

Effect of Chemical Treatment on the Production of Furfural and Active Carbon from Rice Husks

SHAUKAT ALI, FIYYAZ AHMAD CHUGHTAI, NAJIA IRUM AND KIRAN AFTAB
Department of Chemistry, University of Agriculture, Faisalabad-38040, Pakistan

ABSTRACT

The effect of chemical treatment on the production of furfural and active carbon was studied. Rice husks was hydrolyzed with various concentrations of sulphuric and hydrochloric acids and thereafter distilled to get furfural. The residue after the extraction of furfural was directly activated at 700°C to produce active carbon. The active carbon thus prepared was checked for its adsorption capacity using iodine and methylene blue. Significant yield of high quality furfural (19.92%) and active carbon (42.60%) was obtained. The quality of active carbon was compared with commercially available active carbon (E. Merck). Sulphuric acid proved to be a better hydrolyzing/activating agent as compared to hydrochloric acid.

Key Words: Rice husks; Furfural; Active carbon; Hydrolyzing agents; Adsorption

INTRODUCTION

Pakistan has agro-based economy and agriculture accounts for a large share of GDP and employment. Besides, a number of useful products, a large quantity of agro-wastes are also produced which is not properly used at present. These agro-wastes are either burnt as fuel or improperly disposed off causing the problem of waste treatment and environmental pollution. However, in recent years attention has been focused on the proper utilization of these agro-wastes as a potential source of industrially important chemicals such as furfural and active carbon. These two important chemicals can serve as a nucleus for the production of a large number of other chemicals. They also find extensive applications in many industries such as polymer, insecticide, herbicide, petrochemicals, food, pharmaceutical, oil and fat, fertilizer, etc. Rice is one of the major cash crops of Pakistan. Two wastes *viz.* rice straw and rice husks are produced during the processing of rice. Recently some indigenous materials have been recognized as potential sources of furfural and active carbon (Chughtai *et al.*, 1986, 1987, 1990, 1999). The present study is the continuation of previous work done and involves the utilization of rice husks as a potential source of furfural and active carbon.

MATERIALS AND METHODS

Preparation of stock sample. Rice husks was collected from a rice mill near Jaranwala, district Faisalabad, dried to a constant weight in oven at 110°C and stored in a desiccator as stock sample.

Furfural. The stock sample (50 g each) was refluxed with 500 and 250 mL of various concentrations (14 and 16% w/v) of commercial hydrochloric acid and sulphuric acid separately each for 100 and 140 minutes. The refluxed material was distilled (simple and steam distillation), and

the distillate thus obtained was redistilled. The distillate was collected till it gave red colour with aniline acetate. Furfural was, thereafter separated from its aqueous solution according to the method described by Chughtai *et al.* (1986).

Active carbon. The residue obtained after the recovery of furfural was dried in an oven at 110°C and directly activated at 700°C for one hour in a muffle furnace. The product thus obtained was cooled to room temperature, weighed and ground to a fine powder. The adsorption capacity of the resulting activated carbon was tested using iodine and methylene blue (Beg & Usmani, 1985) and was compared with commercial activated carbon.

RESULTS AND DISCUSSION

Production. The average yield (%) of furfural and active carbon obtained by using various concentrations of H₂SO₄ and HCl under different sets of conditions is given in Table I. Taking into consideration the acid concentration it was observed that 14% H₂SO₄ and HCl furnished higher percentage of furfural and active carbon as compared to 16% of both the acids. The results are similar to the findings of Khan and Nawaz (1983) and Chughtai *et al.* (1996, 2000) who also reported H₂SO₄ as a better hydrolyzing agent. This decrease in yield with increase in concentration of the hydrolyzing agent may be attributed to the destructive effect of concentrated acid. An increase in percentage yield of furfural was also observed with increasing digestion time from 100 to 140 min for both simple and steam distillation, keeping all the other factors constant. This observation is in harmony with the findings of Sharma and Sahgal (1983) who reported that percentage yield of furfural increased with time and then decreased after a certain period of time. This may probably be due to the reason that at longer digestion time, greater extent of pentosans of the raw material was converted to furfural. Taking into account

Table I. Comparison between average yield (%) of Furfural and active carbon at various acid concentrations and digestion periods using H₂SO₄ and HCl with simple and steam distillation

