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# Preparation and Characterization of Activated Carbon from Desiccated Coconut Residue by Potassium Hydroxide

Mohd Adib Yahya[1], Z. Al-Qodah[2,*], C.W.Z.C.W. Ngah[1] and Mohd Azman Hashim[1]

[1]Faculty of Science and Technology, Universiti Sains Islam Malaysia, 71800 Nilai, Negeri Sembilan, Malaysia
[2]Department of Chemical Engineering, Taibah University, Medina, Kingdom of Saudi Arabia

*Corresponding author: E-mail: z_alqodah@hotmail.com

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This study investigates the effect of temperature and impregnation ratio on the physicochemical properties of activated carbon prepared from desiccated coconut residue by chemical activation using potassium hydroxide. Desiccated coconut residue sample was first carbonized at three different temperatures for 1 h at 400, 500 and 600 °C, respectively. The resulting chars were impregnated with KOH at three different impregnation ratio; 1:1, 1:2 and 1:3, respectively and activated under nitrogen atmosphere for 1 h at three different temperatures based on its carbonization temperature. The BET surface area and pore volume was strongly affected by temperature in which increased in temperature caused increased in BET surface are and pore volume. The BET surface area also increased with impregnation ratio but then decreased due to pore widening of activated carbons.

**Keywords: Desiccated coconut residue, Activated carbon, Potassium hydroxide.**

## INTRODUCTION

Activated carbon can be defined as carbonaceous materials that have a very high surface area[1]. It can be prepared by two types of treatment which are physical and chemical treatments[2]. The descriptions related to the activated carbon preparations have been described elsewhere. Both procedures could create a very high specific surface area of activated carbon by oxidative generation of porosity with variable size and shape distribution which dependent upon its raw material and process conditions[3]. Nowadays, the preparation of activated carbon derived from agricultural waste materials have been noticed by many researchers. These renewable sources can be converted into very useful products with many applications. This study was done to utilize the desiccated coconut residue (DCR) to produce activated carbon since it is inexpensive, even no cost required to obtain the material. Desiccated coconut residue is basically a byproduct of the coconut industry which is obtained from the milk extraction and generally being left as waste materials. There are large quantities of the desiccated coconut residue that being left as waste materials and only small parts that being used as fertilizer or feed for animals[4]. This conversion of desiccated coconut residue into activated carbon would increase its economic value, reducing the disposal cost of the material and provide low-cost material for activated carbon preparation[5]. In this study, the desiccated coconut residue sample was previously charred and then impregnated with potassium hydroxide. The chemical was chosen due to the fact that it is more eco-friendly as compared to other chemical agents such as $ZnCl_2$ and $H_3PO_4$[6].

## EXPERIMENTAL

**Preparation of samples:** The desiccated coconut residue was collected from local market in Nilai (Malaysia) and was dried for 24 h until use. The desiccated coconut residue was then ground to powder with kitchen grinder (Panasonic MX-337) to obtain particle size of 1-2 mm.

**Preparation of activated carbon:** A two-step activation (carbonization-activation) was performed in this study. A 20 g of desiccated coconut residue sample was first carbonized using tube furnace. The carbonization was done by heating at the rate of 10 °C $min^{-1}$ from room temperature until reaching to the desired temperature (400, 500 and 600 °C) and kept for 1 h under $N_2$ flow (300 mL $min^{-1}$) and let it cooled to room temperature. The process was repeated until substantial amount was achieved. After that, a 1 g of the char produced was impregnated with KOH with the three different ratios (1:1, 1:2 and 1:3) at ambient temperature for 1 h. Then the mixture was dried in an oven at 105 °C for overnight. The mixture was then pyrolyzed for 1 h at three different temperature (400, 500 and 600 °C), based on its char production temperature, respectively. The yield of desiccated coconut residue-activated carbon (DCR-AC) was calculated[7] using eqn. 1;

$$\text{DCR-AC yield (\%)} = X_1/X_0 \times 100 \quad (1)$$

where, $X_1$ is the weight of the DCR-AC, $X_0$ is the weight of the desiccated coconut residue.

