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Purification of synthesized Hydroxy Methyl Furfural (HMF) Extracted from organic waste

Vinutha Moses[1], Dr.Archana[2], Lakshmi priyanka G[1], Dr. M.A Lourdu Antony Raj[1].

[1]Department of Chemical Engineering
R.V College of Engineering, Bangalore, KA, India
[2]Department of Chemical Engineering
MS Ramaiah Institute of Technology, Bangalore, KA, India

Abstract - Organic domestic waste and agricultural waste poses a serious threat, since they ferment, creating conditions favorable to the survival and growth of microbial pathogens. Direct handling of solid waste can result in various types of infectious and chronic diseases with the waste workers and the rag pickers being the most vulnerable. Hence the organic waste was treated in a way that a product hydroxy methyl furfural (HMF) was developed that has a market value. HMF is not only a new resin in the market but has other great potential applications. and it has given a different dimension to the research that exists in a research field. HMF is also classified as a food improvement agent in food industry in the form of food additive as well as a flavoring agent for food products and also produced industrially as a carbon neutral feedstock and other chemicals. Moreover, HMF is termed as the sleeping giant as a solidified and polymerized version of HMF can also be used to make biodegradable plastics, but the yield and purity available in literature is not appreciable to HMF. and there exists only limited amount of purification procedures for HMF. The yield and purity of HMF must be increased to enhance the applications. Therefore, this research focuses on increased the purity of HMF by different methods.

Keywords- Hydroxy Methyl Furfural (HMF), Purity, organic waste, and applications etc.

I. INTRODUCTION

Waste is created in our homes, organizations and mechanical sources. Internationally, waste generation is increasing in volume and consecutively the harm it causes is increasing drastically [1]. The greater part of the waste is biodegradable like nourishment squander, green waste, wood, paper, etc. while the others like plastic compartments, containers and cans are non-biodegradable [2]. Even the biodegradable waste produced at homes is a nuisance and results in foul smells and the aesthetic beauty of the place is destroyed. It becomes plainly undesirable, messy and Unattractive spots to dwell in. It harms living beings by way of diseases transmitted from parasites dwelling and breeding in such areas.

1. Waste Accumulation

Waste accumulation additionally diminishes the employments of the land for other, more helpful purposes [3]. There are various provisions for waste in India and abroad including landfills, incinerators, and a developing number of exchange stations. The economic frameworks to stress the financial estimation of materials and vitality help in estimating the net worth of waste. This way we can estimate the amount of resource that has been spent and waste das the product is waste and the estimation of profits from a new product which is developed by turning waste to value. In this method generation and utilization are also accounted for in terms of money spent and saved respectively. It can be concluded. that waste tends to be very huge loss of resources for the earth be it monitory or resource wise as the requirenormous utilization of normal capital and vitality [4,5]. The assets and space are limited for waste collection and are eventually not going to be sufficient for waste accumulated over years due to generations of waste accumulation. The existing landfills will also degrade in condition [6]. Reusing is an outdated concept. The reused substance will eventually become waste that has reached its final life cycle, and this is dumped, and it accumulates and then causes harm to the environment [7-9]. Waste whether non-biodegradable or bio-degradable will cause very dire situations in the future with respect to health of the biosphere [10-13].

2.Organic Waste Scenario Globally

Asia is seeing a quick increment in urban population with around 35 percent of its aggregate population dwelling in urban territories and they early increase in urban population is almost 4 percent. It is anticipated that by 2025, around 52 percent of the Asians would live in urban zones causing a major stress on resources and on urban land. The fast-financial development has enhanced the ways of life of the urban occupants by empowering them to buy more products and thus an obvious increase in consumption of food, etc. which will lead to an increased amount of organic waste accumulation [14, 15].

3. Organic Waste Scenario in Bengaluru, India

Bangalore is the administrative, cultural, commercial, and industrial and knowledge capital of the state of Karnataka, India with an area of 741 sq. km. Population density has increased from 10,732 (in 2001) to 13,392 (in 2011) persons per sq. km. The per capita GDP of Bangalore is about $2066, which is considerably low with limited expansion to balance both environmental and economic needs. The current estimates indicate that about 3000–4000 tons of Municipal Solid Waste are produced each day in the city, the daily collection is estimated about 3600 tons per day. The increase in the per capita generation from 0.16 (1988) to 0.58 kg/day/person (2009) is due to the changes in consumption patterns. The Changes in composition are noticed recently with the increasing quantity of waste [16].

