



## Effects of Activating Agents on the Activated Carbons Prepared from Lapsi Seed Stone

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### Abstract

Activated carbon has been known as an excellent adsorbent and is widely used due to its large adsorption capacity. Activating agents influence surface area and porosity of the activated carbon produced. In this study, a series of activated carbons were prepared from Lapsi seed stone by chemical activation using different activating agents KOH, H<sub>2</sub>SO<sub>4</sub>, FeCl<sub>3</sub>, MgCl<sub>2</sub>, and CaCl<sub>2</sub>. The Iodine number and maximum adsorption capacity ( $q_m$ ) for methylene blue were determined. Surface functional groups were analyzed by Fourier Transform Infra Red Spectroscopy (FT-IR). Surface morphology was characterized by scanning electron microscope (SEM). The SEM micrographs revealed that the activated carbons were found to be mainly microporous and mesoporous. Among the different activated carbons, the one prepared by using KOH shows highest adsorption capacity for methylene blue and high Iodine.

**Keywords:** lapsi seed stone, activated carbon, methylene blue, iodine number, *Choerospondias Axillaris*.

### Introduction

Adsorption is an important technique used in separation and purification of gas and liquid<sup>1</sup>. Among many types of adsorbents, activated carbons are the most widely used, because of their high adsorptive capacity and inexpensive. Their adsorptive properties are linked to their great surface area, a high micro-porosity, and presence of surface functional groups<sup>2</sup>. Consequently; activated carbons are used for removing organic and inorganic pollutants from water and waste water streams. The demand of activated carbons has increased significantly as a water-purifying agent to reduce the environmental risks caused by the water pollution worldwide<sup>3</sup>.

Activated carbons from carbonaceous materials can be prepared in two steps: the carbonization of raw carbonaceous materials in an inert atmosphere and the activation of carbonized product. The purpose of carbonization is to enrich the carbon content and to create an initial porosity and activation helps to enlarge pores<sup>4</sup>. Activation can be carried out by chemical or physical methods<sup>4</sup>. In physical activation, the precursor is first carbonized in an inert atmosphere then activated using oxidizing agents such as steam or carbon dioxide; whereas in chemical activation, precursor is impregnated with chemical agent such as zinc chloride (ZnCl<sub>2</sub>), potassium hydroxide (KOH) or phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) and carbonized in an inert atmosphere<sup>5</sup>. Advantages of the chemical activation over physical activation are lower carbonization temperature and shorter time of activation. Furthermore, activated carbon obtained by chemical activation is mesoporous with a larger surface area<sup>6</sup>.

The important characteristic of an activated carbon is its adsorption capacity which depends on inherent nature of precursor as well as on the manufacturing process<sup>2</sup>. It is determined by activation conditions such as activation time, activating agent and temperature. Among these, activating agents have a great influence on the influence the pore development and surface characteristics of the activated carbon produced. Activating agents such as phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) potassium hydroxide (KOH), sodium hydroxide (NaOH), calcium chloride (CaCl<sub>2</sub>), zinc chloride (ZnCl<sub>2</sub>), etc have been reported for the chemical activation process<sup>5,8</sup>.

Activated carbons can be prepared from carbonaceous materials such as coal, wood, nut shells and agricultural by-products or wastes. Among them, agricultural by-products are currently being extensively used to prepare activated carbon because of their availability in large quantities at low cost<sup>9,10</sup>. Recently, activated carbons have been commonly produced from waste products such as olive stones<sup>11</sup>, almond shells<sup>12</sup>, walnut shell<sup>13</sup>, cherry stone<sup>14</sup>, apricot stone<sup>15</sup>, sugarcane bagasse<sup>16</sup> etc.

In the present study, the activated carbons were prepared from Lapsi (*Choerospondias Axillaris*) seed stone. Lapsi is native fruit of Nepal. Lapsi fruit is consumed fresh, pickled or processed into a variety of sweet and sour, tasty food products; whereas its seed stones are waste byproduct and used as fuel in brick kilns<sup>17</sup>. Lapsi seed stones were selected as activated carbon precursors because of their availability and only little work has been done on this material<sup>18,19</sup>. To the best of our knowledge, influence of activating agents on the adsorption capacity of

activated carbons made from Lapsi seed stone has not been well investigated. The aim of the present work is to study the effects of activating agents on the adsorption capacity of the activated carbon prepared from Lapsi seed stone.

