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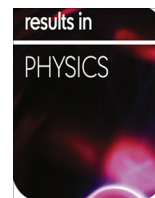
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Preparation and characterization of high surface area activated carbon from Fox nut (*Euryale ferox*) shell by chemical activation with H_3PO_4



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ABSTRACT

Activated carbons were prepared from Fox nutshell by chemical activation with H_3PO_4 in N_2 atmosphere and their characteristics were studied. The effects of activation temperature and impregnation ratio were examined. N_2 adsorption isotherms characterized the surface area, total pore volume, micropore volume and pore size distribution of activated carbons. Activated carbon was produced at 700°C with a 1.5 impregnation ratio and one hour of activation time has found $2636\text{ m}^2/\text{g}$ and $1.53\text{ cm}^3/\text{g}$ of highest BET surface area and total pore volume, respectively. The result of Fourier-infrared spectroscopy analysis of the prepared activated carbon confirmed that the carbon has abundant functional groups on the surface. Field emission scanning electron micrographs of the prepared activated carbon showed that a porous structure formed during activation.

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Introduction

Activated carbon is a porous carbonaceous material with the highly developed surface area and rich surface groups, are widely used in numerous application, including adsorption, catalyst support, gas separation and storage, solvent recovery and decolorizing, super capacitors, and as electrodes [1–4]. The internal porosity and their related properties such as surface area, pore volume, pore size distribution, and the presence of functional groups on pore surfaces play a significant role in the adsorptive capacities of activated carbons [4,5]. Activated carbons with abundance of micropores are extensively used for adsorption of small molecule pollutants, and highly developed mesoporous activated carbons are used for the adsorption of larger molecules such as dyes [6].

The factors such as the nature of raw materials, the activation method, the activating agent and the conditions of activation process affect the textural and chemical characteristics of the activated carbons [3]. In recent years, activated carbon is being produced from a large number of easily available and low-cost materials such as agricultural products like paulownia wood [3], vine shoots [4], fire wood [5], marigold straw [7], candlenut shell [8], corncob [9], coconut shells [10], reedy grass leaves [11], lotus stalks [12], and buriti shells [13]. Activated carbons are usually

produced by two methods: physical and chemical activation. Physical activation includes carbonization of the precursor in an inert atmosphere and activation of the resulting char by an activation agent such as steam, CO_2 or air [14]. The carbonization step consists of a pyrolysis procedure, in which, volatile compounds are released due to several complex, rival, and consecutive reactions leading to the achievement of fixed carbon in an inert atmosphere and at temperature range of $400\text{--}500^\circ\text{C}$. In the second step of the physical activation process, the precursor is placed under a heat treatment at high temperatures from 700 to 1100°C in the presence of oxidizing agents, such as CO_2 , water steam, air, or a mixture of them [13].

In chemical activation, the precursor is mixed with a certain amount of active agents such as KOH [2] and [5], NaOH [10], H_3PO_4 [3,4,11,12], and ZnCl_2 [13,15], which lead to the development of porous structures in the material. Among the various dehydrating agents used for chemical activation, phosphoric acid is preferred recently due to environmental and economic concerns [3]. When compared to zinc chloride, phosphoric acid is the most preferred because of the environmental disadvantages like the problem of corrosion and inefficient chemical recovery associated with zinc chloride. Moreover, the carbons obtained by using zinc chloride as an activating agent cannot be used in pharmaceutical and food industries as they may contaminate the product [8]. Phosphoric acid allows the developing of both micropores and mesopores of the resulting activated carbon [3]. In the chemical activation method, the process is carried out at lower temperatures

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than physical activation process. The activating agents act as dehydrating agents that inhibit the formation of tar as well as volatile substances during the process, which helps to enhance the yield of porous carbon and to decrease the activation temperature and activation time compared with the physical activation method [13,15,16].

In the present study, Fox nutshell has been used as a precursor material for activated carbon preparation. Fox nut (*Euryale ferox*) is a tropical and subtropical climate plant commonly known as Makhana, Black Diamond or Gorgon Nut, which belongs to water lily family (Nymphaeaceae). In turn, the shells of Fox nut become a residue, which has no better commercial value, only used by people to get some fuel value by burning [15]. Fox nutshell is readily available new precursor material for the preparation of activated carbon as it contains low ash (5%) and high volatile matter (70.1%). Thus, activated carbon can suitably be prepared from the Fox nutshell that will lead to its useful utilization. To the best of our knowledge, no study is reported on the preparation of activated carbon from Fox nutshell by chemical activation method with H_3PO_4 . Therefore, the primary objective of the present work was to prepare high surface area microporous activated carbon from Fox nutshell using H_3PO_4 as the chemical activation reagent.

