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Preparation and Characterization of Activated Carbon from Babul *(Acacia arabica)* and Coconut Shells by Physical Activation in a Fluidized Bed Reactor

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Two indigenous raw materials Babul *(Acacia arabica)* and coconut shells were subjected to physical activation for the preparation of activated carbon by a mixture of superheated steam and air in a S.S. fluidized bed reactor. The influence of various operational variables like particle size of the two raw materials, time of activation and pressure of the fluidizing gas on different physical and chemical properties of the Granular Activated Carbon (GAC) samples was investigated. Different operational parameters being optimized in this study were particle size of 1.00 – 2.00 mm, operating pressure of 20 psi in 75 min time of activation. Babul was however not found to be an appropriate precursor for preparing GAC, probably due to its low inherent strength, eventually resulting in quite low yield and ball pan hardness of GAC samples. Further it was concluded, that GAC in quite a good yield and adsorptive capacity in liquid as well as vapour phase systems and standard hardness was obtained from coconut shells and it was much superior, when compared with the product obtained from Babul.

Key words: Physical activation, Fluidized bed, Coconut shells, *Acacia arabica*, Operating variables.

Introduction

Active carbons are unique and versatile adsorbents because of their extensive surface area, microporous structure and high adsorption capacity. They are assuming increasing importance in the control of air pollution, in purifying and controlling the general chemical environment, in certain biomedical applications and for the removal of organic matter from water and waste water (Martin 1980). The use of carbon for purposes other than as a fuel or reductant dates back to the eighteenth century when activated carbon from wood was employed as medicine (Hassler 1963).

It is known from visual and microscopic studies that all carbonaceous materials have a system of pores. The art of making an activated carbon is to enlarge these pores and also to develop new pores by the removal of volatile matter (Bannal *et al* 1988). Activated carbons are generally prepared by either of the two processes namely chemical and physical activation. These authors have done detailed work on the preparation and characterization of powdered and granular activated carbons by chemical activation of various indigenous precursors like inferior woods (Usmani *et al* 1989), rice husk (Usmani *et al* 1994) and low rank coals (Usmani *et al* 1996). In the process of chemical activation, inorganic metallic salts are used to impregnate the raw materials before carbonization. Contrary to that in physical activation, the processes of carbonization and activation take place in two separate steps, and activating mediums most commonly used are superheated steam, CO_2 and other oxidizing gases. The latter is a high temperature process as compared to the former one and mostly used in the preparation of granular activated carbons (GAC). Two type of reactors namely static bed and fluidized bed are generally used in the process of physical activation. Among the two, the latter (fluidized bed), is recognized to be the most efficient reactor, as it offers the advantage of extremely high heat and mass transfer (Kunii and Levenspiel 1969). Still very little R&D work is available in scientific literature on the operational conditions of fluidized bed activation for the preparation of active carbon (Edwards *et al* 1963). The present study aims to utilize and explore certain indigenous raw materials like Babul (*Acacia arabica*) and coconut shells as possible presursors for obtaining granular activated carbon (GAC) by the process of physical activation in a fluidized bed reactor. Babul is an indigenous wood abundantaly available in the rural and urban areas of Pakistan. Coconut has successfully been grown in the local environment, especially coastal areas of the country. Various operational conditions like particle size of the each raw material, activation temperature, time and operating pressure of the fluidizing gas were

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established in these studies, after performing a series of experiments for a set of operational variables. Moreover, the influence of these variable on different parameters, like yield, strength and activity of these products in liquid and vapour phase systems were also determined.

Experimental

i) Preparation of raw materials. Babul *(Acacia arabica)* and Coconut shells were used as the raw materials in these fluidization experiments. These were disintegrated/chipped in a pilot disintegrator and classified to two different particle sizes of 1.00 to 2.00 mm and 2.00 to 3.00 mm on a standard sieve shaker.

ii) Physical activation by fluidization in a fluidized bed reactor. The experiments in connection with the physical activation of Babul *(Acacia arabica)* and coconut shells, disintegrated to a certain particle size, were performed in a fluidized bed reactor. A semi pilot scale fluidized bed reactor was designed, fabricated, installed and got patented for this purpose (Usmani *et al* 1999).

