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Synthesis and Identification of Furfural from Sunflower Husk

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Abstract: This study proposed to optimize the conditions for furfural synthesis from sunflower husk by acid hydrolysis which has become a seasonal source of air pollution. In this route we had taken the different weight percentage from 1wt% to 35 wt% of hydrochloric acid. Sunflower husk characterization was done by using moisture contain and ash contain analysis and separation of furfural is done with the help of distillation. Product was characterized by volumetric process such as bromine- bromide excess method. Identification of furfural was done by using FTIR analysis. Furfural and its derivations are intended chemicals due to several possible applications.

Keywords: Acid hydrolysis, sunflower hulls, furfural, FTIR.

I. INTRODUCTION

Numerous crops generate a huge amount of biomass (lignocellulosic and starch). However, not all of this biomass is used by industry. The substitute is to use this biomass as a feedstock for the production of oligosaccharides and monosaccharides by hydrolysis due to the large availability and lower cost of vegetal biomass and the need to reduce waste production.[1,2] Acid hydrolysis can be done by using concentrated or dilute acid solutions. Dilute acid solutions are principally useful when lignocellulosic biomass has a significant proportion of hemicellulose because the hemicellulosic fraction is more easily hydrolyzed than cellulose. Concentrated acid solutions require lower processing temperatures and pressures, and the hydrolysis reaction occurs at a lower process time.[3] Acid hydrolysis using concentrated acid solutions produces high yield of glucose from cellulose. In lignocellulosic hydrolysis from biomass, sulfuric acid is frequently used, although other mineral acids, such as hydrochloric, nitric and phosphoric acid are also used.[4,]

Furfural is a renewable biochemical produced from lignocellulosic biomass that has many different uses. It is considered an excellent solvent for many organic materials, such as resins and polymers. It is also a precursor to other desired compounds such as furfuryl alcohol (via hydrogenation), furan (via decarbonylation) and tetrahydrofuran (via hydrogenation of furan).[6] Similarly, it can serve as the starting material for the production of 5-hydroxymethylfurfural (by way of hydroxymethylation with formaldehyde).[7]

Furfural can also be used as a feedstock to make both gasoline, diesel or jet fuel.Huber et al. showed that furfural can serve as a precursor for production of liquid alkanes.[8] Furfural is exclusively produced from lignocellulosic biomass by dehydrating pentoses (mainly xylose) which are present in significant amounts in the hemicelluloses of some agriculture residues and hardwoods. The preferred feedstocks are agricultural residues because these are homogeneous and regularly available in large amounts from the food processing plants. The industrial technology for furfural production relies on batch or continuous reactors where the pentosan fraction of the lignocellulosic is converted into monosaccharides (pentoses) by acid hydrolysis. Further dehydration reactions of the pentoses yield furfural.[5]

II. MATERIALS & METHODS

50 grams of dried sunflower husk (dried in the oven temperature of 200^0 C for 24 hours) ground in to minute particles of size 1mm, 250ml of varying wt% of HCl, and 62.4gm NaCl were introduce into a IL three- neck round bottom flask. A vigrux column and a condenser were attached and the reaction mixture was heated and stirred with a mechanical stirrer. Steam distillation was observed after 15 minutes at the distilling temperature of 107^0C. The distillate was set to flow into an extraction flask containing 250ml chloroform.

Two layer were formed / with the aqueous layer at the top & chloroform furfural at the bottom..The bottom chloroform –furfural layer was subjected to the simple distillation or rotary evaporator unit to remove chloroform, and a clear yellowish liquid (F-I) remained. To prepare 12% HCL add 166.6 ml HCL in 333.3 ml water. Take 200 ml 12% HCL solution add 25ml reagent and known quantity of furfural in iodine flask and put it in dark light place for about 30 min. After 30min add 10ml 10% KI solution and 10 ml starch indicator. Titrate the solution with 0.1 N sodium thiosulphate solution. Take observation and calculate concentration of furfural.

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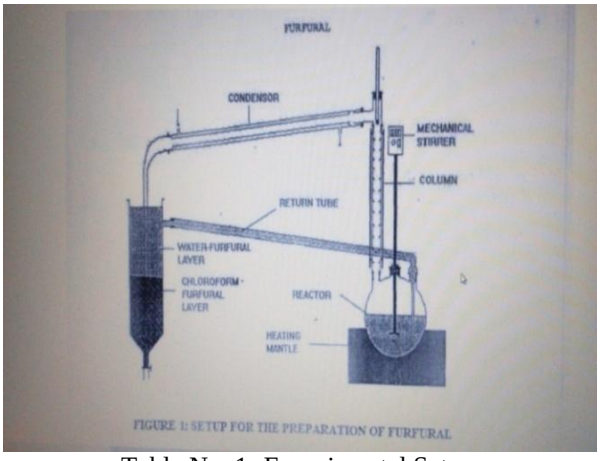


