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SYNTHESIS OF ACTIVATED CARBON FROM *COCOS NUCIFERA* LEAVES AGROWASTE BY CHEMICAL ACTIVATION METHOD

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Abstract. Activated carbon from *Cocos nucifera* leaves agrowaste was derived. The effect of impregnation ratio was studied by chemical activation method using phosphoric acid as an activating agent. Activated carbon was produced at the activation temperature of 673 K by slow pyrolysis. Nitrogen adsorption isotherms study was performed. Effect of impregnation ratio on the yield, methylene blue number, iodine number, and acid adsorption was studied. The FT-IR spectra show the presence of activated carbon. The TGA investigation reveals that activated carbon is thermally stable at 723 K. The SEM images show the incorporation of activated carbon particles which lead to the systematic change in the morphology of activated carbon. Surface chemistry study predicts the acidic and basic functional groups of *Cocos nucifera* leaves activated carbon.

Keywords: *Cocos nucifera* leaves, activated carbon, phosphoric acid, pyrolysis.

1. Introduction

Activated carbon in its broadest sense includes a wide range of processed amorphous carbon-based materials. It is not truly an amorphous material but has a microcrystalline structure. Activated carbon has a highly developed porosity and an extended interparticulate surface area. Their preparation involves two main steps: the carbonization of the carbonaceous raw material at temperatures below 1073 K in an inert atmosphere and the activation of the carbonized product. Thus, all carbonaceous materials can be converted into activated carbon, although the properties of the final product will be different, depending on the nature of the raw material used, the nature of the activating agent, and the conditions of the carbonization and activation processes.

Cocos nucifera leaves are not a consumable part and are usually discarded as wastes. *Cocos nucifera* leaves have become economically important species since their demand domestically and for export has increased tremendously. To make better use of cheap and abundant agricultural waste, it is proposed to convert *Cocos nucifera* leaves waste into activated carbon. This conversion will solve problems of unwanted agricultural wastes being converted into useful, value-added adsorbent and also the use of agricultural by- products which represent potential source of adsorbents. This will solve problems of waste management. However, not many studies have been reported on converting *Cocos nucifera* leaves into activated carbon.

A challenge in activated carbon production is to produce very specific carbons which are suitable for certain application. The most important property of the activated carbon is its adsorption capacity which is highly influenced by the preparation conditions.

The good quality activated carbon should have low ash content as possible. S. Faust [1] suggests that typical values of ash content should be in the range of 5–7 % and carbon content – about 85–90 %. As the carbon content of the activated carbon increases, the surface area also increases. High carbon content value is desired to achieve high surface area.

Activated charcoal produced from residues would reduce the pressure on forests since wood is also commonly used for this purpose [2]. Many agricultural by-products such as coconut shell [3, 4], grain sorghum [5], coffee bean husks [6], rubber wood sawdust [7] and chestnut wood [8] have been discovered to be suitable precursors for activated carbon due to their high carbon and low ash contents. Agricultural wastes are considered to be a very important feedstock because of especially two facts: they are renewable sources and low cost materials.

In the present study, the optimal experimental conditions required to obtain adequate activated carbon with desirable properties in terms of carbon yield, BET surface area, iodine number surface area and ash content, methylene blue number, iodine number, which are critical in determining a good quality of activated carbon, are studied.

2. Experimental

2.1. Preparation of Activated Carbon

First preparation of activated carbon (AC) was done in three batches of 50, 100 and 300 g. Chemically pure H_3PO_4 (Merck) was used as an activating agent. A known mass of activated agent was mixed with distilled water, and biomass waste was impregnated in acidic solution. The amount of phosphoric acid solution used was adjusted to give a certain impregnation ratio (weight of activating agent/weight of raw material) of 1:1, 2:1, 3:1, and 4:1. The impregnated sample was kept for 24 h. After 24 h the residual water was removed and kept in the oven for 383 K. A weighed amount of impregnated samples was kept in a muffle furnace at 673 K. The muffle furnace was purged with high purity nitrogen gas to avoid oxidation. Nitrogen flow was adjusted to 3ml/K at 673 K. The activated carbon was subsequently removed from the furnace and cooled to room temperature.