Experimental

Material

Fox nutshell was collected from a local Fox nut processing unit from Madhubani district of Bihar state in East India. The Fox nutshell was soaked in a 0.5 N NaOH solution and left for 12 h to remove all impurities like mud and ash present in the Fox nutshell during harvesting and processing [15]. The Fox nutshell was properly washed with distilled water until the washed solution pH was achieved about 7. The cleaned material was dried at 110 °C for 24 h for further experimentation. The above method used is suitable for removing ash as well as water-soluble substances [15,17]. The proximate and ultimate analyses of Fox nutshell and NaOH treated Fox nutshell, and chemical component analysis of Fox nutshell are shown in Table 1. After NaOH treatment of the Fox nutshell, the ash content (4.62%) decreased while the volatile content (71.70%) increased [15]. A similar result has been reported by Yang et al., [18]. The dried Fox nutshell was crushed and sieved to obtain a particle size of range 1.4–2.0 mm. Analytical reagents grade Merck chemicals were used in this study.

Preparation of activated carbon

Preparation of activated carbon from Fox nutshell was carried out by chemical activation process with the H_3PO_4 activating agent. The initial concentration and density of the H_3PO_4 solution were 88% (w/v) and 1.75 g/ml, respectively. The impregnation ratio was determined as the ratio of the weight of H_3PO_4 to the weight of the dried Fox nutshell [15]. The Fox nutshell was impregnated with an H_3PO_4 solution in 0.5 to 2.0 ratios with an increment of 0.5. The required H_3PO_4 volumes per 20 g dry raw material were 6.5, 12.99, 19.49 and 25.98 ml for the impregnation ratios of 0.5, 1, 1.5 and 2, respectively. For impregnation, 20 g of Fox nutshell was dissolved in 150 ml solution of H_3PO_4 with different impregnation ratios and stirred at approximately 80 °C. The mixture was left for 24 h at room temperature, during this period the mixture was mixed at a fixed time interval of 6 h. After that, the impregnated sample was evaporated to dryness at 110 °C for 24 h in a hot oven. This method is in practice for preparation of the activated carbons [15]. A weighed amount of impregnated Fox nutshell was placed in a ceramic crucible and inserted at the middle of the horizontal electric tubular furnace tube. The sample was heated from room

Table 1

Proximate and ultimate analyses of raw material and activated carbon (prepared at impregnation ratio of 1.5 and carbonization temperature of 700 °C).

Analysis	Raw material (wt%)	NaOH treated raw material (wt%)	Activated carbon (wt%)	ASTM test standard
<i>Proximate</i>				
Moisture	4.0	3.71	3.12	D1762–84
Volatile Matter	70.1	71.70	21.05	D 5832–98
Ash	5.0	4.62	2.12	D 2866–11
Fixed carbon ^a	20.9	19.97	73.71	
<i>Ultimate</i>				
Carbon	42.30	44.27	64.85	
Hydrogen	4.30	4.32	4.52	
Nitrogen	0.82	0.24	1.24	
Sulfur	0.07	0.08	0.15	
Oxygen ^a	52.51	51.09	29.24	

^a By difference.

temperature to a final preselected temperature ranging from 600 to 800 °C. The final carbonization temperature was achieved with a heating rate of 5 °C/min and 60 min activation time with the N_2 flow of 150 mL/min. After completion of the process, the produced carbonization material was soaked with 0.5 N hydrochloric acid solutions for 24 h [15]. The soaked carbonized material was washed several times with hot double distilled water and finally with cold double-distilled water to the pH of the wash water was neutral. The wet prepared material was dried in an oven at 110 °C for 24 h and stored in plastic containers.

Characterization of the prepared activated carbon

Elemental (CHNS) analysis of prepared activated carbons was performed with an elemental analyzer (Elementar, Germany). The specific surface area and pore structure characteristic of the prepared activated carbons were determined by nitrogen adsorption at −196 °C by the surface area analyzer (Quantachrome, USA). The sample was degassed under vacuum at 300 °C for three hours before the measurement. The surface area (S_{BET}) of prepared activated carbon was estimated by BET (Brunauer–Emmett–Teller) method. The Dubinin–Radushkevich (D–R) method was used to calculate the micropore surface area (S_u). The mesopore surface area (S_m) was calculated by subtracting S_m from S_{BET} ($S_m = S_{BET} - S_u$) [15,17]. The total pore volume (V_T) was estimated to be the liquid volumes of N_2 at relative high pressure ($P/P_0 \sim 0.99$). The micropore volume (V_u) was obtained using the DR method, and mesopore volume (V_m) calculated by subtracting V_u from V_T ($V_m = V_T - V_u$). The pore size distribution of the prepared activated carbon was determined by BJH (Barrett–Joyner–Halenda) model and the mean pore diameter (D_p) was calculated from $D_p = 4V_T/S_{BET}$ [15,19]. The surface functional groups of the prepared activated carbon were studied by Fourier transform infrared spectroscopy (Thermo Scientific Nicolet™ iS10), recorded from a wavenumber of 400–4000 cm^{-1} . The pellet was prepared by mixing a sample (0.1 mg each) with KBr (100 mg) in a mortar pestle and resultant mixtures pressed in the hydraulic pump. The surface morphologies of the raw material and prepared activated carbon were carried out by Field emission scanning electron microscopy (Nova NanoSEM 450).