Table No. 1- Experimental Setup

II. RESULTS AND DISCUSSION

Table No. I Raw Material Analysis

Sr No.	Test	Quantity %
1.	Ash Contain	10
2.	Moisture Contain	25

Table No.II Furfural yield Vs Conc. of HCl

Furfural Yield	Acid Concentration of HCl							
Wt% of acid	1 %	5 %	7 %	12 %	15 %	25 %	30 %	35 %
ml	0	0.3	1.1	2.1	3.4	4.3	5.4	4.9
gm	0	0.34	1.27	2.43	3.94	4.98	6.26	5.68
Wt %	0	0.34	1.27	2.43	3.94	4.98	6.26	5.68

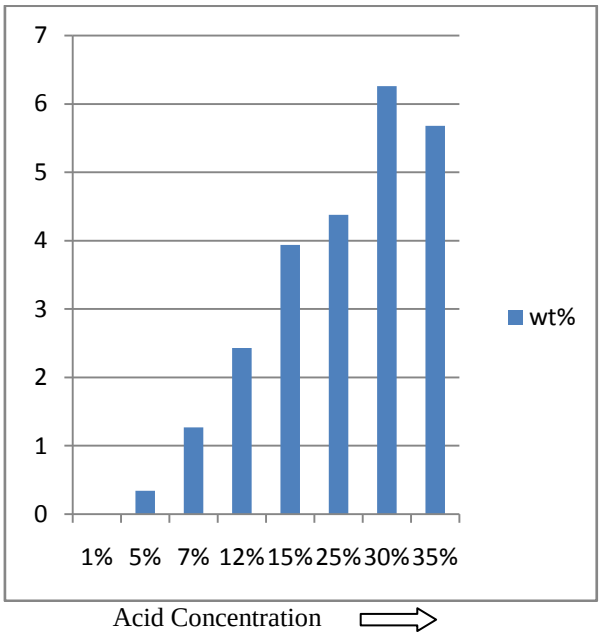
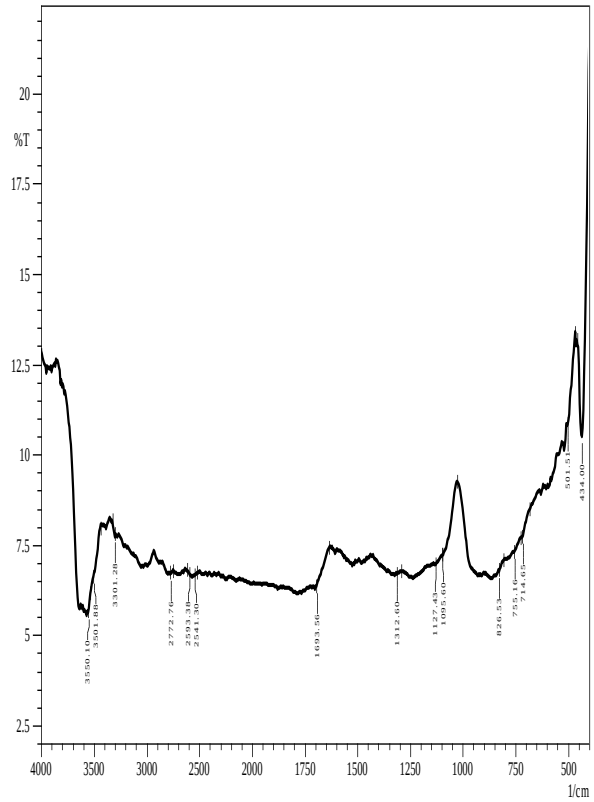


Fig 2- Effect of Acid Concentration on Yield of Furfural in Wt %.



The IR spectrum (Figure 3) shows a very strong absorption at 1,693.56 cm^{-1}. This absorption shows a very significant functional group which is the conjugated carbonyl (C=O). The absorption wave number is slightly lower than usual i.e. 1,740 to 1,720 cm^{-1}. due to internal hydrogen bonding which occurs in conjugated unsaturated aldehydes. But this peak can appear for chemical compounds like carboxylic acid (COOH), ketone, esther. Furthermore, no broad peaks were observed. The hydrolysis of sunflower husks was carried out using hydrochloric acid (HCL) of different concentrations. We compare the rate of hydrolysis of the different concentrations acid on the sunflower husks

IV. CONCLUSION

New possibilities that could flow from a bio- refinery producing furfural and some derived product, which having good market. To acquire use or application of product for Drug and specialty chemical manufacture; replacement of phenol in foundry resins; and specialized polymer are some possibilities. The optimum yield of furfural at 30 Wt% of H_2SO_4 was found to be 6.26 wt%.

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