After activation the samples 3M hydrochloric acid was used to remove the phosphoric acid compounds. The washed samples were dried at 383 K for 6 h in the oven and then grounded to form a porous carbon powder (Fig. 1). The equipment was fabricated to hold the raw sample of 30x9x9 cm size.
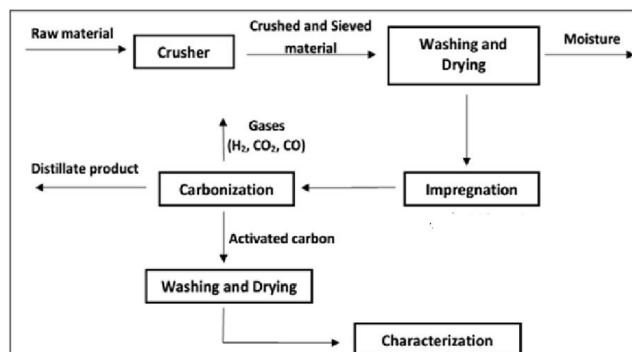

Fig. 1. Schematic diagram for preparation of activated carbon

2.2. Physical Characterization of *Cocos nucifera* Leaves Activated Carbon

The pore structures of the resulting carbons were analyzed using N_2 adsorption and scanning electron microscope (SEM).

SEM images were recorded using Joel JSM 6390LV field emission SEM. A thin layer of platinum was sputter-coated on the samples for charge dissipation during FESEM imaging. The sputter coater (Eiko IB-5 Sputter Coater) was operated in an argon atmosphere using the current of 6mA for 3 min. The coated samples were then transferred to the SEM specimen chamber and observed at an accelerating voltage of 5 kV, eight spot size, four aperture and 15 mm working distance.

2.3. Surface Chemistry Characterization of *Cocos nucifera* Leaves Activated Carbon

The surface chemistry characterization of the activated carbon was performed with pH drift and Fourier Transform Infrared Spectroscopy (FT-IR) to identify its surface functional groups both qualitatively and quantitatively.

2.3.1. pH Drift Method

This method was conducted to determine the pH_{PZC} of *Cocos nucifera* leaves activated carbon. pH_{PZC} (point of zero charge/zero charge point/zero point charge) is pH when the charge in the activated carbon surface is zero. The procedure of pH drift method can be described as follows: 50 ml of 0.01N NaCl were prepared and added into a series of Erlenmeyer. Then, their pH values were adjusted in the range 2–12 with the interval of 0.1 using 0.01N HCl solution and 0.01N NaOH. pH of initial solutions was measured with pH-meter and then noted as pH initial. After the constant value of pH initial was reached, 0.15 g of activated carbon sample was added into each Erlenmeyer and then shaken for 48 h. After 48 h pH of the solution was measured using pH meter and noted as pH final. pH_{PZC} of activated carbon sample is the point when pH initial = pH final.

Cocos nucifera leaves activated carbon shows low pH_{PZC} in the range of 2.5–3.0. These low pH_{PZC} values are consistent with Boehm titration results, which show the dominance of acidic group at the surface of the activated carbons.

2.3.2. Boehm Titration

The Boehm titration method can be described as follows: 0.5 g of activated carbon were placed into a series of flasks which contain 50 ml of 0.05N sodium bicarbonate, sodium carbonate, sodium hydroxide and hydrochloric acid. The flasks were sealed and shaken for 24 h. After 24 h the solutions were filtered, and then 10 ml of each solution were pipetted to a flask and titrated with 0.05N sodium hydroxide and/or hydrochloric acid, depending on the original solution used. The amount of

acidic groups on the activated carbon is calculated under the assumption that NaOH neutralizes carboxylic, lactonic and phenolic groups. The number of surface basic sites is calculated from the amount of HCl that reacted with the carbon. The reaction between the reagents and the acidic oxygenated functional groups on the surface is based on the difference in acid/base strength. The strength of acidic groups is as follows: carboxyl > lactone > phenol [9].

2.3.3. Infrared Spectroscopy

A quantitative analysis of activated carbon was conducted by obtaining FT-IR transmission spectra of carbon samples by KBr technique. The technique was conducted by placing the KBr powder grinded with an agate mortar in the sample cup and then the powder surface was evened using the attached sample pressing bar. Next, the powder was mounted to the instrument to make a background measurement. After that, the activated carbon sample was diluted with the KBr powder with the ratio of 10 % and grounded with the agate mortar until it became fine particles to mix both kinds. Then, the mixed powder was placed in the sample cup and the powder surface was also evened using the sample pressing bar. Finally, the mixed powder was mounted to the instrument to make a sample measurement in the transmittance %T mode. The analysis was carried out by Shimadzu 8400S FTIR instrument in the wave number range of 4000–500 cm^{-1}.

