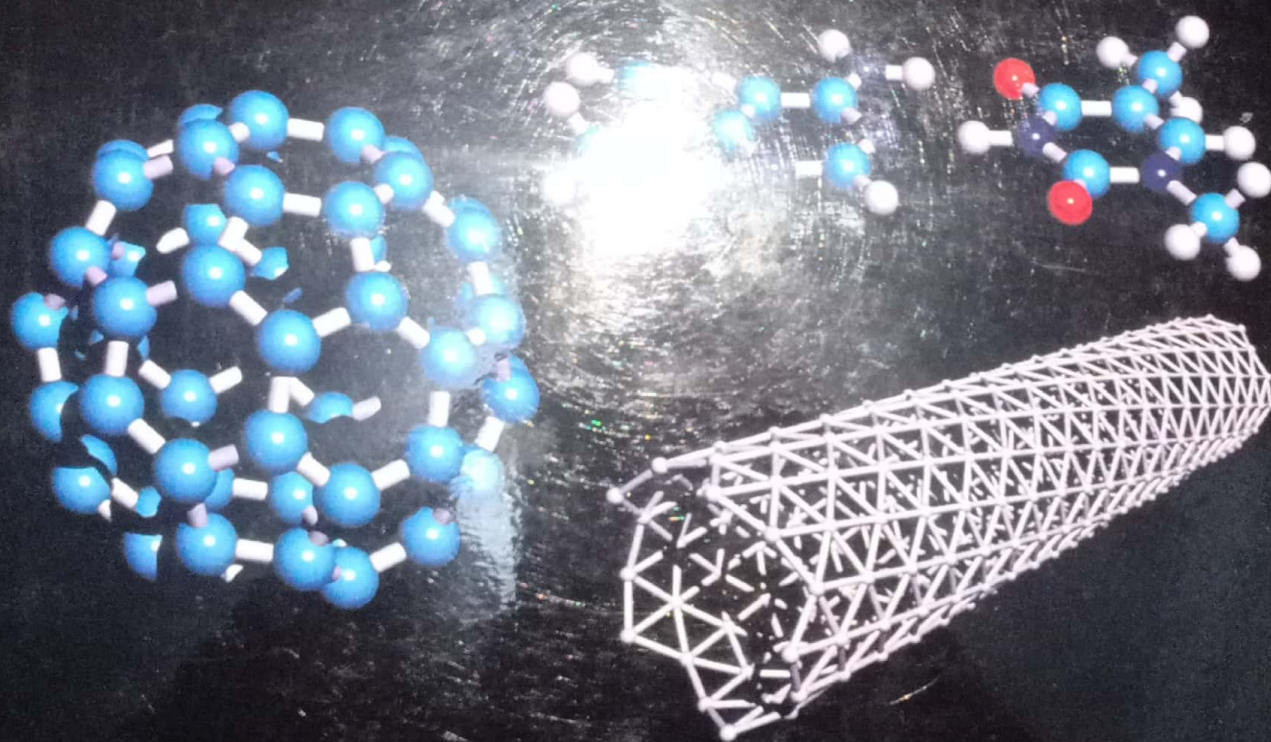


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# International Journal of Applied Chemistry

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## The Effect of Activation Time on The Chemical Structure and Quality of Activated Carbon From Coconut Shell Charcoal Using $ZnCl_2$ Activator

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### Abstract

The study on the preparation and preparation of activated carbon from coconut shell charcoal via the combined chemical ( $ZnCl_2$ ) and physical ( $CO_2$ ) activations has been carried out. This study was aimed to study the chemical change occurred during the activation. The charcoal was immersed in the solution of  $ZnCl_2$ , where the concentration of solution (0.5-2.0 M) and the immersion time (12-24 h) were varied. Then, the physical activation was carried out at  $800^\circ C$  with the variation of time (30-150 min) and it was streamed using  $CO_2$  (20 mL/min) for 2 h. Characterization of the activated carbon include FTIR, SEM, BET and XRD analyses. The FTIR analysis showed that the surface of the activated carbon had the functional groups of -OH, C-H and C-O. The XRD analysis showed that the activated carbon was amorphous. The SEM analysis showed that the pore was more porous, the pore diameter increased by the increasing of activation time. The activated carbon with the best quality was obtained with the condition of concentration of  $ZnCl_2$  of 1.5 M, immersion time of 24 h and activation time of 120 min. At such condition, the activated carbon had the surface area (BET) of  $359.286\text{ m}^2\text{g}^{-1}$ , pore volume of  $1.984 \times 10^{-1}\text{ cm}^3\text{g}^{-1}$  and average pore radii of  $11.04\text{ \AA}$ .

