



Preparation and Characterization of Activated Carbon from Kurdistan Walnut Shell Activated by NaOH

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ABSTRACT

Kurdistan walnut shell (KWS) was examined for preparation of activated carbon using sodium hydroxide as chemical agent. The characterisations of the Kurdistan walnut shell activated carbon by sodium hydroxide (WSAC-NaOH) were studied using different techniques. pH of point of zero charge (pH_{pzc}), iodine number and proximate analysis. The adsorption of methylene blue by prepared activated carbon was analyzed by the Langmuir and Freundlich adsorption isotherms. The data fitted well to the Freundlich isotherm with the maximum adsorption capacity = 344.8 mg/g for Langmuir adsorption isotherm. The analysis showed that the activated carbon derived from walnut shell activated by sodium hydroxide is more capable than commercial activated carbon (CAC) and can be used as a possible adsorbent.

1. INTRODUCTION

Activated carbon is a porous carbonaceous material, whether in a powder or granular form. It is a structure consisting of a network of interconnected micropores, mesopores and macropores that demonstrates a good ability for the adsorption of organic molecules because of its high surface area (Arvind and Hara, 2016).

However, its applications are limited due to high cost. To provide economic solution, the use of low cost of agricultural by-products has been shown to produce activated carbon. Different agricultural by-products such as bean pod (Cabal et al., 2009), almond shell (Demirbas et al., 2008), cherry stone (Jaramillo et al., 2009), rice husk (Sahu et al., 2009), date

palm seed (El Nemr et al., 2008), bamboo (Hameed and El-Khaiary, 2008), sunflower seed hull (Thinakaran et al., 2008), coconut husk (Tan et al., 2008a), and waste apricot (Onal et al., 2007) have been used for preparation of activated carbons. In addition, few studies have been reported in the literature on the use of walnut shell using as agricultural waste-based activated carbon.

Walnut shell is one of the common agricultural by-products with large quantity in Kurdistan. Annually several tons of walnut shells are produced in Kurdistan. Including compounds of walnut shell are lignin, hemicellulose, and cellulose with low ash and a high carbon content which renders it a

appropriate precursor for activated carbon production. Walnut shell because of its availability as a renewable resource and high rigidity has been effectively used for activated carbon production and its use in removing pollutants by adsorption has been studied (Srinivasan and Viraraghavan, 2008; Ayrimis et al., 2013).

In the impregnation process, the precursor is mixed with an amount of chemical agents such as KOH (Ji et al., 2007; Feng-Chin et al., 2005), NaOH (Cazetta et al., 2011), H₃PO₄ (Yorgun and Yildiz, 2015) and ZnCl₂ (Pezoti et al., 2014) which cause the development of porous structures in the raw material. In this study, activated carbon has been produced by using a physiochemical activation method from Kurdistan walnut shell using sodium hydroxide as chemical agent.

2. MATERIALS AND METHODS

2.1. Preparation of the Activated Carbon

The precursor for the preparation of activated carbon was walnut shells were collected from local markets in Erbil, Iraq. To remove impurities, the raw material was washed with hot distilled water and dried. The raw material was cut into small pieces and then sieved to an average particle size of 2.5 – 2.0 mm. Sixty grams of raw material was impregnated in 10 % chemical agent (NaOH) at 1: 2 ratio (w/v) and heated in a water bath at 85-90 °C for 24 h. The raw material was separated from the solution, washed with distilled water until the solution became neutral, and then dried in an oven overnight at 100 °C. The sample was then placed in a stainless steel vertical tubular reactor and put in the furnace. The process of carbonization was conducted under high purity nitrogen gas (99 %) at a flow rate of 150 cm³/ min with a

heating rate to 400 °C under the pressure of 1 atm and held for 2 h. The product was cooled to room temperature (25 °C), washed with distilled water, and then dried in an oven at 100 °C.