2.3.4. Optimization of Impregnation Ratio

Optimization of impregnation ratio is studied. Table 1 depicts the yield obtained in the percentage at 673 K by slow pyrolysis. Study was conducted at different impregnation ratio from 1:1 to 4:1 for different batches of 50, 100 and 300 g. It is observed that the yield increases till 3:1 impregnation ratio and decreases from 3:1 to 4:1. Thus impregnation ratio of 3:1 is referred as an optimised impregnation ratio.

2.3.5. Temperature Optimization

Activation temperature optimisation study was performed at slow pyrolysis temperatures of 523, 573, 623 and 673 K. At the temperature of 673 K the yield remains constant for different batches. Thus the temperature of 673 K was selected as the optimised temperature condition for pyrolysis.

2.3.6. Time Optimization Study

Activation time study is performed by taking run at different carbonisation times: 45, 60, 75 and 90 min. The yield at 90 min remained constant for activated carbon produced.

Thus 90 min were selected as optimised time condition for performing the final run of product. Hence all experiments at different impregnation ratio were done at 90 min.

2.4. Methylene Blue Number

Methylene blue solution was prepared by adding 0.15 g of methylene blue to 100 ml of water. AC (0.1 g) was weighed accurately and transferred to 50 ml glass stoppered flask. First 10 ml of methylene blue solution from burette was transferred to glass stoppered flask and centrifuged for 5 min. As first 10 ml were decolorized, the methylene blue solution was continued to be add (1 ml each time) till the blue colour disappeared for 5 min.

The methylene blue number (MBN) is a measure of mesoporosity (2–5 nm) present in activated carbon.

Decolourizing power of carbon (DP) was determined according to IS: 2230-1962. It is expressed in terms of milligrams of methylene blue adsorbed by 1 g of AC:

$$DP = \frac{15V}{10M}$$

where V is a volume of consumed methylene blue solution, ml; M is a mass of material taken for the test, g .

2.5. Determination of Yield

The yield is defined as the ratio of final weight of the obtained product after washing and drying to the weight of dried precursor initially used. The yield of AC was calculated based on the following equation:

$$Yield = \frac{Weight\ of\ AC\ after\ carbonization \cdot 100}{Weight\ of\ the\ raw\ material}$$

Table 1

Effect of impregnation ratio on the yield

Sample	Yield, % (50 g)	Yield, % (100 g)	Yield, % (300 g)
CL-PA-01 (1:1)	64.0	75.6	61.5
CL-PA-02 (2:1)	72.0	80.5	68.0
CL-PA-03 (3:1)	79.0	90.5	73.0
CL-PA-04 (4:1)	72.0	81.5	65.3

Proximate analysis of *Cocos nucifera* leaves

Cocos nucifera leaves	Weight, %
Ash	2.39
Moisture	8.17
Volatile matter	14.06
Fixed carbon	75.38
Total	100

2.6. Determination of Acid Adsorption Test

50 ml of glacial acetic acid were added to 400 ml of water. 10 ml of prepared solution were titrated by 1N NaOH solution for blank reading (W_1). Then 10 g of AC was added to 400 ml (water and acid sample) and stirred continuously for 0.5 h. 10 ml of activated carbon solution was titrated by 1N NaOH (W_2). The amount of acid adsorbed by activated carbon is $W_1 - W_2$.

2.7. Determination of Iodine Number

The iodine number (IN) is a relative indicator of porosity in an activated carbon. It is a measure of micropore content of the AC (up to 2 nm). The iodine number is determined according to ASTM D4607-94 method.

The iodine number is defined as the milligrams of iodine adsorbed by 1.0 g of carbon when the iodine concentration of the filtrate is 0.02 N (0.02 mol/l). Adsorption of iodine from an aqueous solution has been used to indicate absorptive capacity.

$$IN \text{ (dry basis)} = F \cdot (I_{2o} - I_{2e})$$

where I_{2o} – amount of I_2 at the outset of the test, mg; amount of I_{2e} at the end of the test, mg; F – filtrate normality equal to $VN/50$; V – volume of sodium thiosufate solution, ml; N – normality of sodium thiosulphate solution.

2.8. TGA Analysis

The carbonization behaviour was measured by using thermobalance (DTG-60, Shimadzu Corporation) on the aspect of the weight loss behaviour. Temperature range is maintained from ambient to 1373 K. Heating rate range is from 273 to 333 K/min.