4.Hazardous Effects of Organic Waste

The group at a risk from the unscientific disposal of solid waste includes the population in areas where there is no proper waste disposal method, especially the preschool children; waste workers; and workers in facilities producing toxic and infectious material. Other high-risk group includes population living close to a waste dump and those, whose water supply has become, contaminated either due to waste dumping or leakage from land fill sites. Uncollected solid waste also increases risk of injury, and infection.

5. Resin

The solution is to manufacture bio-composites. Bio-composites consist of a bio- resin and plastic melted to form plastic sheets where the plastic sheets act as the fiber component of composites and the bio-resin acts as the resin/ binder for the fiber. The word Resin begins from French "Resin" and Latin "Resina". It is a characteristic or engineered exacerbate that is exceptionally gooey and later solidifies with treatment. It is dissolvable in liquor however insoluble in water [17].

6.Bio-resin

Bio-resins can be used as another alternative to the conventional polyurethane based plastics as they can be solidified to get biodegradable plastics too. Moreover, using the bio-resin decreases the harmful emissions that occur while using and developing the synthetic resins and utilizes plastic waste if composite laminates are made using plastic sheets and bio-resin. [18].

Vegetable oils have been broadly utilized for the generation of bio-resin; they can be developed on a large scale or small scale or on Nano-scale [19]. Blends of a vegetable oils (soybean oil,corn oil,linseed oil,isomerizes/conjugated oils, and so on.) with Styrene and di vinyl benzene have been fundamentally or cationic ally polymerized within the sight of different fillers and filaments, for example, natural muds, glass, hemp, flax, jute or knead strands, wood flour, sugar can ebagasse, spent germ, corn Stover, wheat straw and recovered cellulose[20]. Greener polymerization strategies, for example, olefin metathesis polymerization have additionally been connected to bio composite creation [21]. Fleece, silk, human hair and plumes and so on can be used as fibers which are naturally available. Fleeces a safe w qualities that recognize it from hair or hide; it is pleated, it is flexible, and it develops in staples. Fiber taken from creatures or furry warm-blooded animals e.g. sheep fleece, goat hair (cashmere, mohair), alpaca hair, horse hair and so forth. Fleece is the material fiber [22].

II. MATERIALS AND METHODOLOGY

1. Materials and equipments

Materials and equipments for the experiment are shown in the given table

Table 1 Details of the Chemicals used

The glassware used are made of borosilicate, volume and the numbers used are presented in Table 2

Table 2 Details of glassware used.

Type of Glassware	Volume (ml)	Numbers
Beakers	200	4
Beaker	500	1
Measuring cylinder	15	1
Plastic funnel	-	1
Petridish	-	1
Glass rod	-	1
Separating funnel	500	2

The equipments used are of ASTM standards the specifications are in Table 3

Table 3 Details of Apparatus

Apparatus	Description and specifications
Ball mill	To convert the organic or kitchen waste into a powder form. Balls were made of cast iron
Mixer	To grind the obtained powder sample into a fine powder
Sieve set	was obtained from techno chemicals and instruments. screens of mesh size 60-100 were used
Electronic weighing machine	The capacity of the weighing machine to Weigh was 1000 grams.
High performance Liquid Chromatography	HPLC equipped with a zorbax SBC 18 reverse phase column (Agilent) was used
Freezer (-40 degree Celsius)	To crystallize the extracted HMF into a solid form.
Fourier Transform Infrared Spectroscope	IR spectra were obtained by FTIR instrument (Bruker model, Alpha Echo ATR) and wave number range was400-4000 cm^{-1}

2. Methodology

The following processes explained in the flowchart The preliminary procedures presented in the first few steps of flowchart are from collection to sieving Collection of organic waste from M.V. Hostel kitchen of R.V. College of Engineering which included banana peels, cabbage, lady's finger, sugarcane fibers,

SL NO	Chemicals used	Purity (%)
1.	Hydrochloric acid	34.5
2.	Dimethoxy Sulfoxide	99.5
3.	Tetra Hydro Furan	96
4.	sec- Butanol	99
5.	Sodium chloride	99.3
6	Distilled water	100
7	Methyl tertiary butyl ether	-
8	n-pentane	-

etc. The collected waste was shade and sun dried to prevent growth of microorganisms and to get completely dried waste. And The dried waste was made powder by crushing it in a ball mill.

