Preparation of Activated Carbon from Desiccated Coconut Residue by Chemical Activation with NaOH

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Abstract

This study investigates the effect of temperature and impregnation ratio on the physicochemical properties of activated carbon prepared from agricultural waste; desiccated coconut residue (DCR) by chemical activation using sodium hydroxide (NaOH). DCR sample was first carbonized at three different temperatures for 1 hour at 400°C, 500°C, and 600°C respectively. The resulting chars were impregnated with NaOH at three different impregnation ratio; 1:1, 1:2, and 1:3 respectively and activated under nitrogen atmosphere for 1 hour at three different temperatures based on its carbonization temperature. The specific surface area was strongly affected by impregnation ratio in which increased with impregnation ratio. The specific surface area also increased with temperature but then decreased at highest desired temperature.

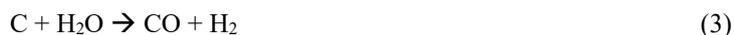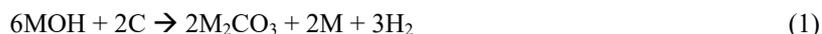**Keywords:** desiccated coconut residue, activated carbon, sodium hydroxide

1. Introduction

Activated carbon is a kind of amorphous carbonaceous material with high porosity and surface area. The production of activated carbon can be divided into two types: physical activation and chemical activation. Physical activation is a two step process which involves carbonization and followed by gasification by an oxidizing agent (typically CO_2 and steam). Meanwhile, chemical activation refers to a single step process in which the carbonization and activation process occurs simultaneously. The most common chemical agents used are $ZnCl_2$, H_3PO_4, KOH and NaOH (ElShafei et al., 2014). The huge cost of the production of activated carbon has been the most challenging parts for commercial manufacturers. Therefore, the use of cheap raw materials with high contents of carbon as well as low inorganic compounds has been a focus of research efforts in the last decades (Yahya et al., 2015). Agricultural by-products and waste materials such as coconut shell (Olafadehan et al., 2012), wood (Choy et al., 2005), and rice husk (Yalcin & Sevinc, 2000) are among the low-cost precursors for activated carbon production. The key criteria in selecting a carbonaceous material as activated carbon precursor includes low inorganic content, low cost, workability of the material, minimum impact upon environmental, and its storage life (Choy et al., 2005). A good precursor for activated carbon should also have high carbon content (Hiremath et al., 2012). Waste materials can be divided into two categories; conventional wastes and non-conventional waste. Conventional wastes refer to waste from agricultural and wood industry, such as coconuts and olives whereas, non-conventional waste refer to waste from municipal and industrial activities such as polyethyleneterephthalate (PET) and polyvinyl chloride (PVC) (Dias et al., 2007). With this respect, our precursor known as desiccated coconut residue (DCR); a by-product of coconut industry that obtained from the extraction of milk is categorized as conventional wastes which fulfill the criteria desirable for activated carbon precursor. It is believed that the use of chemical activation in activated carbon preparation has been more frequent for conventional waste (Dias et al., 2007).

DCR was selected in this study to prepare activated carbon as it is inexpensive, even no cost required to obtain the material. This conversion of DCR into activated carbon would increase its economic value, reducing the disposal cost of the material and provide cheap material for activated carbon preparation (Prahas et al., 2008). Preparation of activated carbon by alkaline hydroxide activation basically involve two steps; carbonization of the precursor under an inert atmosphere followed by activation of the char impregnated with activating agents (Xu et al., 2010). Consequently, in this study the raw DCR was first carbonized into the char prior to activation with NaOH for

activated carbon productions. It suggests that the use of NaOH is very effective for activation of the char of the materials (Prahas et al., 2008).

Activation mechanisms for NaOH comprise the reaction of carbon material with the hydroxides. Carbon molecules depletion from char network gives rise to development of porosity. The activation mechanisms are commonly complex. The reaction mechanisms have been proposed as follows (Hu et al., 2013):

$$6MOH + 2C \rightarrow 2M_2CO_3 + 2M + 3H_2 \tag{1}$$

$$2MOH \rightarrow M_2O + H_2O \tag{2}$$

$$C + H_2O \rightarrow CO + H_2 \tag{3}$$

$$M_2CO_3 + 2C \rightarrow 2M + 3CO \tag{4}$$

$$M_2CO_3 \rightarrow M_2O + CO_2 \tag{5}$$

$$M_2CO_3 + C \rightarrow M_2O + 2CO \tag{6}$$

$$2M + CO_2 \rightarrow M_2O + CO \tag{7}$$

$$M_2O + C \rightarrow 2M + CO \tag{8}$$

Where, $M = Na^+$

Our previous study (Yahya et al., 2015b) has shown that DCR was successfully utilized for activated carbon preparation using potassium hydroxide (KOH) as an activating agent. In this study we used sodium hydroxide (NaOH) as an activating agent instead of KOH. It is hoped that the research would provide a cheap material for the preparation of activated carbon.