TGA study was performed on the activation of activated carbon impregnated with the activating agents H_3PO_4. For each experimental run, a sample with the weight of 6 ± 0.5 mg was placed on a platinum sample pan. The samples were heated from 308 to 1273 K at the rate of 10 K/min in a nitrogen atmosphere. This study defines the remaining amount of sample decomposed in nitrogen as W/W_0, where W_0 and W represent the initial and instantaneous mass of the sample, respectively. TGA characterization was done to know the thermobalance of the samples prepared.

3. Results and Discussion

3.1. Proximate Analysis

Proximate analysis of *Cocos nucifera* leaves is given in Table 2. The precursor used in this study has the high carbon content of approximately 75 % and low ash content of about 6.4 %. It means that *Cocos nucifera* leaves are suitable to be used as activated carbon precursor.

3.2. Yield of Activated Carbon Derived from *Cocos nucifera* Leaves

Fig. 2 depicts the effect of activation temperature and impregnation ratio on the yield of activated carbon. This figure shows that the carbon yield was found to decrease with the increasing of impregnation ratio. The reaction of lignocellulosic with phosphoric acid begins as soon as the components are mixed, the acid first attacks hemicellulose and lignin because cellulose is more resistant to acid hydrolysis [11].
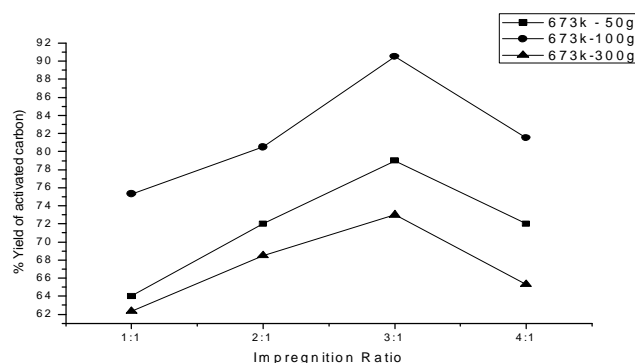

Fig. 2. Effect of impregnation ratio and activation temperature on the yield of activated carbon

At 673 K temperature the aromatic condensation reactions also take place among the adjacent molecules,

that results in the evolution of gaseous products from the hydroaromatic structure of carbonized char leading to the decreased yield of carbon [12]. Impregnation ratio is another critical parameter that affects the quality of the carbon. From Fig. 2 it is obvious that the yield of carbon decreases as the impregnation ratio increases. Here the excess phosphoric acid will promote gasification of char and increase the total weight loss of carbon. The same result is also observed by other researchers [10-12].

3.3. Effect of Impregnation Ratio on Methylene Blue Number

The increase in methylene blue (MB) concentration showed no significant changes. This is due to the fact that with the increase in MB concentration, the driving force for mass transfer also increases. At low concentrations there will be unoccupied active sites on the adsorbent surface. This retards the overall MB adsorption by activated carbon. Methylene blue number is an indication of ability of carbon to adsorb high molecular weight substances like dye molecules. Methylene blue number of greater than 400 indicates that carbon is good for dye adsorption.
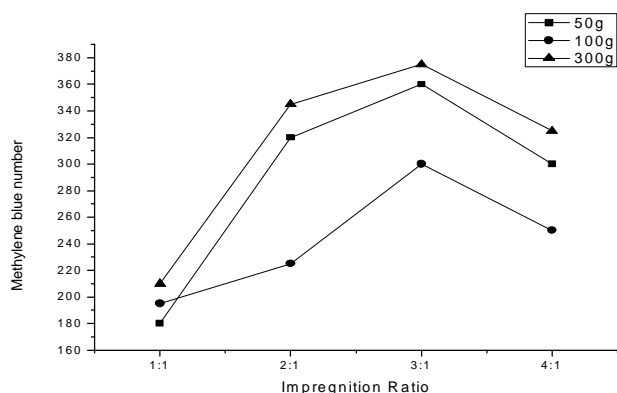

Fig. 3. Effect of impregnation ratio on methylene blue number activated carbon

One can see from Fig. 3 that methylene blue number increases till 3:1 ratio and decreases thereafter to 4:1. Moreover, methylene number is not greater than 400, thus it is not suitable for dye adsorption. Hence, suitability for dye adsorption made us to calculate the effect of impregnation ratio on iodine number.

3.4. Effect of Impregnation Ratio on Iodine Number

Iodine number is the most fundamental parameter used to characterize activated carbon performance. It is the measure of the micropore content of the activated carbon (0–20 Å, or up to 2 nm) by adsorption of iodine from the solution.