## Material and Methods

Lapsi seed stone was used as a precursor for the preparation of activated carbon. The Lapsi fruits were purchased from local market of Kathmandu, Nepal. They were stripped for the pulp by boiling to expose stones. The seed stones were then washed dried and crushed into powder. The seed stone powder was then sieved to obtain the fraction of size 212  $\mu\text{m}$ . The dried powdered Lapsi seed stones were processed to obtain different types of activated carbon as described below.

The analytical grade chemicals potassium hydroxide (KOH), sulphuric acid ( $\text{H}_2\text{SO}_4$ ) magnesium chloride ( $\text{MgCl}_2$ ), ferric chloride ( $\text{FeCl}_3$ ) and calcium chloride ( $\text{CaCl}_2$ ) were purchased from Qualigen, India. Ultra high pure (UHP) nitrogen was used for inert atmosphere during carbonization.

**Carbonization procedures:** Activating agents used for preparation of activated carbons are shown in table-1. For comparison, result of the activated carbon obtained by using phosphoric acid ( $\text{H}_3\text{PO}_4$ ) is also listed together in table-1.

**Table-1**  
**List of activating agents**

S. No.	Activated carbon	Activating agent
1	LAC1	-
2	LAC2	KOH
3	LAC3	$\text{FeCl}_3$
4	LAC4	$\text{CaCl}_2$
5	LAC5	$\text{MgCl}_2$
6	LAC6	$\text{H}_2\text{SO}_4$
7	LAC7	$\text{H}_3\text{PO}_4$

**Direct Carbonization:** 20 gm of dried Lapsi seed stone powder was subjected to carbonization in tubular electric furnace at  $400^\circ\text{C}$  under the flow (75 ml/min) of  $\text{N}_2$  gas for 3 hours. After carbonization, the carbonized product was repeatedly washed with distilled water. The sample was then oven-dried at  $110^\circ\text{C}$  for 24 hrs, sieved to get the particles size 106  $\mu\text{m}$ .

**Carbonization with Chemical Activation:** 20 gm of the crushed Lapsi seed stone was heated in a muffle furnace for 3 hrs at  $300^\circ\text{C}$ . Then, 30 ml concentrated sulphuric acid was added gradually to the charred product with stirring. The sample was then heated in an oven at  $110^\circ\text{C}$  for 24 hrs. After that, sample was washed with distilled water to make it free of acid. The prepared activated carbon was finally oven dried at  $110^\circ\text{C}$  for 24 hrs and sieved to obtain a particle size of 106  $\mu\text{m}$ .

**Carbonization (in  $\text{N}_2$  atmosphere) with Chemical Activation:** 20 gm of dried, crushed Lapsi seed stone was mixed with 20 gm activating agent in the ratio of 1:1 by weight

and sufficient distilled water was added, stirred with magnetic stirrer at  $70^\circ\text{C}$  until excess water has evaporated. Then, sample was oven dried at  $110^\circ\text{C}$  for 24 hours. After drying, the sample was carbonized in a horizontal tubular furnace at  $400^\circ\text{C}$  under a continuous nitrogen flow of nitrogen (75 ml/min) for 3 hrs. After that, the carbon was treated with 1N HCl, subsequently washed with warm distilled water until free of chloride ions. The sample was then dried at  $110^\circ\text{C}$  for 24 hrs and sieved to get the particles of size 106  $\mu\text{m}$ .

**Characterization of Activated Carbon: Adsorptive characteristics:** Methylene blue number and Iodine number were determined to access the absorptive capacity of prepared activated carbon.

**Methylene blue number (MBN):** Methylene blue number is maximum amount of methylene blue adsorbed onto one gram of carbon<sup>20</sup>. Methylene blue number was determined according to the Method (JIS K 1470-1991)<sup>21</sup>. In this assay, 0.025 gm of each, prepared activated carbon was mixed with 50.0 ml of methylene blue (MB) solution in several conical flasks separately at different concentrations (15–100  $\text{mg L}^{-1}$ ). After shaking for 24 hrs, the suspensions were filtered and remaining concentration was measured spectrophotometrically at  $\lambda$  max of 660 nm using an UV /Vis spectrophotometer (UV- CECIL- CE-100). Standard solutions of methylene blue were used for calibration. The amount of methylene blue adsorbed from each solution is calculated by the equation (1) and (2).

$$q_e (\text{mg/g}) = \frac{(C_o - C_e)V}{M} \quad (1)$$

$$\text{Percentage adsorbed} = \frac{100(C_o - C_e)}{C_o} \quad (2)$$

Where,  $q_e$  (mg/g) = methylene blue adsorbed,  $C_o$  (mg/L) = the initial concentration of methylene blue,  $C_e$  (mg/L) = the equilibrium concentration of methylene blue,  $V$  (L) = the volume of methylene blue,  $M$  (gm) = the mass of activated carbon.

