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Characterization of Activated Carbon Prepared from Peach *(Prunus persica)* Seed Stone by Chemical Activation with Zinc Chloride($ZnCl_2$)

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Abstract

The present study explored the investigation of chemical and instrumental analysis of activated carbons prepared from Peach *(Prunus persica)* seed stone by chemical activation with zinc chloride($ZnCl_2$). The activated carbons were prepared at different temperatures: 400°C, 500°C, 600°C and 700°C in the presence of nitrogen atmosphere. The chemical analysis such as iodine number and methylene blue number has been utilized to characterize the activated carbons. The iodine number values of the prepared activated carbons were found to be 895.68 mg/g, 920.67 mg/g, 945.44 mg/g and 783.72 mg/g and the methylene blue number values of the prepared activated carbons were found to be 459.9 mg/g, 496.38 mg/g, 495.99 mg/g and 499.68 mg/g at 400°C, 500°C, 600°C and 700°C respectively. The activated carbons were further analyzed by instrumental analysis like FTIR and XRD. XRD analysis of activated carbons was found to have broad peaks indicating amorphous nature of the activated carbons. Surface morphology of prepared activated carbons were analyzed using Scanning Electron Microscopy (SEM). The results from various analysis indicated that the adsorption properties of activated carbons prepared from Peach seed stones were comparable to commercial activated carbon and thus can be utilized as an alternative adsorbent of commercial activated carbon.

Keywords

Activated carbon – Peach *(Prunus persica)* stone – Chemical activation – Zinc Chloride ($ZnCl_2$) – Iodine number – Methylene blue number

1. Introduction

Activated carbon is a highly porous carbon material which has high adsorption capacity. Activated carbons are widely used as an adsorbent due to its high adsorption capacity and low cost [1]. Activated carbons can be manufactured from olive seed [2], coconut shell [3], bamboo wastes [4] and various other carbonaceous materials.

The activated carbons have high internal surface area. The high surface area results due to the presence of surface pores of variable shape and size [5]. The surface pores can be distinguished as[6]:

- Micropores (radius <1nm)
- Mesopores (radius between 1-25nm)
- Macropores (radius greater 25nm)

The distribution of pores is unique for each type of activated carbon. Activated carbon for adsorbing gas molecules tend to have higher distribution of micropores whereas the activated carbons used for decolorization tend to have higher distribution of mesopores.

On the basis of particles size, activated carbon can be classified as:

- Granular activated carbon
- Powdered activated carbon
- Extruded activated carbon

Granular activated carbons have irregular shaped particles and the size ranges between 0.2 mm to 5 mm. Powdered activated carbons have more fine particles in comparison to granular activated carbon and the

size of particle ranges between 5 to 150 A°. Extruded activated carbons are manufactured by extrusion process which have particles in the shape of cylindrical pellets having diameter ranging from 1mm to 5mm.

There are two methods involved for the manufacturing of activated carbon[7]:

- Physical activation method
- Chemical activation method

Physical activation involves activation via oxidizing gas such as steam where as chemical activation involves activation via chemical agents such as zinc chloride($ZnCl_2$)[8], phosphoric acid(H_3PO_4), potassium hydroxide(KOH)[9], etc. Physical activation is performed at higher temperature than chemical activation. Since chemical activation can be achieved at relatively lower temperature and also the activation period of chemical activation is shorter than physical activation, chemical activation is reported to be more advantageous over physical activation.

2. Experimental

2.1 Materials

The Peach seed stones required for the experiment were obtained from the local market located at Kalimati, Kathmandu, Nepal. Around 40 kilograms of peach fruit were brought and about 4 kilograms of Peach seed stones were extracted for the experiment.

2.2 Sample preparation

The edible soft portion of the procured peach fruit was removed and the Peach seed stones were individually extracted from the fruit. The Peach seed stones were washed several times with distilled water and were dried in oven at 110°C for 24 hours. The dried Peach seed stones were crushed and grinded and then sieved to obtain fine particles of Peach seed stones. The Peach seed stones were then chemically activated using zinc chloride ($ZnCl_2$) in the ratio of 1:1. The activated Peach seed stones were then kept in oven at 110°C for about 24 hours. The mixture was then carbonized in a tube furnace in presence of nitrogen atmosphere at four different temperatures (400°C, 500°C, 600°C and 700°C) for three hours respectively to obtain four samples of activated carbon. Then, the samples were allowed to cool at room temperature for the next 24 hours. Around 100 ml of 0.1 N HCl was added to each sample of activated carbon and then the samples were stored for next 24 hours. Then, the samples were washed and filtered with distilled water several times to remove the impurities present in the prepared samples. After washing and filtering, the samples were dried in oven at 110°C for about 24 hours and then sieved to obtain uniform particles of activated carbon.

