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Biomass Refinery – a way to Produce Value Added Products from Agricultural Biomass

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ABSTRACT: *Production of furfural from pentosan in sunflower husk was carried out in an attempt to produce value-added products from sunflower husk which is commonly burnt around sunflower oil mills of India. This process involved the conversion of sunflower husk into xylose, which was then cyclodehydrated to furfural using dilute sulphuric acid. Product was characterized by volumetric process such as bromine- bromide excess method. Furfural obtained was analyzed using gas chromatography (GC) and gas chromatography with mass spectrophotometer (GC-MS). The product was colourless, but turned yellowish and then dark brown upon exposure to air and light. Furfural obtained was in liquid form, with a molecular weight of 96.2 g/mole and the formula of* $C_5H_4O_2$.

Keywords: *Acid hydrolysis, sunflower hulls , furfural, GC-MS, xylose*

I. INTRODUCTION

The making of fuels and specialty green chemicals from readily available and renewable lignocellulosic biomass is an significant step towards domestic energy sovereignty as well as fall in content of carbon as a output [1]. One route to fulfill this goal is performing a biomass chemical pretreatment followed by enzymatic saccharification and fermentation [2]. Pretreatment is an important step in biofuel manufacture in order to overcome the uncontrollable nature of biomass. It is generally performed using either acids, such as dilute sulfuric or phosphoric acid, or alkaline agents, e.g., sodium hydroxide, ammonia or lime[3]. Lignocellulosic biomass is combination of cellulose, hemicellulose and lignin. Cellulose consists of spacially organized microfibrils, each containing thousands of six-carbon glucose monomers linked with -glycosidic bonds[4]. Hemicellulose is a heteropolymer of both five and six-carbon monosaccharide molecules[4]. Lignin is a complex hydrophobic polymer of p-hydroxyphenyl, guaiacyl, and syringyl residues; it fills in the spaces between the cellulose fibers and hemicelluloseos to provide rigidity [5].

The extraction of chemicals could be an alternative to generate high-value product from this renewable lignocellulosic biomass. Furfural is one of them. It is an aldehyde of pyromucic acid. The global market for furfural is expected to reach about USD 1200.9 million by 2020. The current market price for furfural is around US$1,500 per tonne. It is widely used in industries as a base material for synthesizing a family of derived solvents like furfuryl alcohol and tetrahydrofuran and in the production of resins for molded plastic and metal coatings. Furthermore it plays a big role in the manufacture of insecticide as well. Recently, furfural has been used in the food industry for flavorings purpose too. This paper give information to extract and identify furfural obtained from local sunflower husk. It is sometimes called ‘green chemistry’ [6] in the sense that production of a chemical is achieved with a biomass. There are a numeral of biomass resources, which include wood and wood waste, agricultural crops and their waste products, municipal waste products, municipal solid waste, animal waste, waste from food processing and aquatic plants and algae [7]. Among these biomass sources, agricultural residue and energy crops are identified as good precursors for the production of biogas, bio-oil and bio-char fuels [8].

Sunflower husks are a by-product left after sunflower oil has been extracted from the seed. Sunflower husks offer numerous advantages and opportunities for bio-fuel research, particularly in bio-oil and biochar production. The objective of converting biomass material to biochar or biochemicals for numerous application.[9]

II. MATERIALS AND METHODS

Sunflower husk sampling

100 grams of dried sunflower husks (dried in the oven temperature of 200°C for 4 to 5 hours) ground into particles of size 1.1 mm by using I.S. screen.

Experimental Method

About 100 gms of dried sunflower husks and 133 gm of sodium chloride (NaCl) were mixed together in a big clean basin. Then the mixture was placed in a 1L three-neck round bottom flask. A volume of 250 m of varying % of H_2SO_4 was added into the round bottomed flask. The round bottomed flask was connected to an upright column ie Vigreux column and water condenser. Pretreatment was carried out for approximately 5 hr.

