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Research Article

Utilization of Agricultural Based Material (*Cajanus cajan* L. Mill Spaugh Seed Husk) for Production of Low Cost Activated Carbon

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ABSTRACT

Activated carbon is highly demanded adsorbent and its demand is expected to increase from 2016 by 10% per year (1.9 MMT). Activated carbon is flexible in use and has several industrial applications like for water treatment, separation, deodorization, purification, storage and catalysis but its use is retarded because of its cost. The cost of activated carbon can be lowered by using low cost feedstock such as rice hulls, sugar cane, peach pits, fertilizer waste, waste rubber tire, etc. Need of hour is to have an eco-friendly, economic, effective and sustainable feedstock for production of activated carbon. *Cajanus Cajan* (Toor daal) seed husk is a lingocellulosic material produced through milling industries and it is found to be suitable for activated carbon manufacturing. The effect of different operating parameters such as carbonization time, carbonization temperature and activating agent concentration (H_3PO_4) on yield of activated carbon has been studied. Yield increase as the concentration of activating agent increases. The surface property of activated carbon was characterized by BET and SEM analysis. Under optimum condition, activated carbon with a surface area of 386m^2/g was obtained. Carbonization time has remarkable effect on yield. Optimum carbonization temperature was found to be 500^oC for *Cajanus cajan* seed husk in terms of yield.

Keywords: *Cajanus cajan* seed husk, Activated carbon, Carbonization, Activation.

1. INTRODUCTION

Disposal of Agricultural by-products obtained from various mills (dall, oil etc) are currently a major economic and ecological issue. The conversion of these agro products to adsorbent, such as activated carbon could solve environmental problems such as accumulation of agricultural waste. Presently activated carbon is prepared from fossil fuels or non-renewable source. Utilizing activated carbon from lingocellulosic biomass instead of fossil sources such as coal will reduce global warming's effects. Activated carbon includes a broad range of carbonaceous materials, which exhibits a high degree of porosity and extended inter particulate surface area. Activated carbon possess high surface area and well defined micro-porous structure (average pore opening is about 1.5 nm). It is commonly used in the sugar, chemical, petrochemical, water treatment, and other industries.

The world's activated carbon production and consumption in the year 2000 was estimated to be 0.4 Mega Metric Tons Per Annum. By 2005, it had doubled with the production yield of only 40%[1]. World demand is expected to increase from 2016 by 10% per year (1.9 Mega Metric Tons). Presently activated carbon is produced from carbonaceous material such as wood, coal, lignite, coconut shell, peat etc but these raw materials are unable to accomplish the industrial demand of activated carbon. This may because of low yield and high ratio of raw material to finish product (3:1). Therefore new material has to be search which must be abundant & ease in availability.

With this objective of reducing the cost of producing activated carbon and to fulfill its industrial demand contemporary research is taking a turn towards agro-industrial or vegetable

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(lingocellulosic) material to be used as raw material. Toor Dall husk (*Cajanus cajan* L. Mill spaugh seed husk (CCSH)) is an agricultural based material and is available in huge quantity and has no industrial significance. According to an estimate it is cultivated on 4.79 Mega hectare in 22 countries[2]. The mills produce 15kgs of seed husk per 100kgs of Toor dall processed. Converting byproduct produced from these mills into valuable product like activated carbon will result in safe disposal and reuse of waste and also will enhance profitability.

The objective of the present work was to investigate the feasibility of producing activated carbon from an agricultural based material by chemical activation. Different operating parameters were studied in order to investigate the optimum operating condition. This work was done by keeping in mind not only to lessen the cost of activated carbon, but also to diminish environmental impact.

2. MATERIALS AND METHOD

Toor dall husk was collected from nearby Nagpur (INDIA) region. Chemical activation method was employed for activation with 98%Orth-Phosphoric acid as activating agent. The preparation of activated carbon from lignocellulosic materials involved two processes, the carbonization and the activation, which can be performed in one or two steps depending on the activation method (physical or chemical, respectively).

2.1. Raw material

Raw material must contain high percentage of carbon, low cost, hard enough to sustain under operating condition and good storage. There are several lingo-cellulosic materials like coconut shells, hazel nuts that determine their suitability for the production of activated carbon. Lignocellulosic biomass derived from agricultural by-products has proven to be a promising type of raw material for producing activated carbon, especially due to its availability at a low price.

Toor dall husk is a carbon rich material. It offers significant potential for the preparation of carbonaceous chars which may be activated to obtain higher porous carbonaceous char. This Lignocellulosic material is a byproduct of milling industries, it is sold as cattle food only and has no another usage. Therefore utilizing Toor dall husk for activated carbon production can provide better remedy. This material is quite advantageous because of having high lignin and cellulose content. High lignin containing materials give higher number of macropores while microporous structure can be obtained with high cellulose content[3].

