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Production of furfural and active carbon by chemical treatment of Kai

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ABSTRACT

In order to cope with the current situation and to cater the needs of our industry, it is imperative to exploit our own resources. For this purpose present research work was carried out to study the effect of chemical treatment on the production of furfural and active carbon from Kai (an agro-waste). Kai was impregnated with 14 % HCl and 14% H_2SO_4 for different digestion periods (100 and 140 minutes) by keeping the sample acid ratio of 1:5 and 1:1, using various salt catalysts (NaCl, $CaCl_2$, $ZnCl_2$, $AlCl_3$). Furfural was estimated colorimetrically. The adsorption capacity of active carbon prepared by the residue obtained after the recovery of furfural was evaluated by using iodine and methylene blue method. The results indicated that with increase in digestion time the furfural yield increases upto certain limit. In contrast to yield of active carbon the adsorption efficiency increases with increasing time period. In case of sample acid ratio greater yield of furfural was achieved with 1:5 ratio. While maximum yield of active carbon and its adsorption efficiency is attained with 1:10 ratio. The study proved that H_2SO_4 was better hydrolyzing agent for maximum yield of furfural and to increase the adsorption capacity of active carbon. The use of salt catalyst increases the adsorption capacity. Among the salts $ZnCl_2$ proved to be the best catalyst for furfural yield and for adsorption capacity of active carbon but average yield of active carbon was greater with $AlCl_3$.

Keywords: Chemical treatment, furfural, active carbon, hydrolyzing agents, adsorption efficiency.

INTRODUCTION

Pakistan is an agricultural country, a wide variety of agro-wastes are thus available in large amount. These agro-wastes are either burnt as fuel or improperly disposed off causing the problem of waste treatment and environmental pollution. Attention has been focused to utilize these agro-industrial wastes for the production of commercially important chemicals like, furfural and activated carbon [1]. These two important chemicals can serve as a nucleus for the production of a large number of other chemicals. Furfural (an industrial solvent) is a colourless, inflammable, volatile, aromatic liquid [2]. It is used mainly as a selective extracting solvent in the lubricating oil of the petroleum industry. It is also used in polymer industry and takes part in the production of nylon 66 and resins used for moulding powders [3]. It is employed as an additive in syrup industry. An amorphous form of carbon with high adsorption efficacy is called active carbon [4]. Activated carbon removes organic and inorganic contaminants from water.

Printing industry uses activated carbon to recover solvents. It is used in the production of consumer products such as water, sugar, wine, soup, gelatin and vinegar. Pharmaceutical industry uses highly pure activated carbon, e.g. in the purification of vitamins and paracetamol. A number of successful attempts have been made to produce furfural and active carbon from dhabb *(Typha angustata)*, saw dust, corn cobs and bagasse [5-6]. In a quest for better raw material, present study was designed to produce furfural and active carbon by chemical treatment of kai. Local production of these chemicals will not only solve the problem of waste treatment but will also save the foreign exchange spent on their import.

MATERIALS AND METHODS

Collection and preparation of raw material: Kai was collected from the local area of Faisalabad. After collection it was washed several times with tap water to remove extraneous material followed by washing with distilled water. The washed material was firstly sun dried then oven dried to a constant weight. The dried material was pulverized to a fine powder and was stored in a desiccators as a stock sample.

Digestion and Distillation: The stock samples (50 g each) were digested with 500 mL and 250 mL of 14% commercial sulphuric acid for 100 and 140 minutes with 1 g salt catalyst like NaCl, $AlCl_3$, $CaCl_2$ and $ZnCl_2$. Similar process was followed for preparation of furfural using 14% HCl as hydrolyzing agent. The digested samples were distilled and distillate was collected till it gave negative test with aniline acetate.