2. Experimental

2.1 Activated Carbon Preparation

The preparation of the activated carbon was done following our previous study (Yahya et al., 2015b) except for NaOH. The DCR was collected from local market in Nilai and was dried for 24 hours and then ground to powder with kitchen grinder to attain particle size of 1 to 2 mm. DCR was first carbonized for 1 hour at three different temperatures (400°C, 500°C, and 600°C) under N_2 flow (300 ml min^{-1}) and was cooled to room temperature. The DCR-char was then impregnated with NaOH (Merck, Germany) at three different ratios (1:1, 1:2, and 1:3) at room temperature for 1 hour duration. The impregnated char was then dried in an oven overnight at 105°C, followed by heating for 1 hour at three different temperatures which based on its char production temperature, respectively. The resulting activated carbon was then washed with 1 M HCl and followed by plenty of deionized water to remove any residual organic and mineral matter. The DCR-AC was then dried at 110°C overnight, before being cooled and stored for further studies. The samples were abbreviated 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).

2.2 Proximate and Ultimate Analysis

Proximate analysis was done by using a thermogravimetric analyser (Mettler Toledo TGA-DSC STAR^e system). The ultimate analysis was done to determine the existence of the carbon, hydrogen, nitrogen, and oxygen was determined by CHNS analyzer (Thermo Finnigan, USA).

2.3 Bulk Density Determination

The bulk density was determined following Al-Qodah & Shawabkah (2009). The bulk density was calculated on the dry basis as in Equation (9):

$$\text{Bulk density} = \text{weight of sample (g)/volume of sample (cm}^3) \tag{9}$$

2.4 Physical Properties of DCR-AC

The morphology of the resulting activated carbon was studied using a Scanning Electron Microscope (Phenom, G2 pro) operated at 15 kV. The magnifications were selected at x 1000.

2.5 Nitrogen Adsorption Measurements

The specific surface area (SSA) of the DCR-AC, micropore volume and mesopore volume was studied by N_2 adsorption at -196°C (77 K), using an automatic volumetric sorption analyzer (Quantachrome, Autosorb 2). The instrument was equipped with Dewar flasks to hold the coolant (liquid nitrogen), analysis stations to hold the sample cell during analysis, sample cells with glass filler rods to put the sample inside the glass, cold trap to prevent the diffusion of sample degassing products into the manifold and heating mantles to outgas the sample.

DCR-AC was first degasified at 350°C for a certain period (more than 5h) prior to analysis. In the analysis period, the adsorbate was introduced to the manifold. The valve of the sample cell was opened to allow the adsorbate to interact with DCR-AC samples. The pressure was repeatedly measured until a stable reading was achieved.

The Brunauer, Emmett and Teller (BET) method was used to measure the SSA of DCR-AC. The equation of BET equilibrium adsorption was based on the assumption of multi-layer adsorption, as following Equation (10) (Devnarain et al., 2002):

$$\frac{1}{W[\left(\frac{P_o}{P}\right)-1]} = \frac{1}{CW_m} + \frac{C-1}{CW_m}\left(\frac{P}{P_o}\right) \tag{10}$$

Where, W represents weight of nitrogen adsorbed at a relative pressure, P/P_o, W_m is the weight of adsorbate that constitutes a monolayer of surface coverage, and C is a constant related to adsorption energy in the first adsorbed layer in which its value indicates the magnitude of the adsorbent/adsorbate interactions.

