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Synthesis of activated carbon from Toor dall husk (cajanus cajan seed husk) by chemical activation

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Abstract: Activated carbon was prepared from agricultural material (cajanus cajan L. seed husk) by chemical activation using H_2SO_4 as activating agent. Preparation condition was optimized to produce high surface area activated carbon with highest yield.
The effect of carbonization time, carbonization temperature and activating agent concentration were investigated in terms of yield. Carbonization temperatures were selected in range of 400-600^oC. It is noted that yield decreases with increase in H_2SO_4 impregnation concentration except for 500^oC; highest yield was noted at 500^oC. Carbonization time has linear relationship with yield. Yield decreases by increasing carbonization time and then become constant after 20 min. Activated carbon's yield can be increased by H_2SO_4 activating acid concentration and highest yield was noted at 15N. SEM analysis of produced activated carbon was done and surface area was found out to be 386 m^2/g by BET surface area analysis.
Keywords: Toor dall husk, cajanus cajan seed husk, activation, carbonization, activated carbon, yield.

Introduction:

Lignocellulosic biomass derived from agricultural products has proven to be a promising type of raw material for producing activated carbon, especially due to its availability at a low price. Utilization of lingocellulosic biomass to produce activated carbon is an important approach in pollution control strategy. Activated carbon is a highly consuming adsorbent in removal process of heavy metal, dyes, toxic compound and many other polluting compounds. It has got most outstanding properties and characteristics because of its high porosity, specific surface area, high applicability, thermal stability and good adsorptive capacity[1]. It is commonly used in the sugar, chemical, petrochemical, water treatment, and other industries. In addition, activated carbon provides an effective mean for gas phase applications, such as for separation, deodorization, purification, storage and catalysis[2].

Industrially activated carbon is produced from high range of carbonaceous material such as coconut shell, wood etc. However the demand is much more than the production of activated carbon from these raw materials. Therefore these raw materials are unable to accomplish the industrial demand of activated carbon. The cost of activated carbon is comparatively high because of high industrial demand, the treatment methods used in its production, its low yield, high energy consumption, high cost of raw material and the losses generated meanwhile. High ratio of raw material to finish product is an important factor for high demand and cost of activated carbon.

Activated carbons of different types can be produced with specific characteristics depending on the raw material, carbonisation method and activation techniques used in the manufacture[3]. The activation of carbonaceous char can be done by physical or chemical method. Physical activation involves carbonisation followed by activation whereas activated carbon by chemical activation can be prepared only by one step. The physical activation caused structural alteration, which enhanced the enlargement of micropores and even their degradation, leading to the formation of mesopores. Chemical activation conferred to activated carbon a heterogeneous and exclusively microporous nature[4]. The activating agents used for chemical activation are H_2SO_4, H_3PO_4, $ZnCl_2$ and that for physical activation are CO_2, Steam or inert gas like N_2. The temperature range for both activation methods ranges from 400-1000^oC.

In present article Toor dall husk (Cajanus cajan L. Mill spaugh seed husk) was selected as a raw material for production of activated carbon. Cajanus cajan seed husk is available in abundant quantity as it is a by product of milling industry. H_2SO_4 was utilised as activating agent with different concentration to optimise the activating agent required.

Experimental Procedure:

Cajanus cajan seed husk (Toor dall husk) was collected from locally available dall mill industries in vicinity of Nagpur (INDIA). H_2SO_4 of Laboratory grade was used during activation. Carbonization of material was carried out in muffle furnace in concentric stainless steel container to produce inert atmosphere.

Activated carbon preparation

Cajanus cajan seed husk was washed with distilled water to remove dust and dirt. Then it was kept for sun drying for 3 days. The material was crushed into small pieces and kept in oven for complete removal of moisture. Different concentration of H_2SO_4 solution was prepared (5N, 10N, 15N) and weight & dried Cajanus cajan seed husk was impregnated in the solution for 24 hrs. Material was washed with distilled water to remove excess acid from surface of Cajanus cajan seed husk. Washing and filtration was continued till the pH of filtrate comes out to be neutral. Washed activated sample was placed for sun drying and then for oven drying of 2hr at 105^oC.

