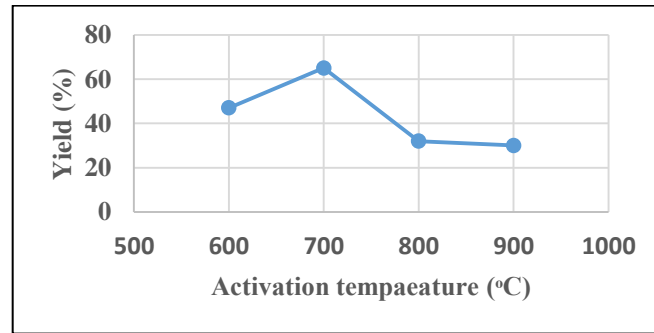


Fig. 3. Effect of activation temperature on the yield of AC.

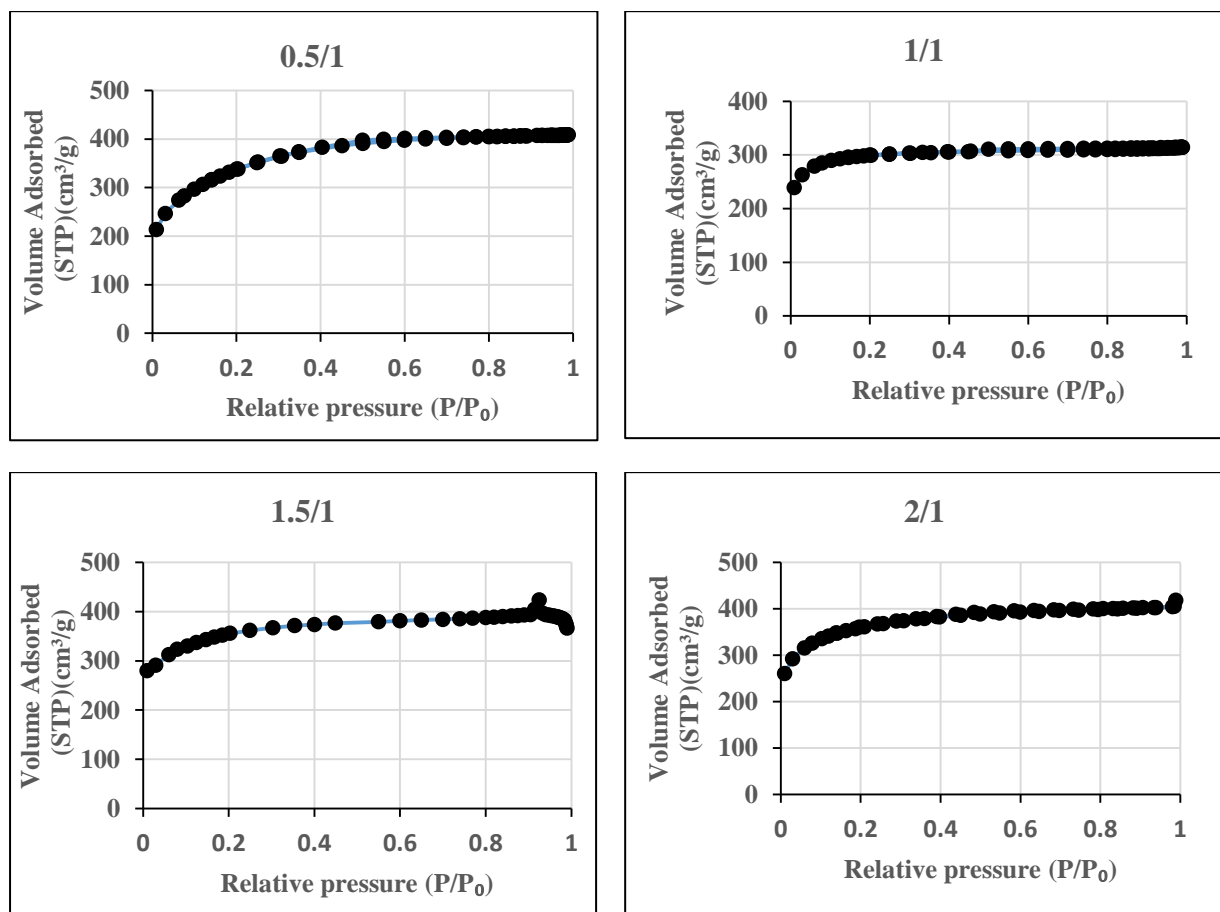


Fig. 4. N₂ gas adsorption-desorption isotherms of AC-SPB prepared at different impregnation ratios.

3.3. Effect of Preparation Conditions on Porosity Characterization

3.3.1. Nitrogen gas adsorption-desorption isotherms

The isotherms of nitrogen adsorption-desorption for AC are shown in Fig. 4. It shows the N₂ adsorption-desorption isotherms of the prepared AC from SPB using different impregnation concentrations of ZnCl₂ to precursor: 0.5, 1, 1.5 and 2 at activation temperature of 700°C for 1 h. The N₂ adsorption capacity of the samples increased from 213.26 to 279.97 cm³/g with the increase of impregnation ratio from 0.5 to 1.5 and then decreased to 260.19 cm³/g with the further increase of impregnation ratio to 2. It can be concluded that the increasing of impregnation ratio from 0.5 to 1.5, the volume of N₂ adsorbed gradually increased. While the volume of N₂ adsorbed decreased with increasing of impregnation ratio from 1.5 to 2, this is because the destruction of pore walls caused by high impregnation of ZnCl₂ to the precursor. It confirms to the findings reported by Rosas et al. (2009) [46] and Diao et al. (2002) [47].

3.3.2. Effect of impregnation ratio on surface area and pore volume

The effect of impregnation ratio on the surface area and pore volume of the prepared AC-SPB is shown in Fig. 5 with the increase of impregnation ratio from 0.5 to 1.5, surface area and pore volume increased from 1348.21 m²/g and 0.486 cm³/g to 1737.72 m²/g and 0.632 cm³/g, respectively. Whereas the trend reversed at higher impregnation ratio for surface area and pore volume.