**Keywords:** coconut shell,  $ZnCl_2$ , activated charbon, FTIR, XRD, SEM and BET.

### Introduction

Activated carbon is porous solid material containing carbon (85-95%) and is produced from carbon-contained-material using the heating at high temperature<sup>[6]</sup>. Several raw materials which could be treated to give the activated carbon includes coconut shell,

coal, wood, peanut shell and humic <sup>[10]</sup>. Pyrolysis of the coconut shell gave charcoal as the main product. The obtain charcoal has the potential to be processed as activated carbon <sup>[21]</sup>. The activated carbon of the coconut shell charcoal has a lot of pores, large surface area and high adsorption capacity <sup>[11, 14]</sup>.

The preparation of activated carbon includes carbonization and activation. In the former process, the volatile compounds would be removed. The later one would build the porosity and increase the surface area <sup>[13]</sup>. The main product of carbonization is the charcoal with the side products of tar and liquid smoke. The obtained charcoal is not relatively active and has low adsorption capacity. Therefore, the activation should be conducted to increase the number of pore and to increase the surface area <sup>[17, 7]</sup>.

The activation process is aimed to enlarge the pore by breaking the hydrocarbon bonding or oxidizing the molecules on the surface, hence the physical and chemical properties of charcoal could be optimized, i.e. the increase of surface area and adsorption capacity <sup>[2, 20, 10]</sup>.

The common activation methods employed in the preparation of activated carbon are physical and chemical activations. The chemical activation is the breaking of carbon chain using chemical as activator such as KOH, NaOH, ZnCl<sub>2</sub>, KCl, H<sub>3</sub>PO<sub>4</sub> and HCl <sup>[18, 5]</sup>. On the other hand, the physical activation is the breaking of carbon chain with the assistance of heat, vapor and CO<sub>2</sub>. By performing the activation, it is expected that the qualities of the activated carbon as an adsorbent could be increased. The charcoal is heated at high temperature, 800-1,000°C <sup>[1]</sup>. In this study, the chemical activation using ZnCl<sub>2</sub> was combined with the physical activation using CO<sub>2</sub> in order to activate the charcoal. The combination of the two methods might increase the quality of the activated carbon. Therefore, the mechanism of chemical change occurred during the activation process should be conducted. This study was also aimed to study the the change of chemical structure of the activated carbon as the function of time of activation.

## Experimental

### A. Materials and tools

The materials employed in this study were coconut shell charcoal produced from pyrolysis (PT. Tropica Nucifera Industry), CO<sub>2</sub> (technique grade), ZnCl<sub>2</sub> (p.a., Merck), aquadest, universal indicator, HCl (p.a., Merck) and Whatman 42 filter paper. The tools utilized in this study were mortar, grinder, sieve (80 and 100 mesh, USA Standard Testing Sieve), oven (Gravity Vonvection), electric furnaces (Carbolite model 2132, maximum temperature of 1,200 °C), tube furnace-thermolyne (Sybron) Type 21100, balance (AND GR-200), hot plate, desicator, pH-meter, activation reactor, electronic balance, electric oven, magnetic stirrer, Fourier Transform Infra Red (FTIR, Shimadzu Prestige 21), Scanning Electron Microscope (SEM, JOEL JSM-6360) and Surface Area Analyzer (NOVA Ver. 2.2).

## **B. Methods**

### **1. Preparation of coconut shell**

The coconut shell obtained from pyrolysis was cleaned and grinded to give powder. The powder was sieved using the sieve to give the particle in 80-100 mesh.

### **2. Chemical activation**

The prepared coconut shell was immersed on the solution of  $ZnCl_2$ , where the concentration of the solution (0.5-2.0 M) and immersion time (12-24 h) were varied. The immersed charcoal was filtered using vacuum filter and heated on the oven at  $120^\circ C$  for 2 h and was placed on the desiccator.

