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PREPARATION OF FURFURAL FROM KAI (*SACCHARUM SPONTANEUM*)

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KAI was utilized, as a raw material, for the production of furfural. The effect of various acid concentrations and different digestion periods was studied at a constant temperature of 100 ± 0.5°C on the yield of furfural. Optimum yield 9.66 per cent was achieved with 15 per cent v/v hydrochloric acid for a digestion period of 150 minutes.

The effect of catalysts on the optimum yield of furfural revealed that sodium chloride and calcium chloride had a little effect whereas aluminium chloride increased the yield from 9.66 per cent to 11.39 per cent.

INTRODUCTION

Furfural (2-furfuraldehyde) is an important organic compound due to its applications in various chemical industries. It is used as a selective solvent in vegetable oil, drying oil and in petroleum refining for the purification of lubricating oil, gas oil and diesel oil. It is employed as paint and varnish remover. Furfural and its derivatives are also used in pharmaceutical insecticide, herbicide and fungicide preparations. It is widely used in plastic industry and for the manufacture of synthetic rubber, nylon "66" fibre and perfumes.

Furfural has been obtained from a variety of vegetative raw materials and agricultural wastes such as bagasse, rice hulls, wheat bran, corn cobs and pericarp but such an attempt has not been made on KAI which is a cheeper and exuberantly available raw material in Pakistan. Therefore, this project was undertaken to prepare furfural from KAI.

MATERIALS AND METHODS

For digestion, dried stock sample of KAI (10 g) was taken in round bottom

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flask of 500 ml and heated at a constant temp. 100 ± 0.5 °C under reflux at various hydrochloric acid concentrations (8, 10, 12, 15, 18 and 20 per cent v/v) and time periods (60, 90, 120, 150 and 180 minutes). Digested material was distilled and the distillate was collected till it gave negative test with aniline acetate.

Distillate was neutralized with sodium hydroxide using phenolphthalein as an indicator. Estimation of furfural from the neutralized distillate was made according to the Stillings and Browning's method (1940). The separation of furfural from the distillate was made by following the techniques and instructions of Ryabenkii *et al* (1936). Colour, odour, boiling point and refractive index of the separated furfural were checked and compared with standard furfural. Furfural phenyl hydrazone, a derivative of furfural was prepared with phenyl hydrazine in glacial acetic acid and its melting point was noted.

RESULTS AND DISCUSSION

The effect of various acid concentrations and digestion periods at 100 ± 0.5 °C on the yield of furfural from KAI has been presented in Table 1.

Table 1. *Average yield (in percentage) of furfural at various acid concentrations and digestion periods*

Digestion time in minutes	Acid Concentrations (% v/v)					
	8	10	12	15	18	20
60	4.49	4.81	5.35	6.06	6.63	6.32
90	4.62	6.03	6.88	7.09	7.48	6.71
120	5.36	6.97	7.48	7.74	8.08	7.21
150	6.96	7.28	7.92	9.66	9.02	7.73
180	4.55	5.21	6.09	6.80	6.54	5.90

From the study it is clear that optimum yield of furfural was 9.66 per cent with 15 per cent hydrochloric acid at a digestion period of 150 minutes while at 8 per cent acid concentration and 60 minutes digestion time yield was minimum (4.49%).

Considering the effect of acid concentrations, it is observed that with increasing acid concentration the yield first increased to an optimum level and

beyond that limit, the yield started decreasing. This trend has also been observed by Sharma and Sahgal (1983) who reported that an increase in acid concentration (within limit) caused an increased yield of furfural. The low yield at 8, 10, 12 per cent acid concentrations may be due to incomplete hydrolysis of pentosans into pentoses whereas low yield at 18 and 20 per cent hydrochloric acid may be attributed to the destruction of furfural at higher acid concentrion.

Taking into account the effect of digestion periods, it is obvious that yield first increased with increasing digestion period upto 150 minutes and beyond that it decreased. Increase in the yield of furfural upto 150 minutes seems to be reasonable because furfural formation from pentosans is a slow process. However, the low yield beyond this optimum level is probably due to formation of polymer at long duration.

Various catalysts like sodium chloride, calcium chloride and aluminium chloride in different concentrations of 0.25, 0,5, 1 and 2 per cent were used to note their effect on the production of furfural at optimum condition of 15 per cent acid concentration and 150 minutes digestion period. Results thus obtained (Table 2) revealed that sodium chloride and calcium chloride showed a little effect whereas 1 per cent aluminium chloride further increased the yield from 9.66 to 11.39 per cent.

Table 2. *Effect of catalysts ($NaCl$, $CaCl_2$, $AlCl_3$) on the yield* 9.66 *per cent of furfural at* 150 *minutes digestion time and* 15 *per cent v/v acid concentrations*

% of Catalysts used	NaCl	$CaCl_2$	$AlCl_3$
0.25	9.69	9.73	9.84
0.50	9.75	9.84	10.13
1.00	9.83	9.92	11.39
2.00	9.81	9.93	11.37
3.00	9.80	9.92	11.38

Recovery of furfural was 56.8 per cent on the basis of estimated amount. Recovered furfural was a colourless liquid which boiled at 162°C (lit. 161.7°C) with refractive index 1.519 at 25° (lit. 1.521 at 25°C). Melting point of furfural phenyl hydrazone was 96 - 97°C (lit. 97 - 98°C).

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