Separation procedure

Distillate collected from the distillation process was subjected to partitioning using chloroform in separation funnel. Aqueous and non-aqueous layers were obtained. The top aqueous layer was charged into the reaction flask by a return tube connected to one of the neck. 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.

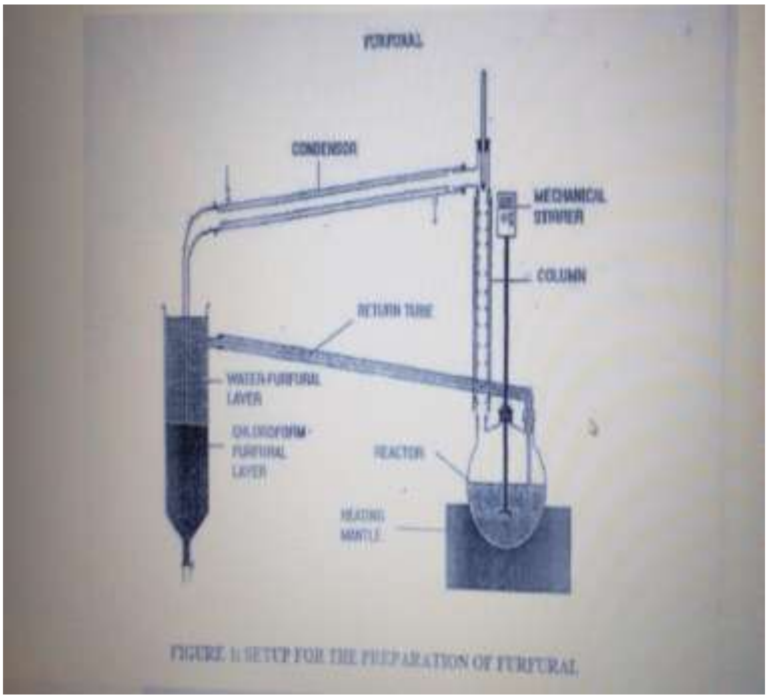
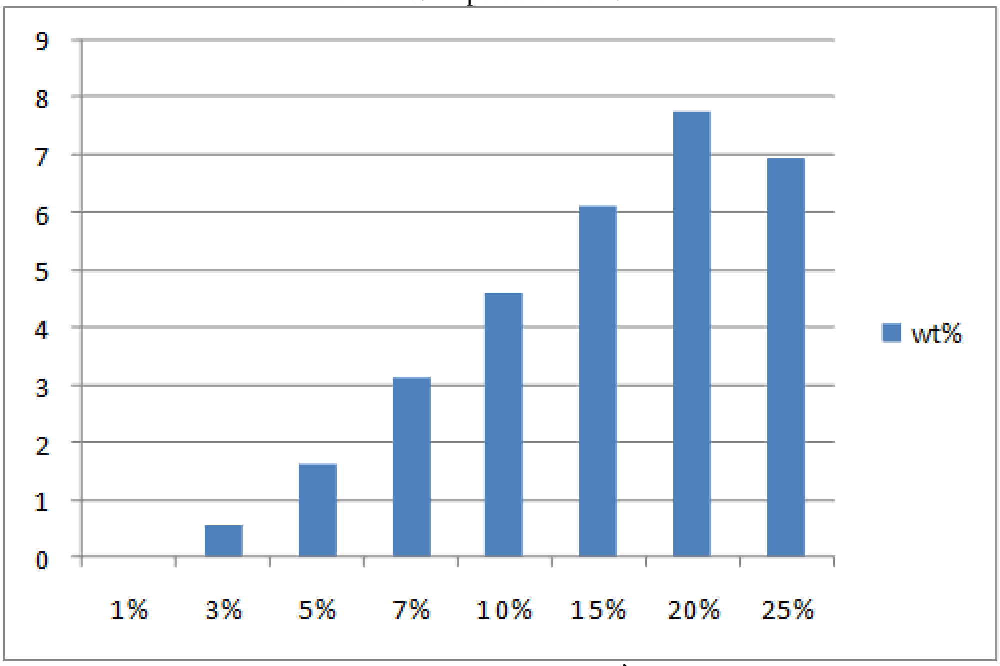