### **3. Physical activation**

The chemical-treated-charcoal was placed on the activation reactor tube furnace at which the glasswool was placed on the bottom and upside of the charcoal. It was then activated at  $800^\circ C$  with the variation of time (30-150 min) and it was streamed with  $CO_2$  (20 mL/min) for 2 h. The charcoal was then immersed on the solution of HCl 1M for 24 h. The charcoal was washed with aquadest until neutral (monitored by pH-meter). It was dried on the oven at  $110^\circ C$  for 2h.

### **4. Analysis of the activated carbon**

Characterization of the activated carbon included the chemical and physical properties. There were FTIR (functional group), XRD (crystallinity), SEM (surface properties), BET (surface area), total pore volume and pore diameter average.

#### **Fourier Transform Infra Red (FTIR)**

The FTIR analysis (Shimadzu Prestige 21, pellet KBr) was performed to determine the functional group of the activated carbon.

#### **X-ray Diffraction (XRD)**

The XRD analysis was used to analyze the coconut shell charcoal and the prepared activated carbon. The analysis was performed using XRD (Shimadzu XRD-6000).

#### **Scanning Electron Microscopy (SEM)**

The SEM analysis was done to analyze the microstructure of the surface of the activated carbons, before and after activation.

#### **Surface area analysis BET**

The analysis of pore structure and the surface area was conducted using Surface Area Analyzer NOVA Ver.2.2. The determination of the surface area was performed using BET method. By employing BET equation, total pore volume and average pore radii could be determined.



## Results and Discussions

### FTIR Spectra

The FTIR spectra of the  $ZnCl_2$ -activated-activated carbon on the concentration of 1.5 M for the immersion time of 24 h at  $800^\circ C$  with the variation time of 30, 60, 90, 120 and 150 min were presented on Figure 1. While the wave number of the infra red absorption band spectra of coconut shell charcoal and activated carbon with a variation of the activation time is presented on Table 1. The intensity of the stretching vibration for hydroxyl group of Z.30 to Z.90 had shifted to higher wavenumber, from  $3,441.0$  to  $3,495.0\text{ cm}^{-1}$ . The pattern of Z.90 to Z.150 showed the decreasing on the wavenumber from  $3,495.0$  to  $341.8\text{ cm}^{-1}$ . It was predicted that the effect of temperature and activation time did not destroy the bonding of carbon site. These vibrations were supported by the absorption at  $1381.0$ - $1342.4\text{ cm}^{-1}$  which corresponded to the stretching vibration from C-O and the bending vibration of OH from alcohol and carboxylic acid. The absorption on the region of  $2939.5$ - $2931.8\text{ cm}^{-1}$  was characteristic for the stretching vibration of C-H aliphatic on  $-CH_3$  and  $-CH_2-$ . Such absorption was not observed in the case of Z.150. It was predicted that the longer activation time and the high activation temperature led the bond breaking via free radical mechanism. According to [8], the higher the temperature of carbonization and the activation time, the more the functional group which was oxidized or decomposed, thus the absorption was disappeared or reduced. In addition, the absorption at  $2337.72\text{ cm}^{-1}$  was the stretching vibration from C=O of carboxylic acid. It was strengthened by the presence of absorption at  $1928.0$ - $1851.66\text{ cm}^{-1}$  on Z.120 and Z.150. The absorption at  $1635.6$ - $1581.6\text{ cm}^{-1}$  indicated the presence of stretching vibration for aromatic C=C bond. The absorption at  $802\text{ cm}^{-1}$  showed the presence of the bending vibration of aromatic C-H.

Carbonization and activation processes on the preparation of activated charcoal using  $ZnCl_2$  with the different activation time, led the change of functional groups on the charcoal, which was followed with the formation of new compounds via the free radical mechanism. Based on the data, it could be stated that the identified functional groups on the activated carbon with the different activation time were relatively the same. However, the intensities was increased or decreased, depended on the activation time. Carbonization of a material at higher temperature and the long activation time might shift the wave number [15]. The absorption of the activated carbon at  $1500$ - $900\text{ cm}^{-1}$  was increased. That region belonged to the absorptions of C=C, bending vibration of OH and stretching vibration of C-O ester groups. The intensities were high and then reduced after the carbonization and the long activation. The decrease on the absorption intensities indicated the formation of aromatic compound [12].