The volume adsorbed was based on the Equation (11):

$$\frac{V}{V_m} = \frac{C(P/P_o)}{[1-P/P_o][1+(C-1)(P/P_o]} \tag{11}$$

The quantity of N_2 gas adsorbed in monolayer was determined by plotting P/P_o vs $1/V[P/P_o)-1]$. The SSA was determined by following Equation (12):

$$S = \frac{[\sigma N_A]\rho V_m}{MW} \tag{12}$$

Where, σ denotes the adsorbate molecule area, ρ is the liquid density of adsorbate, N_A is the Avogadro's number (6.022×10^{23} number/mole) and MW signifies molecular weight of adsorbate molecule (nitrogen). The values of BET surface area (S_{BET}) (also known as SSA) were calculated from the adsorption isotherms at the range of $0.05 \leq P/P_o \leq 0.30$ (Okada et al., 2003). Micropore volume was obtained by t-plot method. Total pore volume was obtained at 0.99 of relative pressure (Hu et al., 2001).

3. Results and Discussion

3.1 Proximate and Ultimate Analysis

The proximate analysis of raw DCR has been reported by our previous study (Yahya et al., 2015b) which showed that the contents of the moisture, volatile matter, ash, and fixed carbon were 3.0 %, 84.20 %, 5.51 %, and 7.29 %, respectively. The high value of volatile matter and low value of ash content in DCR indicated that it was suitable for the biomass to be converted into activated carbon. The ultimate analysis of the DCR and DCR-AC was shown in Table 1. The carbon contents of the activated carbon were generally higher than that of raw materials. It was observed that the carbon contents were decreased with impregnation ratio for all types of temperature which may be attributed to the carbon monoxide that was produced during activation (Choy et al., 2005).

Table 1. Ultimate analysis of DCR and DCR-AC

Ultimate analysis (%)	DCR	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	63.24	53.07	46.00	74.93	65.80	53.34	75.25	72.55	65.70
Hydrogen	12.80	1.83	2.11	1.82	1.70	1.83	3.35	1.21	1.64	2.70
Nitrogen	0.19	3.23	2.48	2.42	3.19	2.12	0.06	2.24	2.28	0.20
Oxygen*	37.53	31.70	42.34	50.24	20.18	30.25	43.25	21.30	23.83	31.40

* Oxygen was calculated by difference.

3.2 Yield (%) of DCR-AC

Table 2 shows yield percentage of the resulting DCR-AC. It was observed that the yield of the DCR-AC in the range 11.20 to 14.63 (%). The low yield of the activated carbon was expected due to low lignin contents in the raw DCR hence resulted in low yield (Yahya et al., 2015b).

3.3 Bulk Density

Bulk density is one of the important characteristics of AC as it is a measure of the amount of the adsorbate that the carbon can hold per unit volume (Abechi et al., 2013). Table 2 shows the bulk density of the DCR-AC. Bulk density of raw DCR was found to be 0.24 g/cm^3. It was found that the bulk density of the DCR-AC was found between 0.16 and 0.21 g/cm^3. The lower bulk densities of DCR-AC compared to the raw DCR were attributed to the porosity development during activation process (Lima & Marshall, 2005).

3.4 Surface Area, Pore Size Distribution and Porosity of DCR-AC

Figure 1 shows the N_2 adsorption isotherms of DCR-AC prepared. All isotherms adsorption exhibited Type I isotherms with horizontal plateau at higher relative pressures. The isotherms showed that all DCR-AC indicated highly microporous materials with a narrow pore size distribution except for AC 1:400, AC 2:400 and AC 3:400. 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 (Demiral et al., 2008). From Table 2, the SSA obtained from N_2 adsorption ranged from 27.15 to 1394.79 m^2/g. The total pore volume was between 0.05 to 0.78 cm^3/g. The SSA was in the order: AC3:500 > AC3:600 > AC2:500 > AC2:600 > AC1:500 > AC1:600 > AC2: 400 > AC3:400 > AC1:400. The total pore volumes were ranged from 0.05 to 0.78 cm^3/g. The SSA values corresponding to all samples were plotted in Figure 2.

