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UTILIZATION OF BARLEY HUSK FOR THE PRODUCTION OF FURFURAL AND ACTIVE CARBON

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The present research work was undertaken to utilize barley husk as a raw material to prepare furfural and active carbon, under different experimental conditions. It was noted that the yield of furfural increased with increasing digestion time. However, yield of active carbon decreased with increasing digestion time. Moreover, H_2SO_4 furnished higher percentage of furfural than HCI at all solid-liquid ratios and digestion times. Among various salt catalysts, $ZnCl_2$ proved to be the best activating agent.. Maximum yield of active carbon was obtained with HCI in the presence of $CaCl_2$ as catalyst at 100 minutes digestion time. It was noted that $ZnCl_2$ along with 14 percent H_2SO_4 at 140 minutes digestion time gave a quality product of high adsorption capacity.

Key words: Barley husk, furfural, active carbon, acid hydrolysis, catalysts, adsorption capacity, Pakistan

INTRODUCTION

In Pakistan a huge quantity of agro wastes are being produced. These agro wastes are either burnt as fuel or improperly disposed off causing the problem of waste management and environmental pollution. Carbonaceous materials can effectively and efficiently be converted into furfural and activated carbon by proper chemical treatment.. Furfural is extensively used in pharmaceutical, petroleum, synthetic fiber, vegetable oil, fertilizers, beverages, food, textile, ceramics and polymer industries (Zeitsch, 2000).

Active carbon is used for clarifying and decolorizing liquids; in benzol recovery plants for the extraction of hydrogen sulphide from coal and coke-oven gases; the production of sulphur dichloride; the removal of fuel oil from alcohol; for assisting in the production of high vacuum; in the removal of odour from water; the removal of disagreeable fumes from various processes, and the recovery of vapours (Meng and Liang, 2000).

In the recent years several attempts have been made by Chughtai *et al* (1996 and 2000) to prepare furfural and active carbon; chemicals of great importance from cheap raw materials like Sawdust, Bagasse, Corn cobs and Khabble grass *(Cynodon dactylon).* The present research work was planned to utilize barley husk for the production of furfural and active carbon.

MATERIALS AND METHODS

Barley husk collected from rural areas of Faisalabad was sun dried for 10 days followed by oven drying at $105^{\circ}C$ to a constant weight.. The dried material was pulverized to a fine powder and was stored in a desiccator as a stock sample.

The stock samples (50g each) were refluxed with 150 mL, 250 mL and 500 mL HCI and H_2SO_4 for 100 and 140 minutes with added salt catalysts; NaCl, $CaCl_2$, $AlCl_3$ and $ZnCl_2$, separately. The refluxed material was distilled and the furfural was extracted from the distillate. All the experiments were conducted in triplicate. The extracted furfural was estimated colorimetrically by the method of Angle (1947). The residue obtained after the extraction of furfural was dried and directly activated at $700^{\circ}C$ for 1 hour in a muffle furnace. After cooling to room temperature, it was ground to a fine powder. The active carbon thus prepared was checked for its adsorption efficiency using iodine and methylene blue by standard method of Beg and Usmani (1985).

RESULTS AND DISCUSSIONS

Furfural

The means of three observations are given in Table 1. Maximum yield of furfural (16.2%) was obtained at 140 minutes digestion time when H_2SO_4 was used as hydrolyzing agent in the presence of $ZnCl_2$ as catalyst.. Sulphuric acid yielded more furfural than HCI at both digestion periods and solid: liquid ratios, thus proving it to be a better hydrolyzing agent than HCI. This may be attributed to the dehydrating nature of H_2SO_4. Sulphuric acid was also reported to be a better hydrolyzing agent than HCI by Bains *et al.* (1977) during thermal hydrolytic studies in wastes.

The results given in Table 1 revealed that by increasing digestion time from 100 to 140 minutes, the average yield of furfural increased both in case of HCI and H_2SO_4. This increase may be due to the reason that at longer digestion time greater conversion of pentoses to furfural takes place. These results are also in accordance with the findings of Chughtai *et al.* (1986).

Considering solid-liquid ratio it was noted that 1:10 ratio furnished better percentage of furfural than 1:5 and 1:3 under all sets of conditions. This enhancing effect may be attributed to the fact that at lower solid-liquid ratio insufficient amount of hydrolyzing agent was available for the conversion of pentoses to furfural..