### XRD analysis

The diffractograms of the  $ZnCl_2$ -activated-activated carbon and the commercial one from Merck were presented on Figure 2 showed the broad peak at  $2\theta = 10^\circ$ . They showed that the two materials were amorphous and the structure had not been well organized yet. The presence of the broad and relatively sharp peak around  $2\theta = 24^\circ$  on the diffractogram of the prepared activated carbon showed that the structure had

started to form a good pattern. Table 2 showed that the prepared activated carbon had the strongest intensities at  $2\theta$  of  $24.82^\circ$ ,  $24.16^\circ$ , and  $23.50^\circ$ , while those of Merck were at  $2\theta$  of  $23.90^\circ$ ,  $24.98^\circ$ , and  $25.44^\circ$ . The results were in line with those of [3] which stated that the broad peak at  $2\theta$  of  $26^\circ$  related to the 002 spacing of graphite, which was characteristic for the activated carbon.

The presence of broad peak with low intensity at  $2\theta$  of  $43^\circ$  showed the significant increase on the organisation of the crystal structure. The increase on the crystallinity occurred due to the shrinkage of the crystallite structure of charcoal, where the gap will be produced between the crystallite and the pore will be bigger [16].

### Analysis of the activated carbon using Scanning Electron Microscope (SEM)

The SEM Image of charcoal before and after the activation were presented on Figure 3. The results showed that there were changes on the surface of the charcoal during the activation. On the activated carbon, the indentation was quite deep where the distance between the pores was closer as the increasing of the activation time. In addition, the activation made the pore became more porous. It was indicated that the activation process could open and form the new pores on the activated carbon [19].

The SEM image (magnification of 1,000-1,500 times) showed that the increasing of time decreased the particle size of the activated carbon. It was indicated that the breaking of carbon chain has occurred during the carbonization and activation. The smaller the particle size of the activated carbon, the higher its surface area.

### Distribution of pore size of the activated carbon

The distribution of the pore size described the pore size of material. The distribution of the pore size of the untreated and treated activated carbon were presented on Figure 4. As displayed on Figure 4, all the treated activated carbons and the commercial one had the similar pattern. The treated activated carbon had smaller pore diameter and higher pore volume than the untreated one. In order to determine the pore homogeneity, the calculation was conducted base on Barret-Joyner-Halenda (BJH) method. The curves on the Figure 4 were quite pointy and had the same pattern, i.e. there was one peak. It was indicated that the activated carbon was produced with relatively good uniformity. It was also showed that the chemical and physical activation using  $ZnCl_2$  could uniformed the pore size.

### Determination of BET surface area of the activated carbon

Surface area, pore size and pore volume are important properties of adsorbent, which is related to the available active site on the surface. The pore parameters of the activated carbon was displayed on Table 3. As depicted on Table 3, the increasing of activation time from 30 to 120 min increased the surface area from 222.05 to 359.28  $m^2g^{-1}$ . The area decreased to 357.35  $m^2g^{-1}$  for the activation time of 150 min. By increasing the activation time, the heavier volatile compounds would evaporate and be released from the pore, then formed the new pore and increased the surface area, pore volume and decreased the average pore diameter. The decrease of area at 150 min was probably due to the occurrence of knocking and the damage on the pore wall, thus inhibited the pore formation [3]. Additionally, the total pore volumes of the activated

carbon for the activation time of 30, 60, 90, 120 and 150 min were  $1.288 \times 10^{-1}$ ;  $1.590 \times 10^{-1}$ ;  $1.815 \times 10^{-1}$ ,  $1.984 \times 10^{-1}$ , and  $1.955 \times 10^{-1} \text{ cm}^3/\text{g}$ , respectively. Those value were higher than the volume of unactivated charcoal,  $0.217 \times 10 \text{ cm}^3 \text{ g}^{-1}$ .

## Conclusion

1. Combination of chemical and physical activation increased the crystallinity of the amorphous carbon, pore volume, surface area and homogenized the pore diameter with the size of mesopore.
2. The activation process could increase the aromatic compound. The presence of -OH and C=O functional groups made the activated carbon to be polar and might be applied as an adsorbent.
3. The activated carbon with the best quality was obtained with the condition of concentration of  $\text{ZnCl}_2$  of 1.5 M, immersion time of 24 h and activation time of 120 min.
4. The variation of activation time gave the activated carbon with the similar properties, such as smaller pore diameter and higher pore volume than the unactivated carbon. The activated carbon had the surface area (BET) of  $359.286 \text{ m}^2\text{g}^{-1}$ , pore volume of  $1.984 \times 10^{-1} \text{ cm}^3\text{g}^{-1}$  and average pore radii of  $11.04 \text{ \AA}$ .