From the results of methylene blue values it becomes necessary to know the adsorptive capacity. Adsorption of iodine from an aqueous solution was used to indicate the absorptive capacity. Iodine number increases for different batches (50, 100 and 300 g) with different ratio of phosphorous acid to activated carbon ((1:1)–(4:1)). As iodine number increases, there is the increase in porosity of activated carbon. It is the measure of activity level. As it is seen from Fig. 4 the iodine number decreases from the ratio 3:1 to 4:1 for all batches, indicating the decrease in degree of activation, *i.e.* in activity level.
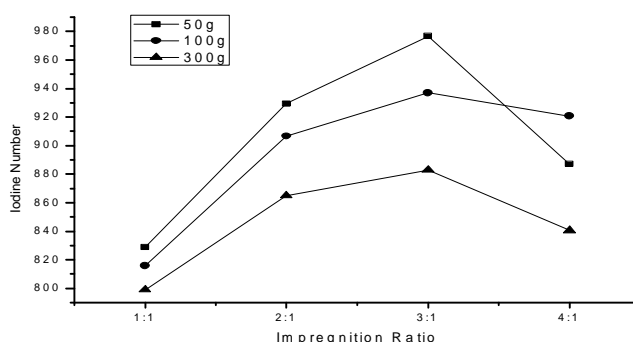

Fig. 4. Effect of impregnation ratio on iodine number

3.5. Effect of Acid Adsorption Value

Water soluble matter and acid soluble matter give the information about the amount of impurities present in carbon which affects the quality of water. But in our analysis the data show that all the carbons contain very low amount of impurities. As impregnation ratio increases from 1:1 to 4:1, water soluble content increases till impregnation ratio of 3:1 and decreases from 3:1 to 4:1 for all batches. It is observed that 300 g batch contains very less amount of impurities as compared to 50 or 100 g batches (Fig. 5).
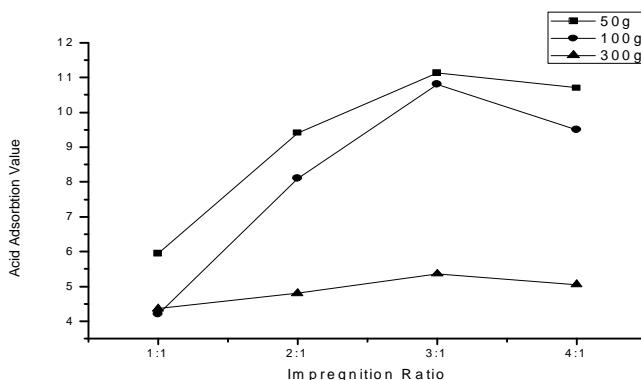

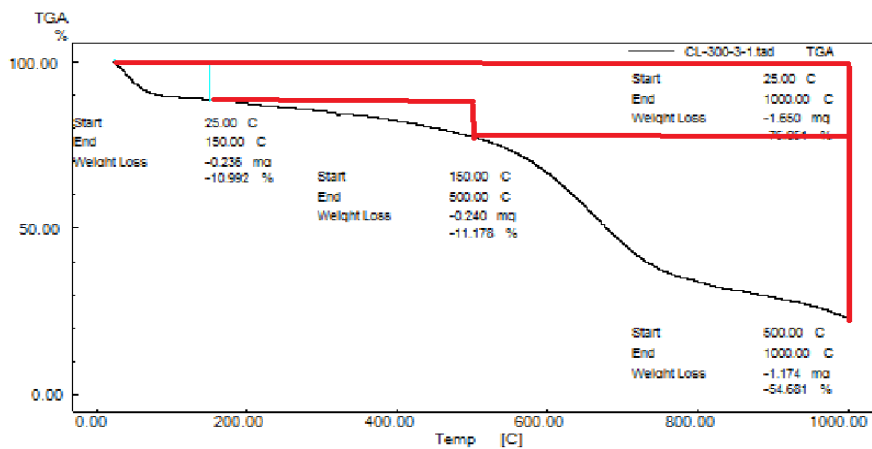**Fig. 5**. Effect of impregnation ratio on acid adsorption