The second pyrolysis basically involves dehydration reactions in which water was eliminated. There were also cross-linking reactions of the carbon, hydrogen and oxygen in the char structure which enhance the aromatization of the char. The cross-linking could inhibit the shrinkage of the structure during pyrolysis and created the porous structure which related to the chemically activated carbon[8]. The DCR-AC was then washed with 1 M HCl followed with deionized water (> 500 mL). The washing steps were done basically to remove any residual organic and mineral matter[9]. The washing step of acid followed by water could expose the porous structure of the activated carbon[8]. All the samples were then dried at 110 °C overnight, cooled and stored for further studies. The process of DCR-AC production was repeated until a substantial amount was achieved[10]. The samples were named as AC 1:400, AC 2:400, AC 3:400, AC 1:500, AC 2:500, AC 3:500, AC 1:600, AC 2:600 and AC 3:600 (1, 2, 3 indicated ratio and 400, 500 and 600 indicated desired temperature).

**Proximate analysis:** Proximate analysis to study the relative amounts of moisture, volatile matter, fixed carbon and ash content was studied using a thermogravimetric analyser (Mettler Toledo TGA-DSC STARe system) following Boonpoke *et al.*[11]. A known amount of sample was heated from 30 to 900 ºC under $N_2$ atmosphere. To obtain the ash content, air was introduced instead of $N_2$ and held for 10 min. at the final step of the process. The loss of weight during pyrolysis was calculated for the percentage of moisture, ash content, volatile matter and fixed carbon. Fixed carbon was calculated by weight fraction basis (fixed carbon = 1 - moisture - ash content - volatile matter)[12].

**Ultimate analysis:** The ultimate analysis was used to determine the existence of the carbon, hydrogen, nitrogen and oxygen was determined by CHNS analyzer (Thermo Finnigan, USA). Oxygen value was determined by difference[11].

**Bulk density determination:** The bulk or apparent density was determined by using method used by Al-Qodah and Shawabkah[13]. A known amount of desiccated coconut residue and DCR-AC was weighed and transferred to a 10 mL graduated cylinder. The cylinder was tamped with a rubber pad while DCR-AC being added until the entire original was transferred to the cylinder. Tamping was continued for 5 min. until no further settling produced. The volume was recorded and the bulk density was calculated on the dry basis in eqn. 2:

$$\text{Bulk density} = \frac{\text{Weight of sample (g)}}{\text{Volume of sample (cm}^3\text{)}} \quad (2)$$

**Physical properties of DCR-AC:** The surface morphology of activated carbon was investigated using a scanning electron microscope (Phenom, G2 pro) which was operated at 15 kV. The samples were mounted on an adhesive carbon tape attached to an aluminium-stub and then sputter coated with platinum in the sputter coater[13]. The SEM magnifications selected were at × 1000.

**Nitrogen adsorption measurements:** The surface area of the DCR-AC, the micropore and mesopore volume was obtained by $N_2$ adsorption at -196 °C (77 K), using an automatic volumetric sorption analyzer (Quantachrome, Autosorb 2). The system basically determined the surface area and pore size characteristics by volumetric adsorption of $N_2$ at 77 K[9]. DCR-AC was degasified at 350 °C for a period of at least 5 h to remove any adsorbed species[8]. The BET method was used to measure the DCR-AC surface area. Micropore volume was obtained from *t*-plot method. Then, the mesopore volume was obtained by subtracting micropore volume from total pore volume. Total pore volume was obtained from nitrogen adsorption[15] at a relative pressure of 0.99.