Table 1: IR absorptions of coconut shell charcoal before and after activation using  $\text{ZnCl}_2$  as the function of activation time

Charcoal from pyrolysis	Charcoal after activation using $\text{ZnCl}_2$ Variation of time (min)					Vibration
	30	60	90	120	150	
3749	-	-	3749	3749	3749	
3487.17	3441.0	3448.7	3495.0	3448.7	3417.8	Stretching vibration of O-H from hydroxyl group
2831	2931.8	2939.5	2939.5	2931.8	-	Stretching vibration of C-H from $\text{CH}_3$ and $\text{CH}_2$ group
2377,72	2337.7	2337.7	2337.7	2337.7	2337.7	Stretching vibration of C=O from carboxylic acid group
-	-	-	-	1851.66	1928	Aromatic C-H bond
1581,6	1627.9	1635.6	1635.6	1635.6	1635.6	Stretching vibration of C=O from aromatic carbonyl group Stretching vibration of C=C from aliphatic double bond
-	1350.1	1342.4	1381.0	1342.4	1381.0	Stretching vibration of C-O and bending vibration of O-H from alcohol and carboxylic acid
1165	1095.5	1111.1	1041.5	1080.1	1103.2	Stretching vibration of C-O-C from 6-membered ring ether
802,39	964.4	956.6	956.6	956.6	956.6	Bending C-H vibration

Table 2: Intensities of diffractogram peaks of ZnCl<sub>2</sub>-activated-activated carbon and commercial activated carbon

The highest peak	ZnCl <sub>2</sub> -activated-activated carbon		Activated carbon from Merck	
	2θ	Intensity	2θ	Intensity
The 1st	24,82	100	23,90	152
The 2nd	24,16	91	24,98	144
The 3rd	23,50	89	25,44	131
Total		280		427

Table 3: Specific surface area, total pore volume and average pore radii of the activated carbons

Sample	Specific surface area (m <sup>2</sup> /g)	Total pore volume (x 10 <sup>-1</sup> cm <sup>3</sup> /g)	Average pore radii (Å)
Charcoal	15,451	0,217	26,478
Z8-1,5-24-30	222,058	1,288	11,60
Z8-1,5-24-60	267,431	1,590	11,89
Z8-1,5-24-90	322,107	1,815	11,27
Z8-1,5-24-120	359,286	1,984	11,04
Z8-1,5-24-150	357,355	1,955	10,94

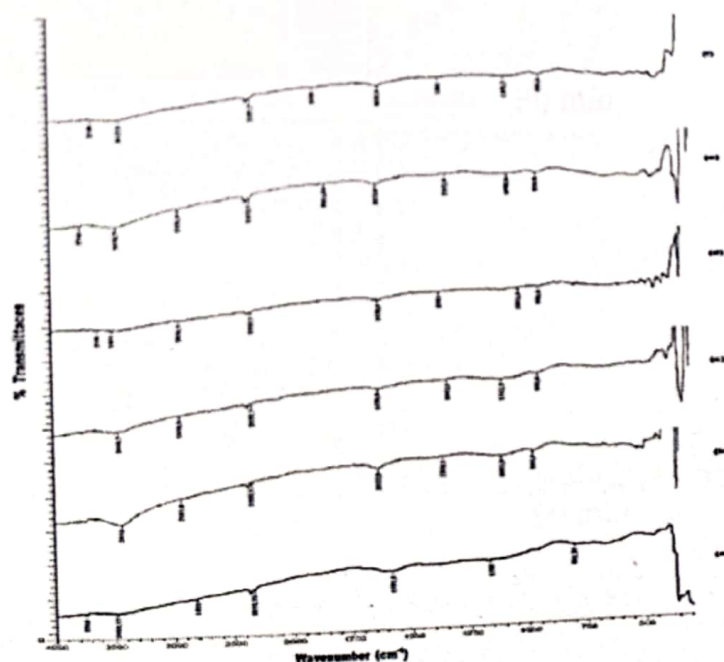


Figure 1: FTIR spectra of coconut shell charcoal before activation (a) and after activation using ZnCl<sub>2</sub> for 30 (b), 60 (c), 90 (d), 120 (e) and 150 (f) min