2.2. Characterization of Adsorbent

2.2.1. Iodine value test

The adsorption of aqueous iodine is measured a simple test for estimating the surface area of activated carbons. However, the relationship between surface area and iodine number cannot be generalized. It varies with changes in raw material of carbon and pore volume distribution. 0.1 g of dry commercial activated carbon and prepared activated carbon was separately taken in dried 100 ml conical flasks. The samples were run in duplicates and added 5 ml of 5% HCl. The flasks were shaken until the carbon was dumped. 10ml of 0.1N iodine solution was added to each flask and was shaken properly for 4 minutes. 10 ml filtrate was titrated against standard (0.1N) sodium thiosulphate solution using starch as an indicator. The concentration of iodine adsorbed by activated carbon was calculated as amount of iodine adsorbed in milligrams. The used solution in this test was performed according to American Society for Testing and Materials Standard (ASTM, 2006).

2.2.2. Methylene test

Methylene blue adsorption tests were conducted by mixing 0.1 g of each, WSAC and CAC separately with 100 ml of 25, 50, 75 and 100 mg L⁻¹ of methylene blue solution. After shaking for 24 hours, the solutions were filtered and methylene blue residual concentration was measured at 660 nm using UV /Vis spectrophotometer for the determination of concentration. The amount of adsorption at equilibrium, q_e (mg/g) was

calculated through the follow equation (Alam et al., 2007) :

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (1)$$

where C_0 and C_e (mg/g) are the initial and equilibrium concentrations of methylene, respectively, V (L) is the volume of the solution, and W (g) is the mass of dry adsorbent used. The percentage removal of methylene was calculated using the following equation (Azam et al., 2009) :

$$\text{Removal (\%)} = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (2)$$

2.2.3. Determination of moisture content

1.0 g of each of WSAC and CAC were taken in dried and weighed crucibles separately. The crucibles were kept in an oven held at a temperature of 105 °C for 4 hours. The crucibles were put in a desiccator for 1 hour and weighed. For determination of moisture content ASTM–D1762 (1990) was used. The percentage of moisture content was measured as follows:

$$\text{Moisture (\%)} = \left[\frac{(A - B)}{A} \right] * 100 \quad (3)$$

Where A = weight of dried sample, B = weight of the sample after drying at 105 °C

2.2.4. Determination of ash content

1.0 g of each of WSAC and CAC were taken in dried and weighed crucibles separately. The samples were run and put in a muffle furnace at 750 °C for 6 h. The crucibles were cooled in a desiccator and weighed. The percentage of ash content was calculated as follows:

$$\text{Ash (\%)} = \frac{D}{B} * 100 \quad (4)$$

Where D = weight of residue left in gram, B = weight of dried sample in gram.

2.2.5. Determination of pH

For determine pH of WSAC and CAC, the standard test method ASTM-D3838 was utilized (ASTM, 1996). 1.0 g of WSAC and CAC were transferred into 100 ml distilled water taken in a beaker and stirred by a magnetic bar for 1 h separately. pH meter was used for measuring pH.

2.2.6. Determination of pH of point of zero charge (pH_{pzc})

pH_{pzc} of activated carbon is significant because it shows the net surface charge of the carbon in the liquid phase. The pH_{pzc} is the point where the curve of pH_{final} vs $\text{pH}_{\text{initial}}$ intersects the line $\text{pH}_{\text{initial}} = \text{pH}_{\text{final}}$. In order to determine the pH of point of zero charge 0.15 g of WSAC and CAC was taken to eleven 100 ml conical flasks containing 50 ml of 0.01M NaCl separately, whose initial pH has been measured and adjusted with NaOH or HCl for each activated carbon. The conical flasks were sealed and placed in a shaker for 24 hours after which the pH was measured.

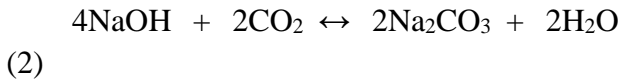
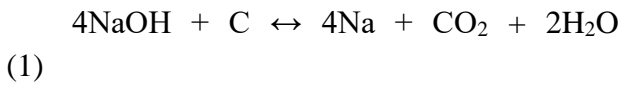
3. RESULTS AND DISCUSSION

3.1. Investigation of sorption parameters

3.1.1 Effect of NaOH

The addition of NaOH solution to the carbon structure creates the feasibility of reactions with all four elements concerned, namely the carbon, the hydrogen, the oxygen and the sodium. The products of these reactions include sodium metal, water, carbon dioxide

and sodium carbonate. Possible general reactions, for NaOH are as follows:



The mechanism of surface reactions will be much less complicated than indicated by these general reactions which can never take place as written. Rather, the processes will take place through a series of much simpler, simultaneous/consecutive reactions. The first state of dispersion of the NaOH is significant to maximize the contact between the carbon surface and NaOH (Marsh and Rodriguez-Reinoso, 2006).

