TECH-CHRONICLE
AN INTERNATIONAL E-JOURNAL ON
EMERGING TRENDS IN SCIENCE, TECHNOLOGY AND MANAGEMENT

Utilization of Solid Waste Generates from Dall Mill by converting it into Activated Carbon

Mr. Yogesh S. Shinde
PG Stud, Department of Petro-chemical Technology,
Laxminarayan Institute of Technology,
Nagpur, Maharashtra, INDIA
swapnaraj49@gmail.com

Dr. V. N. Ganvir (Asst. Professor)
Department of Petro-chemical Technology,
Laxminarayan Institute of Technology,
Nagpur, Maharashtra, INDIA
vnganvir@gmail.com

Mr. A. C. Shende (Asst. Professor)
Department of Petro-chemical Technology,
Laxminarayan Institute of Technology,
Nagpur, Maharashtra, INDIA
anand.shende@gmail.com

Abstract - India is agriculture based country and produces considerable quantities of agricultural by-products. These by-products have shown a great potential as a precursor materials in the manufacturing of activated carbons. Such carbons may replace existing carbons, especially coal based carbons used in many industrial applications. In this study the production of activated carbon from Cicer Arientinum L. seed husk by chemical activation method was examined. The raw material was subjected to a chemical activation using zinc chloride and phosphoric acid as an activating agent. Preparation conditions were optimized to produce high surface area activated carbon with highest iodine number. The effect of carbonization temperature (400 - 800 ^{0}C), carbonization time (15 - 90 min) and impregnation ratio (2:1 - 9:1) were investigated in terms of iodine number.

***Key words:** Activated carbon, Carbonization, Activation, Impregnation ratio, Iodine number*.

I. INTRODUCTION

Activated carbon is a material with high porosity and commercially used as adsorption of gases and solutes from the aqueous solution. It is mainly used a) as a catalyst support b) for the separation of gases c) recovery of solvents d) removal of organic contaminants from drinking water [1]. Commonly used materials for preparing activated carbon are coals and ligno-cellulosic material. The adsorption has been found to be advantageous as compared to the other traditional treatment due to its lower cost, simple design, higher efficiency and a potential to treat dyes and metal ions [2]. The more commonly used adsorbent for adsorption is activated carbon. Many agricultural by-products produce high quality activated carbon. Such carbons may have the potential to replace existing commercial activated carbons, especially coal based carbons used in many industrial applications.

Solid wastes disposal of agricultural by-products are currently a major economic and environmental issue and conversion of these agro-products into value added activated carbon could solve environmental problems [3]. Apart from pollutant removal, activated carbon is also economical in nature, because it is sourced from agricultural sector wastes and is abundantly available. Production and regeneration of commercial activated carbons is still expensive and the target of recent research for environmental protection by cost effective alternative adsorbent. The materials used for production of activated carbon are primarily industrial and agricultural by-products and forest wastes, such as coconut shell, rice husk [4], bagasse [5], plant waste [2], bamboo [6] and date stones [7]. The manufacture of activated carbon involves two main stages, the carbonization of carbonaceous precursor and the activation of the resulting char.

II. EXPERIMENTAL PROCEDURE

Cicer Arientinum L. seed husk was obtained from locally available dal mill on the district of Nagpur in India. Zinc chloride and phosphoric acid of laboratory grade was used during the activation of precursor.

Carbonization of the material was carried out in the muffle furnace in silica crucible in an inert atmosphere.

Activated carbon preparation-

Cicer Arientinum seed husk was double washed with the help of distilled water to remove the dust. Then it was kept for sun drying for 4-5 days. Then this material was kept in oven for 2 hrs. at 110 ^{0}C for complete removal of moisture.

For the comparative study, two different chemical activating agents were used. Zinc chloride and phosphoric acid solutions were prepared and weigh. Dried sample was impregnated in the solution for 24 hrs. Keeping mixture for prolong time of digestion will lead to ash formation [7]. The mixture was filtered and washed with plenty of distilled water to ensure removal of excess acid from surface. The material was washed and filtered again and again till the pH of filtrate water comes out to be neutral. Washed activated sample was kept for sun-drying for 2 days. To ensure complete removal of moisture, activated sample was kept in oven for 2 hrs. at 110 ^{0}C.

Activated sample was carbonized in muffle furnace at different operating temperatures in order to obtain the optimized temperature. Carbonization of sample in an open atmosphere produces more amount of ash and also reduces carbon yield and surface area of carbon. Activated precursor, was carbonized at constant temperature (400 ^{0}C to 800 ^{0}C) and sample was allow to carbonized for certain time period. After carbonization, sample is allowed to cool to room temperature in desiccator.