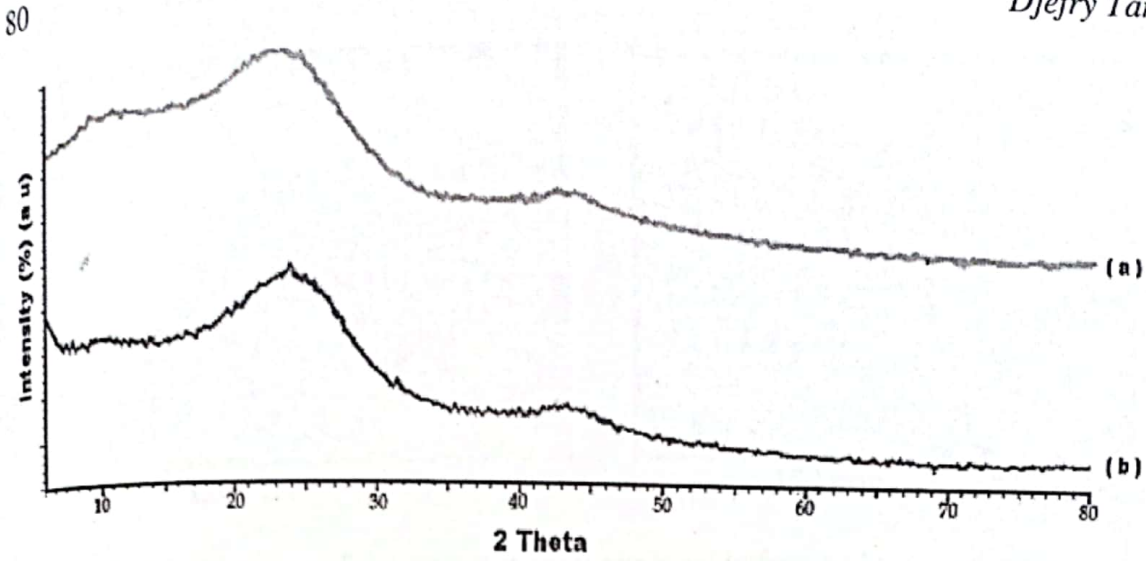
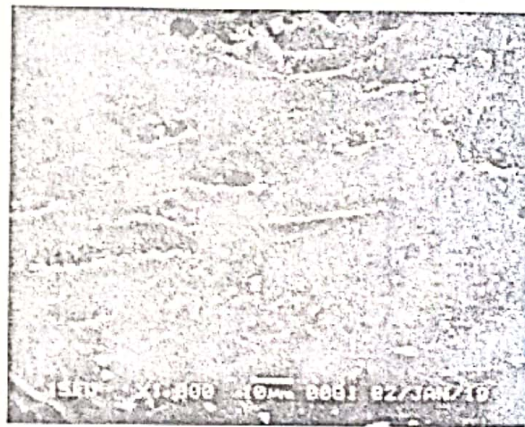


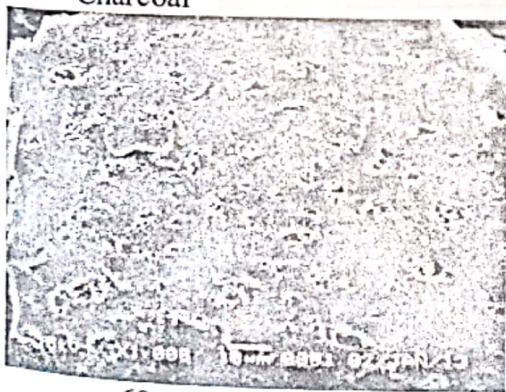
Figure 2: Diffractogram of ZnCl<sub>2</sub>-activated-activated carbon (a) and commercial activated carbon (b)