Estimation, Separation and Identification of Furfural: The prepared furfural was estimated colorimetrically as described by Angel [7]. Furfural percentage on the basis of dry matter sample of Kai was calculated. Furfural was separated from aqueous solution by using the technique of Chughtai *et al*. [8]. Physical and chemical methods were performed to ensure that the recovered chemical was furfural. Physical tests included the comparison of color, odor, and boiling point of purified furfural with that of standard sample. Chemical tests included the formation of furfural phenyl hydrazone.

Preparation of active carbon: The residue after the extraction of furfural was used as starting material for the preparation of active carbon and it was activated directly in the absence of air for an hour in muffle furnace at $700^{\circ}C$. The activated samples of carbon were removed from furnace and grinded to fine powder of 200 sieve mesh. The prepared active carbon was weighed then packed in plastic bottles and stored in desiccator for measuring adsorption efficiency.

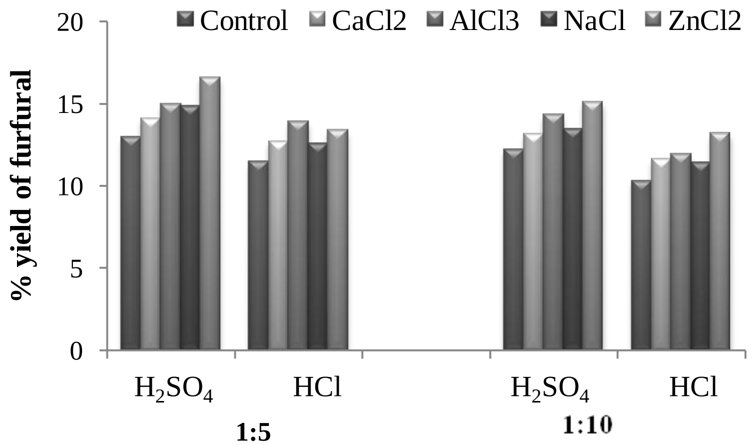
Adsorption efficiency: The adsorption efficiency of the prepared active carbon was checked by using comparison method. The adsorption showed by various samples was compared with each other and with commercially available active carbon (E-Merk) against methylene blue and iodine by following the methods of Beg and Usmani [9].

Statistical Analysis: The data obtained both from furfural and active carbon was analyzed statistically using Variance Technique in Completely Randomized Design with factorial arrangement of treatment by the method of Steel and Torrie [10].

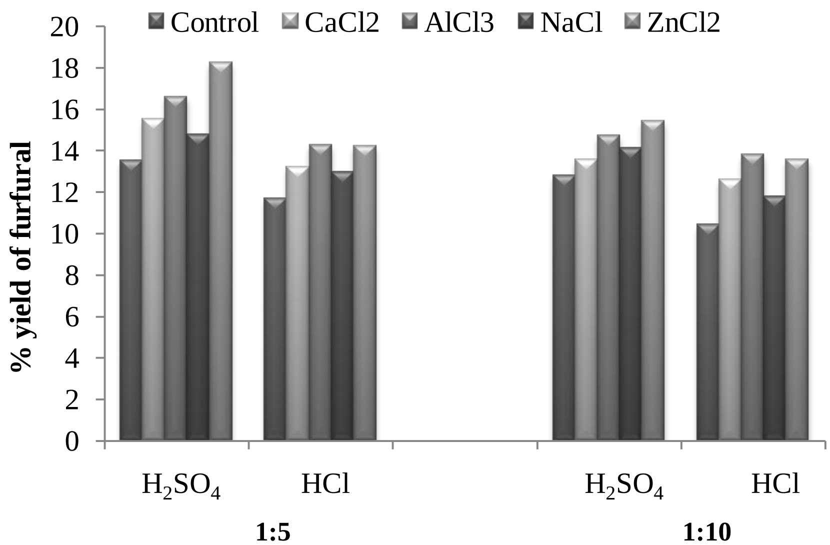
RESULTS AND DISCUSSION

For the present work, Kai an agro-material has been hydrolyzed to prepare furfural and active carbon under various experimental conditions. Effect of digestion time, acids, salt catalysts and sample acid ratio on the percentage yield and adsorption capacity of active carbon has been studied.