The methylene blue number is determined by plotting  $q_e$  as a function of  $C_e$  according to Langmuir model and Langmuir parameters  $q_m$  and  $K_L$  are evaluated by least square method. The  $q_m$  is methylene blue number.

**Iodine number (IN):** Iodine number is the milligrams of iodine adsorbed by one gram of carbon<sup>20</sup>. The Iodine number are determined according to ASTM D4607-94 method<sup>22</sup>. 0.1 gm of dry activated carbon was separately taken in several conical flasks and added 5 ml of 5 % HCl, boiled and cool. To the cold solution, 10 ml of 0.1N Iodine solution was added. The contents are shaken vigorously for about 4 minutes and filtered. Then 10 ml filtrate was titrated against standard (0.1N) hypo solution using starch as an indicator. The concentration of iodine adsorbed in milligrams was calculated.

Iodine number = C \* Conversion factor (f)  
The conversion factor (f) is calculated as:

$$\text{Conversion factor} = \frac{\text{Molecular weight} * \text{Normality of Iodine} * 10}{\text{Weight of Activated Carbon} * \text{Blank reading}}$$

Where,

$$C = \left( \frac{\text{Blank reading} - \text{volume of hypo consumed}}{\text{after adsorption of activated carbon}} \right)$$

**Characteristics: Surface functional groups:** Surface functional groups of activated carbon were determined by taking FTIR spectra on the Thermo Electron Corporation, Nicolet 4700 at room temperature. The % of transmission of samples was recorded over 500–4000  $\text{cm}^{-1}$ .

**Scanning Electron Microscope (SEM) micrographs:** Surface morphology of activated carbons was studied using SEM micrograph. SEM micrographs of carbon samples were prepared on a carbon tape and images were taken using U-8000, Hitachi Co. Ltd. Japan.

## Results and Discussion

**Iodine number and methylene blue number:** Methylene blue and Iodine adsorption are easy and commonly used methods to characterize activated carbon performance. By comparing the adsorptive characteristics of activated carbon for methylene blue and Iodine, the information on pore size can be obtained. Iodine is a small sized molecule so it indicates the capacity of carbon to adsorb smaller molecules. Similarly, the size of methylene blue molecule is much larger than the size of iodine molecule, and its adsorption is restricted mainly to the mesopores however, a small portion is also found in larger micropores<sup>23</sup>. Iodine number and methylene blue number (maximum adsorption capacity) of the activated carbons are listed in table-2.

**Table-2**

**Iodine number and Methylene blue numbers of activated carbons**

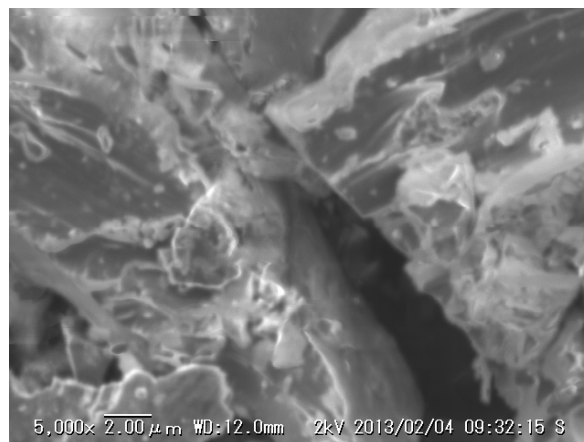
S. No.	Activated carbon	Iodine number (mg/gm)	% Removal of Methylene blue solution (25 ppm)	Methylene blue number ( $q_m$ ) mg/gm
1	LAC1	249	76	97
2	LAC2	510	90	158
3	LAC3	502	78	108
4	LAC4	381	85	144
5	LAC5	330	80	135
6	LAC6	431	99	156
7	LAC7	845	100	277