Activated Cajanus cajan seed husk was carbonized in muffle furnace at different temperature in order to optimize the carbonization temperature. Carbonization of material in open atmosphere may lead to combustion of material and ash formation. Open atmosphere carbonization reduces yield and surface area of preparing activated carbon. To have an inert atmosphere, concentric container of stainless steel were utilize with annular space between them was filled with sand as heat transferring media and sample was kept for carbonization in inner stainless steel container. The whole assembly was kept in muffle furnace after attaining constant temperature (400^oC-600^oC) and material was allow to carbonized for a particular time. After carbonization material is allowed to cool to room temperature and then yield was calculated for each run.

Figure 1 – Cajanus cajan seed husk converted to activated carbon

Result and Discussion:

Activating acid concentration:

Carbonization of Cajanus cajan seed husk without activation will produce low yield (23%) and lower surface area activated carbon. Activating material with acids like H_2SO_4, $ZnCl_2$ will enhance yield and porosity in activated carbon[5]. Cajanus cajan seed husk was digested with different concentration of H_2SO_4. From figure 2, it is noted that yield increases with increase in H_2SO_4 concentration as shown in figure, however carbonizing material at 600°C with different H_2SO_4 concentration shows no change in yield.

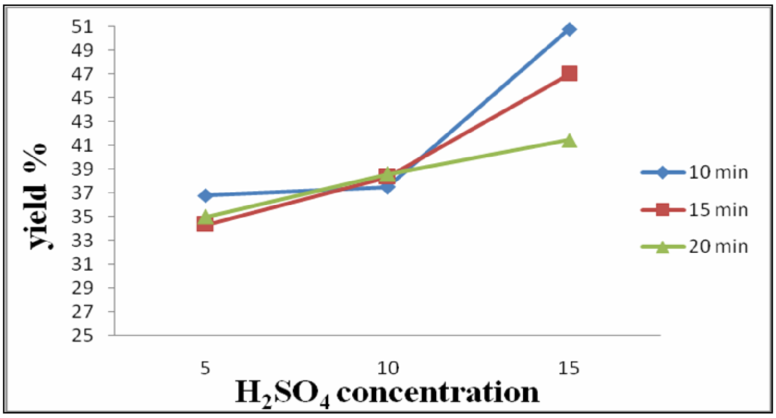

Figure 2: yield vs H_2SO_4 acid concentration carbonized at 500°C

Carbonization time

Carbonization time depends upon the type of precursors being used for activated carbon preparation. Resident time for carbonization will affect the overall texture, quality and quantity of the carbonized product with the attendant effects on the ash, moisture and possibly metal contents[6]. Physical characteristics of material determine the period required for fully carbonization of precursor. Cajanus cajan seed husk is a thin material with high lignocellulose content therefore it requires less time for carbonization. The yield is found to be consistently decreased with carbonization time and at particular time it get constant as shown in figure 3. In figures 3, we could elaborate that the 10N activated material carbonized at 500°C have no effect of carbonization time however fluctuating results were seen for materials carbonized at 600°C (figure 4). Results reveal that the 15min carbonization time can be taken as optimum because beyond that time there is not much change in material property and yield will almost remain same, which signifies that Cajanus cajan seed husk was get fully carbonized at 15 min of carbonization.