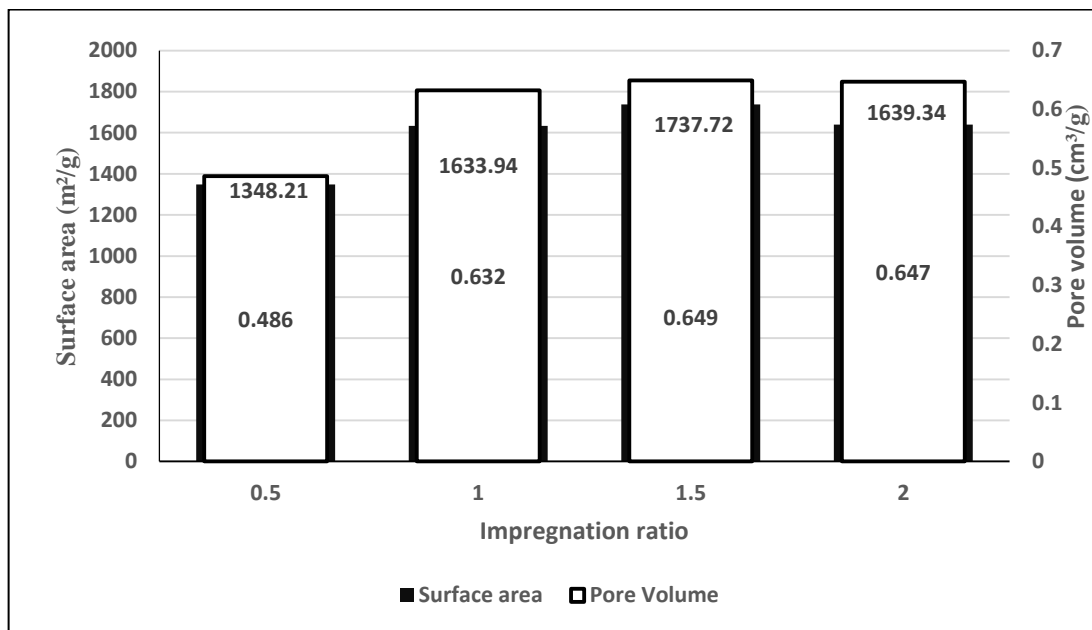


Fig. 5. Effect of impregnation ratio on surface area and pore volume of AC-SPB activated with ZnCl₂.

3.3.3. Effect of impregnation ratio on micropore surface area and micropore volume

The effect of impregnation ratios of prepared AC-SPB activated with ZnCl₂ for 1 hour at 700°C is shown in Fig. 6. Since the contribution is very high