## RESULTS AND DISCUSSION

**Thermal analysis of raw material:** Fig. 1 shows the result of the TGA for the raw material desiccated coconut residue. It showed that there were three stages of thermal decomposition of the desiccated coconut residue. The first stage 50 to 120 °C was attributed to the moisture content of the desiccated coconut residue. A significant loss was seen from 250 to 420 °C which was attributed to the volatile released[14,15], including organic compounds such as cellulose and lignin[16]. Cellulose basically decomposed at temperature between 299.85-429.85 °C. Meanwhile, lignin decomposed at temperature range of 249-549 °C and hemicellulose decomposed at much lower temperature[17]. Gonzalez *et al.*[18] reported that hemicellulose was the first to decompose at around 200-260 °C and followed by cellulose at around 240-350 °C. The author also stated that lignin is a more stable as compared to the former ones. Its decomposition range was wider and overlapped with the weight loss of the hemicellulose and cellulose components and its contribution to weight loss was difficult to determine. The decomposition of the lignin has been identified at around 280-500 and 175-800 °C. It has been identified that, the decomposition zones were divided into two zones *i.e.*, active and passive zone. Active zone was attributed to the volatile compounds that produced from hemicellulose and cellulose decompositions. Passive zone was attributed to the lignin (and some cellulose) decomposition. Li *et al.*[19] reported that lignin basically starts to decompose at low temperature; 160-170 °C and its decomposition continues at slow rate until around 900 °C. The author also stated that hemicellulose was the second component to decompose and followed by cellulose, at temperature range of 200-400 °C. At this range, the main decomposition occurs which involves degradation reactions. From the figure, at temperature of beyond 400 °C, there was a slow weight loss takes place which was comparable in this study. Hence, the temperatures of 400, 500 and 600 °C were selected in this study to produce char and activated carbon.

**Proximate analysis:** There are some criteria to select a carbonaceous material as activated carbon precursor. It includes low inorganic content, low cost, workability of the material, minimum impact upon environmental and its storage life[20]. A good precursor for activated carbon should also have high content carbon[21]. Results showed the results of the proximate analysis for raw desiccated coconut residue were as follows; moisture (3 %), volatile matter (84.20 %), ash (5.51 %)

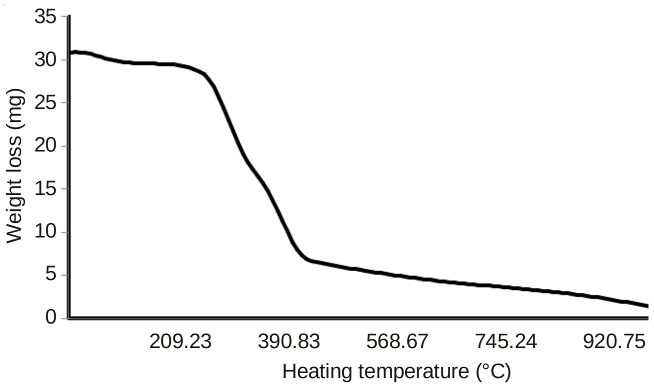

Fig. 1. TGA analysis as a function of heating temperature

and fixed carbon (7.29 %), respectively. It showed that desiccated coconut residue has a high value of volatile matter and low value of ash and moisture content. The high volatile matters of the desiccated coconut residue were a sign that the biomass can be used as activated carbon precursor[22]. As reported by Williams and Reed[8], biomass generally has a high value of oxygen and volatile matter and a low value of ash content, respectively. Ash content is defined as a measure of inorganic impurities in the activated carbon. Its present is basically reducing the activities of the activated carbon[23]. Hence, the low value of the ash content in the desiccated coconut residue could result in minimum impact on pore development of activated carbon during activation[24].

**Ultimate analysis:** Table-1 shows the ultimate analysis of desiccated coconut residue and desiccated coconut residue-AC prepared. As can be seen from the table, raw desiccated coconut residue contained 49.48 % C, 12.80 % H, 0.19 % N and 37.53 % O, respectively. The carbon contents of the DCR-AC generally higher than that of raw material of desiccated coconut residue. The non-carbon elements (H, O, N and S) were removed in the form of gaseous during the chemical activation, resulting low values of the elements for all DCR-AC[25]. Hydrogen content reduced was attributed to the rupture of the organic molecular chains occur during activation process[26]. The carbon contents decreased with impregnation ratio for all types of temperature. This reduction of the carbon content of the DCR-AC was attributed to the production of the carbon monoxide during the activation process[11].

