



Research article



Hydroxymethyl furfural (HMF) a high strength cellulose resin for wood composite laminates

Vinutha Moses^{a,*}, Archna Narula^b, N. Chetan^c, Ranjeet Kumar Mishra^b^a Department of Chemical Engineering, Rashtriya Vidyalyaya College of Engineering, Bengaluru-560059, India^b Department of Chemical Engineering, M S Ramaiah Institute of Technology, Bengaluru-560054, India^c Department of Industrial Engineering and Management, Dr. Ambedkar Institute of Technology, Bengaluru-560056, India

ARTICLE INFO

Keywords:

Bioresin
Cellulose
Hydroxymethyl furfural (HMF)
Vegetable peels
Bagasse
Banana peels
Glucose
Sulphonated charcoal

ABSTRACT

The organic wastes though biodegradable is harmful and pose a serious threat to the environment. This research focused to; harness the cellulose existing in the peels of vegetables, banana and bagasse to establish Hydroxymethyl furfural (HMF) bioresin. Eleven experiments were performed with different w/w% of DMSO and Sulphuric acid to find the best solvent extract. The FTIR-ATR validated sample 6 extract (10 w/w% at 98 contact h) as the prime compared to the other extracts. The conversion of cellulosic glucose to HMF was performed on 10 ml of sample 6 extract. The filtrate was further concentrated to 90% at 80°C. The HPLC analysis displayed 51% conversion of HMF from glucose. The universal testing machine (UTM) test executed on the HMF resin laid on the wood veneers exhibited good curing and tensile strength (18.5 MPa). The physical property of HMF lies close to the commercially available wood resin in the market.

1. Introduction

The environment faces serious threat due to the growth of microorganisms and pathogens resulting from the fermentation of domestic and agricultural waste under favourable conditions (Dursun and Maaroo, 2010). The rag pickers and waste workers also suffer from chronic infectious diseases while directly handling the fermented waste. In addition, land filling, untreated waste disposal sites and waste treatment methods are also hazardous (Abbina et al., 2017) These sites should be ideally located at distant places far away from human location and activities. The landfills should be well constructed and lined to prevent leachate seeping into the ground water sources. This construction is expensive and time consuming (Francucci et al., 2014; Lin, 2018). Therefore, every country faces critical problems in waste treatment processes. Hence, protecting and saving environment has become a matter of concern. The organic waste is resourceful, and processes must be developed to extract or convert these *valuable compounds* into a natural *bio resin* (Manthey et al., 2013). The most common use of biomass is energy generation (Mishra, 2022). The energy consumption and carbon emissions from the sector of building have been increasing with rapid development of urbanization and the improvement of living standards of residents in China. Sustainability have been a hot topic in terms of product devel-

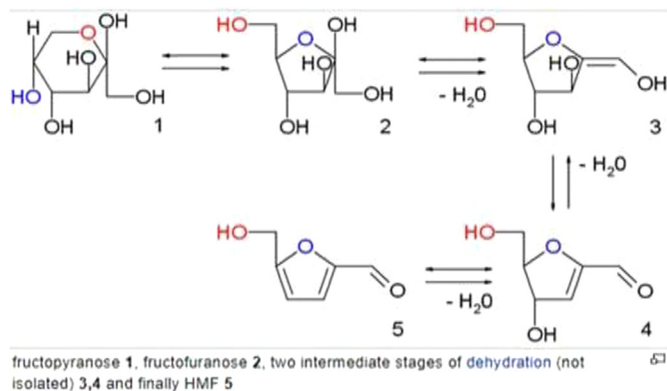
opment, buildings and energy. Preserving environment by developing sustainable products and eradicating poverty is UN's 2030 Sustainable Development Goals for 2030 (Pieter et al., 2019).

1.1. Bioresins

Bio-resins reduce the harmful emissions resulting from the use and development of synthetic resins and are preferred more than the synthetic polyurethane resins; it also finds its application in composite laminates made from reused plastic sheets sandwiched with bio-resin (Cochran et al., 2020; Lligadas, 2013; Francucci et al., 2014). Macro to nano scale production of bioresin from vegetable oil, sugarcane bagasse, spent germ, kenaf strands, natural mud, Stover, flax, jute flax, wheat straw, glass, hemp, wood flour, and cellulose are the primarily used bioresins (Wang, 2012; Manthey et al., 2013), but not economical (Luo et al., 2012; Cochran et al., 2020; Idris et al., 2015). Naturally occurring fibers in plants and animal sources are an exclusive material for developing products and fabricating advanced functional materials (Han et al., 2021). Olefin metathesis polymerization is an example of greener polymerization strategies and materials with better efficiency (Zhao, 2016; Rosatella et al., 2011; Liu et al., 2019; Sengupta and Ray, 2017).