Digestion time (min)	Acid used	% age	Simple distillation				Steam distillation			
			1:5 W/V		1:10 W/V		1:5 W/V		1:10 W/V	
			Furfural (%)	Active carbon (%)	Furfural (%)	Active carbon (%)	Furfural (%)	Active carbon (%)	Furfural (%)	Active carbon (%)
100	H ₂ SO ₄	14	13.68	33.80	11.55	36.00	19.92	29.80	13.20	34.60
		16	12.98	34.00	4.80	40.30	13.24	32.40	7.44	36.40
		14	9.48	35.50	8.40	38.70	10.56	35.10	9.67	34.00
	HCl	16	8.60	37.00	7.69	4.00	9.44	36.00	8.80	35.10
		14	12.76	34.28	7.09	42.60	17.12	31.00	12.24	38.10
		16	14.04	35.46	6.00	39.00	14.90	32.00	9.42	36.64
140	H ₂ SO ₄	14	8.10	36.20	4.08	39.20	10.42	35.60	7.50	35.34
		16	5.40	38.40	3.69	41.28	7.56	36.40	5.92	36.00
	HCl	16	5.40	38.40	3.69	41.28	7.56	36.40	5.92	36.00

solid-liquid ratio it was noted that 1: 5 ratio furnished higher percentages of furfural as compared to 1: 10 under similar conditions. Distillation process also affected the percentage yield of furfural and higher percentages of furfural were produced by steam distillation as compared to simple distillation. The maximum yield of furfural 19.92% was thus obtained when the raw material was hydrolyzed for 100 minutes using 14% H₂SO₄ keeping solid liquid ratio 1:5 and there after steam distilled.

No prominent variation in percentage yield of active carbon was observed under different sets of conditions. However hydrochloric acid as compared to sulfuric acid gave comparatively better results. It was further observed that percentage yield of active carbon increased with increasing acid concentration from 14 to 16%, digestion time from 100 to 140 minutes and solid liquid ratio from 1:5 to 1:10. From the residue left after the extraction of furfural by simple or steam distillation. The results are further substantiated by the finding of Chughtai *et al.* (2000) who reported steam distillation to be better for the extraction of furfural from corn cobs, bagasse, pericarp of peanut and sawdust. Comparing the two distillation methods it was observed that higher percentage of active carbon was obtained from the sample left after the extraction of furfural by simple distillation as compared to steam distillation. This may be due to the reason that increased break down of

pentosan material to give high yields of furfural by using the steam distillation thereby accompanied by a decrease in the yield of active carbon.

Quality evaluation. The results shown in Table II indicated that the adsorption capacity of active carbon for both iodine and methylene blue decreased by increasing the acid concentration from 14 to 16% for both the acids (HCl, H₂SO₄). A uniform increase in adsorption capacity was observed by increasing digestion time from 100 to 140 min for both the acids (H₂SO₄, HCl) with and without steam distillation. This may imply that longer digestion time caused more break down of the carbonaceous material. The results depicted in Table II make it clear that the steam distillation has negative effect on adsorption efficiency of active carbon and better results were obtained with simple distillation. It was further noted that the adsorption capacity of active carbon was lower than the standard sample (E. Merck). Maximum adsorption capacity (89 mg of methylene blue and 495.30 mg of I₂/g of active carbon) was given by the sample treated with 14% H₂SO₄ for 140 min digestion time keeping solid liquid ratio 1:10. The results are in accordance to the findings of Chughtai *et al.* (2000) who also reported an enhancement in adsorption capacity by increasing digestion period, when they extracted active carbon from Khubble grass. The results also indicated that more adsorption of methylene blue and iodine was observed

Table II. Comparison between average adsorption capacities expressed as mg of iodine and methylene blue adsorbed by 1 g activated carbon (Powder form)

Digestion time (min)	Acid used	% age	Simple distillation				Steam distillation			
			1:5 W/V		1:10 W/V		1:5 W/V		1:10 W/V	
			Methyl-ene blue	Iodine	Methyl-ene blue	Iodine	Methyl-ene blue	Iodine	Methyl-ene blue	Iodine
100	H ₂ SO ₄	14	50	282.61	83	462.49	47	266.88	74	409.29
		16	39	223.70	57	321.75	39	210.60	39	216.57
		14	35	199.95	33	191.95	35	194.27	32	186.31
	HCl	16	35	198.50	31	182.78	32	185.50	33	191.34
		14	66	365.58	89	495.30	48	268.47	76	426.32
		16	44	247.42	62	347.89	41	228.49	41	228.53
140	H ₂ SO ₄	14	41	228.00	33	192.40	37	209.47	40	225.00
		16	49	275.25	32	191.34	37	208.76	36	203.45
	HCl	16	49	275.25	32	191.34	37	208.76	36	203.45

Standard: 1g of commercial active carbon (E. Merck) adsorbed 663 mg of Iodine and 120 mg of Methylene blue

by the samples obtained keeping solid-liquid ratio at 1:10 as compared to the active carbon samples obtained with 1:5 ratio under all sets of conditions.

CONCLUSIONS

It can thus be concluded that 16% H₂SO₄ is a better hydrolyzing/activating agent, 140 min optimum digestion time and 1:10 the best solid-liquid ratio. Simple distillation proved to be a better process than steam distillation. When the results were compared with the previous work it was noted that rice husks furnished lower percentage of furfural but higher amounts of better quality active carbon as compared to Khabble grass, corn cobs, bagasse, pericarp of peanut and sawdust.

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