2.3 Characterization of prepared activated carbon

2.3.1 Iodine Number

Iodine number represents the extent of iodine adsorbed (in mg) by 1 gram of adsorbent (activated carbon). It is provides an estimation of amount of micropore content in an activated carbon. The iodine number for the prepared activated carbon can be calculated as:

Iodine Number $= (B-A) \times CF$

Where

B = Volume of standard Hypo solution required to determine the strength of Iodine solution

A = Volume of Hypo solution consumed after the adsorption by activated carbon

CF = Conversion factor

Conversion factor can be calculated as:

$$CF = \frac{N \times E \times 10}{W \times B}$$

where N represents Normality of Iodine, E represents Equivalent Weight of Iodine and W represents Weight of activated carbon

2.3.2 Methylene Blue Number

Methylene blue number represents the extent of the dye adsorbed by 1 g of adsorbent (activated carbon). It provides an estimation of amount of mesopore content in an activated carbon.

Methylene blue number can be calculated by the following formula:

$$MB = \frac{(C1-C2) \times V}{M}$$

where C1 and C2 represents initial and equilibrium concentration of Methylene blue, V represents volume of solution and M represents Mass of adsorbent or activated carbon

2.3.3 Fourier Transform-Infrared (FTIR) Spectroscopy

FTIR spectroscopy was used to analyze the surface functional groups of the prepared activated carbon.

2.3.4 X-Ray Diffraction (XRD)

XRD analysis was used to study the structural properties (amorphous or crystalline nature) of the prepared activated carbon. It is a non-destructive and highly accurate instrumental analysis method to characterize the prepared activated carbon.

2.3.5 Scanning electron microscopy(SEM)

Scanning electron microscopy was used to study the surface morphology of the prepared activated carbon.

3. Results and discussion

3.1 Iodine number and methylene blue number

Table 1: Iodine number and methylene blue number of prepared activated carbons

Temperature(°C)	IN(mg/g)	MBN(mg/g)
400	895.68	459.9
500	920.67	496.38
600	945.44	495.99
700	783.72	499.68

Iodine number (IN) and methylene blue number (MBN) values for commercial activated carbon (CAC) was found to be 845.92 mg/g and 499.087 mg/g respectively.