A typical composition of selected precursor (Toor dall husk) was found in Anacon Laboratory, Nagpur (India),

Component	% (wt/wt)
Lignin	11.30
Cellulose	8.40
Hemocellulose	15.60
Ash	3.5
Moisture content	8.00

2.2 Preparation of precursor

Toor dall husk was first sun dried for more than a week in a Petri dish covered with a fine net. The net was provided to avoid foreign contaminants. Further it is oven dried for 2 hrs at 105°C to ensure moisture removal. For chemical activation, material has to be pretreated. Phosphoric acids of different concentrations have been made with normality 5N, 7.5N, 10N, 15N, 20N.Toordall husk is immersed into these acids for digestion. Impregnation ratio has a diverse effect on quality of activated carbon produced. 3:1 impregnation ratio can provide adequate results[4]. Digestion was done for 24 hrs, keeping mixture for prolong time of digestion will lead to ash formation[5]. The precursor was washed and filtered again and again till the pH of filtrate water comes out to 6 or 7. Washed activated precursor was kept for sun drying for 48hrs. To ensure complete removal of moisture, activated precursor was kept in oven for 2hrs at 110°C.

2.3 Purpose of chemical activation

The objective of activation is to eliminate tarry product from bulk of precursor in order to develop porosity and increase internal surface area. The presence of phosphoric acid during activation promotes de-polymerization, dehydration and redistribution of constituent biopolymers and also favors the conversion of aliphatic to aromatic compound[6]. Digestion of precursor in diluted acid helps to break lignin and cellulose into fragments. Concentrated acid may lead to sample agglomeration therefore dilute solution of acid was utilized for activation. The chemical agents that are generally used are phosphoric acid, zinc chloride and sulfuric acid.

Fully dried Toor dall husk is subjected to chemical activation by phosphoric acid. Digestion of precursor in acid or base before carbonization will lead to chemical activation of precursor. Increase in initial concentration of H_3PO_4 solution decreases the yield of activated carbon but pore size increases[7]. Different concentrations of phosphoric acid have been taken to investigate the optimum concentration of acid required for efficient and highly porous carbon activation.

2.4 Carbonization process

Two concentric stainless steel container were used with annular space between them was filled with sand for heat transfer medium. Washed and dried Activated precursor (CCSH) was kept in closed inner stainless steel container to maintain inert atmosphere. Though carbonization can be performed open atmosphere but inert atmosphere can provide better results in term of higher surface area. The carbonization was carried out by keeping sample in muffle furnace at high temperature. Material was kept in furnace after attaining constant temperature. For the purpose of finding optimum condition, activated precursor was subjected to carbonization at different temperature for different time of intervals in muffle furnace. Carbonized material is then cooled to room temperature slowly in desiccators to avoid direct contact of prepared activated carbon with surrounding humidity.

3. RESULTS AND DISCUSSION

3.1 Effect of concentration of activating acid (H_3PO_4) on yield of activated carbon

Activation was done by taking different H_3PO_4concentrations. It is noted that activated carbon yield of acid impregnated samples increases with increase in H_3PO_4 concentration till 15 N and then start decreasing for all temperature (400-600°C). From figure 1 we can conclude that, H_3PO_4increases the yield (till 15N) irrespective of carbonization temperature. Hence ortho-H_3PO_4 used for activating Toor dall husk is found to be suitable.

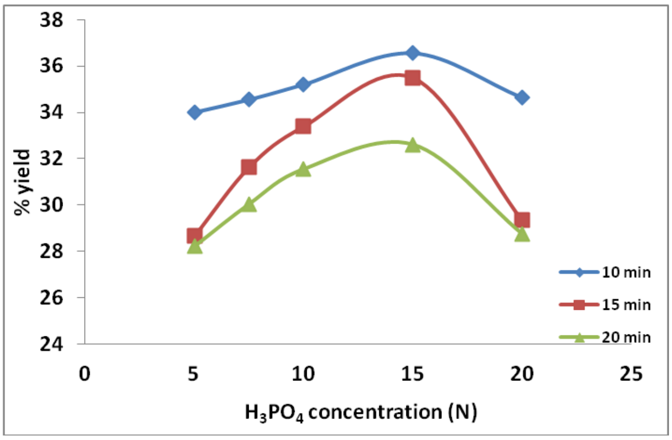

Figure 1: Effect of H_3PO_4 concentration on yield of activated carbon at 500°C.

3.2 Effect of Carbonization temperature on yield of activated carbon

Plenty of runs were taken to investigate temperature required for carbonization of Toor dall husk (CCSH). It is noted that at 400°C, material didn't get fully carbonized. This may because of volatile matters preset in Toor dall husk, which require higher temperature. Figure 2 represents carbonization of 15N H_3PO_4impregnated material at different temperature and same result was noted by taking different acid concentration (5-20N). It is noted that carbonizing material at 500°C and 600°C gives same results in terms of yield. 500°C is found to be optimum temperature for carbonization because at this temperature material gets fully converted into carbon and once carbon gets prepared further heating doesn't make any change.