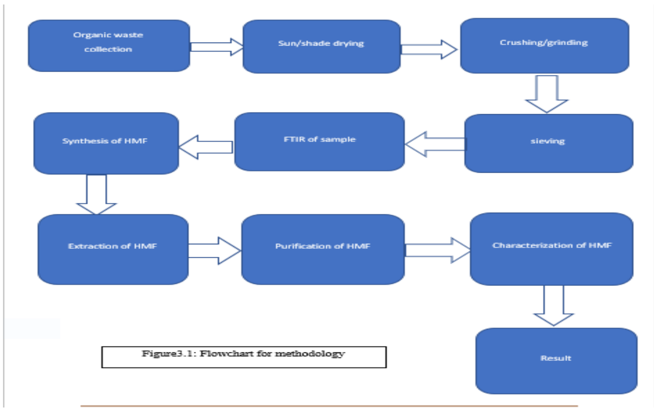


Figure 1 Flowchart for methodology

The powder collected after passing through 100 mesh of sieve was the powder used for further reactions. Hence, sieving was carried out to get a fine powder for reaction and reducing solid content followed by FTIR analysis of the powdered sample.

3. Synthesis of Powdered Sample

The organic waste that is cellulose and fibrous (green kitchen and agro waste) was collected from kitchen and sugarcane vendors. collected waste was subjected to pre- treatment processes to remove contaminants and soil. The fibrous organic waste was dried using oven at 110°C to remove the moisture content and to prevent microbial growth. The dried waste is crushed with cast iron balls in a ball mill and sieved using a sieve of mesh size 100 according to British Standards (152micrometers). The sieved sample is stored at -5°C to prevent contamination as shown in Figure 2.As the raw material being used to synthesize bio-resin is a grow as there were no proper methods to follow as a result a most appropriate protocol was chosen by trial and error method suitable for our source to develop bio-resin.

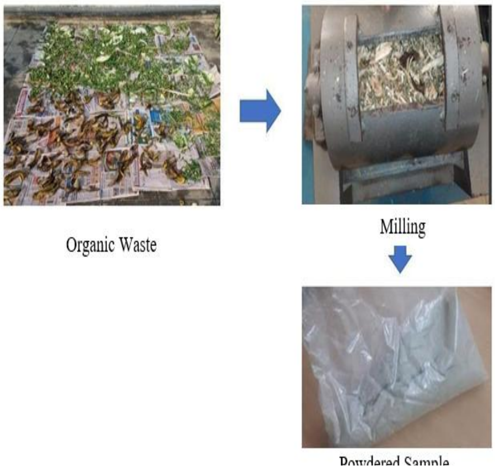


Fig. 2 Synthesis of powdered sample.

IV. CONVERSION OF POWDERED SAMPLE TO HYDROXYMETHYL FURFURAL

Direct conversion method which involves the addition of quantities of HCl, water and DMSO to the powdered sample at boiling temperatures is a feasible and cost-effective method to synthesize HMF.In this method HMF resin of 55 % concentration (by weight) is seen. The reaction was carried out at 1 Atmospheric pressure and 170°C. Time taken for the reaction is 10 minutes. The quantities of raw material taken were as follows 10 g of organic waste, 2 ml of HCl 55ml of DMSO and 29 ml of water. The experiment is as can be seen from Figure 3. To increase the yield from 55%, optimization studies were carried out by varying temperature conditions and concentrations of raw materials taken

Fig. 3 Synthesis of HMF resin from organic waste by Direct Conversion method.

1. Extraction of HMF

Extraction of HMF resin was carried out using a mixture of the ratio of 4:2:1(by volume) of 2-butanol,20% of NaCl solution: Tetrahydrofuran (THF) was taken. The mixture was added to the product solution. After 48hrs of mixing, extraction mixture and HMF resin, the mixture was kept in a separating funnel. Two separate layers were collected from the separating funnel and the organic bottom layer was collected and heated to 80 degree Celsius.

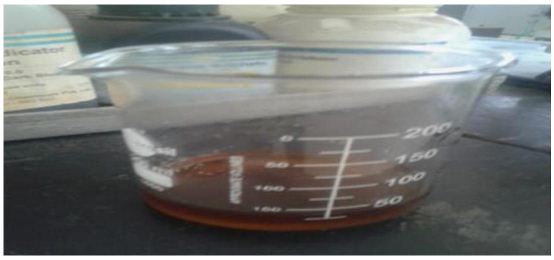


Fig. 4 Extracted HMF.

2. Purification of HMF resin by crystallization- Purification of HMF was increased where the crude HMF was dissolved in 4 volumes of Methyl Tertiary Butyl Ether (MTBE) at room temperature. When the temperature was decreased to -30 degree Celsius. After 30 minutes, the crystal formation was observed. The mixture was left to crystallize for 12hrs. after which the crystals were filtered off at -30 degree Celsius. Crystals were washed with the 1-pentane(1volume) and sucked dry. The purity will be obtained about >>99% pure according to the HPLC.