to the total surface area and pore volume, the trends of micropore surface area and volume also followed the same fashion of total surface area and pore volume. Preferable results of micropore surface area (1148.58 m²/g) and micropore volume (0.335 cm³/g) were obtained at 1.5 impregnation ratio.

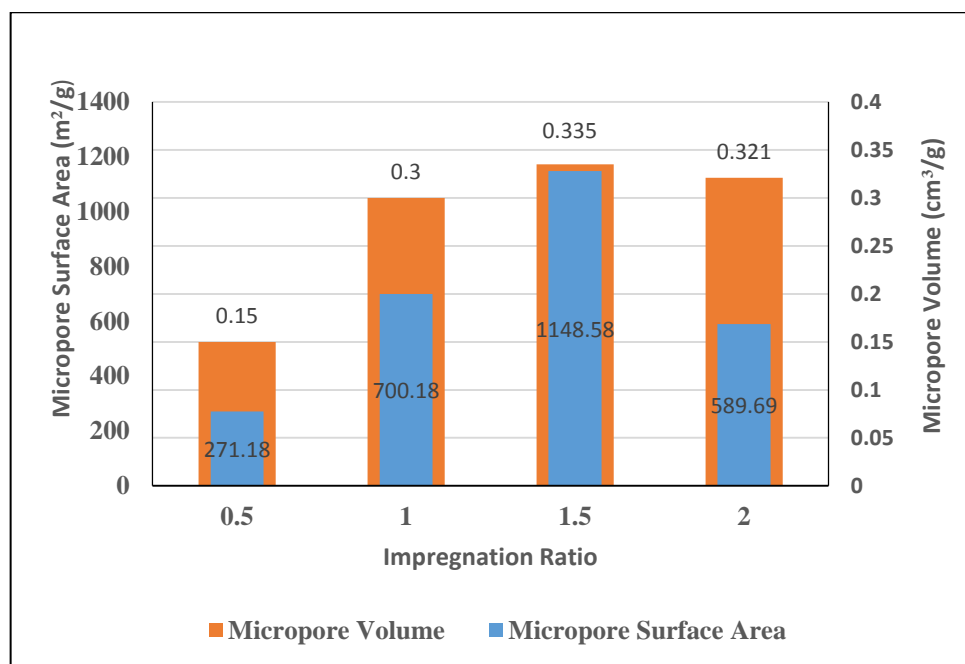
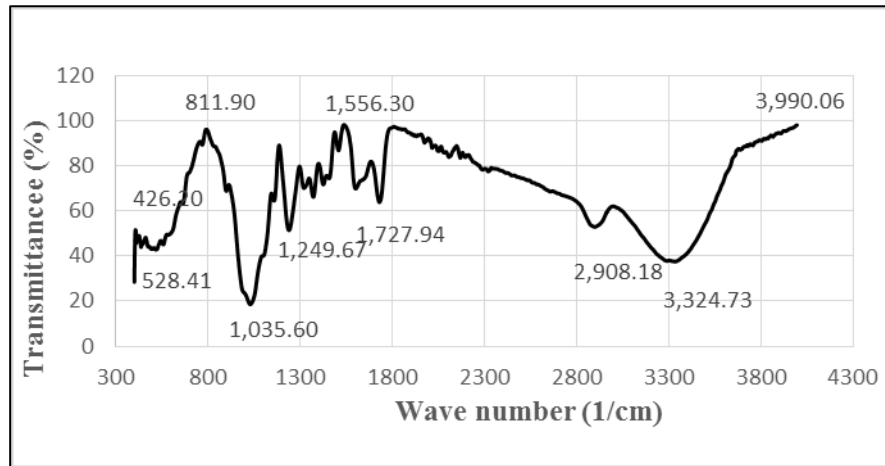


