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ACTIVATED CARBON FROM INDIGENOUS INFERIOR WOODS

Part-V. Granular Activated Carbon Preparation and Liquid Phase Characterization

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The effect of zinc chloride in different impregnation ratios on various physical and chemical properties of granular activated carbon prepared from saw dust (0.250-0.473 mm) of 'Babul' *(Acacia arabica)* has been studied. It has been found that zinc chloride acts both as an activating agent as well as a binder during this process. The suitability of gelatinous mass used for granulization with different impregnation ratios has been studied and an appropriate ratio established. The activity of the product has been evaluated in liquid phase against adsorbates of different molecular dimensions. Granular carbon suitable for adsorption of small and larger molecules (micro and meso) may be prepared by chemical impregnation method.

Key words : Activated carbon, Granules, Inferior wood, Impregnation ratio, Liquid phase.

Introduction

The use of activated carbon in diversified fields is increasing rapidly due to its vital role in combating environmental degradation. According to a recent survey [1], the annual per capita consumption (in kilograms) of active carbon is about 0.5 in Japan, 0.4 in USA, 0.2 in Europe and 0.03 in rest of the developing world, generally reflecting the degree of awareness among the comity of nations for a more safe and cleaner environment.

Activated carbon is generally used in two forms, namely powdered and granular. The use of powdered activated carbon (PAC) is comparatively older and it is generally used in food processing and water purification technologies. The principal uses of granular activated carbon (GAC) are in air purification, solvent recovery, recovery of gold and in cigarette filters. Liquid phase applications consume about 80% of the total production of powdered and granular activated carbons, whereas gas phase applications consume exclusively the granular form [1]. GAC is generally associated with small pore diameter and large internal surface contrary to that of its powdered form where large pores and smaller internal surface are the main features.

As regards the processes for the preparation of GAC, two are generally in practice. One involves grinding and mixing of the mineral based raw materials (coals etc.) with a binder, reconstitution of the resultant mass by briquettig or extrusion and thereafter its physical activation. The second one is based on chemical impregnation of raw materials of vegetable origin (woods, agrowastes, nutshells etc.) by cooking and afterwards its extrusion. This process involves calcination of chemically impregnated raw materials with charring and aromatization of the carbon skeleton thus creating a porous structure [2].

The present study is in continuation of our previous work [3-6] and it describes the preparation and characterization of granular form of activated carbon by chemical impregnation of saw dust of an indigenous inferior wood.

Experimental

For the experiment described here, a sample of saw dust of 'Babul' *(Acacia arabica)* was procured from a saw mill. The portion passing through 0.473 mm and retained on 0.250 mm was collected and dried in an oven at 105° to a constant weight. This powdered raw material (100 gram) was mixed with 25,50,75 and 100 g of zinc chloride dissolved in 300 ml of 5% HCl in separate sets of experiments. These samples were then placed in porcelain dishes and slowly heated on a sand bath with continuous stirring to a dark gelatinous mass of penetration value in the range of 3.0-6.0 mm as measured by a Seta Universal and Standard Penetrometer [7]. Each sample was then extruded through a stainless steel die having holes of 2.5 mm diameter under a hydraulic press at a pressure ranging from 2810-5620 kg/cm^2 [8]. The mass in the granular form was carbonized in a S.S. vessel in an inert atmosphere at 500-550° for 2 hrs. This carbonized sample was then refluxed using 750 ml of 10% HCl solution, washed with distilled water till free from acid and chloride ions and dried. The loss in weight, yield and activating agent recovery in each case was then calculated [9].

Different physical characteristics like true and bulk density, pore space per 100 g and ball pan hardness of these samples were also determined [10,11]. The adsorptive capacity of these granular carbons against certain adsorbates in liquid phase *viz.* iodine, methylene blue and molasses was determined which generally correspond to their micro, meso and

macroporous structure. Thereafter, surface areas of these carbon samples correlated with pore diameter of 10, 15 and 28 A°, respectively, were calculated from their adsorption data [12-14].

Results and Discussion

A series of experiments have been performed to select an appropriate impregnation ratio of activating agent (zinc chloride) suitable for the preparation of GAC by extrusion technique. The saw dust of 'Babul' with a particle size 0.250-0.473 mm was used as a raw material.

Table 1 describes different observations obtained during the preparation of granular carbon. It may be seen here that as the impregnation ratio (I.R.) increases, the consistency or degree of density of the gelatinous mass/putty used for granulization gradually decreases with a subsequent increase in its penetration value. After a series of experiments with different I.R., a range of 25-100% activating agent was found to be suitable for granulization whereas an I.R. of 50% at a penetration value in the range of 3.6-3.8 mm was found to be most appropriate for this purpose. It also shows that recovery of activating agent increases with the increase in the ratio of impregnating agent and ranges between 65 to 78%. Table 1 also depicts the influence of activating agent on percent yield and ash content of the finished product and the decrease in the former is gradual, less pronounced in lower impregnation ratios. The ash content of these carbons also shows a gradual decrease with the increase in I.R. which may presumably be due to the ash reducing action of this particular activating agent [15].

TABLE 1. DIFFERENT CHARACTERISTICS OBSERVED DURING THE PROCESS.

Samples code	Impregnation ratio (%)	Penetration value*(mm)	Activating agent recovery (%)	Yield (%)	Ash contents(%)
GR_1	25	2.9-3.2	64.4	41.1	4.18
GR_2	50	3.6-3.8	66.5	40.5	3.55
GR_3	75	4.4-4.7	70.6	36.0	3.39
GR_4	100	5.6-6.0	78.3	30.0	3.35

* Weight used = 50 g , Temperature = 25°, Time = 5 Sec.