Table 2. Physicochemical properties of 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
Yield (%)	13.05	15.28	14.54	14.63	12.31	11.20	12.48	11.60	11.52
Bulk density (g/cm^3)	0.17	0.20	0.16	0.17	0.19	0.18	0.20	0.21	0.20
S_{BET} (m^2/g)	27.15	112.94	65.53	244.20	702.98	1394.79	189.68	563.71	1199.73
V_{mi} (cm^3/g)	0.00	0.03	0.02	0.09	0.31	0.60	0.08	0.26	0.52
V_{me} (cm^3/g)	0.05	0.10	0.04	0.05	0.10	0.18	0.04	0.06	0.16
V_t (cm^3/g)	0.05	0.13	0.06	0.14	0.41	0.78	0.12	0.32	0.68
Porosity (%)	0.00	23.08	33.33	64.29	75.60	76.92	66.67	81.25	76.47

S_{BET} = BET surface area, V_{mic} = micopore volume, V_{mes} = mesopore volume, V_t = total pore volume.
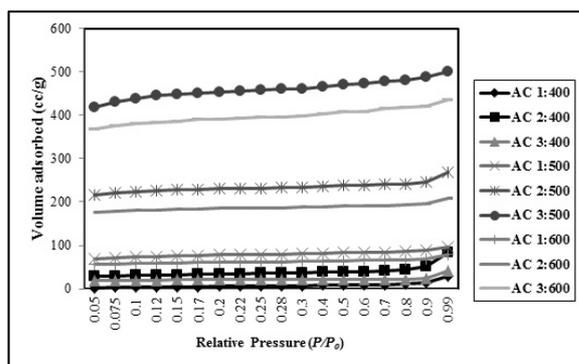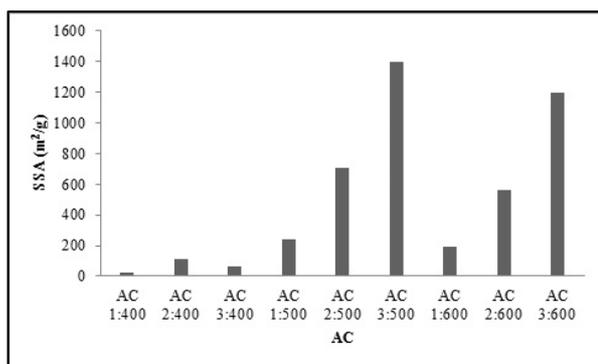

Figure 1. N_2 adsorption isotherms of DCR-AC

Figure 2. SSA corresponding to all activated carbon prepared

3.5 Effect of Temperature

Figure 3 shows the effects of temperature on the SSA of DCR-AC. It was observed that the SSA for DCR-AC was significantly increased with the increased of temperature from 400 °C to 500 °C and then decreased at 600 °C for impregnation ratio from 1 to 3. It was reported that the initial increased of the SSA was attributed to the increased in the reaction between DCR-char with the chemical agent (NaOH) during activation process hence developing new pores. However, the increased of temperature from 500°C to 600°C caused widening of the micropores thus the SSA to be decreased. Xu et al. (2010) reported this situation was due to mechanism that involved in NaOH activation. Some of active sites in the char were selectively etched by NaOH at high temperature to generate porous structure. During carbonization, the carbon contents in the char were increased and the porous structure was built by eliminating non-carbon atoms. The crystal structure in the char was rearranged resulted in well-ordered structure. These rearrangements obstructed the subsequent activation reactions of the char with NaOH to generate

porous structure. Therefore, the higher the temperature resulted in more ordered of the crystal structure of the char and became more difficult to be etched by NaOH. This caused the SSA and pore volume of NAC to decrease with increasing temperature. Results also showed that at temperature of 400°C, NAC had shown very low SSA. This finding was in agreement with a study on preparation of AC from rice husk by Isoda et al. (2014) in which the author also reported that this phenomenon was attributed to the mechanism involved in NaOH activation. NaOH was first reduced to Na^+ and introduced into the structure of the char and expanded it. With the increased in temperature, Na^+ was moved forcefully separating the grapheme type-layers hence creating the porosity. The reaction was severely a function of temperature, therefore at low temperature (400°C) it was not effective for the formation of AC.