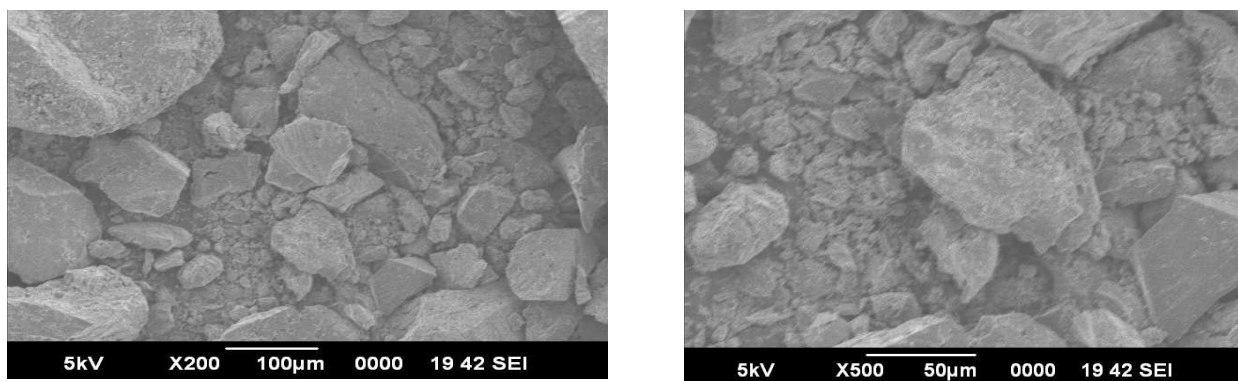**Fig. 6.** TGA curve of *Cocos nucifera* leaves

Fig. 7. SEM images of carbons produced from *Cocos nucifera* leaves at 673 K

Table 3

Surface chemistry of *Cocos nucifera* leaves derived activated carbon at 673 K

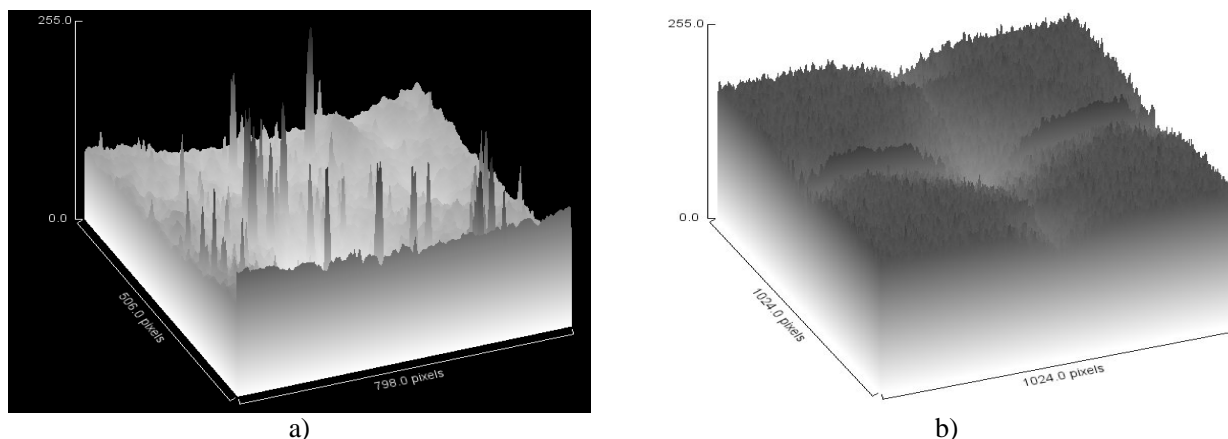| Sample	pH$_{PZC}$	Carboxylic, meq/g	Lactonic, meq/g	Phenolic, meq/g	Acidic, meq/g	Basic, meq/g
CL-PA-01 (1:1)	3.0	1.50	0.70	1.02	1.80	0.88
CL-PA-02 (2:1)	3.0	1.62	0.90	1.05	1.90	0.92
CL-PA-03 (3:1)	3.0	1.80	1.40	1.20	2.10	0.96
CL-PA-04 (4:1)	2.5	1.60	1.25	0.99	1.85	0.85

Fig. 8. Surface area plots of *Cocos nucifera* leaves: total particles (a) and individual particle (b)

3.6. TGA Study

The thermogravimetric analysis of the samples is presented in Fig. 6. The samples weight was 0.01 g, temperature range – 303–1273 K and rate of heating – 10 K/min. 12% weight loss have been observed for *Cocos nucifera* leaves. It is seen that activated carbons are thermally stable up to 423 K without appreciable weight loss. However the samples show weight loss of 10 % in the first step, followed by the weight loss of 11 % in the second step (423–773 K) and the major weight loss of 50–55 % forms char residues of 0–55 % in the third step ascribed to the thermal decomposition of polymeric backbone and carbonisation of the fragments, as well as AC particles.