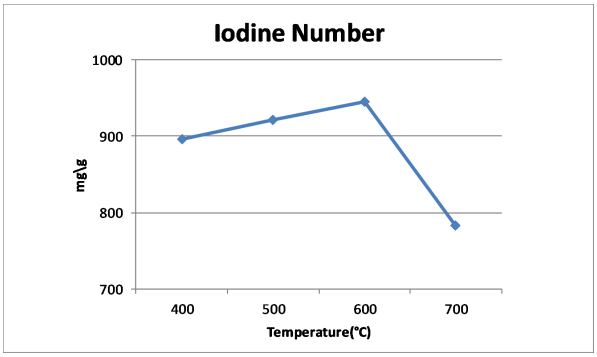

Figure 1: Effect of carbonization temperature on iodine number

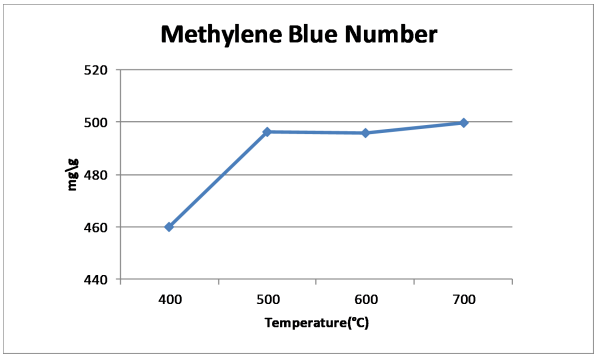

Figure 2: Effect of carbonization temperature on methylene blue number

Figure 1 and 2 represent the graphs of iodine number and methylene blue number of the activated carbons prepared at 400°C, 500°C, 600°C and 700°C respectively. At 400°C, the iodine number value was found to be low which can be ascribed due to the incomplete decomposition of lignocellulosic material. With the increase in temperature, gradual increase in iodine number value of the prepared activated carbon was observed until 600°C, after which the value was found to decrease significantly. The increase in iodine number value until 600°C can be observed due to increase in amount of micropores by distillation of tars from the lignocellulosic precursor. The distillation of tars occurs by pyrolysis process during which moisture and various non-carbon hetero-elements in the lignocellulosic precursor are removed. The increase in microposity increases the surface area which results in the higher adsorption of iodine molecules. Above 600°C, significant decrease in iodine number value can be observed which might be due to the degradation of micropores at higher temperature causing decrease in adsorption capacity of the prepared activated carbon. In case of methylene blue test, the methylene blue number value increased from 400°C to 500°C. Above 500°C, it can be observed that the methylene blue number values are almost uniform. The increase in methylene blue value from 400°C to 500°C can be ascribed due to the increase in amount of mesopores. From 500°C to 600°C, the almost uniform value of methylene blue number can be inferred due to minimal carbonization of tars in the mesopores.[10]

3.2 Fourier Transform-Infrared (FTIR) Spectroscopy

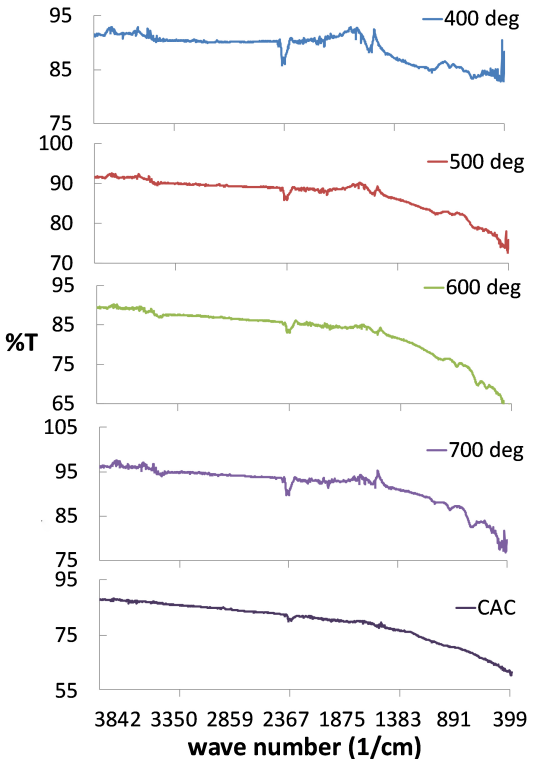

Figure 3: FTIR spectrum of prepared activated carbons from Peach seed stones at various carbonization temperatures and commercial activated carbon (CAC)

Fourier transform infrared (FTIR) spectroscopy was used to analyze the surface functional groups of the prepared activated carbons. The activated carbons contain wide varieties of surface functional group which have significant role in determining the adsorption properties of the activated carbons. Various bonds present in various surface functional group absorb the infrared light incident upon the sample and generate a spectrum for instrumental anaylsis in FTIR technique. The adsorption capacity of the activated carbon not only depends upon the porosity but also depends upon the chemical reactivity of surface functional group.

FTIR spectrum of prepared activated carbon can be observed in figure 3 which indicates presence of various surface functional groups. Strong peaks at around 2328 cm^{-1} can be observed in the FTIR spectra which can be ascribed due to stretching of C-H bond. The weak band at around 1557 cm^{-1} can be due to the unsaturated stretching of C-C bonds caused due to aromatic C=C vibrations. Furthermore, the weak peaks in the region of 737 cm^{-1} and 640 cm^{-1} can be associated with aromatic C-H bending.