Results and discussion

Proximate, yield and ultimate analysis of activated carbon

The proximate analysis was performed according to the ASTM methods, are mentioned in Table 1. Proximate analyses of the

materials are in terms of moisture content, ash content, volatile matter and fixed carbon. The fixed carbon content of the materials was calculated by mass difference. From Table 1, the proximate composition of the raw material was 4 wt% moisture, 5 wt% ash, 70.1 wt% volatile matter, and 20.9 wt% fixed carbon. Low ash and high carbon content, which makes it suitable to be used as activated carbon precursor [8]. After NaOH treatment (base leaching), the ash content decreased while the volatile content increased of the precursor material (Table 1). The results are consistent with the previous study carried out by Yang [18]. The proximate analysis of activated carbon prepared at 700 °C carbonization temperature and a 1.5 impregnation ratio is shown in Table 1. From Table 1, the proximate composition of the prepared activated carbon was 3.12 wt% moisture, 2.12 wt% ash, 21.05 wt% volatile matter, and 73.71 wt% fixed carbon.

Fig. 1 displays the yield of the prepared activated carbons at different activation temperatures and impregnation ratios. The yield of activated carbon was calculated by dividing the mass of the resultant activated carbon by the initial mass of the raw material used for activation [15]. From Fig. 1, it is observed that the activated carbon yield was in the range of 41.13–24.31%. The yield is comparable to that obtained in other studies [3,8,20], 33.18–25.16% from paulownia wood, 42.15–56.25% from jackfruit peel, 37.2–42.3% from chestnut wood, 31.9–48.5% from fruit stones.

Fox nutshell is a complex composite material formed of natural polymers (cellulose, lignin, and hemicellulose). During activation or carbonization at high temperatures, the polymeric structures of the biomass decompose and liberate most of the non-carbon elements, mainly hydrogen, oxygen and nitrogen in the form of liquid (called as tars) and gasses, leaving behind a rigid carbon skeleton in the form of aromatic sheets and strips [8]. The yield of activated carbon is increased due to the presence of phosphoric acid during activation promotes depolymerization, dehydration, and redistribution of constituent biopolymers, and also favoring the conversion of aliphatic to aromatic compounds [8].

Fig. 1 shows the effect of activation temperature and impregnation ratio on the yield of activated carbon. Activation with a low H_3PO_4 impregnation ratio of 1:1 resulted in a higher yield range of 41.13–37.84%. At each carbonization temperature, yields of the activated carbon decreased with increasing impregnation ratio above 1:1 ratio. During the activation process, the phosphoric acid reacted with the char and volatile matter and diffused quickly out of the surfaces of particles. Therefore, with a high impregnation

ratio, the gasification of surface carbon atoms became primary, leading to an increase in the weight loss and a low carbon yield during activation process [3]. Similarly activated carbon yields are previously reported [3,8].

The results of the elemental analysis of the activated carbons at various carbonization temperatures and impregnation ratios are tabulated in Table 2. An increase in carbon content of activated carbons from 63.89 to 64.25 wt% at 1.5 impregnation ratio with an increase in activation temperature from 600 to 700 °C has been observed; with further increase in temperature to 800 °C the carbon content decreases to a value of 55.60 wt%. The hydrogen and oxygen content is found to decrease from 4.74 to 4.52 wt%, and 29.53 to 29.24 wt% respectively. The increase in carbon content with increase in activation temperature from 600 to 700 °C may be due to an increasing degree of aromaticity [21]. The effects of impregnation ratios on the elemental composition of activated carbons studied at 700 °C activation temperature are tabulated in Table 2. With increasing impregnation ratio, the carbon content of activated carbons is increased, and hydrogen, nitrogen, and oxygen contents are decreased. Variation of the carbon content of the activated carbons with temperature shows a good agreement with the results reported by Fierro et al., and Angin [21,22].

BET surface areas and distribution of pore sizes of prepared activated carbons

N_2 adsorption–desorption isotherms

The nitrogen adsorption–desorption isotherms of the activated carbons prepared at various carbonization temperatures and impregnation ratios are shown in Figs. 2 and 3. The shape of N_2 adsorption–desorption isotherms are different at different carbonization temperatures and impregnation ratios. These isotherms belong to a mixed type (i.e. isotherms I and IV) of IUPAC classification [23]. A type I isotherm is associated with microporous structures and a type IV isotherm indicates a mixture of microporous and mesoporous materials. The initial part of the isotherms is of type I with significant uptake at low relative pressures, which corresponds to adsorption in micropores. At intermediate and high relative pressures, the isotherms are of type IV with a hysteresis loop of type H4 associated with monolayer–multilayer adsorption followed by capillary condensation in narrow slit-like pores [9]. Nitrogen uptake increases with increasing activation temperature from 600 to 700 °C and impregnation ratio from 0.5 to 1.5 indicating the gradual development of the porous structure. Further increase in activation temperature from 700 °C and impregnation ratio from 1.5 caused decreasing adsorption of nitrogen which is connected with the deterioration of porous structure (Figs. 2 and 3). The deterioration of porous structure is most probably due to the destruction of the wall between adjacent micropores by reaction with phosphoric acid [9]. With the increase in impregnation ratio, it is found that the hysteresis loop gradually widens indicating that the activated carbons get populated with mesopores (pores are enlarged) with a rise in impregnation ratio. For all impregnation ratios a plateau is not apparently reached rather adsorption occurs over the entire pressure interval indicating the presence of wide range of pore diameters (Fig. 3). Thus, in all the prepared activated carbons there is simultaneous presence of micro and mesopores. Similar results are reported by many investigators [3,8,9,15].