Table-2 shows the variation of iodine number from carbons prepared by the different activating agents used. The Iodine number of carbons prepared by various processes are in order of LAC2 > LAC3 > LAC6 > LAC4 > LAC5 > LAC1. From Table-2, KOH impregnated activated carbon has the highest iodine number (510 mg of iodine/gm of carbon), and the lowest iodine number was obtained for activated carbon impregnated with  $\text{MgCl}_2$  (330 mg of iodine/gm of carbon). However, the activated carbon prepared by activation with  $\text{H}_3\text{PO}_4$  process has still higher Iodine number when compared to KOH impregnated

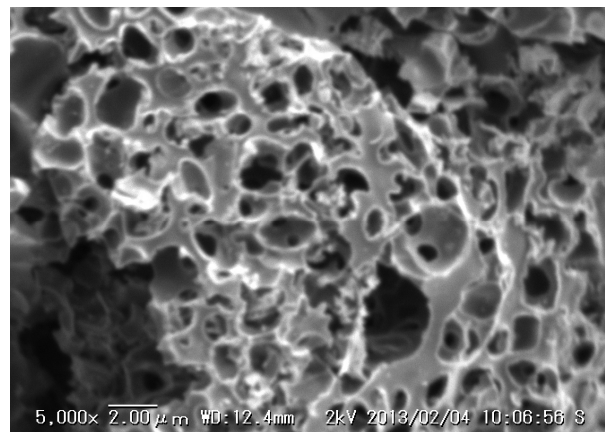
activated carbon<sup>18</sup>. The higher Iodine number of these carbons has been attributed to the presence of large micropore structure which may due to chemisorption taking place in the pores of the carbons during activation and might have resulted in the differences in their reactivity with the different activating agents and the enlargement of their pore structure<sup>8</sup>.

Activated carbon impregnated with KOH and  $\text{H}_2\text{SO}_4$  gave fairly high methylene blue value of 158 and 156 mg/gm (table-2). All the other carbons show comparatively lesser adsorption for methylene blue. The adsorption capacity of methylene blue by activated carbons prepared by various processes are in order of LAC2 > LAC6 > LAC4 > LAC5 > LAC3 > LAC1. The high adsorption capacity of KOH and  $\text{H}_2\text{SO}_4$  impregnated activated carbon (LAC2 and LAC6) for methylene blue suggests that, KOH and  $\text{H}_2\text{SO}_4$  impregnated activated carbon have a well-developed mesoporosity and favorable to adsorb larger molecules in comparison to other carbons prepared. However, the methylene blue value for  $\text{H}_3\text{PO}_4$  treated activated carbon was higher among all<sup>18</sup>.

**Scanning Electron Microscope (SEM) micrographs:** Scanning Electron Microscopy (SEM) has been extensively used to characterize the microstructure of activated carbon. The morphological study by SEM micrographs of activated carbons is shown in the figure-1.



