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LOW ASH ACTIVATED CARBON FROM RICE HUSK

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Production of activated carbon as described here is achieved by leaching silica in rice husk char using caustic soda solution of different concentrations. It has been observed that 90% reduction in ash content is obtained by using 10% - 15% caustic solution. The process yields low ash, soft and activated carbon. The activity of the carbon has been determined against iodine, acetic acid and methylene blue. It has been found that the product activity against low molecular weight substances is considerably upgraded as compared with the char. This property, it has been suggested, can be utilized for gas and vapour adsorption from air and in upgrading the quality of water containing low molecular weight organic impurities.

INTRODUCTION

The name active carbon is applied to a heterogeneous group of products consisting essentially of amorphous carbon with varying amounts of ash [1] and possessing, in an enhanced degree, adsorbent properties of wood and animal charcoals. It can be prepared from any carbonaceous material which can be charred e.g. peat, wood, coal etc.

Rice husk is a cellulosic agricultural residue which can be used for obtaining furfural, activated carbon, activated silica, cellulose etc. [2]. It is rough in texture and abrasive in character. It has almost negligible digestible protein and hence is not suitable for animal feed. Because of its high ash and lignin content [3], it is also not suitable for making paper products. At present it only finds use as a fuel for brick kilns or chicken litter or animal roughage [4].

The process for the conversion of agro residues to activated carbon involves heating and activation [5, 6]. On heating rice husk, out of contact with air, the quantity of silica in the char enhances at least twofold compared with the former. This large quantity of silica is undesirable if carbon of good adsorption capacity is desired.

The present studies have been carried out (a) to show that the adsorption quality of carbon from rice husk can be improved if the silica content of char can be reduced and (b) to develop this method further to establish a process for the manufacture of low ash activated carbon. The data reported here are based on production on a semi commercial unit but those described here are on one kg scale.

A process for the manufacture of high silica active carbon from rice husk was developed and patented by PCSIR [7]. The process of activation in general com-

prises either (a) production of a char and then activation with steam, carbon dioxide etc. or (b) pretreatment of the carbonaceous material with an activating solution such as zinc chloride after which it is heated out of contact with air and subsequently leached with water, acid etc. [8].

The present studies involve heating and activation as stated in (b) above in a single step. The raw material itself contains 17-23% ash which on pyrolysis increases to 40%. A large quantity of ash such as this is undesirable because if it is predominant, it can drastically alter the adsorbing characteristics of a carbon. This effect is especially evident in the adsorption of strong electrolytes [9].

A few methods are available for upgrading the quality of high ash carbon. One of these could be the volatilization of silica while the other may be its selective leaching. The former process is quite expensive; the latter, on the other hand, appears to be economically feasible and could be extended to the utilization of other agricultural residues.

EXPERIMENTAL

For the experiments described here, a sample of high ash active carbon obtained from rich husk was utilized. The ash content of this carbon was 41.36%.

Rice husk was pyrolyzed out of contact with air at different temperatures ranging from 600 to 1050° at 50° interval for 2 hr. It was noticed that the carbon obtained at high temperature was rather crystalline while that at lower temperature of 600 - 800° it was amorphous. The latter was employed for the experiments described hereafter.

1 kg sample of high ash active carbon was weighed for each set of experiments and poured into a flask containing 3 litres of caustic soda solution with its strength varying from 5 to 20%. The contents of the flask were boiled in a glass lined vessel with a condenser and stirring arrangement to reduce evaporation losses. Heating and stirring operations were continued for a specified period of time and samples were drawn at regular intervals.

The mixture was cooled and vacuum filtered to separate the carbon from resultant sodium silicate solution. The active carbon so obtained was washed thoroughly with a stream of tap water. Washings were checked by pH paper for the presence of alkali. The washing process was continued until low ash carbon free from alkalinity was obtained. The filtrate obtained after refluxing was kept stored separately from washings which were recycled for further concentration of the sodium silicate solution.

The low ash carbon samples obtained after each set of experiments were dried in an oven at 110–115° to completely remove the moisture. The total ash of the carbon samples was determined separately by the ignition of a weighed quantity of sample in a muffle furnace at 750–800° [10].