**Yield (%) of DCR-AC:** Table-2 shows yield percentage of the resulting DCR-AC. Yield is defined as the percentage of the carbon mass that left after pyrolysis and activation, while burn-off is defined as the weight loss percentage of the pyrolyzed char that occurs during activation[27]. It indicated that DCR-AC had lower yield which ranged from 11.85 to 14.80 %. Suhas *et al.*[28] reported that lignin has been the most responsible component in influencing the resulting char and activated carbon. In general, higher lignin content produces higher char and activated carbon yield. Lignin composition in desiccated coconut residue was found to be 1.18 %[4], hence resulted in lower DCR-AC yield. In general, increasing the ratio of KOH caused the yield to be increased. It was attributed to the potassium ions that attached to the carbon surface. It ultimately acted as catalyst which accelerates the reaction between carbon and KOH[6].

**Bulk density:** Density of activated carbon may vary with the starting material. Different activation method could also produce different density of the activated carbon[29,30]. Therefore, bulk density of an activated carbon is of paramount to be considered in the activated carbon preparations. It has also been found that the nature of the activating agents could also influence the density of the activated carbon[31]. The smaller the density of the activated carbon could result in greater in porosity and surface area[32]. Bulk density of desiccated coconut residue was found to be 0.24 g/cm$^3$. Table-2 indicated DCR-ACs had low values of the bulk density. In general, bulk density of DCR-ACs was lower than that of original desiccated coconut residue due to the development of the porosity[33].

**Physicochemical properties of the DCR-AC:** The most important property for activated carbon was the adsorptive capacity which was related to its specific surface area and pore volume[34]. From Table-2, the surface area obtained from $N_2$ adsorption ranged from 222.53 to 823.81 m$^2$/g. The BET surface area was in the order: AC 2:600 > AC 3:600 > AC 2:500 > AC 3:500 > AC 1:600 > AC 1:500 > AC 2: 400 > AC 1:400 > AC 3:400. The total pore volumes were ranged from 0.13 to 0.50 cm$^3$/g. Total pore volume also called Gurvitch

TABLE-1
ULTIMATE ANALYSIS OF DESICCATED COCONUT RESIDUE AND DCR-AC

| Ultimate analysis (%) | Desiccated coconut residue | AC 1:400 | AC 2:400 | AC 3:400 | AC 1:500 | AC 2:500 | AC 3:500 | AC 1:600 | AC 2:600 | AC 3:600 |
|---|---|---|---|---|---|---|---|---|---|---|
| Carbon | 49.48 | 72.48 | 70.10 | 61.49 | 71.95 | 64.77 | 54.28 | 72.31 | 64.33 | 57.76 |
| Hydrogen | 12.80 | 2.22 | 2.45 | 2.05 | 1.56 | 2.19 | 2.23 | 1.27 | 1.78 | 2.26 |
| Nitrogen | 0.19 | 3.54 | 3.64 | 3.15 | 3.08 | 2.54 | 2.24 | 2.54 | 2.03 | 1.74 |
| Oxygen* | 37.53 | 21.76 | 23.81 | 33.31 | 23.41 | 30.50 | 41.25 | 23.88 | 31.86 | 38.24 |

* Oxygen was calculated by difference

TABLE- 2
PHYSICOCHEMICAL PROPERTIES OF THE DCR-AC

| Property | AC 1:400 | AC 2:400 | AC 3:400 | AC 1:500 | AC 2:500 | AC 3:500 | AC 1:600 | AC 2:600 | AC 3:600 |
|---|---|---|---|---|---|---|---|---|---|
| Bulk density (g/cm$^3$) | 0.16 | 0.13 | 0.19 | 0.22 | 0.19 | 0.16 | 0.21 | 0.22 | 0.20 |
| Yield (%) | 11.85 | 12.00 | 13.00 | 13.00 | 13.60 | 14.8 | 12.00 | 14.00 | 13.60 |
| SBET (m$^2$/g) | 348.85 | 348.97 | 222.53 | 352.28 | 552.24 | 475.45 | 384.05 | 823.81 | 584.80 |
| Vmi (cm$^3$/g) | 0.13 | 0.10 | 0.08 | 0.14 | 0.25 | 0.21 | 0.18 | 0.38 | 0.25 |
| Vme (cm$^3$/g) | 0.11 | 0.15 | 0.05 | 0.08 | 0.05 | 0.05 | 0.06 | 0.12 | 0.10 |
| Vt (cm$^3$/g) | 0.24 | 0.25 | 0.13 | 0.22 | 0.30 | 0.26 | 0.24 | 0.50 | 0.35 |
| Porosity (%) | 54.17 | 40.00 | 61.54 | 63.63 | 83.33 | 80.77 | 75.00 | 76.00 | 71.43 |