Fig. 6. Effect of impregnation ratio on micropore surface area and micropore volume of AC-SPB activated with $ZnCl_2$.

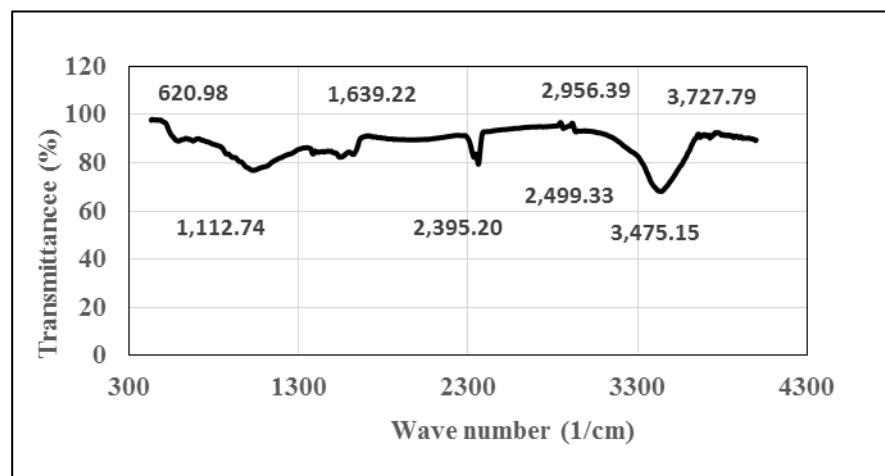
As a result, with the increasing of impregnation ratio, surface area and pore volume gradually increased and achieved its peak value at 1.5 whereas at higher impregnation ratio, the trend reversed. $ZnCl_2$ acts as a dehydration agent during activation process that inhibits tar formation and any other liquids that can clog up the pores of the sample [48, 49].

3.4. FTIR Analysis

Figure 7 shows the surface chemistry of SPB and the AC prepared sample. The raw material is made up of different groups such as mines, hydroxyl groups, aldehydes, carbonyl compounds. The broad peak at 3324 cm^{-1} is due to the presence of amine groups (N-H). The band observed at 2908 cm^{-1} indicates the presence of C-H stretching vibrations of methyl group [50]. The peaks found at 1727 cm^{-1} and 1556 cm^{-1} were due to primary amine group. The adsorption at 1035 cm^{-1} can be ascribed to C-N stretching and adsorption 811 cm^{-1} ($<1000\text{ cm}^{-1}$) represents 'fingerprint' region. FTIR analysis revealed that it was simpler and consisted of a considerable amount of functional groups. As seen on the FTIR analysis of the AC, the broad peak observed between 1544.72 and 1673.94 cm^{-1} due to the primary amine groups. The methylene group (C-H) is responsible for the sharp peak at 2219.70 cm^{-1} . Many functional groups which were observed in raw material were disappeared in case of prepared AC in the range 1727 cm^{-1} to 811 cm^{-1} . The presence of amine group (N-H) and methylene group (C-H)) in prepared AC played very important role in adsorption of heavy metals from solution samples and the surface functional groups have their own importance to drive adsorbate ions to adsorption sites on the adsorbent [38]. The amine group can remove the hydrogen sulfide gas efficiently from a gas stream. Table 2 shows various functional groups found for AC and their frequency ranges.