Effect of Digestion time on average yield of furfural: Results obtained regarding the effect of digestion time on % yield of furfural are shown in Table Fig. 1(a & b) which indicated that maximum yield (18.23%) of furfural is obtained when material was digested for 140 minutes than 100 minutes. This result is in accordance with Chughtai *et al*. [8, 11] who observed that with increasing digestion time the furfural yield first increased and the decreased. The decrease in yield was due to severe decomposition of product at longer digestion time.



(a)



(b)

Fig. 1 Comparison b/w average furfural yield with various solid liquid ratios, different acids and with different salt catalysts at 100 (a) and 140 (b) minutes digestion time

Effect of ratio on average yield of furfural: Considering the effect of sample acid ratio it is clear that in case of H_2SO_4 there is clear cut difference in the yield of furfural. Yield was greater when digestion occur with H_2SO_4 with ratio 1:5 w/v solid-liquid ratio but with HCl there was no much difference as shown in Fig. 1 (a and b). Furfural yield seemed to be decreased due to destructive effect of acid.

Effect of Digestion time on average yield of furfural: Fig. 1(a and b) indicated that H_2SO_4 furnished higher percentage of furfural than HCl proving it to be better hydrolyzing agent as compared to HCl. These results are in accordance with the results of Khan *et al*. [12] who reported H_2SO_4 to be better hydrolyzing agent than HCl.

Effect of catalyst on average yield of furfural: The data presented in Fig. 1 (a and b) showed that the maximum yield of furfural is obtained with $ZnCl_2$ by using both the acids H_2SO_4 and HCl. The maximum yield (18.23 %) was obtained with H_2SO_4 and $ZnCl_2$. $AlCl_3$ also improve the quantity of furfural but NaCl and $CaCl_2$ are not much effective salt catalysts.

Combined effect: Results pertaining to the combined effect of digestion time, ratio, acids and salt catalysts showed that the maximum average yield 18.23 % of furfural was obtained by use of H_2SO_4 as hydrolyzing agent in 1:5 solid liquid ratio with $ZnCl_2$ as catalyst and digestion time of 140 minutes.

Minimum yield was obtained at 100 minutes of digestion time, HCl used as hydrolyzing agent in 1:10 solid ratio and using no salt catalyst.

Active Carbon

Yield of active carbon: From each experiment the residue left after the extraction of furfural was activated at 700°C in the absence of air in muffle furnace and percentage yield of activated carbon was determined. Temperature, solid liquid ratio, digestion time and salt catalysts had a great influence on percentage yield.

Effect of Digestion time on average yield of active carbons: The data compiled in Fig. 2 (a and b) revealed that % yield of active carbon was decreased by increasing the digestion time for both acids. Maximum yield of activated carbon (39.5 %) was obtained from the residue which was refluxed for 100 minutes with 14 % HCl.