volume ($V_{0.99}$) was calculated by converting the amount of $N_2$ adsorbed at a relative pressure ($P/P_o$) of 0.99 to liquid volume of the adsorbate ($N_2$)[15]. Total pore volume is the sum of the volume of micropore and mesopore[35]. Results showed that pore volumes were generally increased with the increasing temperature.

The experimental conditions of the chemical activation in activated carbon preparation can be largely changed. There are many parameters that can affect the activation process, hence, the resulting activated carbon. However, the most important factors that mainly affect the activated carbon preparations are both activating agents and starting materials. As mentioned by Li *et al.*[19], even the same starting material could result in different characteristics in the resulting activated carbon. Different mechanisms also involved in different activating agents used. Okada *et al.*[36] had reported that BET values were also different for different activating agents which were alkali based. The author suggested that the differences were attributed to the ionic radii of the activating used. Baccar *et al.*[37] had also stated that the concentration of activating agents, impregnation ratio and temperature generally influence the activated carbon properties. Subsequently, those properties could determine its best possible applications. Studies have shown that the development of porosity depends on the temperature at which the char was produced[38].

Fig. 2 shows the $N_2$ adsorption isotherms of DCR-AC prepared. The standard classifications of isotherms are as followed; Type I isotherm means there is an enhanced interactions of adsorbent-adsorbate in micropores of molecular dimensions. The narrow range of relative pressure that required to attain the plateau means there is a limited pore size range. In Type II isotherm, it occurs due to the formation of an adsorbed layer in which its thickness increases continually with the increased of relative pressure. This is generally obtained with non-porous or macroporous adsorbent with the complete reversal of the isotherm at higher relative pressure. Type III isotherm indicates there is a weak interactions between adsorbent and adsorbate. Type IV is basically related to Type II isotherm. However, in Type IV there is a hysteresis loop which related to the filling and emptying of mesopores by capillary condensation. In Type V isotherm, it closely related with Type III isotherm but it exhibits hysteresis loop which indicates pore filling and emptying mechanism. Finally, Type VI isotherm is related with adsorption of layer-by-layer on a highly uniform surface and basically depending on temperature[8]. From Fig. 2, it was observed that all DCR-AC exhibited Type I isotherms with an almost horizontal plateau at higher relative pressures. This indicated highly microporous materials with a narrow pore size distribution. Some of the isotherms indicated an increasing slope at values of $P/P^o > 0.9$. This shows there was a development of the wider pores[39, 40].