Effect of carbonization temperature on surface area and pore volume

The carbonization temperature accelerates thermal degradation and the volatilization process of the impregnated sample. This process leads to increases in the surface area and pore development [3]. The surface areas and pore volumes were determined for the prepared activated carbons at different activation temperatures

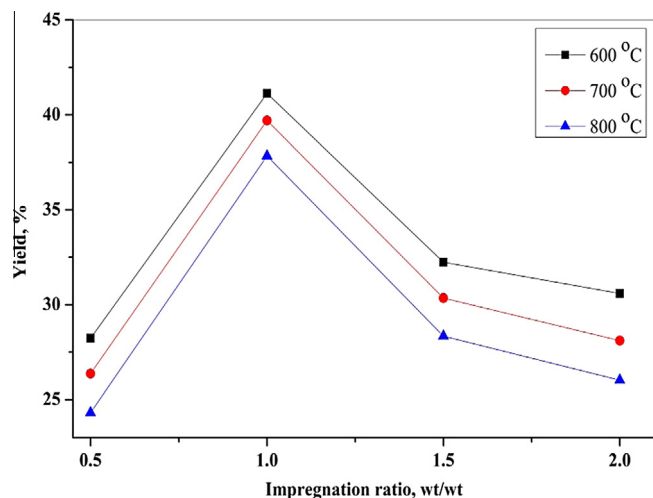
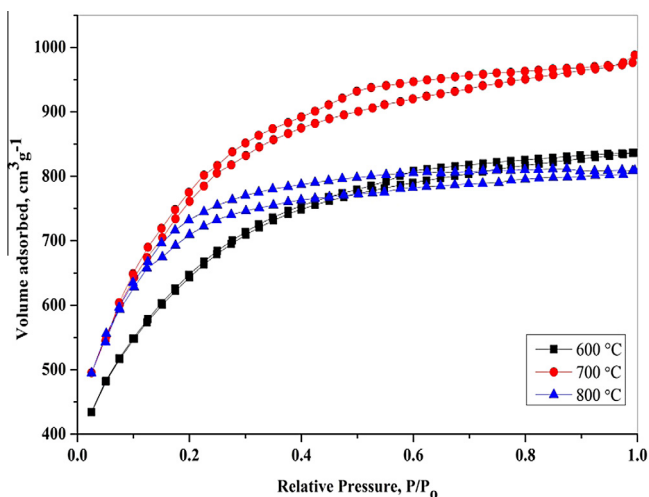
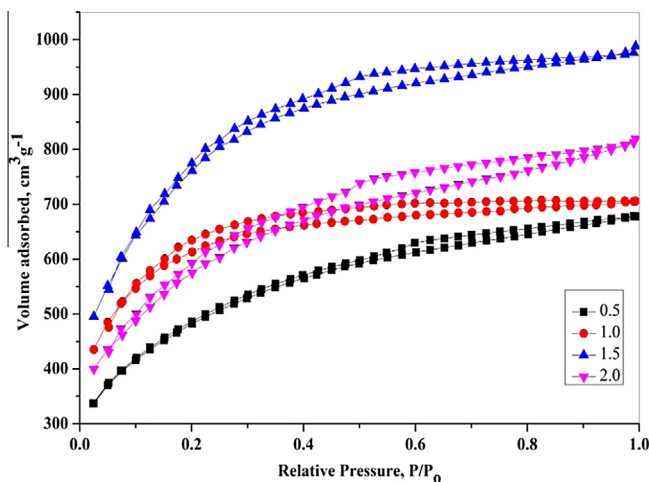


Fig. 1. Effects of carbonization temperature and impregnation ratio on the yield of activated carbon.