3.1.2. Surface characterization of activated carbon

The low moisture and ash content showed in Table 1 revealed that the raw material is capable for the preparation of activated carbons. A small increase in ash content causes a reduce in adsorptive properties of adsorbent. Thus, the activated carbon content of low ash better for use in adsorption process (Quershi et al., 2007). The activated carbon prepared from the raw materials of walnut shell can be used as a good adsorbent instead of commercial activated carbon. The pH of WSAC and CAC was found to be 6.9 and 6.5 respectively. Activated carbon pH between 6 and 8 is suitable for most application (Ahmedna et al., 2000). pH is affected by the reaction of carbon dioxide and organic-inorganic solutes present in water. Any change in water pH is accomplished by the modification in other physicochemical parameters (Parihar et al., 2012). If the solution pH of activated carbon has greater than pH_{pzc} , the surface of the carbon will be negatively

charged and cations may be adsorbed on the surface. On the other hand, at solution pH less than their pH_{pzc} activated carbon surface is that it will have a positive charge and hence be a surface on which anion may be adsorbed. The pH_{pzc} of WSAC and CAC were found to be 6.5 and 6.2 respectively.

Iodine adsorption from liquid phase was assumed to gain further information of the porous structure and characterization of activated carbon. The adsorption of iodine is measured a simple test for assessing the surface area of activated carbons associated with pores larger than 1 nm (Benadjemia et al., 2011). Iodine number is a suggestion of the adsorption capacity in micropores; so it is often employed to study the adsorption capacity of the activated carbons by researchers. Table 1 shows the iodine number of WSAC (976 mg/g) and CAC (805 mg/g). A higher value of iodine number for WSAC in comparison to CAC is due to greater surface area and available pore sites for adsorption of iodine molecule on the surface. Adsorption is a natural process by which molecules of a dissolved compound collect and adsorb to the adsorbent solid surface (Bhise et al., 2012). The activated carbon prepared from walnut shell, it is cheap and available material can be used as activated carbon.

Scanning electron microscopy at Kurdistan Institution for Strategic Studies and Scientific Research (KISSR) was used to characterise the morphology of the adsorbent surface. Figs. 1(a) and 1(b) demonstrations the surfaces of the walnut raw material and walnut activated carbon, respectively, prepared by impregnation with NaOH solution for 24 h and a pyrolysis temperature of 400 °C. Many developed pores can be seen in Fig. 1(b) compared with Fig. 1(a), which shows a smooth surface and these pores are not present

in the walnut raw material. The development of pores is due to the effect of the NaOH concentration. The expansion of pore sizes during carbonization is essential for increasing the surface area of the activated carbon. (Tan et al., 2008b).

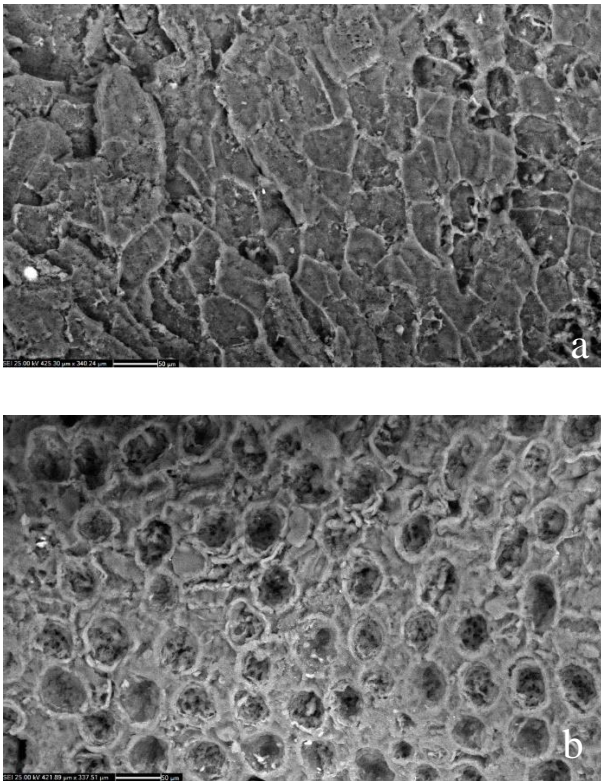


Figure1. SEM micrographs of the Kurdistan walnut shell (a) before and (b) after carbonization at 400 °C

3.2. Adsorption isotherms

In order to optimize the design of an adsorption model, it is significant to set up the most appropriate correlation for the equilibrium curves. In this study, two adsorption isotherms which are the Langmuir and Freundlich isotherms were used to describe the relationship between the amount of methylene adsorbed onto WSAC and CAC and its equilibrium concentration in the solutions.