The concentration of sodium silicate, obtained as a by-product, was determined separately by means of a hydrometer. The sodium oxide content of the solutions, obtained after successive leaching in each set of experiments was determined by the direct sulphuric acid method [11].

The bulk density of high ash active carbon utilized for this process and of low ash carbon samples was determined separately by Standard Analytical Methods [12]. The activity of low ash carbon prepared from rice husk (ash content, 4.6%) was compared with high ash carbon (ash content, 41.3%) and a reference low ash carbon from E. Merck against iodine, acetic acid and methylene blue by the standard method [13]. The adsorption isotherms were plotted between $\log X/M$ and $\log C$ for the different weights of carbon.

RESULTS AND DISCUSSION

Upgradation of high ash carbon can be carried out by extracting silica. For this purpose treatment with hydrofluoric acid could be suggested. This acid may react with silica to form hydrofluorosilicic acid which being volatile could free the carbon from silica. The remaining ashes, being soluble would, on washing, be driven off and pure carbon could be obtained.

The above process is not economically feasible. On the other hand, selective leaching with caustic soda can reduce the ash content to the desired level. Accordingly the

method described here could be developed for the production of low ash carbon. The pyrolyzed carbon is subjected to reduction with alkalis of various concentrations. The reaction yields sodium silicate of high sodium oxide ratio. It may be mentioned that the reaction is facile only if the silica is in amorphous form [14]. Controlled low temperature pyrolysis gives amorphous silica [15]. There was crystallinity on the surface of the particles at high temperature and leaching was therefore difficult. Temperatures of 600–800° gives best results. Table 1 shows the quantitative ratios of materials and reactants used, the time employed for reflux, reduction in the percentage of silica or total ash content and percentage composition of sodium silicate solution obtained as a by-product after each successive leaching. It may be noticed that there is a reduction from 41 to 4% in the total ash content depending upon the concentration of caustic soda solution and time employed for refluxing.

It could be noted from Fig. 1 that 5% caustic soda solution acts very slowly and is almost non-functional in

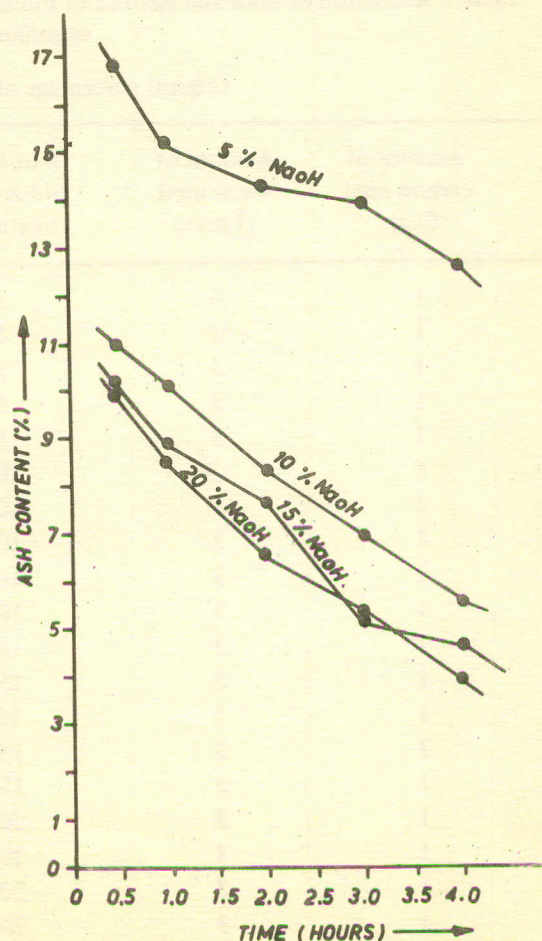


Fig. 1. Graph between ash content and time of refluxion.

silica removal after the initial reaction. Further reaction takes place after continuous refluxing, most probably because of the formation of an equilibrium mixture of sodium silicate and hydroxide. The downward slope of the curve is quite likely due to the loss of water vapour during refluxing, thus increasing the concentration which enables further leaching. The lines corresponding to the other solution concentration have the same slope and that the 15 and 20% curves are not too distant from one another; rather they are intermeshed as compared with that for 10%. This suggests that there is little advantage, if any, in exceeding the concentration of caustic soda beyond 15%. The cut-off point lies at 15% concentration and 3.0 hr. refluxing.