The apparatus comprised a fluidized bed reactor of stainless steel, provided with a cyclone in which carbonized acacia or coconut shells, classified to a certain particle size, were introduced through a hopper. The reactor was placed inside a bricklined furnace, being heated by two burners fixed at different positions, provided with a blower and mixer arrangement. The reactor was then heated to a certain temperature, monitored by a Ni – Cr thermocouple with guage, having a release valve remaining open throughout the activation process. Steam generated from a boiler at a certain pressure was then passed through an air-steam mixer in which air at the same pressure as that of steam was fed from an air compressor. Steam-air mixture was then passed through a superheater heated by a burner, also provided with a blower and mixer. The mixture of superheated steam and air was then fed through the bottom cone of the fluidized bed reactor. Activating gas after fluidizing the carbonized particles of acacia or coconut shells escapes through the release valve during the physical activation process. This process of fluidization / activation was continued for a specified period of time, after which the burners were switched-off and the temperature of reactor was brought down to 450-500^{0}C by an air blower and the steam supply was temporarily suspended by control valve. The product (granular activated carbon), was simultaneously collected in a product storage tank by closure of release valve and opening of discharge valve. After cooling to ambient temperature, different samples were kept in airtight bottles for their evaluation.

iii) Establishment of different process parameters. A series of experiments in connection with the establishment of different process variables were designed and performed with the two different raw materials under investigation. The temperature of the fluidized bed was kept in the range of 900 – 950^{0}C (Smisek and Cerny 1970), as it is the most appropriate temperature commonly practiced in this process. The temperature was monitored by a Ni-Cr thermocouple during the course of all these experiments.

iv) Characterization of the products. The granular products obtained by physical activation of *Acacia arabica* and coconut shells, under different set of experimental conditions, were then evaluated for their liquid and vapour phase adsorptive capacity towards molecules of different molecular dimensions like iodine, methylene blue, molasses and carbon tetrachloride (Snell and Hilton 1969; Jain and Sharma 1971). Moreover, different physical and chemical properties of these products, like ash content, bulk and true density, ball pan hardness etc. were also determined (ASTM 1979). Surface areas of pores of these carbon samples > 10, 15 and 28 Å were also calculated (Svendsen 1975).

Results and Discussion

Babul *(Acacia arabica*, denoted as ‘B’) and coconut shells (denoted as ‘C’) were used as possible indigneous raw materials for preparing GAC, by activation with superheated steam-air mixture,in a pilot scale fluidized bed reactor. The influence of different process parameters, like particle size (D) of the raw materials, operating pressure (P) of the fluidizing medium and activation time (T) etc. on the different physical and chemical characteristics of the GAC samples obtained therein was studied. It may be noted in Table 1, that particle size ‘D’ utilized for detailed investigation was different in both cases of samples ‘B’ and ‘C’. Comparatively, smaller particle size of 1.00 – 2.00 mm was utilized for detailed studies in case of sample ’C’ due to better inherent strength of coconut shells as compared to Babul (Sample ‘B’), where larger particle size of 2.00 – 3.00 mm was initially chosen for the studies. This variation in particle size of the two raw materials ‘B’ and ‘C’ was maintained in initial designing of experiments to minimize undue losses during fluidization and achieving optimum yield and activity with the two raw materials under investigation (Kirubakaran *et al* 1991).