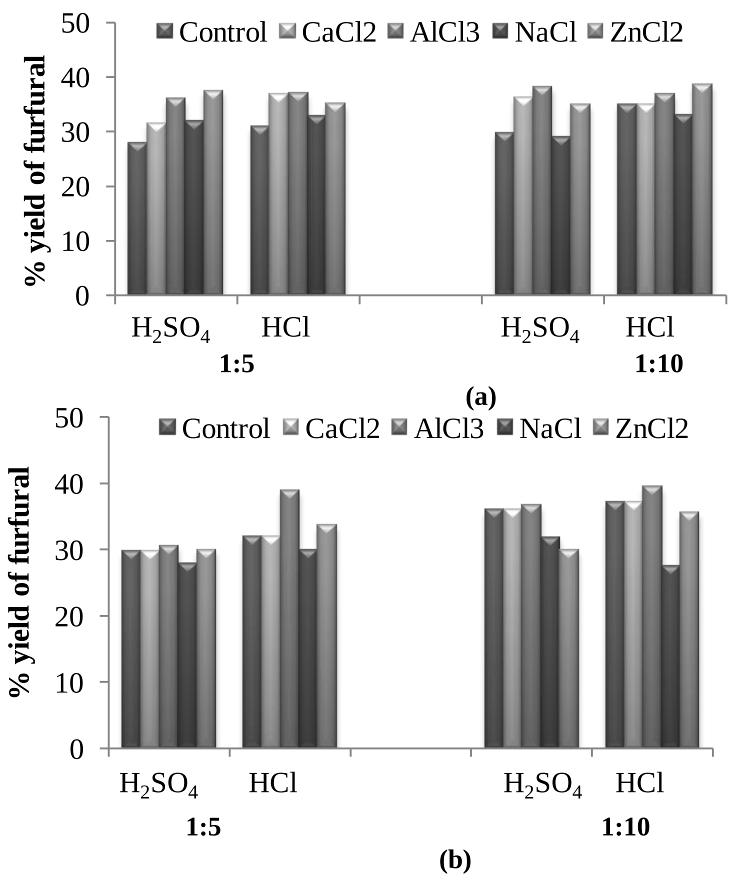


Fig. 2 Comparison b/w average yield of active carbon with various solid liquid ratios, different acids and with different salt catalysts at 100 (a) and 140 (b) minutes digestion time

Effect of Acid on average yield of Active Carbons: Fig. 2 (a and b) illustrated that the maximum yield (39.5 %) was furnished by HCl. The overall study of showed that the % yield of active carbon was greater with HCl as compared to H_2SO_4.

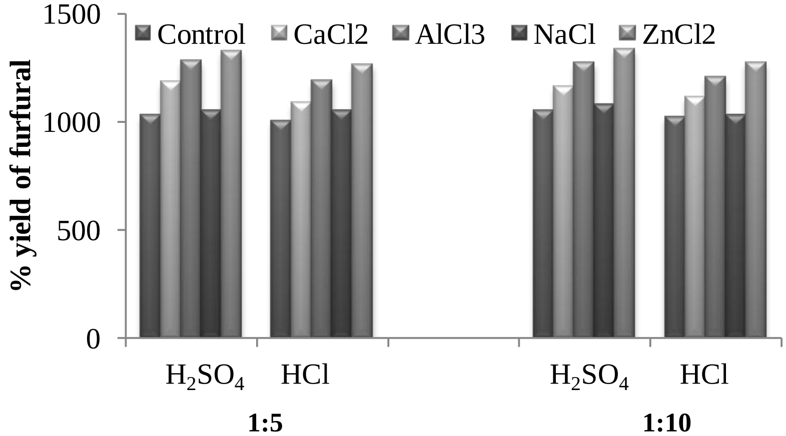
Effect of ratio on average yield of Active carbon: Results obtained regarding the effect of solid liquid ratio on % yield of active carbon shows that % yield increased by increasing the ratio 1:10 w/v for both acids and salt catalysts.

Effect of Salt Catalysts on average yield of Active carbons: The data showed in Fig. 2 (a and b) revealed that $AlCl_3$ and $ZnCl_2$ were the better activating agent for HCl as compared to H_2SO_4. NaCl and $CaCl_2$ proved to be poorer than the other two. $AlCl_3$ gave the maximum yield of activated carbon i.e. 39.5