3.2.1. Langmuir isotherm model

According to Langmuir isotherm, the adsorption occurs at homogenous sites and the nature of the adsorption is monolayer and once the adsorbate is attached on the site, no further adsorption can take place on that site (Hall et al., 1996). The linear equation of Langmuir isotherm is given in Eq. 5.

$$\frac{C_e}{q_e} = \frac{1}{q_{max}b} + \frac{C_e}{q_{max}} \quad (5)$$

where q_e and C_e are the equilibrium concentration of methylene in the adsorbed and liquid phases in mg/g and mg/L, respectively; q_{max} (mg/g) and b (L/mg) are Langmuir constants, which are associated with the maximum adsorption capacity and energy of adsorption, respectively, which can be calculated from the slope of the linear plot of C_e/q_e vs. C_e as shown in Figs. 2 and 3.

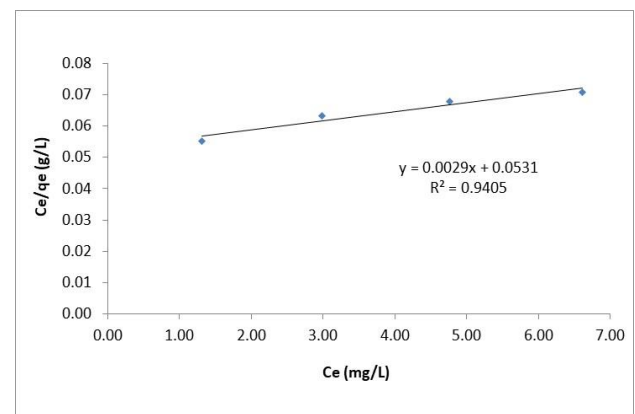


Figure 2. Langmuir isotherm for methylene adsorption onto WSAC

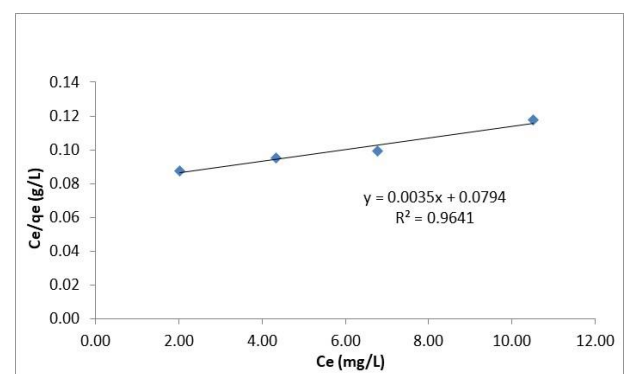


Figure 3. Langmuir isotherm for methylene adsorption onto CAC

3.2.2. Freundlich isotherm model

The Freundlich isotherm model describes the heterogeneous multilayer surfaces of adsorption due to the different trends supporting the surface sites. It supports that occupation will occur at the first stage through stronger binding, and will later decrease with the increasing degree of sites occupation (Tan et al., 2008b). The well-known logarithmic form of Freundlich isotherm is given in Eq. 6.

$$\ln q_e = \ln k_f + \frac{1}{n} \ln C_e \tag{6}$$

where C_e and q_e are the equilibrium concentrations of methylene in the liquid and adsorbed phases, in mg/L and mg/g, respectively. The K_f (mg/g) (L/mg)^{1/n} and n (L/mg) are the Freundlich constants associated with the adsorption intensity and capacity, respectively, which can be measured from the intercept and slope of the linear plot of $\ln q_e$ vs. $\ln C_e$ as shown in Figs. 4 and 5.