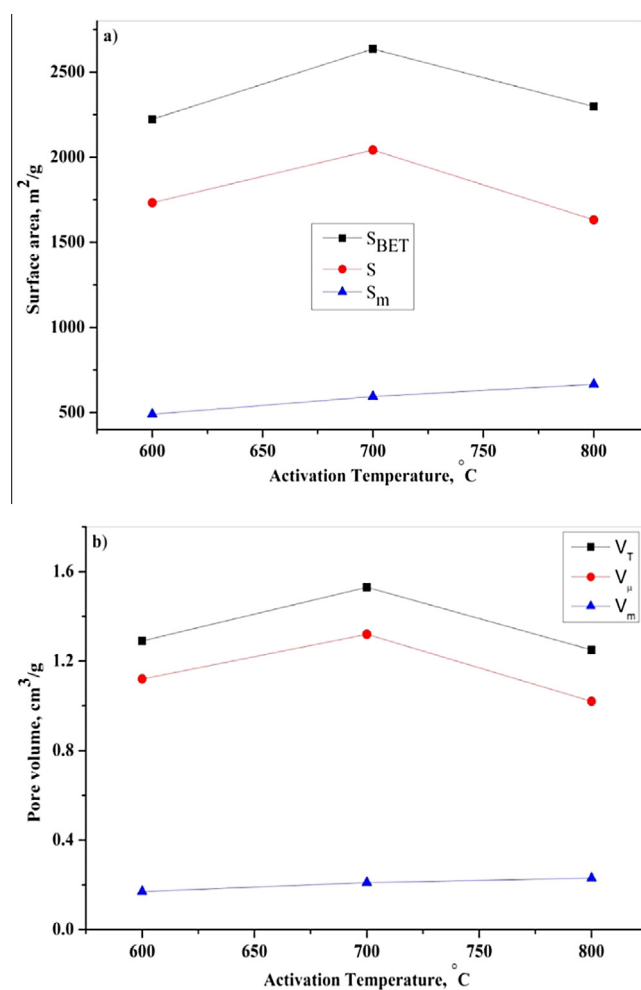
Table 2

Ultimate analysis of prepared activated carbons.

Prepared ACs	Ultimate analysis (wt%)				
	C	H	N	S	O (by difference)
H ₃ PO ₄ impregnation ratio: 3:2 (wt/wt)					
Carbonization temperature (°C)					
600	63.89	4.74	1.68	0.16	29.53
700	64.85	4.52	1.24	0.15	29.24
800	55.60	4.68	1.45	0.13	38.14
Activation temperature: 700 °C					
Impregnation ratio (wt/wt)					
1:2	51.29	4.80	1.42	0.09	42.40
1:1	61.28	4.59	1.30	0.13	32.70
3:2	64.85	4.52	1.24	0.15	29.84
2:1	68.75	4.29	1.16	0.15	25.65

**Fig. 2.** Nitrogen adsorption-desorption isotherms for prepared activated carbons with variable activation temperature (impregnation ratio = 1.5).**Fig. 3.** Nitrogen adsorption-desorption isotherms for prepared activated carbons with variable impregnation ratio (activation temperature = 700 °C).

(600–800 °C), and impregnation ratios (0.5–2.0) are shown in Fig. 4 (a) and (b). The BET surface area and the total pore volumes of the prepared activated carbons are increased by 2223–2636 m²/g and 1.29 to 1.53 cm³/g, respectively, from 600 to 700 °C of the activation temperature. The rise in carbonization temperature from 700 °C to 800 °C may have induced shrinkage in the carbon

**Fig. 4.** Effect of activation temperature on surface areas (a) and pore volumes (b) of activated carbons produced at impregnation ratio of 1.5.

structure, resulting in a reduction in surface area and a pore volume by 2636–2298 m²/g and 1.53–1.25 cm³/g, respectively. A similar trend is also reported by various researchers while showing the influence of activation temperature on the BET surface area and total pore volume of activated carbon obtained through H₃PO₄ activation of other lignocellulosic precursors [3,8,21,25].

Table 3 and Fig. 4 show the micro-mesopore surface area and volume of the activated carbons prepared at different carbonization temperatures. The micropore surface area of the prepared activated carbon rapidly increased at activation temperatures of

Table 3

Summary of BET surface area, total pore volume, micropore volume of prepared activated carbons.

ACs	S_{BET} (m^2/g)	S_{μ} (m^2/g)	S_{m} (m^2/g)	V_{T} (cm^3/g)	V_{μ} (cm^3/g)	V_{m} (cm^3/g)	V_{μ}/V_{T} (%)	S_{μ}/S_{BET} (%)	D_{p} (nm)
H ₃ PO ₄ impregnation ratio: 3:2 (wt/wt)									
Activation temperature (°C)									
600	2223	1733	490	1.29	1.22	0.07	94.57	77.95	2.32
700	2636	2042	594	1.53	1.32	0.21	86.27	77.46	2.32
800	2298	1632	666	1.25	1.02	0.23	81.60	71.02	3.06
Activation temperature: 700 °C									
Impregnation ratio (wt/wt)									
1:2	1622	1193	429	1.05	0.97	0.08	92.38	73.55	2.60
1:1	1989	1362	627	1.09	0.95	0.14	87.15	68.47	2.19
3:2	2636	2042	594	1.53	1.32	0.21	86.27	77.46	2.32
2:1	1943	1496	447	1.27	1.00	0.27	78.74	76.99	2.61

S_{BET} : BET surface area, S_{μ} : micropore surface area, S_{m} : mesopore surface area, V_{T} : total pore volume, V_{μ} : micropore volume, V_{m} : mesopore volume, $V_{\mu}\% = (V_{\mu}/V_{\text{T}}) \times 100$, $S_{\mu}\% = (S_{\mu}/S_{\text{BET}}) \times 100$, D_{p} : average diameter.

600–700 °C and decreased at activation temperatures of 700–800 °C. At 700 °C carbonization temperature and 1.5 impregnation ratio of prepared activated carbon, the S_{μ} and S_{m} were found to be 2042, and 594 m^2/g , respectively. The micropore volume (V_{μ}), and mesopore volume (V_{m}) were found to be 1.32 and 0.21 cm^3/g , respectively. The microporosity percentages of the prepared activated carbons at the studied temperatures show a decrease in microporosity with increasing carbonization temperatures (Table 3). The microporosity decreased from 94.54% to 81.6%, as the temperature rose from 600 to 800 °C, respectively. The prepared activated carbon is mainly microporous, but it also contains mesopores that are crucial in facilitating the access of the adsorbate molecules to the interior of the carbon particles [3]. The prepared activated carbons at all carbonization temperatures (600–800 °C) show both micro and mesoporous. Similar results are reported by other researchers [3,8,9,15], i.e. presence of both micro and mesopores in all activated carbons prepared at higher carbonization temperatures.