3.3 X-Ray Diffraction (XRD)

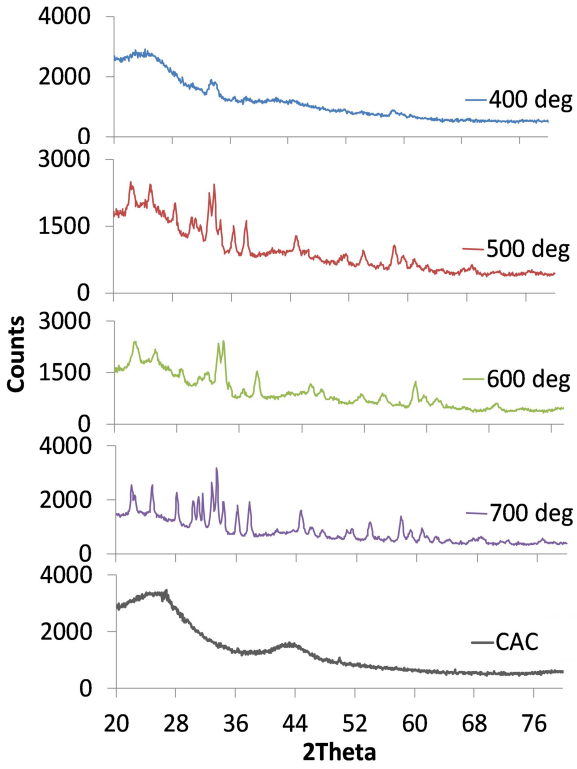

Figure 4: XRD patterns of prepared activated carbons from Peach seed stones at various carbonization temperatures and commercial activated carbon (CAC)

Figure 4 illustrates the XRD patterns of prepared activated carbons. Investigation of the diffraction patterns of the prepared activated carbons were performed using XRY diffractometer. The XRD technique is based on the Bragg's Law of diffraction.

The XRD pattern of activated carbon prepared at 400°C exhibits two distinctive broad reflections in 2theta range of approximately at 25° and 34°. The broad peaks indicate the amorphous nature of the prepared activated carbon. With the increase in carbonization temperature, the observed broad peaks seem to diminish and sharp peaks at various regions of 2theta axis can be observed. The appearance of sharp peaks at higher carbonization temperature can be ascribed due to the formation of crystalline graphite.[11] The degree of crystallinity increases with the increase in carbonization temperature and thus maximum sharp peaks in the XRD pattern can be observed at 700°C.

3.4 Scanning Electron Microscopy (SEM)

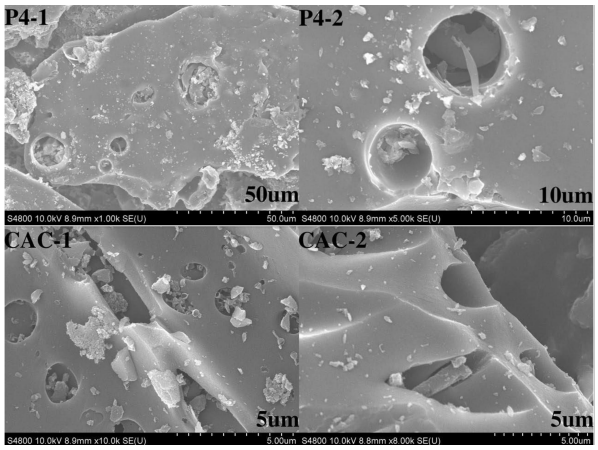

Figure 5: SEM images of activated carbon prepared from Peach seed stones at 400°C (P4-1 and P4-2) and commercial activated carbon (CAC-1 and CAC-2)

Above figures represents the SEM images of commercial activated carbon and activated carbon prepared at 400°C. The SEM images illustrate the presence of surface pores of variable size and shape. The pore size seem to be very small and the arrangement of the pores are found to be random all over the surface of the activated carbon. The formation of surface pores in the activated carbons takes place during the activation process. These surface pores are responsible for high surface area and high adsorption capacity of the activated carbon as the activated carbon adsorb molecules by capillary action through these pores. Surface pores at 400°C seem to have more regular cross-section than the surface pores at higher carbonization temperature than 400°C. With the increase in carbonization temperature gradual deterioration of surface pores take place which leads to decrease in surface area as well as adsorption capacity of the prepared activated carbon.

4. Conclusion

This study discussed the preparation of activated carbon from Peach seed stones using zinc chloride as activating agent at different carbonization temperatures. Different parameters like iodine number, methylene blue adsorption as well as instrumental analysis like FTIR and XRD were carried out to characterize the prepared samples of activated carbon and the results were further analyzed to compare the adsorption properties of the prepared activated carbons with the commercial activated carbon (CAC). The adsorption properties of the prepared activated carbons were found to be comparable to the commercial activated carbon (CAC) which have iodine number and methylene number values as 845.92 mg/g and 499.087 mg/g respectively. Thus, activated carbon from a low cost source having desirable adsorption properties was prepared from Peach seed stones. Therefore, it is concluded that the prepared activated carbon from Peach seed stones can be used as a cheap alternative adsorbent for the commercially available activated carbon(CAC).

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