Tables 1 and 2 detailed different physical and chemical characteristics of GAC samples prepared from raw materials ‘B’ and ‘C’ under different set of operating variables. It may be seen in Table-1, that samples $BP_2t_1D_1$ (B_5) and $CP_3t_1D_1$ (C_3) have the highest pore space out of all the samples of their own particular class. It may also be observed that these two

Table 1

Effect of different process variables on various physical and chemical characteristics of granular activated carbon samples prepared by physical activation of Babul *(Acacia arabica)* and coconut shells

Sample *	Code	Bulk density (g c c^{-1})	True density (g c c^{-1})	Pore space (c c 100 g^{-1})	Ash content (%)
$BP_3t_1D_1$	B_1	0.19125	2.1090	472	1.69
$BP_3t_2D_1$	B_2	0.1769	1.890	512	2.32
$BP_3t_3D_1$	B_3	0.16203	1.6208	553	2.61
$BP_1t_1D_1$	B_4	0.1265	1.098	701	2.30
$BP_2t_1D_1$	B_5	0.1200	2.2869	790	2.10
$BP_3t_1D_2$	B_6	——	——	——	2.53
$CP_3t_1D_1$	C_1	0.4085	1.6014	182	2.0
$CP_3t_2D_1$	C_2	0.4046	1.7014	188	2.6
$CP_3t_3D_1$	C_3	0.3904	1.6590	195	3.7
$CP_1t_3D_1$	C_4	0.4150	1.6843	181	2.0
$CP_2t_3D_1$	C_5	0.4154	1.7253	182	2.1
$CP_3t_3D_2$	C_6	——	——	——	2.2

* B, Babul; C, Coconut shells; P, Operating pressure (P_1, 10 psi; P_2,15 psi; P_3,20 psi); t, Fluidization time (t_1,45 min; t_2, 60 min; t_3,75 min), D, Particle size (D_1, 2.00-3.00 mm; D_2, 1.00-2.00 mm for 'B', D_1, 1.00-2.00mm, D_2, 2.00-3.00 mm for 'C').

samples also have the lowest bulk and highest true density. Further it may be noted in Table 2 that Ball Pan Hardness Number (BPH) of "B" class samples prepared from *acacia* was quite low as compared to samples prepared from coconut shells. Hence BPH which is the prime and most desired property of any GAC sample, is not at all achieved in 'B' class samples and only 'C' class samples attained the desired standard hardness (ASTM 1979).

The data on percent yield and particle size retention presented in Table 2, also reveals that average yield ranges between 45-60 percent in samples produced from coconut shells, whereas in *acacia* it is merely 13-35 %. Similar trend is also observed in parameter determined of particle size retention, which is also comparatively on the higher side in case of former. All these observations are indicative of the fact, that higher BPH, yield and particle size retention obtained in case of samples prepared from coconut shells may be due to the higher inherent compact strength of this particular precursor, probably the most contributing factor towards enhancement of these characteristics.

Tables 3 and 4 depict the influence of process variables of activation time and operating pressure of activating gas, maintained during fluidization, on certain adsorptive characteristics of GAC samples prepared from the two precursors 'B' and 'C', in liquid as well as vapour phase systems (Bevia *et al* 1984). It is quite apparent in a review of these Tables, that the variable of operating pressure of fluidizing gas, as depicted in Table-4, was found to be more influential in increasing the adsorptive capacity of samples B_4, B_5 & C_4, C_3, as compared to that of activation time. It is further evident however, that in case of *Acacia arabica* ('B'), highest adsorptive activity was obtained in case of sample '$BP_2\ t_1\ D_1$' (B_5), obtained in minimum activation time of 45 minutes at an operating pressure of 15 psi. Moreover, this sample also has the highest surface area of pores>10, 15 and 28 Å, correlated with the adsorption data of the three adsorbates being investigated in the liquid phase system (Svendsen 1975). However, in the case of coconut shells ('C'), higher activity against either of the four adsorbastes in liquid and vapour phase

Table 2

Different parameters obtained during physical activation of Babul and coconut shells