3.7. Comparison of the External Surfaces of Resulting Carbon using SEM

Scanning electron micrographs of the surface morphology of several samples of the activated carbons are given in Fig. 7. This figure shows the differences of the external surfaces of the prepared activated carbons. From this figure, it is obvious that the activated carbons produced at 673 K have cavities on their external surface. It can be predicted that the cavities on the surfaces of carbons are the result of activating agent evaporation. In this case it is phosphoric acid during carbonization, leaving the space previously occupied by the activating agent.

3.8. Surface Chemistry of the Carbon

The matrix in carbon consists of not only carbon atoms, but it is also formed by other heteroatoms like hydrogen, oxygen, nitrogen, halogen, sulfur, phosphorus, *etc*. These atoms are bonded on the carbon edges, and govern the surface chemistry of activated carbon. The surface functional groups such as carboxyl, lactone, phenol, carboxylic anhydride, *etc*., are the source of surface acidity. The basic properties of activated carbon are associated with two types of structures: (i) the presence of oxygen-containing groups, *i.e.* pyrone, chromene and carbonyl structures at the edge of carbon crystallite; and (ii) oxygen free Lewis basic sites on the graphene layers. The Lewis basicity of delocalized electrons is influenced by the aromatic system on the carbon surface [13, 14].

Table 3 shows quantitative surface chemistry analysis, which consists of the amount of acidic and basic functional groups of *Cocos nucifera* leaves activated carbons. All activated carbons show low pH_{PZC} in the range of 2.5–3.0. These low pH_{PZC} values are consistent with Boehm titration results, which show dominance of acidic groups at the surface of the activated carbons. From Table 3 it can be predicted that at the same activation temperature with the increase in impregnation ratio the amount of acidic functional groups is reduced and the basic surface groups of the carbon is increased. The same activation temperature will decompose several functional groups to form CO and CO_2. This phenomenon occurs due to the instability of acidic groups at high temperatures [15]. On the other hand, basic groups are increased at the constant temperature. These groups can be formed during cooling of the activated carbon after the heating process. Cooling process enables the fixation of oxygen in the active sites [15]. Similar results were also obtained by Guo and Rockstraw [16] for phosphoric acid activated cellulose.
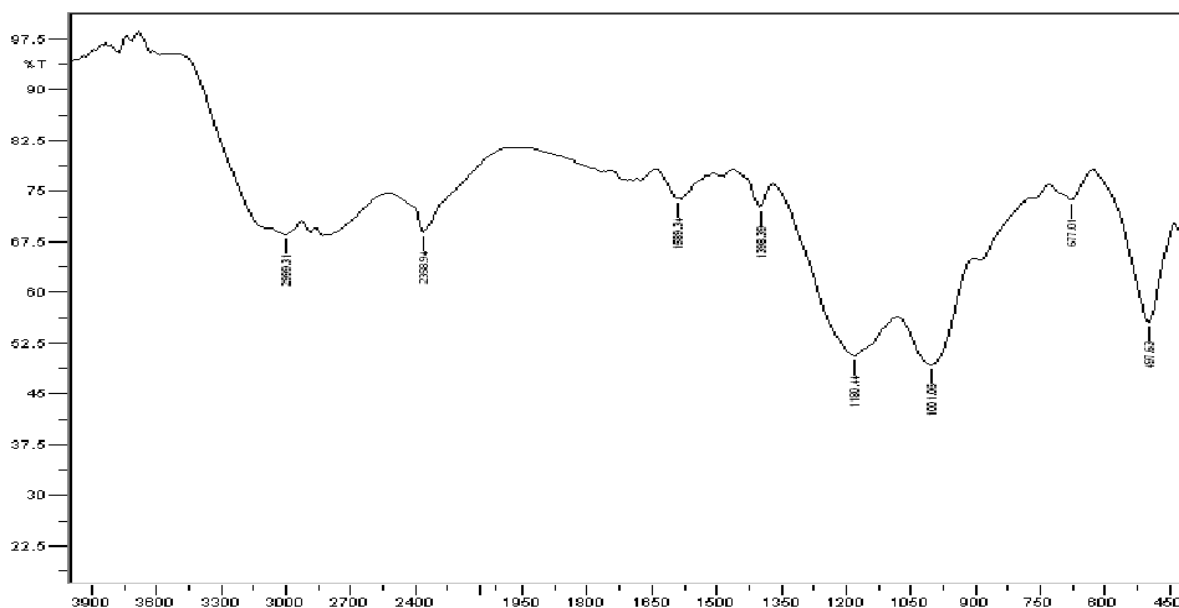

Fig. 9. FT-IR spectra for *Cocos nucifera* leaves activated carbon at the activation temperature of 673 K

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Fig. 8a shows the sharp fibrous structure of *Cocos nucifera* leaves. One can observe from Fig. 8 that individual particle surface is rough and have convex shape.