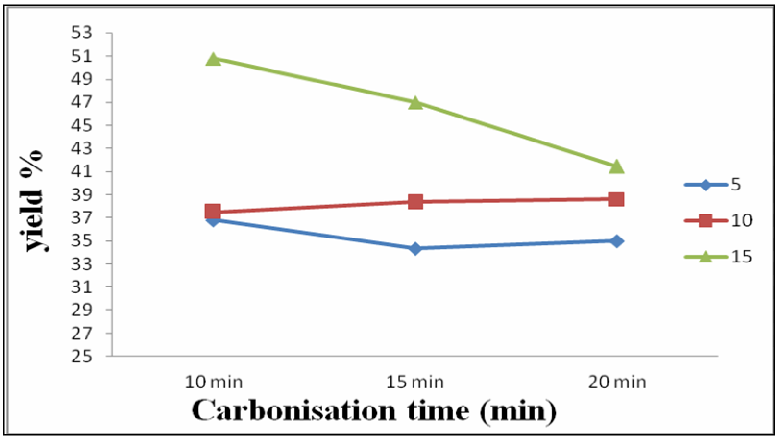

Figure 3: yield vs Carbonization Time of material carbonized at 500°C

Carbonization temperature

Carbonizing the material at higher temperature will increase release of volatile matter and provide low yield. Different carbonizing temperature will have severe effect on structural properties of prepared activated carbon therefore finding the optimum temperature of carbonization is essential[7]. From figures 5, it can be concluded that carbonizing material at 500°C will provide better results. For material impregnated with 5N H_2SO_4, yield decreases with increasing temperature whereas for 10N and 15N impregnated material it increases till 500°C and than decreases or become constant as shown in figure 6,7.

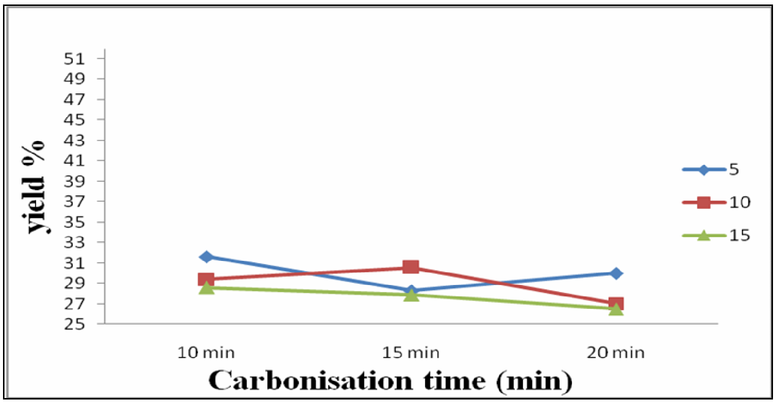

Figure 4: yield vs Carbonization Time of material carbonized at 600^oC

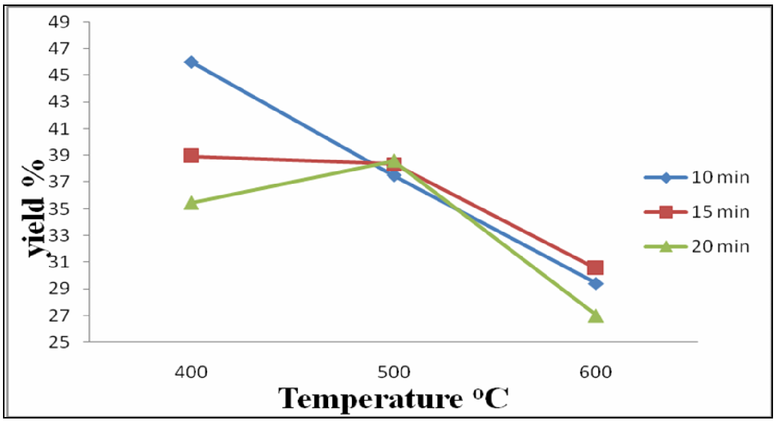

Figure 5: yield Vs Carbonization Temperature at 10N concentration

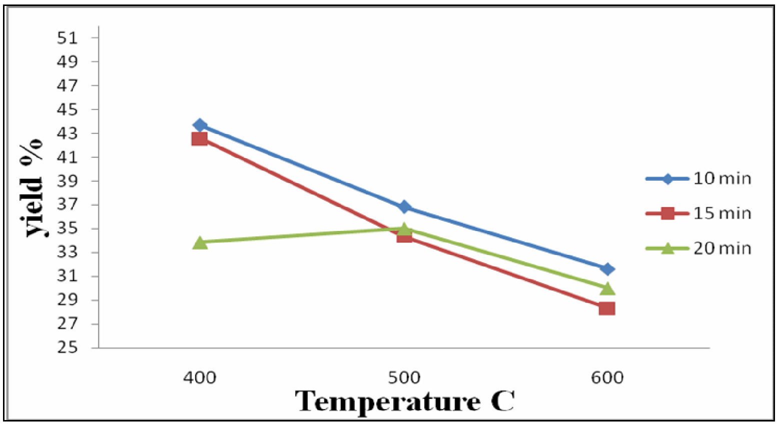

Figure 6: yield vs carbonisation Temperature at 5N concentration.