3.6 Effect of Impregnation Ratio

Figure 4 shows the effects of impregnation ratio on the SSA of the DCR-AC. It was observed that the SSA was significantly increased with impregnation ratio from 1:1 to 1:3 except for all temperatures except for 400°C. The pore volume was also increased with the increased of the impregnation ratio. It has been reported that the particle size of the resulting activated carbon could change depending on the impregnation ratio, hence increased the SSA and pore volume of the DCR-AC. In this study, activating agent (NaOH) may react as templates for porosity development in activated carbon (Molino-Sabio & F. Rodriguez-Reinoso, 2004). Our previous study using KOH had shown quite different findings in which SSA was increased from impregnation ratio of 1:1 to 1:2 and finally decreased at impregnation ratio of 1:3. We have reported that this situation was attributed to the initial template reaction by KOH and widening of micropores that caused the increased and decreased of SSA, respectively. These differences indicate that different activating agents could promote different mechanisms which could probably attributed to the ionic radii differences and concentration of both types of activating agents.
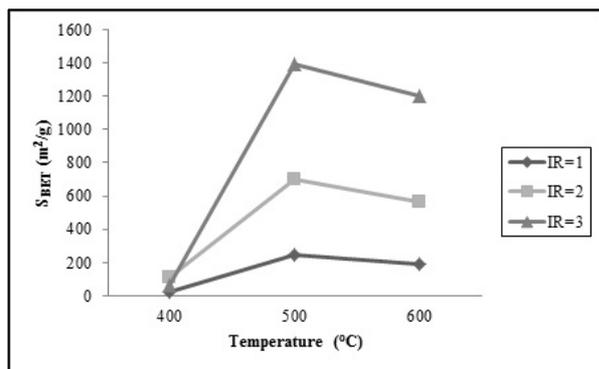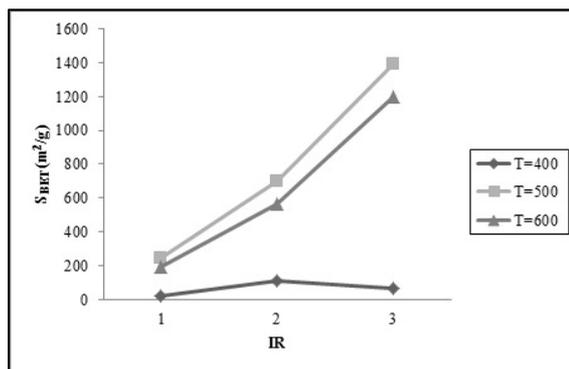

Figure 3. SSA value as a function of temperature

Figure 4. S_{BET} value as a function of impregnation ratio

3.7 Effect of Temperature on Porosity

Figure 5 shows the effect of temperature on the porosity of the DCR-AC. In general, porosity of the DCR-AC was increased from 400°C to 600°C for all types of IR. Porosity is defined as the ratio of micropore volume to total per volume (Boonpoke et al., 2011). Temperature has been identified as a determinant factor in the structure of the activated carbon. Carbonization of the starting material (DCR) which resulted in char production basically had low porosity and disordered structure. Subsequently, activation would further develop the porosity. Carbonization removed the volatile matter in the DCR, whereas activation with chemical agents (NaOH) produced a well developed porosity of DCR-AC (Ketcha et al., 2012). As temperature increased, the diameter of the sample also decreased and resulted in the development of the micropore volume (Mohamed et al., 2010).

3.8 SEM Morphology of DCR-AC

Figure 6 shows the SEM morphology of the selected DCR-AC for all temperatures studied. It was observed that DCR-AC had a wide variety of pores with honey-comb look like structure. There were cavities and regular at the external surface of the DCR-AC. Results also showed that NaOH played an important role in porosity developments. It was also found that all selected DCR-AC had homogenous types of pore structure distribution. In summary, the activation process with NaOH was effective in creating well developed pores on the DCR-AC surfaces.
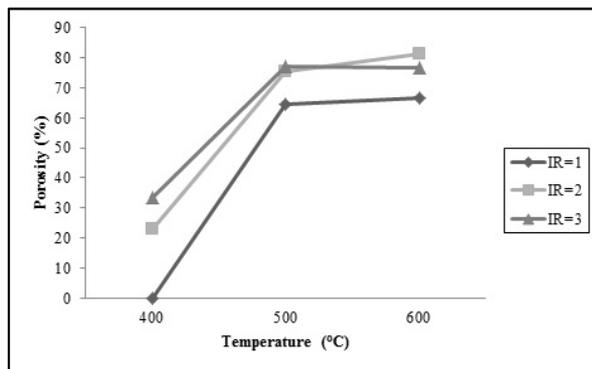
Figure 5. Effect of temperature on particle porosity at different IR value
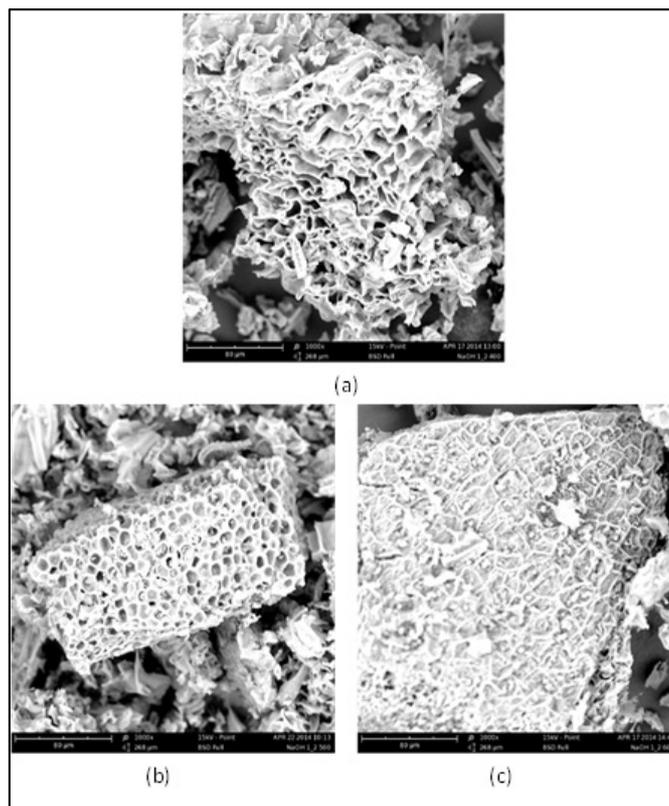