Fig. 5 purified HMF.

3. Characterization of HMF Resin

Various characterization tests were used to find out the synthesized product is HMF. tests such as FTIR was carried out. HPLC was carried out to know the yield and the purity of HMF.

4. Fourier Transform Infrared Spectroscopy analysis (FTIR)

Samples were given for FTIR analysis to confirm the presence of HMF or its derivative .IR spectra were obtained by FTIR instrument (Bruker model, Alpha Echo ATR) and wavenumber range was 400-4000 per cm. The FTIR analysis was conducted at M.S Ramaiah Institute of Technology. Results are discussed in the heading 3

5. High Performance Liquid Chromatography analysis (HPLC)

Organic sample was converted to HMF was given for HPLC Characterization to know the yield of HMF was analyzed. Mobile phase was methanol: water (8:2).

Wavelength for UV detection was set as 283nm at retention time at this condition for HMF is 1.627min. The HPLC analyses was conducted at B.M.S College of Engineering. Results are discussed in the heading 3 figure7 and figure 8.

V. RESULT AND DISCUSSION

1. Characterization of the Powdered Sample

The FTIR analyses results of the powdered sample can be seen in Figure 6. The obtained powder was sieved, and it was given for FTIR test so that a proper solvent could be selected. the peak at 3200, 2700,2900 and no peak at 1800 cm-1 shows that the sample had sucrose, cellulose, lignin, flavonoids that were essential for the synthesis of bio-resin. Therefore, a reaction pathway where the cellulose was converted Glucose and then that was converted to HMF was considered.

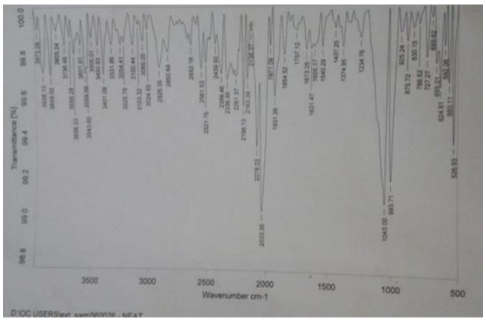
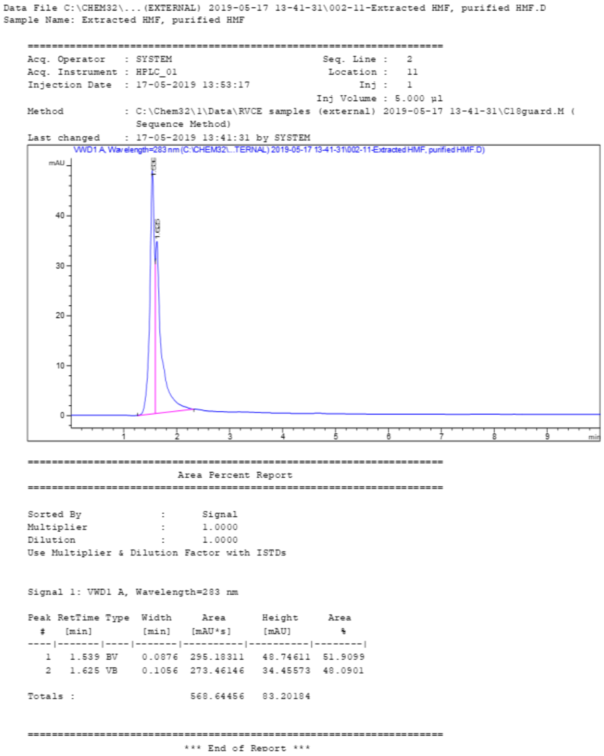


Fig. 6 FTIR of the powdered sample.

2. High Performance Liquid Chromatography Analysis

The area under the peak at the retention time for HMF was 62% as can be seen from the Figure 7. It is an indication of the amount of HMF present in the product which was only 62% (by weight) of the product after extraction of HMF. This means that the rest of the sample consists of impurities which may constitute extraction solvents, unconverted Glucose, etc.

The chromatogram seen in Figure5.8 has some discrepancies like the baseline is not straight. This is due to unstableness in injection since it was done manually; meaning a steady volumetric flow rate of the injection fluid was not maintained as it was manually injected. Unexpected elements like Antimony and Lithium were found during HPLC whose source was found to be in the vegetable source that was milled.