(a)



(b)

Fig. 7. FTIR analysis (a) raw material (b) AC activated with $ZnCl_2$ at 1.5/1 impregnation ratio.

Table 2. Various functional groups found for AC-SPB and their frequency ranges.

Group frequency (cm^{-1})	Functional group
3770	Free OH groups
3360-3310	N-H stretch (Amines)
2935 - 2915	C-H stretch (Methylene)
2900 -2880	C-H stretch (Methyne)
2360 -2330	C-N stretch (Nitrile)
1650-1550	N-H bend (Amines)
1190 -1130	C-N stretch (Amines)

3.5. The Surface Morphologies of SPB

The surface morphology of AC was performed using SEM test as shown in Fig. 7. The SPB raw material's texture was observed to be rigid and non-porous (Fig. 7(a)). When observed under higher magnification, the prepared AC had a well-developed porous surface which was considered as channels to microporous network (Fig. 7(b)).

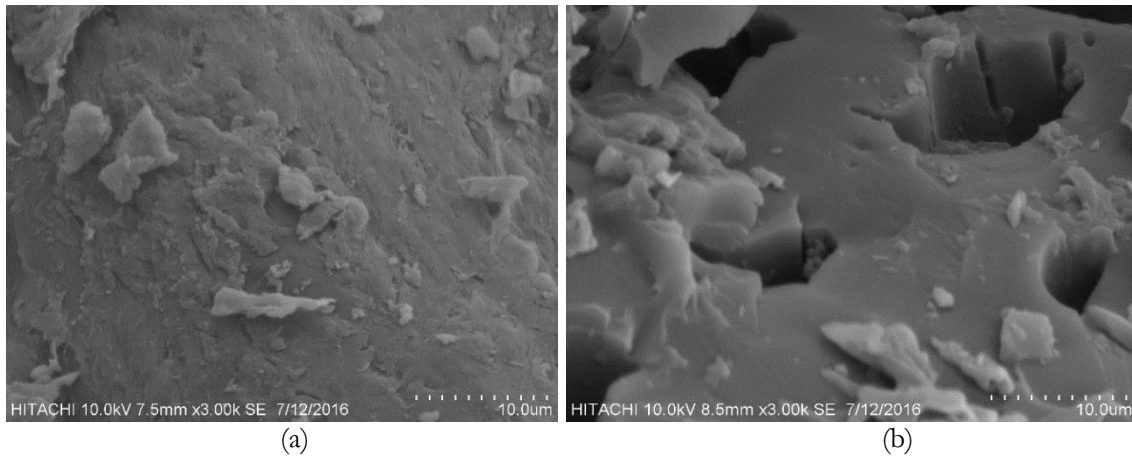
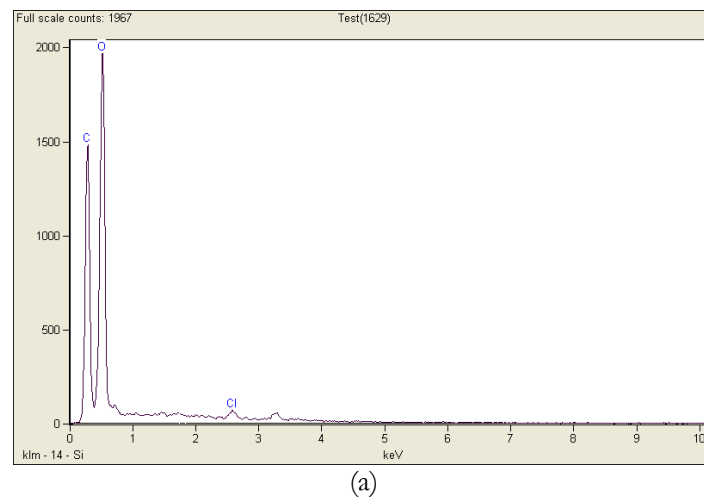
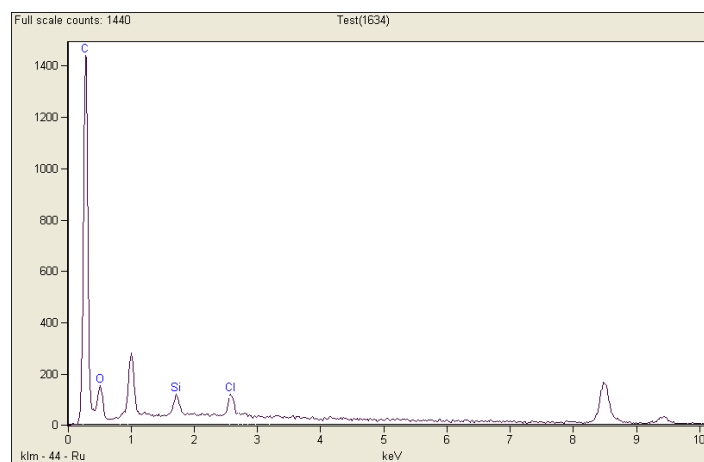