Table 2 show Langmuir and Freundlich constants for methylene blue adsorption onto WSAC and CAC. The values of coefficient of determination (R^2) were 0.999 and 0.994 for WSAC and CAC respectively, which is indicated that the Freundlich adsorption isotherm model was representing the adsorption for removing of adsorbate. On the other hand, the maximum adsorption capacity for WSAC was calculated for Langmuir isotherm equal to (344.8 mg/g) was higher than other adsorbents as a comparison of methylene blue adsorption onto different adsorbents as shown in Table 3.

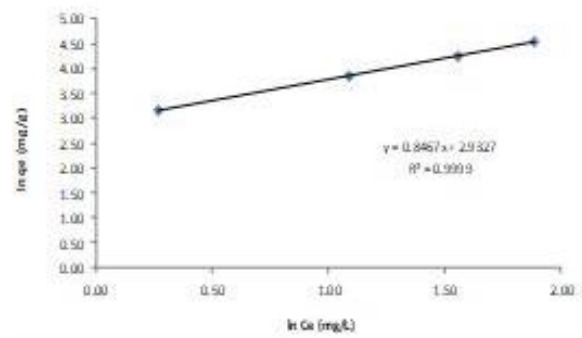


Figure 4. Freundlich isotherm for methylene adsorption onto WSAC

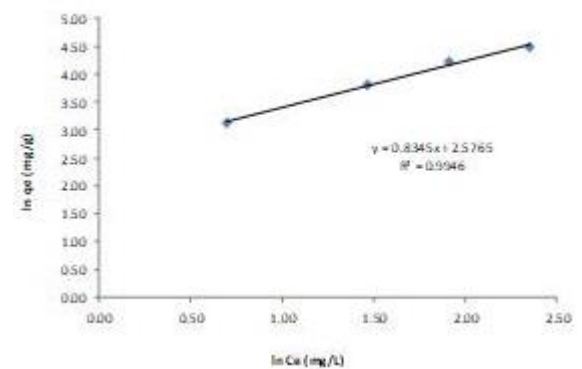


Figure 5. Freundlich isotherm for methylene adsorption onto CAC

Table 1: Physicochemical properties of WSAC and CAC

| Property | WSAC | CAC |
|--|-------|-------|
| pH (mole/L) | 6.9 | 6.5 |
| pH _{pzc} (mole/L) | 6.5 | 6.2 |
| Moisture (%) (g/m ³) | 0.21 | 0.28 |
| Ash (%) (g/m ³) | 2.37 | 2.49 |
| Iodine number (mg/g) | 976 | 805 |
| Adsorption capacity of Methylene blue (mg/g) | 344.8 | 285.7 |

Table2: Langmuir and Freundlich constants for methylene blue adsorption onto WSAC and CAC

| Adsorbent | Langmuir | | | Freundlich | | |
|-----------|----------|----------------|----------------|------------|------|----------------|
| | b | q _m | R ² | Log K | 1/n | R ² |
| CAC | 0.07 | 285.7 | 0.964 | 2.57 | 0.82 | 0.994 |
| WSAC | 0.05 | 344.8 | 0.940 | 2.93 | 0.84 | 0.999 |

Table 3: Comparison of adsorption capacities of methylene blue on the studied adsorbent with that reported in the literature

| Precursor | Adsorption capacity (mg/g) | References |
|--------------------|----------------------------|-----------------------|
| Sunflower oil cake | 16.43 | Karagöz et al., 2008 |
| Cashew nut shell | 68.72 | Kumar et al., 2011 |
| Pine wood powder | 200.00 | Wang et al., 2009 |
| Lapsi seed stone | 277.00 | Shrestha et al., 2012 |
| Bamboo | 286.10 | Liu et al., 2010 |
| Cotton stalk | 315.45 | Deng et al., 2009 |
| Walnut shell | 344.80 | Present study |

4. CONCLUSIONS

Depend on the experimental results and the related discussions, the walnut shell agricultural by-product was showed to be good raw material for preparation of activated carbons using physicochemical activation methods consisting of NaOH impregnation plus N₂ gasification. The Freundlich adsorption isotherm was the best model for representing the adsorption for removing of adsorbate.

In conclusion, the present investigations showed that walnut shell activated carbon could be used successfully in the removal of pollutants from aqueous solutions at the adsorption equilibrium.

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