**Figure-1A: AC prepared by direct pyrolysis**



**Figure-1B: AC prepared by activation with KOH**

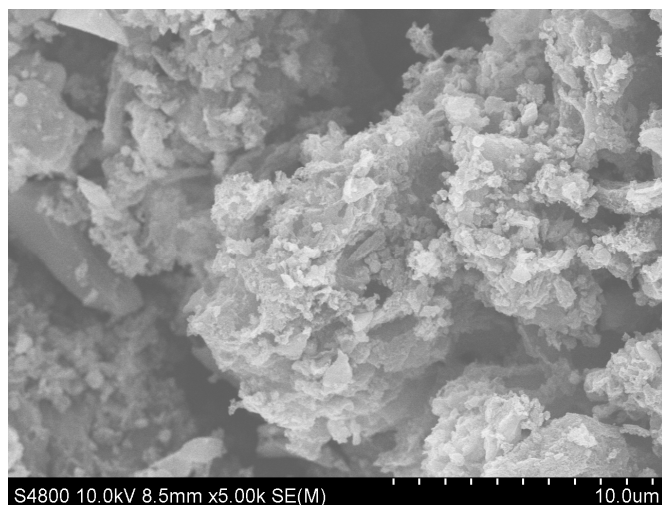


Figure-1C: AC prepared by activation with  $\text{CaCl}_2$

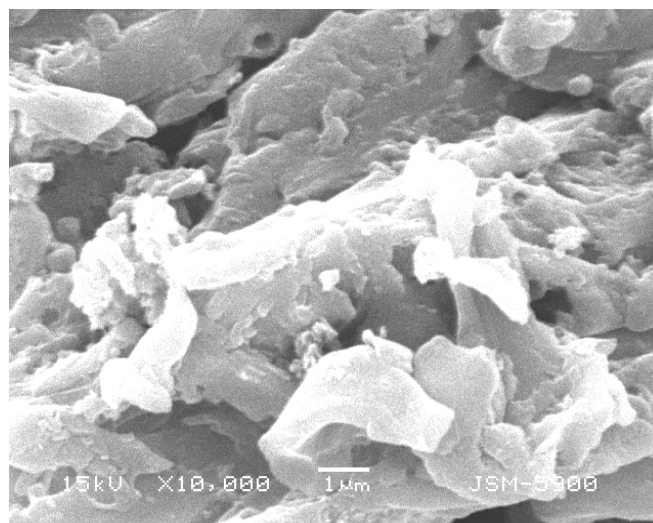


Figure-1F: AC prepared by charring with conc.  $\text{H}_2\text{SO}_4$

### Figure-1 SEM micrographs of activated carbons

The SEM micrographs (figure-1A to 1F) of activated carbon particles showed cavities, pores and rough surfaces on the prepared carbon samples. It shows that, activating agent influenced the topographical characteristics of the carbon surface.

The micrograph for pyrolyzed char, (figure-1A) shows that the surface barely consists of pores. Among all the micrographs, surface of the activated carbon prepared by  $\text{CaCl}_2$ , and  $\text{KOH}$  processes shows the presence of pores.  $\text{KOH}$  impregnated activated carbon (figure-1 B) clearly showed partially developed honey comb like highly defined pores and cavities than pyrolyzed char. However, the pores are non-uniform. Similarly, surface of  $\text{CaCl}_2$  impregnated carbon (figure-1 C) is filled with numerous small pores. On the other hand, activated carbon impregnated with  $\text{MgCl}_2$  and  $\text{FeCl}_3$  and  $\text{H}_2\text{SO}_4$  (figure-1 D, 1 E and 1 F) do not show porous structure. Pore structures of activated carbon prepared using different activating agents point to the occurrence of different reaction mechanisms. The mechanism by which potassium hydroxide activates an existing carbon are more complex and involves the disintegration (almost explosively) of structure following intercalation as well as some gasification by oxygen molecules of hydroxide whereas activation with metal chloride promotes the extraction of water molecules from lignocellulosic materials leading to the generation of porosity<sup>24</sup>.

The SEM result shows that the surface of  $\text{KOH}$  impregnated activated carbon contains well-developed pores where there is a better possibility for absorption into the surface of the pores.

**Fourier Transform Infrared (FTIR) Spectra:** Oxygen containing surface functional groups plays important role in influencing the surface properties and adsorption behavior of