Table I shows that leaching with 10% caustic soda solution produces a 45% solution of sodium silicate as a by-product.

Table 2. shows the density and concentration of sodium silicate solution obtained after each successive leaching.

It may be seen that the highest density of sodium

silicate solution, viz. 1.29, is obtained in the case of leaching with 20% caustic soda. This density of sodium silicate solution is conveniently obtained, if weak liquor from washings is recycled or the refluxed liquor is concentrated. Sodium silicate of high sodium oxide (Na_2O) content is desirable for use in detergent and soap industry.

Table 3 shows gradual reduction in the bulk density of high ash carbon by leaching with increasing concentrations of caustic soda. It may be seen from Table 3 that the bulk density reduction almost levels off above 15% concentration.

These data therefore suggest that bulk density of a carbon sample is directly proportional to its ash content. The latter gradually decreases from 41 to 3.9% and in proportion its bulk density also decreases from 0.735 to 0.281 g/ml. The bulk or apparent density of active carbon varies from 0.20 to 0.8 g/ml and it largely depends upon the percentage of ash content present in it [9].

Table 4. shows the adsorption characteristics of high and low ash carbon of rich husk and a reference low ash

Table I. Reduction of silica and amount of sodium silicate obtained after treatment with different percentages of sodium hydroxide solution

Original percentage of silica in active carbon = 41.36%

S.No.	Amount of carbon used (Kg)	Amount of water used (Litres)	% of NaOH utilized for treatment	Time of refluxing with NaOH (Hr.)	Percentage of ash content after treatment	% of Na_2SiO_3 solution obtained
1.	1	3	5	0.5	16.8	35.2
2.	1	3	5	1	15.1	37.3
3.	1	3	5	2	14.3	39.1
4.	1	3	5	3	13.9	40.2
5.	1	3	5	4	12.6	41.2
6.	1	3	10	0.5	11.0	38.5
7.	1	3	10	1	10.1	40.3
8.	1	3	10	2	8.3	42.3
9.	1	3	10	3	6.9	44.1
10.	1	3	10	4	5.5	45.5
11.	1	3	15	0.5	10.2	38.6
12.	1	3	15	1	8.9	40.02
13.	1	3	15	2	7.6	42.0
14.	1	3	15	3	5.1	45.2
15.	1	3	15	4	4.6	46.3
16.	1	3	20	0.5	9.9	40.6
17.	1	3	20	1	8.5	42.03
18.	1	3	20	2	6.5	44.6
19.	1	3	20	3	5.3	45.0
20.	1	3	20	4	3.9	48.5

Table 2. Density of sodium silicate solution obtained after treatment with different percentages of sodium hydroxide solution

S.No.	% of NaOH solution used for treatment	% of Na ₂ SiO ₃ solution obtained	Density of Na ₂ SiO ₃ solution
1.	5	41.02	1.110
2.	7.5	40.35	1.109
3.	10	45.5	1.139
4.	12.5	41.31	1.112
5.	15	46.3	1.178
6.	17.5	56.2	1.230
7.	20	58.5	1.29

Table 3. Variation of low ash carbon density on treatment with sodium hydroxide solution

S.No.	NaOH solution used for treatment (%)	Ash content (%)	Bulk density of low ash carbon (g/ml)
1.	0	41.36	0.735
2.	5	12.6	0.421
3.	7.5	9.3	0.395
4.	10	5.52	0.371
5.	12.5	7.2	0.325
6.	15	4.6	0.312
7.	17.5	4.2	0.302
8.	20	3.9	0.281

Table 4. Adsorption characteristics of activated carbon samples

Sample	Iodine adsorption, mg/g carbon	Acetic acid adsorption, mg/g carbon	Methylene Blue adsorption, mg/g carbon
High ash carbon (rice husk)	161	144.4	94
Low ash carbon (rice husk)	191	163.8	286
Low ash carbon (e. Merck)	229	192.6	604

carbon from E. Merck. Using the data shown in Table 4, the concentration of iodine (Fig. 2), acetic acid (Fig. 3) and methylene blue (Fig. 4) on the carbon, $\frac{X}{M}$ is plotted against their residual concentration in solution. It may be seen from Table 4 that the adsorption capacity of low ash carbon (191 mg/g for iodine, 163.8 mg/g for acetic acid and 286.0 mg/g for methylene blue) has improved substantially from that of high ash carbon (161 mg/g for iodine,