Effect of impregnation ratio on surface area and pore volume

The Fox nutshell char prepared at the temperatures of 600, 700 and 800 °C with NaOH soaking and without H₃PO₄ activation have surface areas of 203, 452 and 186 m^2/g , respectively [15]. The surface area of the Fox nutshell char prepared at 700 °C temperature with 0.5 N NaOH solution of the raw material was higher (452 m^2/g) than the Fox nutshell char (326 m^2/g) obtained without 0.5 N NaOH solution soaking [15]. The NaOH soaking is also important step during the preparation of high surface area activated carbon [15]. The NaOH soaking is a significant step during the preparation of high surface area activated carbon [15,18]. When the biomass are treated with a low concentration of NaOH, the ester bonds between lignin and carbohydrate are broken due to saponification, causing the destruction of lignin structure and increasing the porosity of material, which can help the activating agent enter the interior of the raw material and increase the contact area between the activating agent and the carbon precursor. Furthermore, the hemicellulose can partially be dissolved in alkali solution and washed away in the base-leaching process, which may also result in a more porous structure of the raw material, helping the activating agent enter the interior of the raw material [18]. Similar achievements of the higher surface area are reported by Kumar and Jena [15], and Yang et al. [18], for the pre-treatment of raw materials with NaOH solutions. The obtained result is in the same trend as Kumar and Jena, and Yang et al., prepared activated carbon from Fox nutshell and herb residues, respectively with ZnCl₂ activation [15,18].

The impregnation ratio is the larger variable for the development of porosity in activated carbon [3]. H₃PO₄ has two important functions: it promotes the pyrolytic decomposition of the original

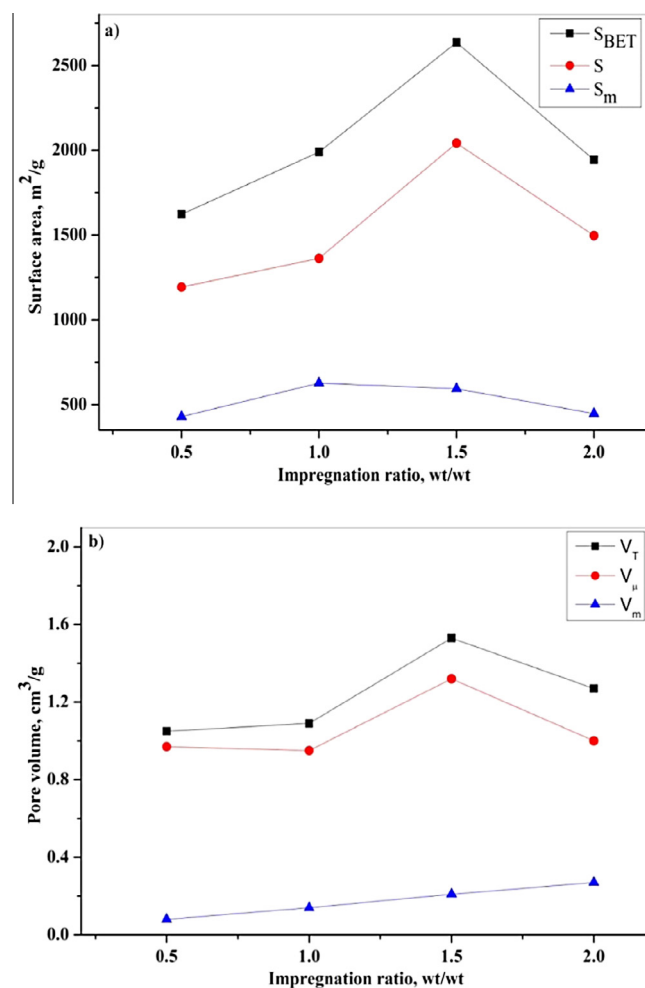


Fig. 5. Effect of impregnation ratio on surface areas (a) and pore volumes (b) of activated carbons produced at 700 °C activation temperature.

material and the formation of the cross-linked structure [26]. However, H₃PO₄ allows the development of both micropores and mesopores of the prepared activated carbon. In chemical activation with H₃PO₄, the effect of different impregnation ratios on the surface area and pore volume of the prepared activated carbons were investigated, and the results are presented in Fig. 5(a) and (b), and Table 3. From Fig. 5 and Table 3, the BET surface areas and the total pore volumes of the activated carbon is found to be increased rapidly with increasing the impregnation ratio from 0.5