The catalysts have remarkable positive effect on the percentage yield of furfural. Among the four catalysts employed $ZnCl_2$ and $AlCl_3$ furnished better results than others.

Table 1. Average yield of furfural from barley husks obtained under various conditions.

Digestion Time (Min)	Solid liquid ratio	Acid used 14 (%)	Salt catalysts (2g/50 g sample)				
			Nil	NaCl	$CaCl_2$	$AlCl_3$	$ZnCl_2$
100	1:10	H_2S0_4	8.3	12.4	13.3	14.9	15.5
		HCl	6.1	9.3	12.2	12.8	14.8
	1:5	H_2S0_4	6.3	6.4	7.1	9.7	10.9
		HCl	5.7	5.9	6.1	7.1	6.8'
	1:3	H_2S0_4	4.0	4.3	3.9	5.9	4.5
		HCl	3.2	3.3	3.5	4.9	5.1
140	1:10	H_2S0_4	8.7	13.2	12.7	15.1	16.2
		HCl	7.2	10.3	9.2	13.3	11.9
	1:5	H_2S0_4	7.7	6.5	7.4	10.7	11.2
		HCl	6.2	5.9	6.9	7.4	9.9
	1:3	H_2S0_4	6.4	3.9	4.8	4.2	5.5
		HCl	3.7	5.4	5.2	6.1	6.5

Table 2. Average yield of active carbon obtained from the residue left after the extraction of furfural from barley husks.

Digestion Time (Min)	Solid liquid ratio	Acid used (14%)	Salt catalysts (2g/50 g sample)				
			Nil	NaCl	$CaCl_2$	$AlCl_3$	$ZnCl_2$
100	1:10	H_2S0_4	9	9.4	10.2	8.1	9.3
		HCl	10.3	12.6	13.4	9.2	8.9
	1:5	H_2S0_4	10.0	10.4	11.3	10.2	10.3
		HCl	11.2	11.7	15.6	10.8	9.8
	1:3	H_2S0_4	17.2	18.4	18.9	15.2	14.3
		HCl	18.3	20.2	21.5	14.2	13.3
140	1:10	H_2S0_4	8.1	8.3	9.3	7.2	8.1
		HCl	9.2	11.5	12.4	8.2	7.9
	1:5	H_2S0_4	9.1	9.3	10.5	8.3	9.4
		HCl	10.2	10.5	14.5	9.4	8.9
	1:3	H_2S0_4	15.2	16.3	16.9	14.2	13.6
		Hel	17.5	18.9	19.5	13.2	12.2

Active carbon

The maximum percentage yield of active carbon (21.5%) was obtained with HCl at 100 minutes digestion time in the presence of $CaCl_2$ as catalyst at 1:3 solid liquid ratio. Taking into account the effect of acids on the yield of active carbon it was observed that HCl is a better activating agent than H_2S0_4. Average yield of active carbon decreased with increasing solid-liquid ratios and digestion times.

The results reported in Table 2 showed that the percentage yield of active carbon decreased with increasing digestion time for both the acids in the presence of salt catalysts. A similar trend has been reported by Junfeng *et al.* (1999). Presence of all salt catalysts increased the percentage yield of active carbon. Moreover, $CaCl_2$ and NaCl were noted as better catalysts than $AlCl_3$ and $ZnCl_2$.

The adsorption capacity of active carbon was evaluated with iodine and methylene blue decolourization (Beg and Usmani, 1985). The results presented in table 3 evidently indicate that $ZnCl_2$ has given maximum adsorption capacity of active carbon in terms of methylene blue and iodine. i.e 56mg/g and 309.68mg/g respectively using H_2SO_4 keeping solid liquid ratio 1:10 at 140 minutes digestion time.

[illegible] from barley husks

[illegible]	[illegible] 0-10	[illegible]	[illegible]									
			[illegible]		[illegible]		[illegible]		[illegible]		[illegible]	
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Taking into consideration, the effect of digestion period, it is evident from Table 3 that adsorption activity of active carbon increased with increasing digestion periods keeping all the other factors constant. The maximum adsorption capacity of active carbon for methylene blue and iodine *(56mg/g* and *309.68mg/g,* respectively) was obtained at 140 minutes digestion period. It can thus be concluded that H_2SO4 is a better hydrolyzing/activating agent as compared to HCI for the preparation of good quality furfural and active carbon from barley husk.

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