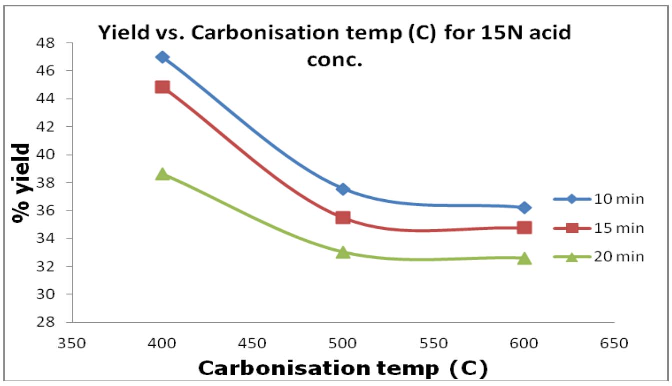

Figure 2: Effect of carbonization temperature on yield of activated carbon (15 N H_3PO_4)

3.3 Effect of Carbonization time on yield of activated carbon

By changing the time for carbonization of precursor, yield of activated carbon vary. As shown in Figure 3, yield inversely proportional to carbonization time. At 500°C it is noted that, there is consistency decrease in yield of activated carbon by increasing carbonization time. Yields are approximately same at 500°C for 15 min and 20 min of carbonization but not for 10 minute. Hence, carbonization time of 15min can be taken as optimum.

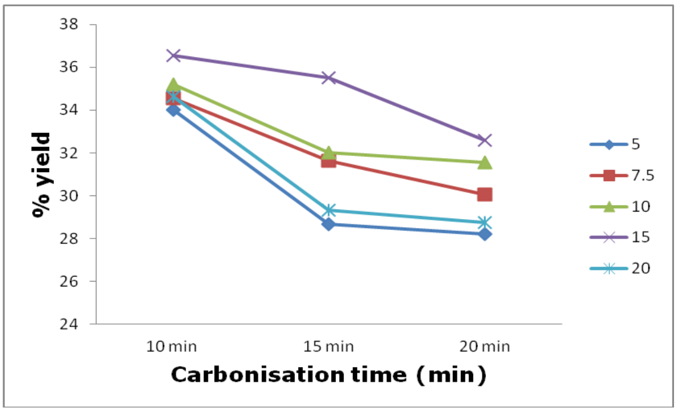

Figure 3: Effect of carbonization time on yield of activated carbon (H_3PO_4) at 500°C

3.4. Characterization of activated carbon

The BET surface area of activated carbon was calculated from the adsorption isotherms by using Brunauer–Emmett–Teller (BET) equation. The BET surface area measurement was obtained from nitrogen adsorption isotherms at 77K and calculated to be 386 m^2/g. The surface characteristics of prepared activated carbon were visualized by scanning electron microscopy (SEM). SEM images of activated carbon are shown in figure 4.

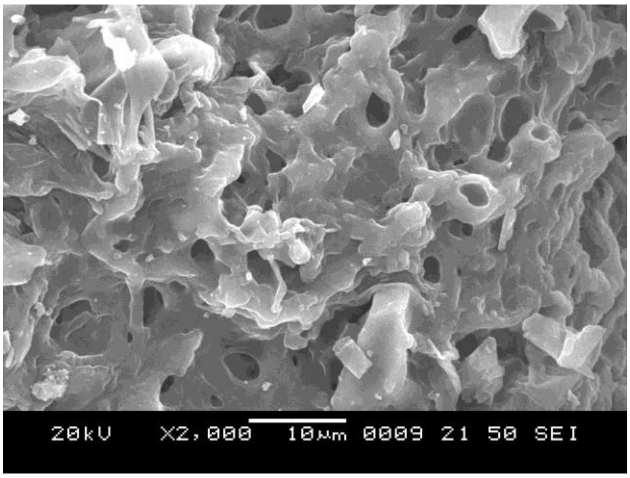

Figure 4: SEM images of activated carbon

5. CONCLUSION

Cajanus cajan L. Mill spaugh seed husk (Toor dall husk) was utilized for activated carbon preparation. Different operating parameters were studied to investigate optimum condition. H_3PO_4 is found to be good impregnating agent; which results in high yield with low ash formation. Highest yield of 35.5% was noted at 15N acid concentration. Time of carbonization has a remarkable effect on yield. Yield consistently decreases with respect to carbonization time. Optimum carbonization temperature was found to be 500°C for Toor dall husk. CCSH can subsist as a good precursor for activated carbon with surface area 386 m^2/gm. It has low cost and huge availability.

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