Figure 6. SEM micrographs of DCR-AC, (a) A2:400, (b) A2:500, (c) A2:600

3.9 Micropore Size Distribution

The evolution of the micropore size distributions (MPSD) was observed by plotting the normalized nitrogen adsorption isotherms. The curves were acquired by dividing the amount of N_2 adsorbed at a given relative pressure by the amount of N_2 adsorbed at a relative pressure near to saturation pressure (0.99) (Lozano-Castello et al., 2003). Figure 7 shows the plot of normalized N_2 adsorption for all DCR-AC. It was observed that the narrowest MPSD for NAC correspond to AC 3:500, AC 3:600 and AC 2:500. These results generally were in agreement with results found in Table 2 associated with the micropore volumes. Figure 8 shows the effects of micropore contributions for DCR-AC as a function of impregnation ratio and temperature studied. These micropore contributions were calculated by dividing the amount of N_2 adsorbed at relative pressure of 0.1 to amount of N_2 adsorbed at relative pressure of 0.95 (Ahmadpour & Do 1997). Results showed that at temperatures of 500°C and 600°C for all types of impregnation ratio, the microporosity contributions had higher values as compared to temperature of 400°C. As previously mentioned in this study it showed that temperature has been important factors that could determine microporosity development in DCR-AC.
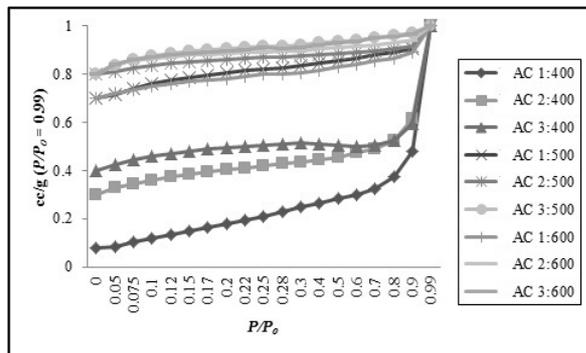
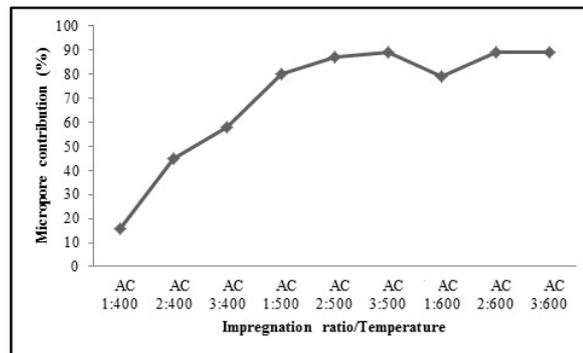Figure 7. Normalized N_2 adsorption isotherms at 77 K for NAC

Figure 8. Effects of impregnation ratio and temperature on the relative micropore contribution of DCR-AC

4. Conclusion

A low cost activated carbon from waste material can be converted into activated carbon which a value added product. Chemical activation by using sodium hydroxide produces activated carbon with high porosity.

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