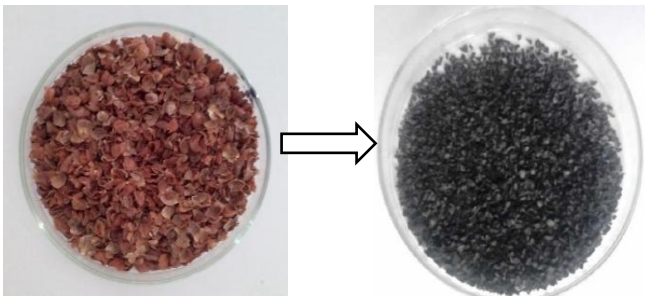

Fig 1. Activated Carbon from Cicer Arientinum seed husk

Proximate analysis-

According to ASTM D121, proximate analysis is the determination of moisture content, volatile matter, fixed carbon and ash content by prescribed methods.

2.1 Moisture content-

Small amount of activated carbon sample weight was measured and then taken in a petri dish. It was spread nicely on the dish. It was then heated in an oven at a temperature of (105-110) ^{0}C for 1.5 hr. (ASTM D-3173). The petri dish was left open or not covered during heating process. After heating petri dish was removed and cooled in a desiccator. After cooling the weight of dried sample was measured.

Moisture content, $M\ (\%) = \frac{(B-F)}{(B-G)} \times 100$ … (1)

Where, B = weight of petri dish + original sample,
F = weight of petri dish + dried sample,
G = weight of petri dish.

2.2 Ash content-

1 gm. of sample was taken in a silica crucible. It was heated in a muffle furnace to 750^{0}C for 1.5 hr. (ASTM D-3174). During this heating process the crucible was left open. After the required heating, the crucible was cooled in a desiccator and then weight of the ash was measured.

Ash content, $A\ (\%) = \frac{(F-G)}{(B-G)} \times 100$ … (2)

Where, G = Mass of empty crucible,
B = Mass of crucible + sample,
F = Mass of crucible + ash sample.

2.3 Volatile matter content-

A known quantity of sample was taken in cylindrical crucible closed with a lid. It was then heated to 925^{0}C for exactly 7 minutes in a muffle furnace. Then the crucible was cooled in a desiccator and weighted.

Volatile matter on dry basis,

$$VM = \frac{[100\ (B-F)-M(B-G)]}{[(B-G)(100-M)]} \quad \text{… (3)}$$

Where, B = Mass of crucible, lid and sample before heating
F = Mass of crucible, lid and contents after heating
G = Mass of empty crucible & lid,
M = % of moistures determined above.

2.4 Fixed carbon content-

Fixed carbon FC = 100 – (% moisture content + % volatile matter + % ash content)

Iodine number-

Iodine number is the milligrams of iodine adsorbed by 1gram of activated carbon from a standard 0.1 N iodine solution when the equilibrium iodine concentration is exactly 0.02 N. Iodine number is a measure of the micro-pore content of the activated carbon. A higher iodine number signifies higher micro-porosity of the sample.

ASTM D4607-94 (2006) gives the standard procedure for the determination of the iodine number of the activated carbon. 0.7 – 2 gram of dried activated carbon was mixed with 10 ml of 5 % by weight of hydrochloric acid in a conical flask. The nit was swirled until the activated carbon was wetted. The conical flask was boiled for 30 sec not directly but by placing it on a hot plate. The contents of the flask were cooled to room temperature and then 100 ml 0.1 N iodine solution was added to it. The flask was shaken vigorously for 30 sec. The contents were filtered through a filter paper. Initially 20-30 ml of the filtrate was discarded and the remaining filtrate was collected in a clean beaker. Then 50 ml of this filtrate was titrated against 0.1 N sodium thiosulphate solution until yellow colour just disappeared. After that 1 ml starch solution was added into it and titration was continued till blue colour just disappeared.

Table 1- Physical properties of Cicer Arientinum husk

Moisture (%)	8%
Ash content (%)	4.79%
Volatile matter (%)	54.81%
Fixed carbon (%)	32.4%

III. RESULT AND DISCUSSION

3.1 effect of carbonization temperature

It is noted that at 400 ^{0}C, material didn't get fully carbonized. This may be due to presence of volatile matter in sample which require higher temperature. The carbonization temperature and impregnation ratio are the two most

important parameters which effect on iodine number of activated carbon. The yield of activated carbon decreases as temperature increases. It is noted that carbonizing material at 500 ^{0}C gives higher value of iodine number in terms of zinc chloride as shown in fig.2. But in case of phosphoric acid, iodine number has significant effect at higher temperature. With increase in temperature up to 700 ^{0}C, iodine number also increases and after that it decreases as shown in fig.3.