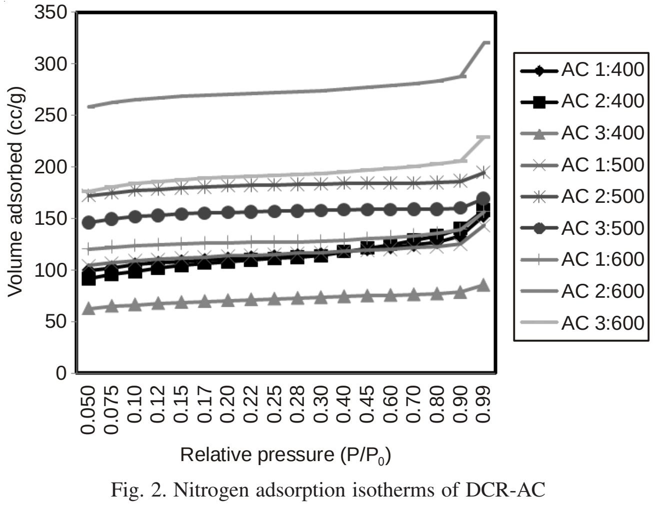

Fig. 2. Nitrogen adsorption isotherms of DCR-AC

**Effect of temperature:** Fig. 3 shows the effect of temperature on the porosity of the DCR-AC. It shows that the increased in temperature from 400 to 600 °C, the BET surface area also increased for all types of impregnation ratio. The increased in temperature caused the increased in reaction between char and KOH, hence creating the new pores[16]. Temperature has been identified as a determinant factor in the structure of the activated carbon[41] and hence influencing the creation of the optimum surface area[42]. The carbonization of the starting material which results in char production basically has low porosity and disordered structure. Subsequently, activation with KOH would further develop the porosity. Carbonization removes the volatile matter in the desiccated coconut residue, whereas activation with chemical agents would produce a well developed porosity of activated carbon[41]. Mohamed *et al.*[43] reported that as temperature increased, the diameter of the sample decreased and causes the micropore volume to be developed. The total pore volume ($V_t$) were basically increased with the increased of activation temperature. It indicated that at higher temperature, a well-developed porosity was achieved[39]. Results also showed that KOH-activating agent has shown to have microporous structure with high surface area[31].

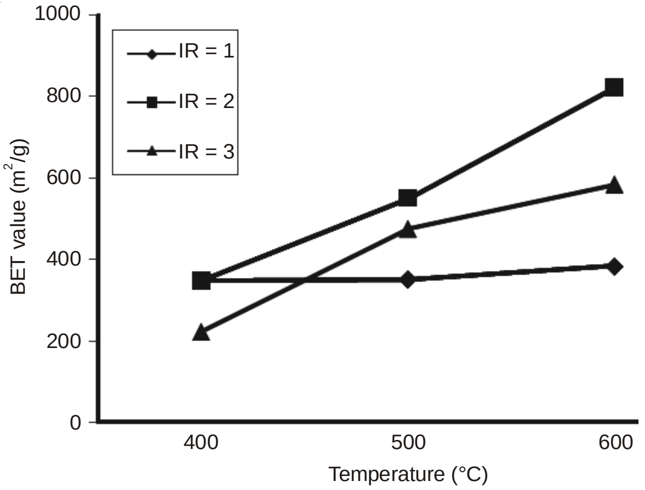

Fig. 3. BET values as a function of temperature

**Effect of impregnation ratio:** Impregnation ratio is defined as the amount of activating agents that incorporated into the desiccated coconut residue sample[16]. Fig. 4 shows the effect of impregnation ratio on the porosity of the DCR-AC. It shows that, the BET surface area increased from ratio 1:1 to 1:2 and then decreased at impregnation ratio of 1:3. This trend has also been reported by Azargohar and Dalai[44]. This phenomenon was attributed to the widening of the micropores in the DCR-AC with the increased of the impregnation ratio.

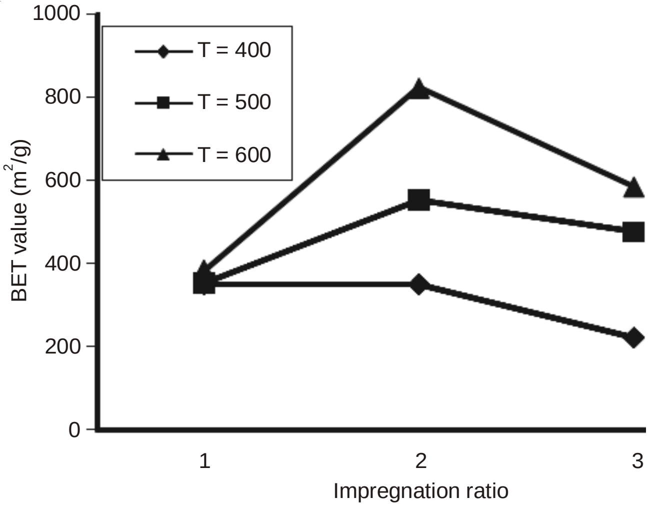

Fig. 4. BET value as a function of impregnation ratio

**Effect of temperature on porosity:** Fig. 5 showed the effect of temperature on the porosity of the DCR-AC. Porosity is defined as the ratio of micropore volume to total per volume[11]. With the exception of impregnation of 1:1, results showed that DCR-AC porosity was initially increased from 400 to 500 °C and finally decreased at 600 °C. The initial increased in porosity was attributed to the released of the volatiles matters. As temperature increased there were sintering and shrinkage of the pore structures of the DCR-AC hence decreasing the porosity[34].