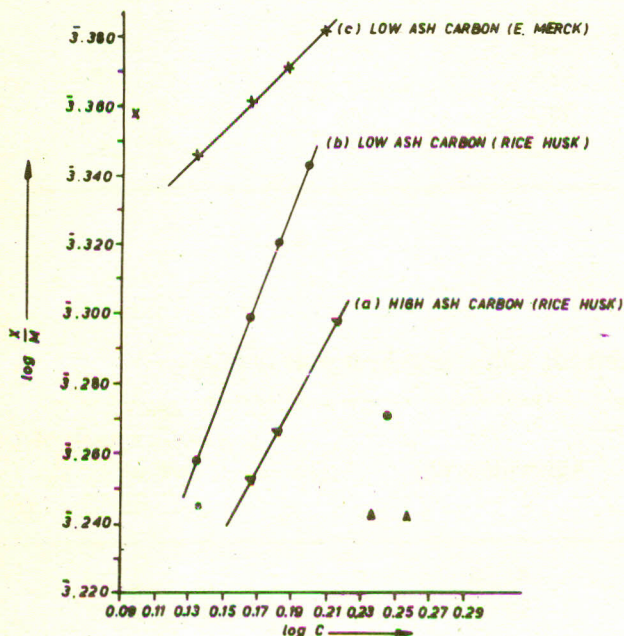


Fig. 2. Adsorption of iodine on activated carbon.

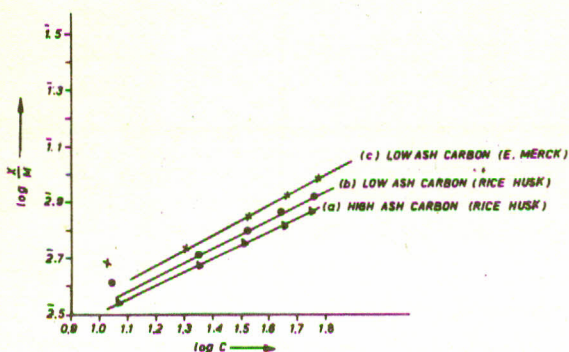


Fig. 3. Adsorption of acetic acid on activated carbon.

144.4 mg/g for acetic acid and 94 mg/g for methylene blue) and is within acceptable range for iodine and acetic acid as indicated by the standard low ash carbon from E. Merck (229 mg/g for iodine, 192.6 mg/g for acetic acid and 604 mg/g for methylene blue).

The data presented here suggest that for the removal of odorous substances and organic matter having high

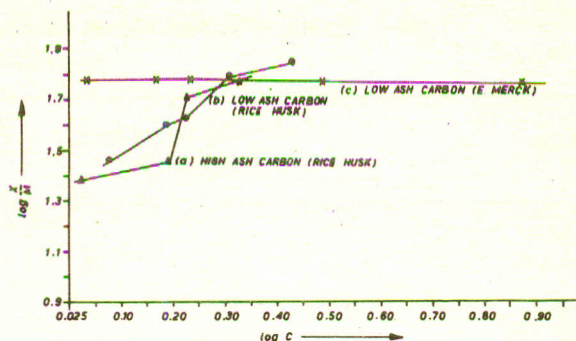


Fig. 4. Adsorption of methylene blue on activated carbon.

molecular weight from polluted water low ash carbon from rice husk can be accepted for taste and odour control. Activated carbon prepared by the above method can be applied to water at any treatment stage before filtration. The period of contact depends on the adsorption capacity and 10 to 15 min. or even more as observed here have been recommended [17]. Pressure type filters consisting of activated carbon are used extensively by the bottling industry and such systems offer promise to small municipalities. Pilot plant studies have successfully demonstrated that such filters can simultaneously filter the raw water and remove taste and odorous substances to produce a water of consistently high quality [18].

Properties of low ash carbon obtained from rice husk suggest that the same belongs to the class of low density soft carbon which may be used for liquid phase adsorption requirements. An added advantage of this process is that it yields sodium silicate. The solution obtained on leaching may be concentrated to the desired density of the silicate solution.

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