Fig. 8. (a) SEM image of raw material (b) AC activated with ZnCl_2 at 1.5/1 impregnation ratio.

The EDX analysis of the prepared AC and the raw material SPB is shown in Fig. 8. The raw material's essential elements, which were O, C, and Cl with weight percentage of 62.35, 37.5, and 0.15% respectively, as shown in Fig. 9(a). Figure 9(b) shows the data for the prepared AC. It reveals that due to carbonisation process, the weight percentage of C reached 97.30%. Furthermore, the weight percentage of O was at 0.955% along with little amounts of silica (Si) and Cl at 0.83 and 0.93%, respectively.



(a)



(b)

Fig. 9. (a) EDX analysis of raw material (b) EDX analysis of AC activated with ZnCl_2 at 1.5/1 impregnation ratio.

3.6. Comparison of Porous Characteristics of Various ACs Prepared from Different Precursors by Activating with ZnCl₂

Table 3 presents the porous characteristics of various ACs prepared from different raw materials by activating with ZnCl₂. The prepared AC from SPB by activation with ZnCl₂ in this study showed maximum surface area more than the previous studies which used the different precursors in preparation of AC by activation with ZnCl₂.

Table 3. Comparison of porous characteristics of prepared AC from different precursors by activation with ZnCl₂.

Precursor	Surface area (m ² /g)	References
Apricot stones	728	[51]
Tamarind wood	1322	[52]
Hazelnut bagasse	1489	[53]
Pistachio nut shell	1635	[28]
Rice husk	750	[54]
Enteromorpha prolifera	1416	[55]
Bael fruit shell	1488	[38]
Sago palm bark	1737	Present study

4. Conclusions

SPB's high cellulose percentage (44.13%) makes it a promising precursor for AC development, and is influenced by the impregnation ratio. When the AC was prepared through physicochemical activation with zinc chloride at an activation temperature of 700°C for 1 hour, a precursor with high surface area (1737 m²/g), pore volume (0.632 cm³/g), micropore surface area (1148.58 m²/g) and micropore volume (0.335 cm³/g) were obtained. Highly porosity characterization was obtained under an activation ratio of 1.5/1 weight of chemical activated agent to weight of precursor. The resulting prepared AC can have various applications in water and wastewater treatments, such as polishing the colour and removing multi-pollutants (e.g. heavy metals). It is a cheaper and more sustainable technique that allows for the utilisation of raw materials in the production of a useful substance.

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