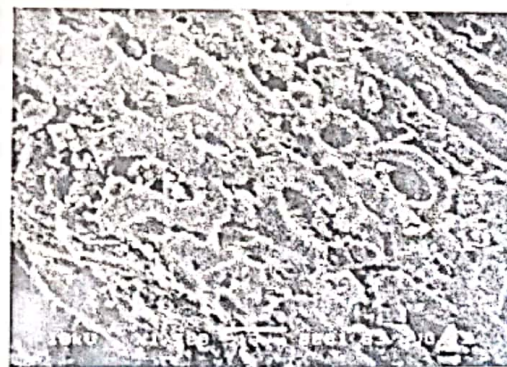
Charcoal



30 min



60 min



90 min

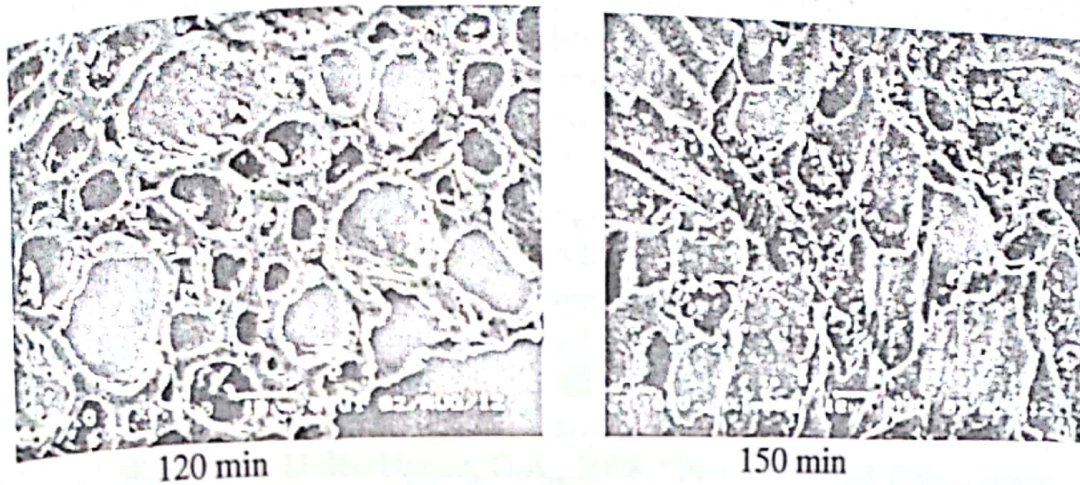


Figure 3: SEM images of the coconut shell charcoal before and after activation using  $ZnCl_2$  (1.5 M) for 24 h at  $800^\circ C$  as the function of activation time

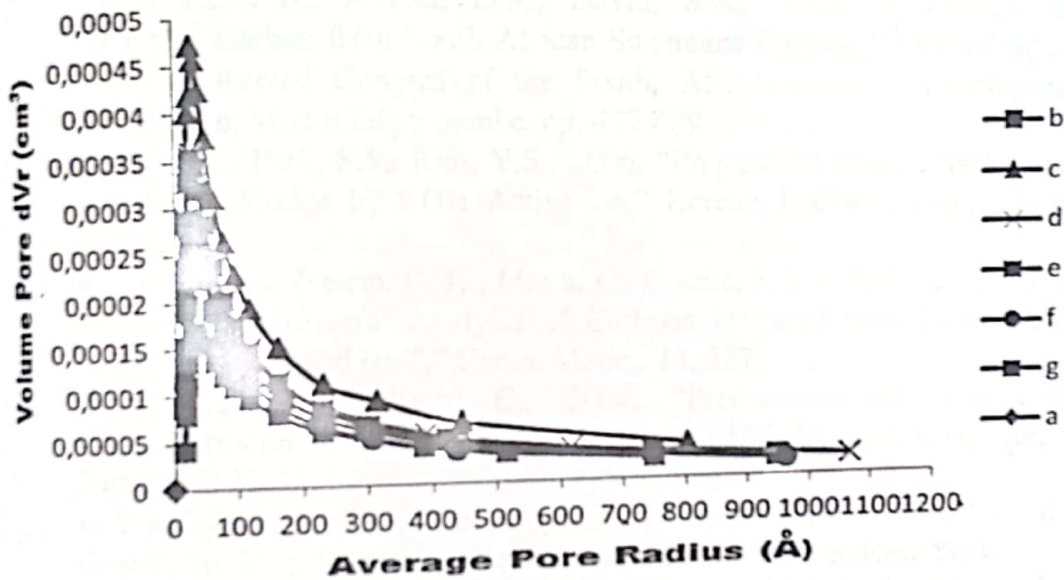


Figure 4: Distribution of pore size of coconut shell before activation (a) and after activation using  $ZnCl_2$  for 30 (b), 60 (c), 90 (d), 120 (e) and 150 (f) min and commercial activated carbon from Merck (g)

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