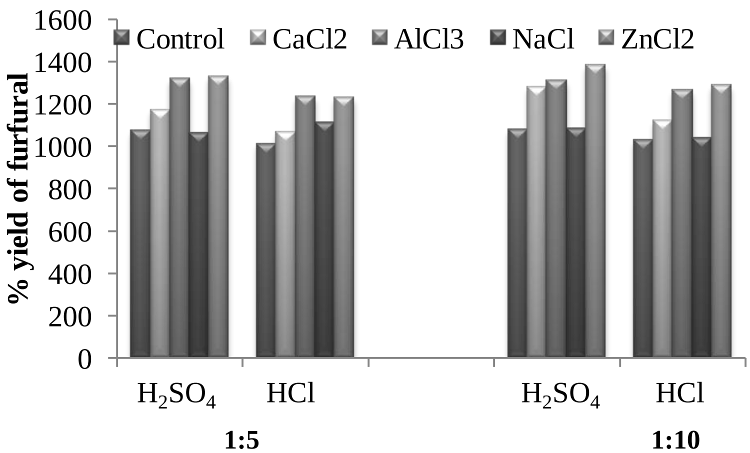
% with 14 % HCl and $ZnCl_2$ gave 37.5 % of active carbon with 14% H_2SO_4. All the four catalysts showed increasing effect on the yield than without any salt catalysts.

Combined effect: Fig. 2 (a and b) showed that greater yield of active carbon was obtained when we use 1:10 solid liquid ratio and was digested for 100 minutes in contrast to yield of furfural for both the acids. In case of acids the HCl proved to give the better yield. Higher yield in case of HCl indicated that the raw material was less prone to decomposition with HCl as compared to H_2SO_4. Moreover the catalysts improved the yield of active carbon to some extent.

Adsorption Efficiency Of Active Carbon : The adsorption capacity of active carbon was checked with iodine and methylene blue method [9]. The effect of different factors on adsorption capacity of active carbon is discussed below



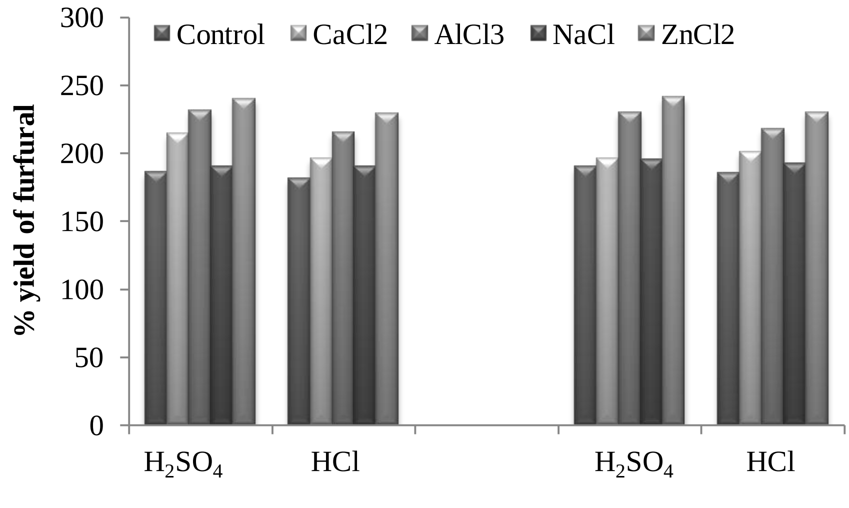
(a)



