



Activated Carbons Derived from Date (*Phoenix dactylifera*) Seeds with Excellent Iodine Adsorption Properties

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Abstract: Activated carbons were prepared from date seed powder by chemical activation method using potassium hydroxide (KOH) as activating agent at different temperatures (400, 500, 600 and 700 °C). Date seed powder was impregnated with KOH (1:1 weight ratio) and carbonizations were carried out under a constant flow of nitrogen (120 cc/min) in a tubular furnace. The activated carbons thus obtained were characterized by powder X-Ray diffraction and Raman scattering. XRD patterns revealed amorphous carbon structure, which was further confirmed by the Raman scattering. Surface morphology of ACs was studied by scanning electron microscopy (SEM). Date seed derived activated carbons showed good iodine adsorption properties giving iodine number value of 850mg/g, which is much higher than the commercial activated carbons. Therefore, KOH activated date seed derived carbons would have potential in dye adsorption and waste water treatments.

Key words: Date seeds, activated carbon, XRD, raman scattering, iodine number, KOH

1. Introduction

Activated carbon is a porous material prepared from carbonaceous materials with simultaneous activation by chemical or physical methods [13,14,15]. Physical activation requires rather high temperature (800–1100 °C) treatment with a constant flow of steam, carbon dioxide, nitrogen or air [7, 19]. While in chemical activation, the precursor material is mixed with certain dehydrating (activating) agents such as phosphoric acid (H_3PO_4), potassium hydroxide (KOH), sodium hydroxide (NaOH), zinc chloride ($ZnCl_2$), sodium carbonate (Na_2CO_3) [2, 8, 12] etc. Then, carbonization occurs at relatively lower temperatures compared to the physical activation temperature [4, 3, 18, 16, 10]. These activating agents induce important changes in the pyrolytic decomposition of the lignocellulosic materials promoting depolymerization and dehydration of constituent biopolymers. Chemical activation is preferential over physical activation for enhancing porosity at a low energy consumption. Maximum pores can be created by optimizing the synthetic conditions such as activation temperature, time, and proper selection of the activating agent and the impregnation ratio with a precursor [17, 9]. Recently, there has been a wide realization of the importance of agricultural waste as a cheap and renewable precursor for the preparation of activated carbons. Not much information is available regarding the preparation of activated carbon from date stones

using potassium hydroxide as an activating agent in existing literature. Date [*Phoenix dactylifera*] also known as date palm is grown in tropic region is a flowering plant species in palm family, Areaceae, cultivated for its edible sweet fruit. Activated carbons prepared from date stones by chemical activation with zinc chloride [1] and phosphoric acid are reported [6, 5, 8,11]. Therefore, the activated carbons from date stones chemically activated with potassium hydroxide carbonized at different temperatures and characterization by Iodine number, Raman Spectroscopy, X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM) images are presented. Thus prepared activated carbons with high surface area from date seeds would be potential use for the adsorption of dyes and waste water treatment technology.

2. Experimental Details

2.1 Materials

Date seeds were collected from local market of Lalitpur, Nepal. Potassium hydroxide (KOH) with purity >98% was purchased from Quailigens, India. The nitrogen is of ultra high pure (UHP).

2.2 Preparation of Activated Carbon(AC)

The precursor, Date seeds were collected from local market of Lalitpur, Nepal. These seeds were washed several times with distilled water and dried at 110 °C for 24 hrs. The dried seeds were crushed and grinded in an electric grinder and sieved through 300 µm sieve. The sieved date seed particles were mixed with KOH in 1:1 weight ratio and then place in horizontal furnace (Accumax,India) under nitrogen flow of 120 ml/min, carbonized at different temperatures at 400,500, 600 and 700° C for 3 hours represented as D-4,D-5, D-6 and D-7. The activated carbons were then cooled to room temperature and washed several times with distilled water to remove remaining chemicals. Then the carbons were dried in an oven at 110° C for 24hours, cooled and were sieved to get the activated carbons of upto106 µm for further study.

2.3. X-Ray Diffraction (XRD) Measurements

X-ray diffraction (XRD) measurements were carried out on Rigaku X-ray diffractometer, RINT, Japan and operated at 40 kV and 40 mA with Cu-K α radiation at room temperature.

2.4. Raman Scattering

Raman Scattering measurements were performed on Jobin-Yvon T64000. The activated carbon is excited using green laser of 514.5 nm with 0.5 mW power. The sample for Raman scattering were prepared on clean silicon wafers.