Table 2 shows different physical properties like bulk and true density and thereafter pore space per 100 g of the GAC samples. A regular pattern of gradual decrease in bulk and increase in true density with the increase in I.R. emerges, resulting in an increase in their pore space. The ball pan hardness of these GAC samples has been tested and a hardness number of 95.68 has been found in GR_2 (Fig. 1) which meets the standard specifications [11] whereas samples GR_1 and GR_3 show a nominal decrease in their hardness numbers. However, with higher I.R. of 100% (GR_4), a sudden decrease in its hardness number (70.36) has been observed. Figure 1 further shows that higher I.R. (above 75%) with corresponding penetration value above 4.7 mm (Table 1) has altogether an adverse effect on the strength properties of GAC.

Table 3 gives some adsorptive characteristics of these granular carbons against certain adsorbates of different molecular dimensions like iodine, methylene blue and molasses (Fig. 2) as well as their receptive micro, meso and macro pore areas. It shows that micropore volume attains an optimum value at lower I.R. of 50% whereas in the case of meso, this stage is reached at 100% I.R. As far as macropores are concerned, these show a gradual increase with the increase in I.R. However, it is quite clear that this activating agent has proven to be ineffective in creating reasonable macropore capacity during the process of making GAC. Although an optimum activity is found in sample GR_3, Fig. 1 and 2 shows that GR_2 has been found to be quite appropriate in respect of reasonable activity and standard hardness of the resulting GAC samples.

In an earlier study [3], impregnation ratio influence of the same activating agent was observed in the preparation of PAC

TABLE 2. DIFFERENT PHYSICAL CHARACTERISTICS OF GRANULAR ACTIVATED CARBONS.

Sample code	Bulk density (g/cc)	True density (g/cc)	Pore space per 100 g	Ball-Pan hardness number
GR_1	0.4193	1.8372	184.06	92.63
GR_2	0.4000	1.8458	195.83	95.68
GR_3	0.3870	1.8479	204.29	93.33
GR_4	0.3396	2.0346	245.31	70.36

TABLE 3. ADSORPTIVE CHARACTERISTICS OF GRANULAR ACTIVATED CARBONS.

Sample code	Iodine No. (mg/g)	Methylene blue No. (mg/g)	Molasses value	Surface area of pores >10A° (m^2/g)	Surface area of pores >15A° (m^2/g)	Surface area of pores > 28 A° (m^2/g)
GR_1	900	106	96	825	303	46
GR_2	1058	265	108	937	758	52
GR_3	1205	352	130	1110	1007	63
GR_4	1253	397	148	1155	1136	71

from the same raw material. A correlation of the present study with the previous one shows that in GAC, an I.R. of 50% is far better in creating overall better activity in respect of micro and meso pores as compared to that of PAC. As far as macropores in PAC are concerned, contrary to that of GAC where a nominal increase is found, these go on increasing rapidly with an increase in the concentration of activating agent. However, an identical behaviour of attaining optimum mesopore volume by GAC and PAC has been observed at an I.R. of 100%.

A review of Table 3 further shows that pore size distribution of a particular activated carbon is largely dependent upon the degree of impregnation. Moreover, the difference in pore volume distribution among PAC and GAC at a certain I.R. may be, mainly, due to the difference in their methods of preparation [16]. Consequently, GAC at a lower I.R. attains comparatively better activity than PAC due to a better mixing and dehydrating action of the activating chemical during the putty making and extrustion process.

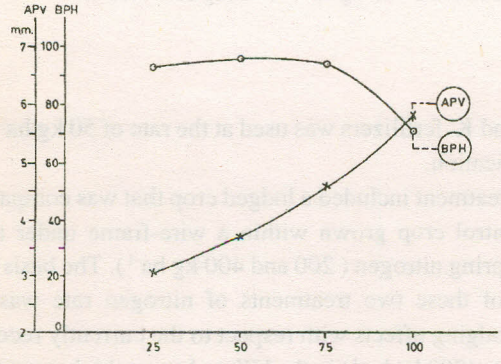

Fig. 1. Effect of I.R. on average penetration value (APV) and ball pan hardness No. (BPH) of GAC.

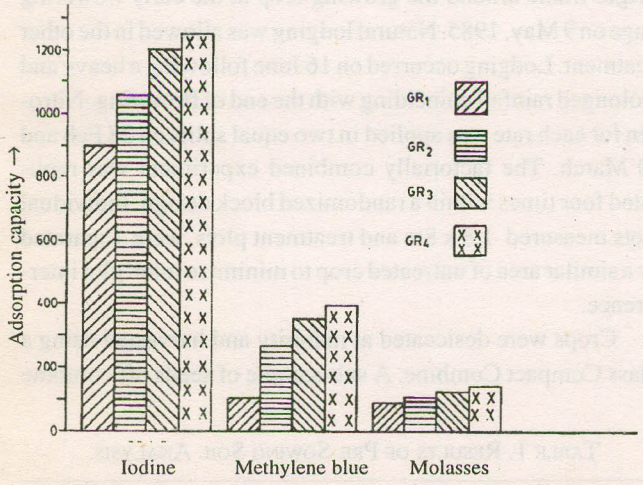

Fig. 2. Adsorptive characteristics of granular activated carbons.

Conclusion

1. Saw dust of 'Babul' is an appropriate raw material for granular activated carbon by chemical activation.

2. Zinc chloride acts both as an activating chemical as well as a binder during the process of making GAC.

3. GAC prepared by chemical impregnation method is found to be more active against small and larger adsorbate molecules.

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