Sample code	Yield (%)	Particle size retention (%)	Ball pan hardness no.
B_1	29.6	49	46
B_2	19.2	38	44
B_3	16.0	31	43
B_4	37.1	62	51
B_5	25.2	58	48
B_6	11.2	29	34
C_1	52.0	51	94.6
C_2	47.7	45	94
C_3	43.2	42	95.7
C_4	55.2	56	93
C_5	56.8	59	94
C_6	60.5	62	96

Table 3

Effect of fluidization time on different adsorptive characteristics of granular activated carbon samples prepared from Babul and coconut shells

Sample code	Iodine no. (mg g^{-1})	Methylene blue no. (mg g^{-1})	Molasses no. (mg g^{-1})	CCl_4 adsorption (%)	Surface area of Pores > 10 Å	Surface area of Pores > 15 Å	Surface area of Pores > 28 Å
B_1	780	234	170	40.2	713	669	82
B_2	814	262	215	54.1	745	749	104
B_3	825	270	225	56.6	755	772	108
C_1	1082	342	290	62.4	995	973	140
C_2	1100	360	310	69.6	1012	1030	149
C_3	1232	387	335	80.8	1135	1107	161

Table 4

Effect of operating pressure of different adsorptive characteristics of granular activated carbon samples prepared from Babul and coconut shells

Sample code	Iodine no. (mg g^{-1})	Methylene blue no. (mg g^{-1})	Molasses no. (mg g^{-1})	$CC1_4$ adsorption (%)	Surface area of Pores > 10 Å	Surface area of Pores > 15 Å	Surface area of Pores > 28 Å
B_4	735	180	140	38.8	671	515	67
B_5	940	290	305	50.6	863	829	147
C_4	1020	315	225	68.9	937	901	108
C_5	1165	345	275	70.8	1073	987	132
B_6*	865	265	240	56.2	792	758	116
C_6*	930	280	190	61.2	853	801	92

* Samples prepared with particle size 'D_2'

systems was achieved in case of sample '$CP_3 t_3 D_1$' (C_3), which was obtained at maximum operating pressure and activation time of 20 psi and 75 minutes respectively. It should be noted here, that comparatively higher activation time and operating pressure of fluidizing gas are required for achieving optimum activity in activated carbon samples prepared from precursor 'C' as compared to that of 'B'. It may be due to the solid compact nature of coconut shell particles, which require comparatively more severe operating conditions than that of *acacia* for the phenomena of pore development during the process of activation. The same is true in case of other parameters like yield, BPH No.etc., where the pattern originated corresponds, well with that of operating variables established separately for the two raw materials. Tables 2-4 also depict different physical and chemical characteristics obtained in the case of samples 'B_6' and 'C_6' obtained with the other particle size 'D_2' of the two raw materials, with the operating process variables, as established in the experiments described earlier. These results reveal that in *acacia*, although activity against all the adsorbates was quite reasonable, but due to lower inherent strength and smaller particle size (1.00 – 2.00 mm) of this particular raw material, percent yield obtained in case of 'B' was quite low (11.2 %), as compared to 25.2%, obtained with the larger particle size D_1 (2.00 – 3.00 mm). In the experiments performed with the second particle size (D_2) of coconut shells, it is quite evident from the data presented in Tables 2-4, that although yield in case of 'C_6' was quite higher but adsorptive activity prominently declined, probably due to lesser penetration of fluidizing gas and eventually lower burn off in comparatively larger size particles of coconut shells (2.00 – 3.00 mm).

It has therefore been concluded from these studies, that GAC of quite reasonable activity in liquid and vapour phase systems may be prepared from coconut shells in quite higher yield and ball pan hardness by physical activation with a mixture of superheated steam + air in a fluidized bed reactor. The operating variables found to be appropriate, as established from this study, were particle size of 1.00 – 2.00 mm, operating pressure of 20 psi in 75 min of activation time. However, in case of Babul *(Acacia arabica)*, not only yield and optimum adsorptive activity were comparatively lower, but ball pan hardness or resultant strength of the GAC samples was also far below the standard requirements.

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