2.5 Scanning Electron Microscopy (SEM)

SEM images of samples were taken to investigate the surface morphology of the activated carbon using SEM: U-8000, Hitachi Co. LTD. Japan. EM images were taken at 5-10 kv.

2.6 Iodine Number

Iodine number is the most commonly used parameter to characterize activated carbon .The adsorption of iodine solution is considered a simple and quick test for determining the surface area of activated carbons. It is a measure of micro pore (0 to 20A or up to 2nm) content of the activated

carbon by absorption of iodine from solution. The iodine number, defined as the amount of iodine adsorbed per gram of activated carbon at an equilibrium concentration was measured according to the procedure established by the American Society for Testing and Materials (ASTM 2006). 0.1 g of dry activated carbon was taken in clean and dried test tube and 5 ml of 5% HCl was added and boiled. Then 10 ml of 0.1N iodine solution was added and shaken for one minute and filtered. The filtrate was titrated against standard sodium thiosulphate using starch as an indicator. The concentration of iodine adsorbed by activated carbon was calculated as amount of iodine adsorbed in milligrams per gram.

The Iodine Number can be calculated as.

$$\text{Iodine number} \left(\frac{\text{mg}}{\text{g}} \right) = C \times \text{Conversion factor} \quad (1)$$

where C is the difference between Blank Reading and Volume of hypo solution consumed by adsorption of hypo solution by activated carbon. The Conversion Factor is determined from the equation (2) as

$$\text{Conversion factor} = \frac{\text{Equivalent weight of Iodine} \times \text{Normality of Iodine} \times 10}{\text{Weight of Activated carbon} \times \text{Blank reading}} \quad (2)$$

3. Results and Discussion

3.1 X-Ray Diffraction (XRD)

Fig. 1 shows XRD patterns of activated carbons from date seeds particles prepared by activation with KOH at same mixing in 1:1 weight ratio carbonized at 400°C to 700°C. From the XRD graph the broad peaks for the activated carbon from date seeds can be seen between the angle (2θ) of 20° to 27°. In XRD pattern, appearance of broad peaks indicates the amorphous nature of the activated carbon and sharp peaks-crystalline nature [3]. This is the good property of activated carbon for the adsorption. The anomaly in the XRD graph as seen in the case of sample D-6, KOH activated carbon at 600°C, is due to the presence of impurities [4]. This indicates that ACs contains only carbon materials with some oxygenated functional groups.

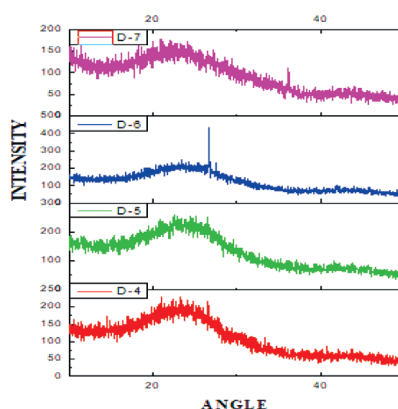


Fig. 1: XRD patterns of activated carbons derived from date seeds by KOH activation at different temperatures from 400°C to 700°C (D- 4) ,(D- 5),(D - 6), and (D - 7)

3.2 Raman Scattering

Fig. 2 shows the Raman spectra of the prepared activated carbons from date seeds (D- 4), (D- 5), (D - 6) and (D - 7). The Raman spectra of all the samples exhibited strong D and G bands approximately at 1345 and 1588 cm^{-1} respectively. Usually, appearance of D band in Raman scattering is associated with a disordered carbon structure and the intensity of D band is correlated with the defects; D band intensity increases with the number of defects [4, 20]. The D band is absent in single wall carbon nanotubes or pure graphitic carbon as these materials possess highly ordered carbon structure.