* Corresponding author.

E-mail address: vinuthamoses@rvce.edu.in (V. Moses).



Scheme 1. Reaction mechanism to get HMF product (Rosatella et al., 2011).

1.2. Hydroxymethyl furfural (HMF) bio-resin

The formaldehyde is the most produced synthetic resin its carcinogenic that is not favourable to the environment (Vasudevan and Mushrif, 2015; Zhou et al., 2017). It causes serious health issues on human. The environment protection activist and the politician have condemned the production of petroleum-based phenol that is the PF (Phenol-Formaldehyde), UF (Urea Formaldehyde) and melamine resin (MF). Bio-based 5-HMF is an environmentally sustainable material, economical and an important substitute for formaldehyde resin (Paridah et al., 2019; Rosatella et al., 2011; Wang et al., 2012).

Kusumah et al. (2017) stated that 75% of biomass contains glucose the fundamental building block of carbohydrate, Zhou et al. (2016) stated that glucose can be converted to Hydroxymethyl furfural (HMF) and replace synthetic resins.

Bio-based 5-HMF is a highly promising and economically important alternative for formaldehyde and its many applications (Zhou et al., 2017). The extreme objective is the advancement and generation of feasible 100% biobased HMF resins (Rosatella et al., 2011; Wang et al., 2012).

Hydroxymethylfurfural (HMF), is also known as furfural 5-hydroxymethyl, formed by the degradation of polysaccharide sugar in an organic compound. The furan ring of the molecule consists of the functional group's aldehyde and alcohol (Rosatella et al., 2011), is seen in Scheme 1.

2. Experiment

2.1. Materials required

Ethanol, chloroform, dimethyl sulfoxide (DMSO), Molisch reagent, HCl, NaOH, Meyer's reagent, CuSO_4 , ninhydrin solution, alcoholic potassium hydroxide solution, benedict's reagent, FeCl_3 , $\text{Pb}(\text{CH}_3\text{COO})_2$, 2-butanol, NaCl solution, tetrahydrofuran (THF), methyl tertiary butyl ether (MTBE) were purchased from Sigma-Aldrich (Merck) Pvt. limited India. All the chemicals were used without any treatment in the present study.

Instruments/Glass wares used are Mechanical shaker, 250 ml borosilicate conical flask, 100 ml borosilicate conical flask, 10 ml test tubes, 100 ml borosilicate glass beaker, oven, specific gravity bottle, tongs, China crucible, spatula, weighing balance, funnel, filter paper, pH paper and Buckner funnel.

2.2. Methodology

The vegetable peels, banana peels and bagasse were collected from researcher's kitchen Bengaluru, India and Bagasse from the sugarcane vendors near RV College of Engineering, Bengaluru. Approximately 2 kg of the waste sample was collected washed with distilled water to remove possible impurities. The collected wastes were sundried around

2-3 days (subjected to the atmospheric conditions). The dried waste sample was weighed using a digital weighing balance and the final weight was found to be 970 g. The waste sample gained after the sun drying was placed in a hot air oven at 110°C for 10 min for preventing fungal growth [Abbina et al., 2017]. Further, vegetable peels, banana peels and bagasse were mixed in the ratio 1:1:1 (wt. basis) and pulverised, to 0.150 mm in size. The powdered sample was stored in airtight bags at -20°C for further experimental studies. Fig. 1 gives the pictorial presentation of the collected wastes and the powdered sample stored in airtight bag. The powdered sample was sent for FTIR-ATR (Attenuated Total Reflectance) to observe the presence of the required bonds and functional groups in the powdered sample. The FTIR-ATR (Thermo fisher make) was performed at MS Ramaiah Institute of Technology, Bengaluru. The moisture less powdered sample was placed on the surface of diamonds crystal and scanning was done with $4000\text{--}400\text{ cm}^{-1}$ with a step size of 2 cm^{-1} , and at a scanning rate of 40.

2.3. Solvent extraction using alcohol, acetone, chloroform, sulphuric acid and DMSO

Lignin and carbohydrate moieties are chemically bound in native biomass forming a lignin-carbohydrate complex (LCC). Due to its strong bonding, the presence of LCC affects the overall extraction of cellulose.