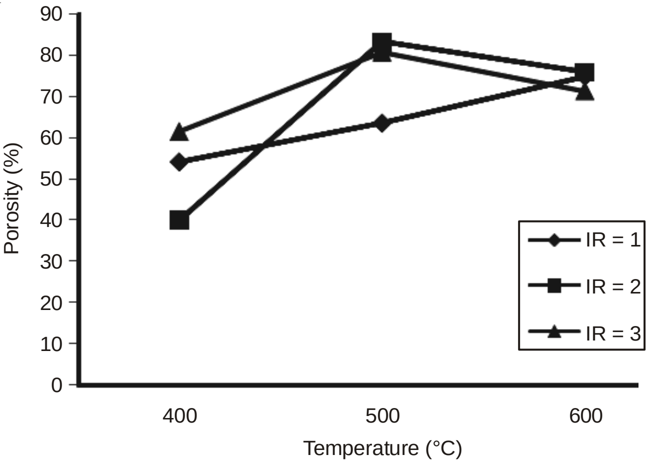

Fig. 5. Effect of temperature on particle porosity at different IR value

**SEM morphology of DCR-AC:** Fig. 6 showed the SEM morphology of the selected DCR-AC with impregnation ratio of 1:2 for all temperatures studied[45]. Results showed that DCR-AC had a wide variety of pores with honey-comb look like structure. There were also full of cavities at the external surface of the DCR-AC[46].

## Conclusion

Activated carbon from desiccated coconut residue was prepared in this study. Chemical activation by using potassium hydroxide produces activated carbon with high BET surface area and porosity. BET surface area was basically affected by temperature and impregnation ratio of the KOH.

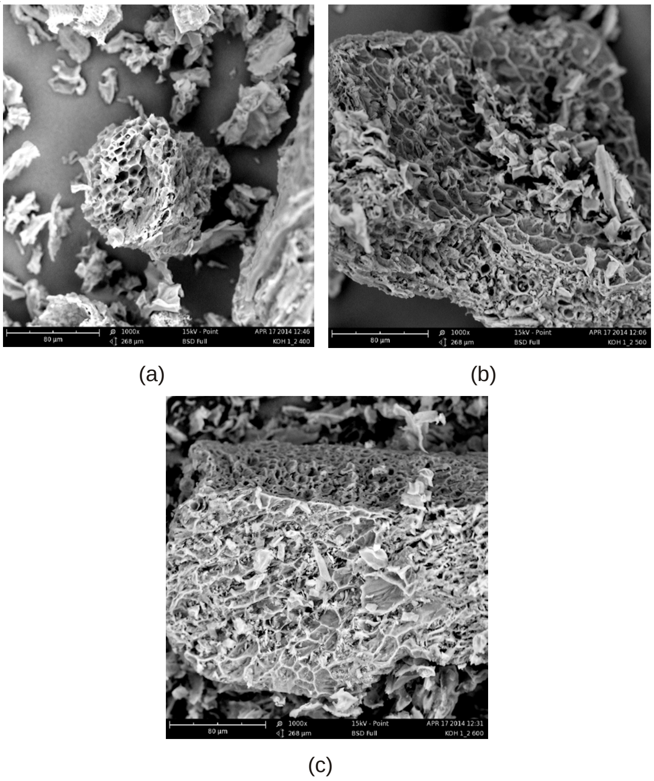

Fig. 6. SEM micrographs of DCR-AC, (a) A 2:400, (b) A 2:500, (c) A 2:600

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