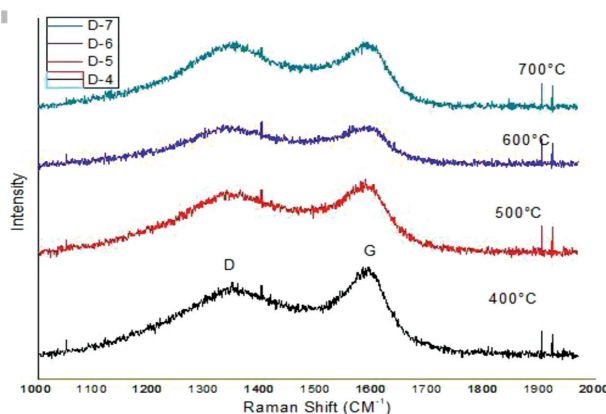


Fig. 2: Raman scattering patterns of activated carbons (D-4), (D-5), (D-6) and (D-7)

3.3 Iodine Number

Iodine Number determination for the activated carbon samples (D-4), (D- 5), (D - 6) and (D - 7) results are given in Fig. 4. The figure clearly shows that with the increase in carbonization temperature from 400°C to 700°C the iodine number also increases gradually. This indicates that the activated carbon derived from date seeds chemically activated with potassium hydroxide with increase in carbonization temperature the microporosity also increases.

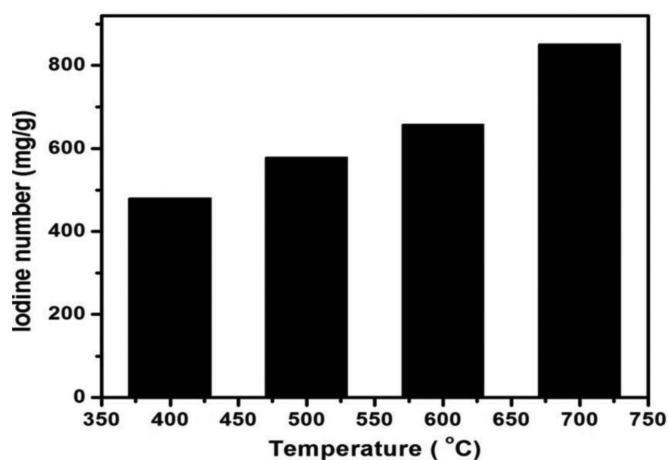


Fig. 3: Iodine Number of ACs prepared at different temperature

3.4 SEM Images of Activated Carbons

Fig. 4 shows the surface morphology of the carbon sample carbonized at 700°C (D-7). Observation of the images illustrates a large development of micro and mesopores. These micro and/or mesopores are responsible for the higher surface area of the material.

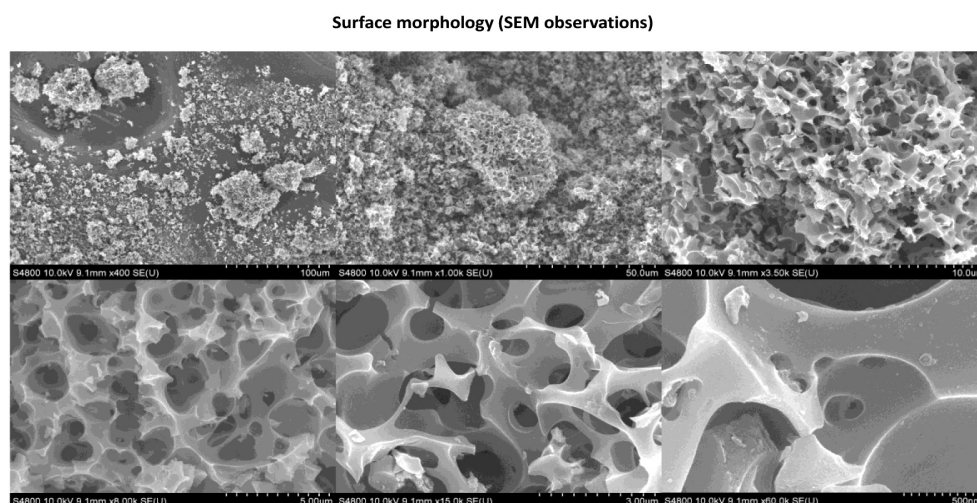


Fig. 4: SEM images of D-7

4. Conclusion

Activated carbons from date seed using potassium hydroxide as activating agent was successfully prepared. XRD pattern at different carbonization temperature of activated carbon indexed to amorphous in nature. For all sample Raman spectra exhibited strong D and G bands approximately at 1345 and 1588 cm^{-1} respectively. Iodine Number is highest for alkali activated carbonaceous material at 700°C indicating a high microporosity level. However the SEM images of D-7 seem to contain micro and mesopores. Thus prepared activated carbons with high surface area from date seeds would be use for the adsorption of dyes and waste water treatment technology.

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