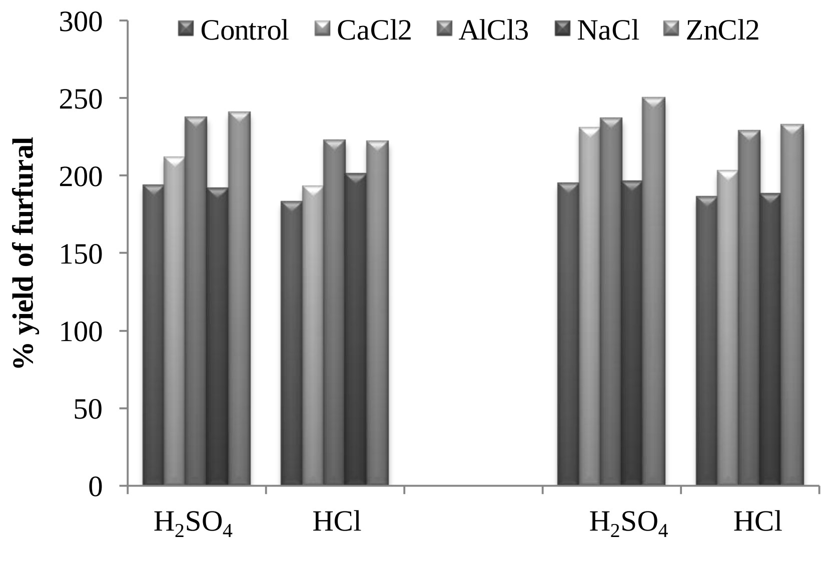
(b)

Fig. 3 Comparison b/w adsorption efficiency of active carbon of iodine with various solid liquid ratios, different acids and with different salt catalysts at 100 (a) and 140 (b) minutes digestion time

- 1g commercial active carbon adsorbed 663 mg of iodine



(a)



(b)

Fig. 4 Comparison b/w adsorption efficiency of active carbon of methylene blue with various solid liquid ratios,different acids and with different salt catalysts at 100 (a) and 140 (b) minutes digestion time

- 1g commercial active carbon adsorbed 120mg of methylene blue

Effect of Digestion time on adsorption capacity of active carbon: The effect of four factors on the adsorption capacity of active carbon is summarized in Fig. 3 and 4 (a and b). The results indicated that the adsorption efficiency increased by increasing the digestion time for both acids and with all salt catalysts. These finding are in harmony with the results reported by Chughtai *et al*. [11, 13-14].

Effect of acids on adsorption capacity of active carbon: The adsorption capacity of active carbon for iodine and methylene blue increased with H_2SO_4 and maximum adsorption capacity of active carbon for iodine and methylene blue was 1333.28 mg and 250 mg/g respectively, with 14 % H_2SO_4.

Effect of salt catalysts on adsorption capacity of active carbon: Taking into account the salt catalysts it was obvious that the catalyst helped a lot in improving the adsorption efficiency of active carbon $ZnCl_2$

was found to be the best catalyst when used with both H_2SO_4 and HCl. However gave maximum yield with H_2SO_4. The order of salt catalysts to increase the adsorption efficiency was NaCl □ $AlCl_3$ □ $ZnCl_2$. This order was in agreement with Chughtai *et al*. [13].

Combined effect: The results reported in Fig. 3 and 4 (a and b) showed that the adsorption capacity of active carbon increases with increase of digestion time as well as with 1:10 w/v sample acid ratio. Moreover the sample obtained with H_2SO_4 gave higher adsorption of iodine and methylene blue at all concentrations and digestion time proving itself to be a better activating agent as compared to HCl. Among the catalysts the sample treated with $ZnCl_2$ gave better results showing that it is best catalyst.

APPLICATIONS

Furfural can be used as a selective extracting solvent in the lubricating oil of the petroleum industry and can be used in polymer industry. Activated carbon prepared by Kai can remove organic and inorganic contaminants from water more efficiently. It can be used in the production of consumer products such as water, sugar, wine, soup, gelatin and vinegar.

CONCLUSIONS

The maximum average yield of furfural was obtained by use of H_2SO_4 as hydrolyzing agent in 1:5 solid liquid ratio with $ZnCl_2$ as catalyst and digestion time of 140 minutes. While greater yield of active carbon was obtained with 1:10 solid-liquid ratio and 100 minutes digestion time by using HCl. But H_2SO_4 showed higher adsorption capacity as compared to HCl. This project is of commercial nature and may be extended to other agro industrial wastes. The basic purpose of this study was to use cheap and easily available indigenous material for the production of furfural and active carbon. This attempt would helpful in local production of these chemicals.

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