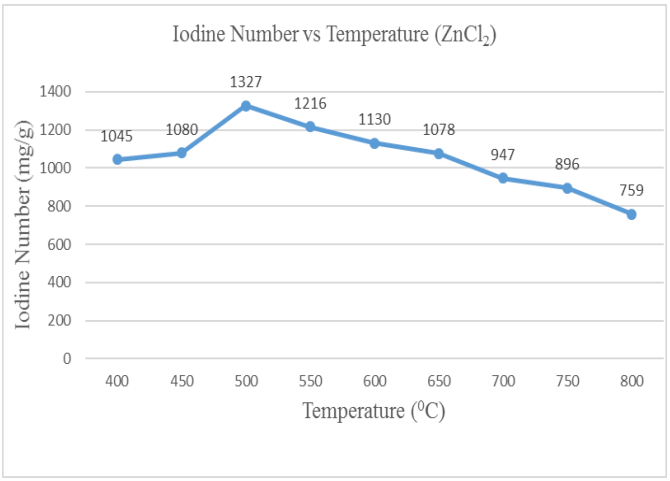

Fig. 2. Effect of Carbonization temperature for $ZnCl_2$ activating agent

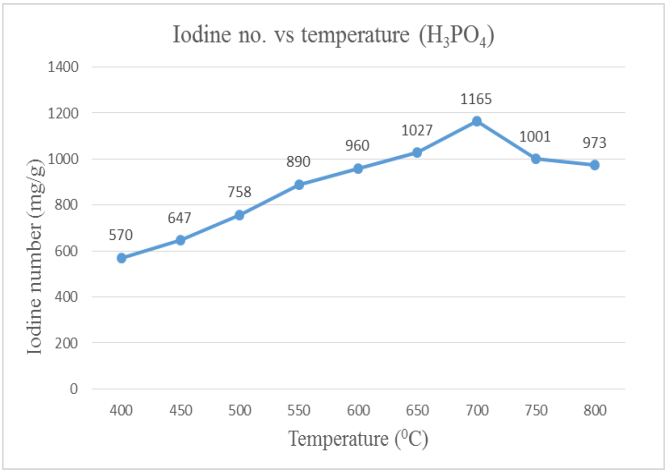

Fig. 3. Effect of carbonization temperature for H_3PO_4 activating agent

3.2 effect of carbonization time

Carbonization time mainly dependent on the type of precursor used for preparation of activated carbon. Since, Cicer Arientinum seed husk required less time of carbonization, due to thin material with high lingo-cellulosic content. By changing time for carbonization of precursor, iodine number of activated carbon vary. As shown in fig.4 and fig.5 iodine number inversely proportional to carbonization time. It is noted that there is consistency decrease in iodine number of activated carbon by increasing carbonization time. In case of $ZnCl_2$, 30 min carbonization time can be taken as optimum because beyond that time there is no increase in iodine number, which signifies the Cicer Arientinum seed husk was get fully carbonized at 30 min of carbonization time.