FT-IR spectra of *Cocos nucifera* leaves activated carbon with various impregnation ratios at each activation temperature are illustrated in Fig. 9. All spectra show broad absorption band around 2999–2400 cm^{-1}. A peak around 1500 cm^{-1} shows the presence of stretching vibration of CO in ketones, aldehyde, lactone and carboxyl. The broad band around 2999–2400 cm^{-1} and peak around 1510 cm^{-1} indicate the presence of carboxylic acids [16, 17]. A relative low intensity peak at wave number around 3100 cm^{-1} may also represent OH stretching vibration in phenol. Very weak peak around 2900 cm^{-1} is CH stretching vibration in methyl group [17]. A strong band at 1590 cm^{-1} can be ascribed to C–C aromatic ring stretching vibration enhanced by polar functional groups [8]. A broad band between 1300 and 1000 cm^{-1} is with the strong band. The peak at 1220–1180 cm^{-1} may be ascribed to the stretching mode of hydrogen bonded PO, OC stretching vibrations in P–O–C linkage and POOH; and the shoulder at 1080–1070 cm^{-1} can be ascribed to ionized linkage P+–O– in acid phosphate esters.

4. Conclusions

Activated carbons were prepared from *Cocos nucifera* leaves using phosphoric acid as the chemical activating agent. The effect of impregnation ratio and activation temperature on pore structure and surface chemistry of resulting carbons was studied. Optimised impregnation ratio for the yield, methylene number, iodine number and acid adsorption value is determined as 3:1. *Cocos nucifera* leaves have the highest N$_2$ adsorption characteristics. SEM and surface chemistry were determined by Boehm titration method and FT-IR to know the dominance of acid group. Thus it can be concluded that carbons with well-developed pores are produced at activation temperature of 673 K which is visible from surface area plot. At constant activation temperature (at different impregnation ratio) the amount of acidic functional groups increases up to the impregnation ratio of 3:1 and decreases thereafter to the impregnation ratio of 4:1. The same phenomenon is observed with the basic surface groups of carbon. This happens due to the increase in yield till impregnation ratio of 3:1 and the decrease for impregnation ratio from 3:1 to 4:1. Thus the impregnation ratio 3:1 is considered as the optimised ratio. Iodine adsorption number comprises the measurement of iodine amount in the adsorption layer of an activated carbon sample. Methylene number value is below 400, which depicts that *cocos nucifera* leaves activated carbon is not suitable for removal of dye particle. Thus, it can be concluded that all carbonaceous materials can be converted into activated carbon, although the properties of the final product will be different, depending on the nature of the used raw material, the nature of the activating agent and the conditions of the carbonization and activation processes.

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СИНТЕЗ АКТИВОВАНОГО ВУГІЛЛЯ З ВІДХОДІВ ЛИСТЯ КОКОСОВОЇ ПАЛЬМИ МЕТОДОМ ХІМІЧНОЇ АКТИВАЦІЇ

Анотація. Отримано активоване вугілля з відходів листя кокосової пальми (Cocos nucifera). За допомогою методу хімічної активації вивчено вплив ступеня просочення з використанням фосфатної кислоти як активуючого агента. Активоване вугілля отримано за температури активації 673 К сповільненим піролізом. Проведені дослідження ізотерм адсорбції азоту. Вивчено вплив ступеня просочення на вихід, кількість метиленового синього, йодне число і кислотну адсорбцію. Наявність активованого вугілля підтверджено ІЧ-Фур'є спектроскопією, його термічна стабільність за 723 К – термогравіметричними дослідженнями. За допомогою скануючої електронної мікроскопії доведено факт включення частинок активованого вугілля, що призводить до систематичної зміни морфології вугілля. Дослідження хімії поверхні активованого вугілля передбачають наявність кислотних і основних функціональних груп.

***Ключові слова**: листя кокосової пальми, активоване вугілля, фосфатна кислота, піроліз.*