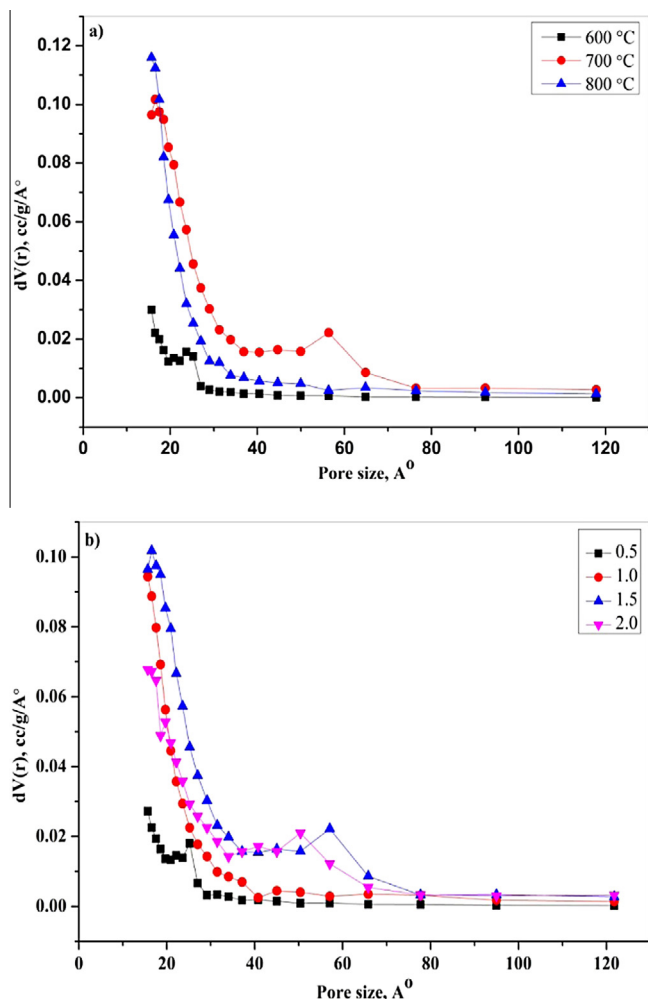


Fig. 6. Pore diameter distribution of activated carbons prepared at (a) different activation temperatures (impregnation ratio = 1.5) and (b) different impregnation ratios (activation temperature = 700 °C).

to 1.5 by 1622–2636 m²/g and 1.05–1.53 cm³/g, respectively. Effect of increasing the impregnation ratio on the BET surface area and total pore volume is clearly stronger at 700 °C. At this carbonization temperature with 1.5 of impregnation ratio, the maximum surface area, and total pore volume were obtained as 2636 m²/g and 1.53 cm³/g, respectively. From Fig. 5, it was observed that the impregnation ratio has a significant effect on the S_{BET} , S_{p} , and V_{p} of the activated carbon. From Table 3, it is decreased that the percentages of the microporosity varied from 92.38% to 78.74%. Similar trends have also been obtained for the influence of impregnation ratio on the surface area and pore volume of the prepared activated carbon with H₃PO₄ activation of other lignocellulosic materials [3,8,19,21,26].

Pore size distribution of prepared activated carbons

In the present work, the pore size distribution curves of the prepared activated carbons were obtained by the BJH method using the pore volumes in the measurement of N₂ desorption isotherms. The prepared activated carbons have a narrow pore size distribution with a significant range less than 20 Å as shown in Fig. 6(a) and (b), which is indicative of micropores present in the prepared activated carbons. At 700 °C of the carbonization temperature, as the impregnation ratio was increased from 0.5 to 2.0, the average pore diameter (D_p) increases from 2.19 to 2.61 nm.

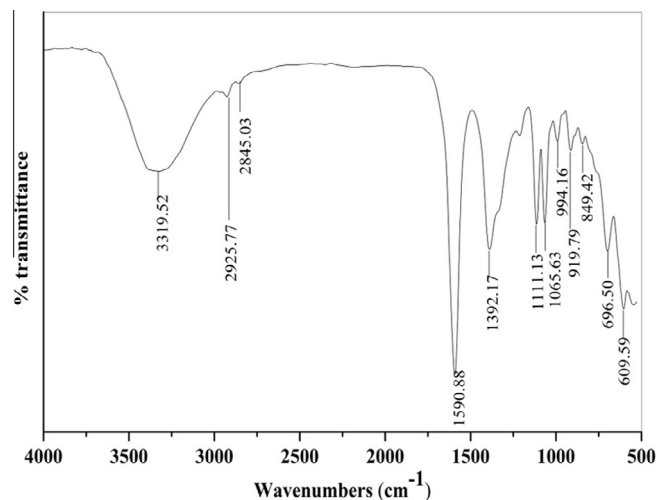


Fig. 7. FTIR spectra of prepared activated carbon at 700 °C of activation temperature and 1.5 impregnation ratio.