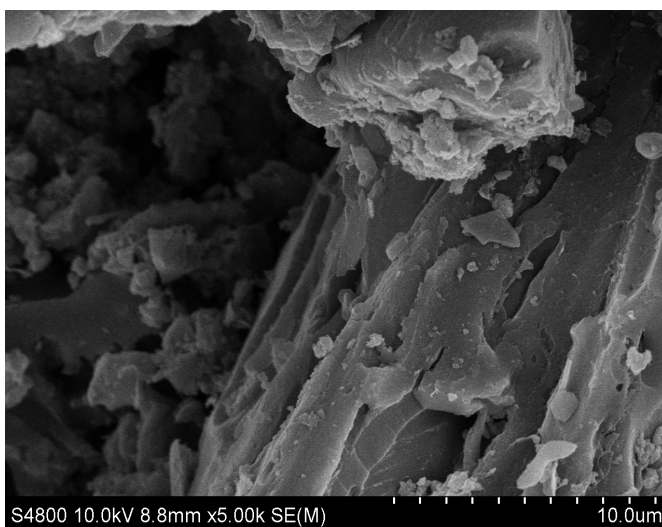


Figure-1D: AC prepared by activation with  $\text{FeCl}_3$

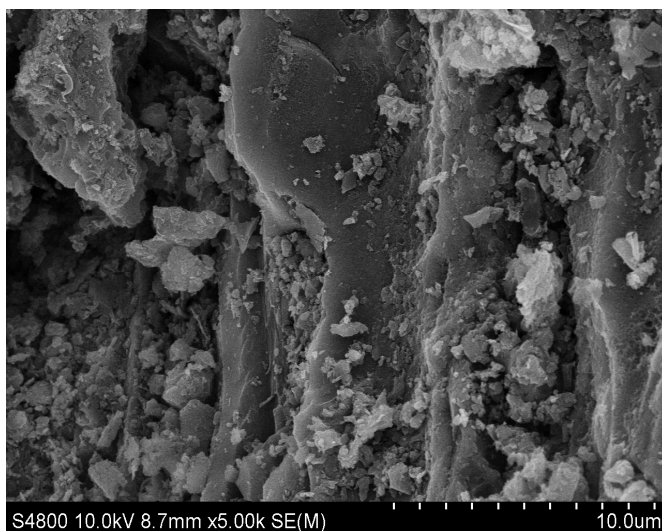
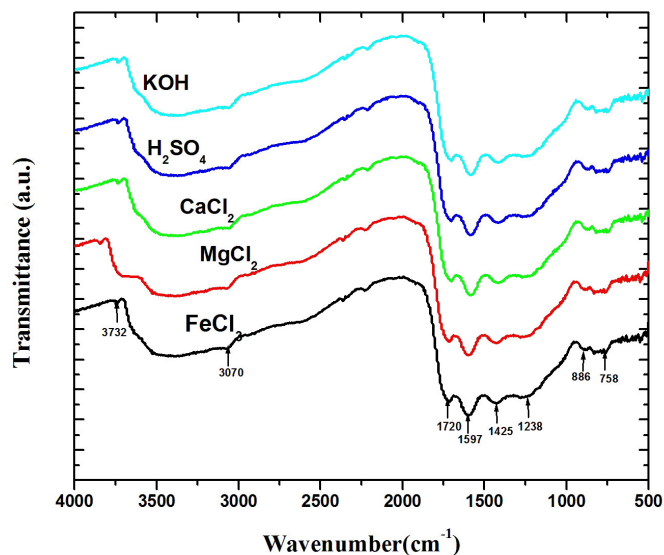


Figure-1E: AC prepared by activation with  $\text{MgCl}_2$

activated carbons. These groups can be formed during activation process or can be introduced by oxidation after preparation of activated carbon<sup>25</sup>. The FTIR spectra obtained for the prepared activated carbons are given in Figure-2.



**Figure-2**  
**FTIR spectra of activated carbons**

Although samples were prepared via various activation methods, there is a similarity in the absorption patterns (figure-2). The spectra show that the surface functional groups of the activated carbons do not exhibit significant differences, independently of the activating agent used. Only slight differences on the intensity of the bands were detected. The samples showed three major absorption bands in the region 1400-1700  $\text{cm}^{-1}$ .

The strong band around 1700  $\text{cm}^{-1}$  refers to the stretching vibrations of C = O bonds which is found in activated carbon prepared from fruit stones<sup>26</sup>. Similarly, the strong peak seen at 1425  $\text{cm}^{-1}$  has been assigned to stretching vibrations of C-C bonds<sup>27</sup>. The strong peak around 1600  $\text{cm}^{-1}$  has not been clearly interpreted. It refers to stretching vibrations of bonds in the aromatic ring coupled to that of highly conjugated carbonyl groups (C=O)<sup>28</sup> and the bands in the region 1100-1200  $\text{cm}^{-1}$  indicates the stretching of C- O bonds<sup>29</sup>. Similarly, Weak peak appeared slightly above 3000  $\text{cm}^{-1}$  apparently in all Carbons is an indication of unsaturation; alkenyl C= C stretch and the peaks in the region 860-600  $\text{cm}^{-1}$  in FTIR spectra indicate aromatic C-H bending<sup>26</sup>. Likewise Weak band around 3740  $\text{cm}^{-1}$  is assigned to stretching vibration of O-H bonds in alcohol or phenol<sup>30</sup>. From the spectra of activated Carbon samples, it can be noted that all carbons contain oxygenated surface functional groups such as hydroxyl, carbonyl, carboxyl and lactones. It seems that the nature of activating agents do not have much effect in the type of oxygenated surface functional groups.

## Conclusion

The effect of activating agents on the properties of activated carbon prepared from Lapsi seed stone by chemical activation has been investigated. Activating agents had no significant effect on the nature of surface functional group. FTIR results indicate that all the activated carbon prepared contains hydroxyl, carbonyl, carboxyl and lactones as oxygenated surface functional groups. From SEM micrograph, activated carbon impregnated with potassium hydroxide showed well developed pores structure compared to other carbons. Iodine Number and methylene blue adsorption also indicates that potassium hydroxide is the best activating agent among the various activating agents tested. Hence this study shows that Lapsi seed stone can be used as a source of lignocellulogic material for the preparation of low cost, high surface area activated carbon with well developed porosity using KOH as an activating agent.

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