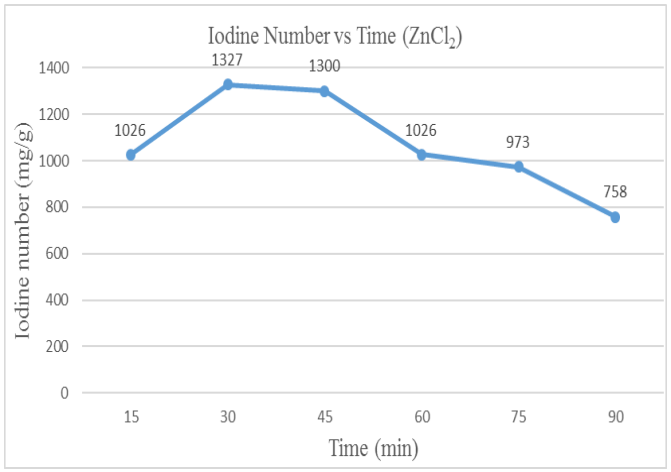

Fig.4. Effect of Carbonization time for $ZnCl_2$ activating agent

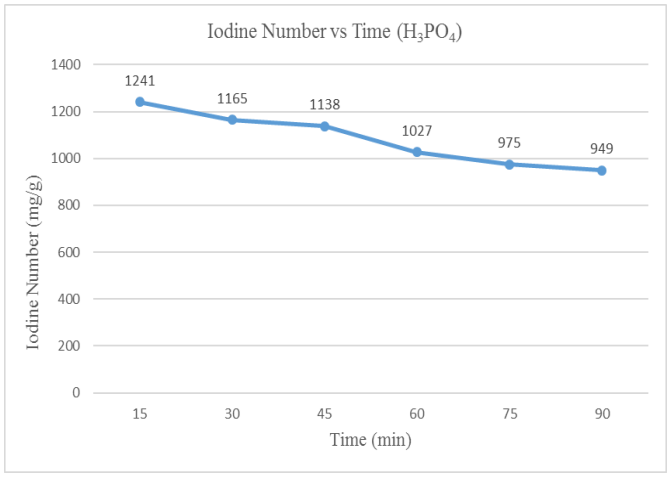

Fig. 5.Effect of Carbonization time for H_3PO_4 activating agent

3.3 effect of impregnation ratio

Effect of impregnation ratio on the iodine number of activated carbon were evaluated under carbonization temperature of 500 ^{0}C and 30 min. The samples were impregnated in the range of 2:1 to 9:1. It is noted that iodine number of activated carbon increases with impregnation ratio up to 5:1. On the other hand it decreases with increase in impregnation ratio from 5:1 to 9:1 due agglomeration of sample in activating solution. Higher iodine number is observed at 5:1 impregnation ratio. Since, from fig.6 we can observe that the iodine number of activated carbon has highest value at 5:1 impregnation ratio. It signifies that activated carbon has more micro pores due to high pore it acts as better adsorbent for adsorption process.

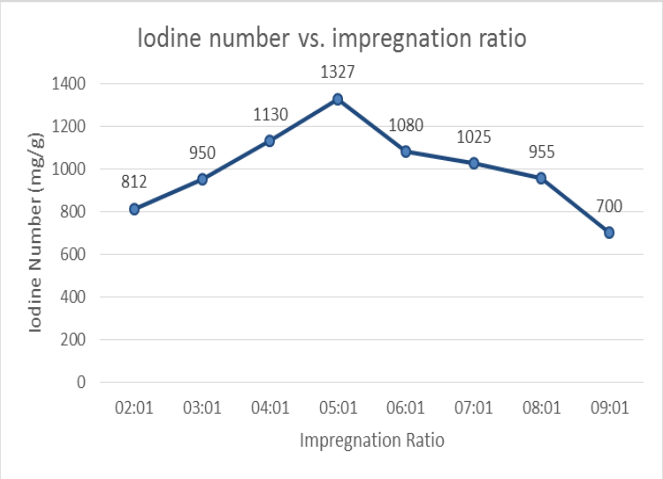

Fig. 6. Effect of impregnation ratio on Iodine number

IV. CONCLUSION

This work shows that Cicer Arientinum seed husk can be used as a precursor to produce activated carbon. The maximum iodine (1327) number of activated carbon prepared by $ZnCl_2$ activation observed at 500 ^{0}C and 30 min. In contrast, the maximum iodine number of activated carbon prepared by H_3PO_4 activation observed at 700 ^{0}C. The experimental results indicated that the prepared activated carbon were economical. Therefore it can be concluded that these activated carbon samples can be employed as effective adsorbents with low cost and huge availability.

ACKNOWLEDGMENT

Special appreciation goes to *Department of Surface coating Technology, Laxminarayan Institute of Technology, Nagpur* for accessing their laboratory and giving training for how to use equipment's. Also thanks to library staff for their contribution in using books and journals. Thanks for supporting us in every aspects.

REFERENCES

[1] Jun'ichi Hayashi, A. K. Preparation of activated carbon from lignin by chemical activation. *Carbon 38* (2000) 1873-1878.

[2] Metin Acikyildiz, A. G. Preparation and characterization of activated carbon from plant waste with chemical activation. *Microporous and Mesoporous Materials, 198* (2014) 45-49.

[3] Suzuki, R. M. Preparation and characterization of activated carbon from rice bran. *Bioresourse Technology, 98* (2006) 1985-1991.

[4] Bouchelta, C. Preparation and characterization of activated carbon from date stones by physical activation with steam. *J. Anal. Appl. Pyrolysis, 82* (2008) 70-77.

[5] Dimitrios Kalderis, S. B. Production of activated carbon from bagasse and rice husk by a singlr-stage chemical activation method at low retention times. *Bioresource Technology, 99* (2008) 6809-6816.

[6] Liu, Q.S. Preparation and characterization of activated carbon from bamboo by microwave-induced phosphoric acid activation. *Industrial Crops and Products, 31* (2010) 233-238.

[7] Timur, S. Characterization and application of activated carbon produced from oak cups pulp. *Journal of Analytical & applied Pyrolysis, 89* (2010) 129-136.

[8] Caturla, F. Preparation of activated carbon by chemical activation with $ZnCl_2$. *Carbon, 29* (1991) 999-1007.