FTIR analysis of the prepared activated carbon

The carbon matrix does not consist of carbon atoms alone but is also formed by other heteroatoms like hydrogen, oxygen, nitrogen, halogen, sulfur, phosphorus, etc. The surface chemistry of the activated carbon governs by the heteroatoms that are bonded to the edges of the carbon layers [25]. The FTIR spectra of the prepared activated carbon of maximum surface area are shown in Fig. 7. The figure shows a broad band at 3319.52 cm⁻¹, which is assigned to O–H stretching of hydroxyl groups from carboxyls, phenols or alcohols and adsorbed water [16,24]. The band at 2925.77 cm⁻¹ and 2845.03 cm⁻¹ is assigned to the asymmetric C–H representing such as methyl and methylene groups [16]. A high band at 1590.88 cm⁻¹ can be allocated to C=C aromatic ring stretching vibration enhanced by polar functional groups [8]. The bands in the region between 1300 and 900 cm⁻¹ is assigned to C–O stretching in acids, alcohols, phenols, ethers, and esters, usually found in oxidized carbons [24]. However, it was also a characteristic of phosphorous and phosphorous carbonaceous compounds present in the phosphoric acid activated carbon in the band region between 900 and 1300 cm⁻¹ [24]. The bands at 1300–900 cm⁻¹ are tentatively assigned to the following phosphorous species: hydrogen-bonded P=O, O–C stretching vibrations in P–O–C of aromatics and P=OOH [24,26]. The bands at 696.50 and 850 cm⁻¹ may be assigned to aromatics substituted by aliphatic groups [27]. The band at 609 cm⁻¹ is assigned to O–H out-of-plane bending vibrations [16].

Surface morphologies of the Fox nutshell and prepared activated carbon

Field emission scanning electron micrographs (FESEM) technique was used to investigate the surface morphology of the Fox nutshell and prepared activated carbons. Fig. 8 illustrates the FESEM photographs of Fox nutshell and prepared activated carbon at 700 °C activation temperature and 1.5 impregnation ratio. It represents significant differences between the external surface topographies. Fig. 8(a) shows the dense structure, smooth and no pores were present on the Fox nutshell. The prepared activated carbon has well-developed pores during activation with H₃PO₄ during carbonization of the Fox nutshell at 700 °C (see Fig. 8(b and c)). Fig. 8(b) and (c) are the images at different magnifications of the same activated carbon. From Fig. 8(b and c), the external

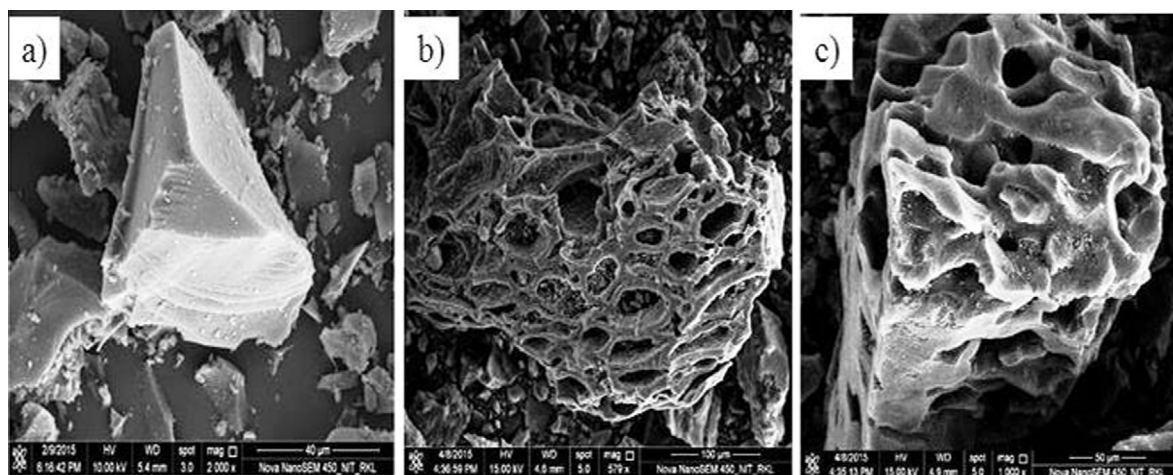


Fig. 8. Field Emission Scanning Electron micrographs of samples: (a) Fox nutshell, (b) and (c) prepared activated carbon at 700 °C of activation temperature and 1.5 impregnation ratio.

surfaces of the activated carbons have the pores which are of different sizes and different shapes. Due to well-developed pores, the activated carbon possessed high BET surface area. The pore development on the surfaces of the activated carbon resulted from the evaporation of the activating agent of H_3PO_4 during carbonization, leaving space previously occupied by the activating agent [8,28].

Conclusions

In the present study, Activated carbon with a well-developed pore structure was prepared from Fox nutshell by chemical activation with phosphoric acid. Activation of Fox nutshell impregnated with the phosphoric acid result in reasonable carbon yield and produced microporous AC with high surface area. Microporosity of the prepared activated carbon is found to decrease with increasing the activation temperature from 600 to 800 °C due to the increment of the mesoporosity. BET surface area and total pore volume are found to decrease with increasing activation temperatures higher than 700 °C due to the shrinkage and the oxidation of the material. The phosphoric acid to Fox nutshell ratio strongly affects the pore structure. Low impregnation ratio, i.e., 0.5, promote the creation of micropores. Impregnation ratios higher than 1.5, the surface area, and the total pore volume decreases due to the attack of the polymeric matrix by the excess of phosphoric acid. The optimum activation temperature and impregnation ratio for the high surface area AC was found to be 700 °C and 1.5, respectively. The prepared activated carbon can be effectively used as an adsorbent for removing hazardous compounds from industrial waste gasses and wastewaters.

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