Fig.1 Experimental Set-up

III. RESULTS AND DISCUSSION

Crude Furfural Yield	Acid Concentration of H_2SO_4							
Wt% of acid	**1 %**	**3%**	**5%**	**7%**	**10%**	**15%**	**20%**	**25%**
ml	**0**	**0.5**	**1.4**	**2.7**	**4**	**5.3**	**6.7**	**6**
gm	**0**	**0.58**	**1.62**	**3.13**	**4.64**	**6.14**	**7.77**	**6.96**
Wt %	**0**	**0.58**	**1.62**	**3.13**	**4.64**	**6.14**	**7.77**	**6.96**

Table no.1 – particle size 1.1 mm



Acid Concentration ⇨

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

The hydrolysis of sunflower husks was carried out using sulfuric acid (H_2SO_4) of different concentrations. We compare the rate of hydrolysis of the different concentrations acid on the sunflower husks. The results indicate that with H_2SO_4, furfural was seen to produce even after 1.30 hr of hydrolysis. The yield of product effect with change with acid concentration. The optimum yield of Furfural at 20% concentration of H_2SO_4 was found to be 7.77 wt %.

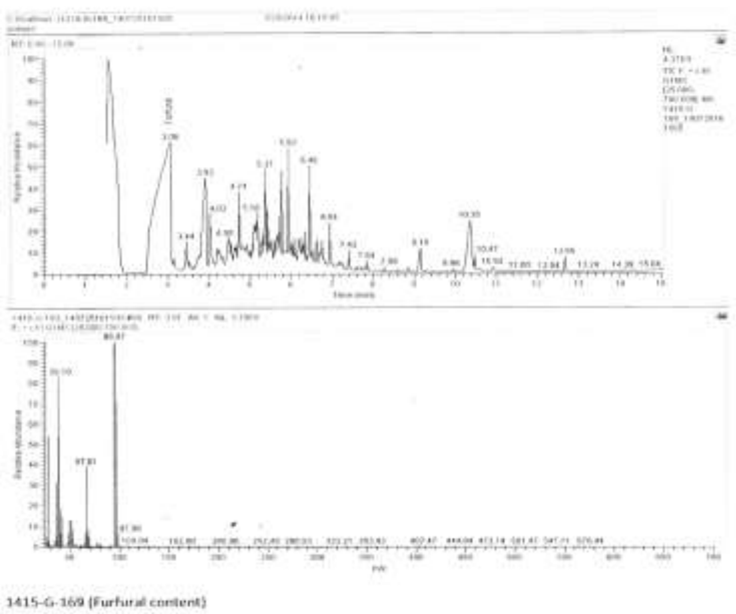


Fig3:- GCMS analysis of furfural

Results obtained from GC-MS are shown in above Figure3. Furfural has a retention time of 3.06 min with relative abundance of 60 %. Sample is proven as furfural after comparing the data obtained with NIST

Library. The spectrum showed a molecular ion peak at m/z 97.96 which correlates to a molecular formula of furfural (C5H4O2). A peak at m/z 95.97 (M-1)+ was obtained due to the loss of hydrogen to from a carbonium ion. Electron was given away by the aldehyde carbon to hydrogen to form this fragmentation because it can form a more stable cation.

IV. CONCLUSION

Furfural has been successfully extracted from Sunflower hull by acid hydrolysis method. Its functional groups and molecular weight were identified using GCMS equipment. It has a formula of $C_5H_4O_2$ and a molecular weight of 96.2 g/mole. It is in a flammable liquid form, which smells like bitter almond. It is colourless but turns yellowish and then brown upon exposure to light and air. From this experiment it is found that the concentrations of acid will increases the yield of furfural also go on increasing. The optimum yield of Furfural at 20% concentration of H_2SO_4 was found to be 7.77 wt%.

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