Data File C:\CHEM32\...(EXTERNAL) 2019-05-17 13-41-31\002-11-Extracted HMF, purified HMF.D
Sample Name: Extracted HMF, purified HMF

Acq. Operator : SYSTEM Seq. Line : 2
Acq. Instrument : HPLC_01 Location : 11
Injection Date : 17-05-2019 13:53:17 Inj : 1
Inj Volume : 5.000 µl
Method : C:\Chem32\1\Data\RVCE samples (external) 2019-05-17 13-41-31\C18guard.M (Sequence Method)
Last changed : 17-05-2019 13:41:31 by SYSTEM

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

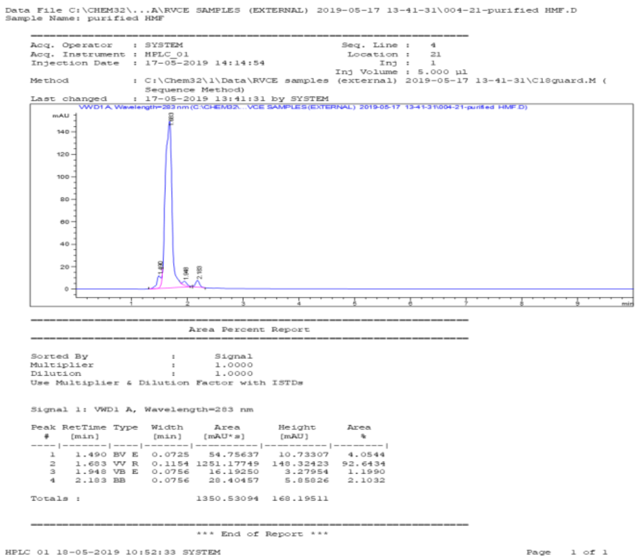
Signal 1: VWD1 A, Wavelength=283 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.539	BV	0.0876	295.18311	48.74611	51.9099
2	1.625	VB	0.1056	273.46146	34.45573	48.0901
Totals :				568.64456	83.20184	

*** End of Report ***

Fig. 7 HPLC results of Extracted HMF resin.

The area under the peak at the retention time for HMF was 92% can be seen. It is an indication of the amount of HMF resin present in the product which was only 92% (by weight) of the product after the purification of HMF. This means that the purity was increased from 62% to 92%. The HMF was purified from the obtained solution.



Data File C:\CHEM32\...A\RVCE SAMPLES (EXTERNAL) 2019-05-17 13-41-31\004-21-purified HMF.D
Sample Name: purified HMF

Acq. Operator : SYSTEM Seq. Line : 4
Acq. Instrument : HPLC_01 Location : 21
Injection Date : 17-05-2019 14:14:54 Inj : 1
Inj Volume : 5.000 µl
Method : C:\Chem32\1\Data\RVCE samples (external) 2019-05-17 13-41-31\C18guard.M (Sequence Method)
Last changed : 17-05-2019 13:41:31 by SYSTEM

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=283 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.490	BV E	0.0725	54.75637	10.73307	4.0544
2	1.683	VV R	0.1154	1251.17749	148.32423	92.6434
3	1.948	VB E	0.0756	16.19250	3.27954	1.1990
4	2.183	BB	0.0756	28.40457	5.85826	2.1032
Totals :				1350.53094	168.19511	

*** End of Report ***

HPLC 01 18-05-2019 10:52:33 SYSTEM Page 1 of 1

Fig. 8HPLC results of purified HMF resin

VI.CONCLUSION

The kitchen waste was sun and shade dried and powdered in a ball mill. From the FTIR spectroscopy results of the sample, it was concluded that cellulose is present in the sample. The bio-resin synthesized was extracted from the solution. From the literature survey it can be concluded that THF and 2- butanol in the volume ration 1:4 respectively with 20% weight by weight composition of Sodium Chloride solution is the best extracting agent to extract HMF resin from organic solution. The extraction procedure with this extraction solution was conducted. The synthesized sample was separated from the impurities using solvents to increase yield and purity of the HMF resin. The product obtained was tested for HMF to confirm whether it is HMF. Based on FTIR, NMR and HPLC results, it was concluded that the product is HMF resin with a purity of 62% after the extraction and after the purification of HMF with a purity of 92%. Hence the purity of HMF was increased from 62% to 92%.

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Author's details

1. Vinutha Moses, Lakshmi priyanka G,Dr M.A Lourdu Antony Raj Department of Chemical Engineering, R.V College of Engineering, Bangalore, Karnataka, INDIA,