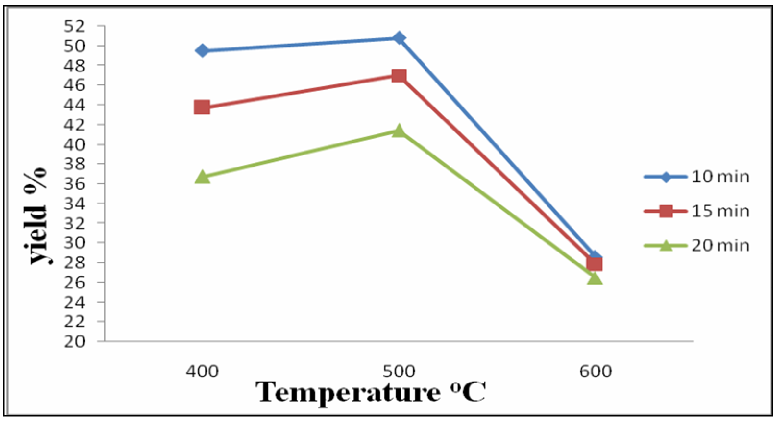

Figure 7: yield vs carbonisation Temperature at 15N concentration.

Scanning Electron Microscope analysis:

SEM analysis was carried out at VNIT Nagpur (India). Figure 8 shows SEM result of activated carbon prepared from cajanus cajan seed husk by chemical activation. The material was noted to be highly porous, amorphous and fully carbonized.

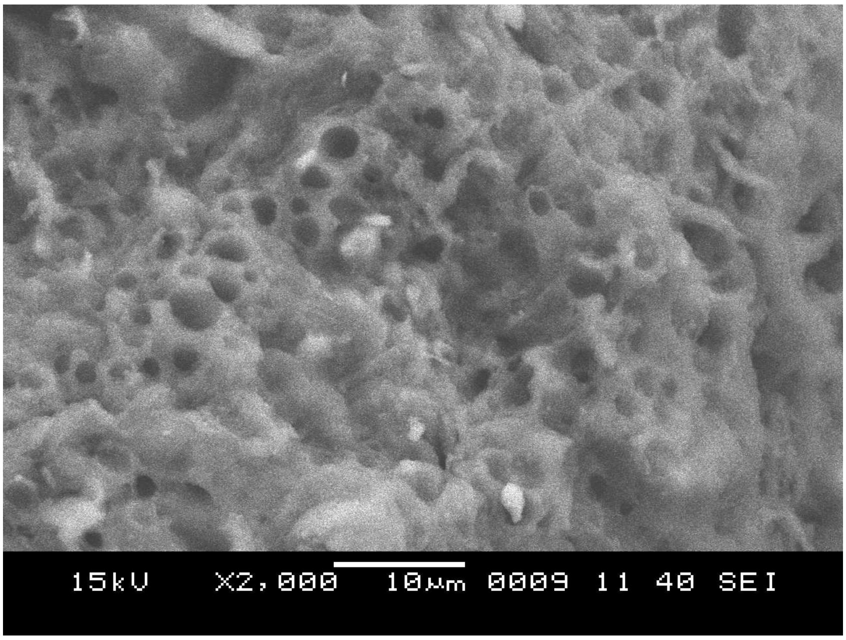

Figure 8: SEM analysis of activated carbon prepared

Conclusion:

Cajanus cajan seed husk was consumed for activated carbon preparation. Operating parameters like carbonization time, carbonization temperature and acid concentration were optimized. Highest yield was noted for material carbonized at 15N H_2SO_4 concentration. Increasing carbonization temperature will decrease yield hence 500^oC can be taken as optimum. H_2SO_4 impregnated material when carbonized for different time of interval results in constant yield. Hence it can be concluded that time of carbonization of H_2SO_4 impregnated material has no effect on yield.

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