In order to separate LCC bonds and make cellulose rich, the powdered sample of 2 g was weighed using a weighing balance (0.0001 precision) in six test tubes, namely T1, T2, T3, T4, T5, T6, T7 and T8, to each of this test tubes 10 ml of chloroform (92% purity), acetone (99.9% purity), methanol (99% purity), ethanol (90% purity), 60% sulphuric acid (98% purity) and DMSO (99.9% purity), were added separately as shown in Fig. 2 at 45°C .

The results of good extraction were seen in sulphuric acid and DMSO observed in the FTIR-ATR spectra, therefore solvent extractive studies were carried out at different w/w%. The finely powdered sample about 1 g was taken in eleven conical flasks separately to which different w/w% of DMSO and Sulphuric acid were added to each conical flask (2wt/wt%, 5% wt/wt%, 10% wt/wt%) and time of contact in h (24, 48, 98) and as presented in Table 1. The temperature was maintained at 35°C (room temperature during experimentation) and 1 atmospheric pressure on a mechanical shaker at 120 rpm with magnetic stirrer as shown in Fig. 3. After the time of contact the extract were collected in small bottles and sent for FTIR-ATR, to detect the presence of required bonds and functional groups present in the extractives.

2.4. Conversion of cellulose present in the finely powdered sample to glucose using sulphated charcoal catalyst

In this method, charcoal was treated with sulphuric acid for 6 h in hot air oven at 130°C and then the solution was filtered using a funnel with What man filter paper (grade-41) to be used as a catalyst as presented in Fig. 4 for the conversion of cellulose to glucose (Rosatella et al., 2011). Then 10 g of the powdered sample was weighed using a weighing balance and transferred to 150 ml conical flask to that 50 ml of distilled water was added and heated on a water bath at 80°C using the filtered sulphated charcoal as the catalyst. This conversion was carried out for 5 h. The glucose converted was concentrated by heating at 50°C for 15 min. The total amount of the concentrated glucose obtained was 5.81 g after heating at 50°C for 15 min. The maximum glucose conversion was first confirmed by FTIR-ATR before further studies.

The catalytic reaction of glucose-to-HMF conversion was carried out at different temperatures 110°C , 120°C and 130°C with a loading 50 mg of concentrated glucose in 50 ml of DMSO and 10 mg of $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ as the solvent and catalyst, respectively (Van Nguyen et al., 2016). The reaction time for the mixture was 2, 4 and 6 hr. The mixture was cooled down to room temperature and was diluted into 25 ml using deionized



Fig. 1. Collected wastes and the powered sample stored.



Fig. 2. Solvent extraction using Alcohol, Acetone, Chloroform.

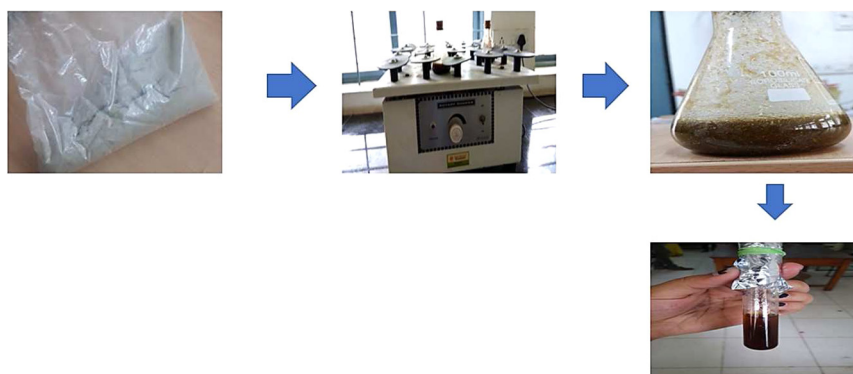


Fig. 3. Extraction of Glucose in DMSO and H₂SO₄.

Table 1. Finely Powdered sample in different wt/wt% of DMSO and Sulphuric acid and contact Time.

Sl. No.	Sample No.	Time h	Sample + Solvent	Composition wt/wt%
1	S1	48	Sample and Sulphuric acid	5
2	S2	48	Sample and DMSO	10
3	S3	98	Sample, DMSO and sulphuric acid	2
4	S4	24	Sample DMSO and sulphuric acid	2
5	S5	98	Sample and DMSO	10
6	S6	98	Sample and DMSO	10
7	S7	24	Sample and Sulphuric acid	10
8	S8	48	Sample and Sulphuric acid	10
9	S9	48	Sample and DMSO	5
10	S10	48	Sample and DMSO	2
11	S11	24	Sample and Sulphuric acid	5

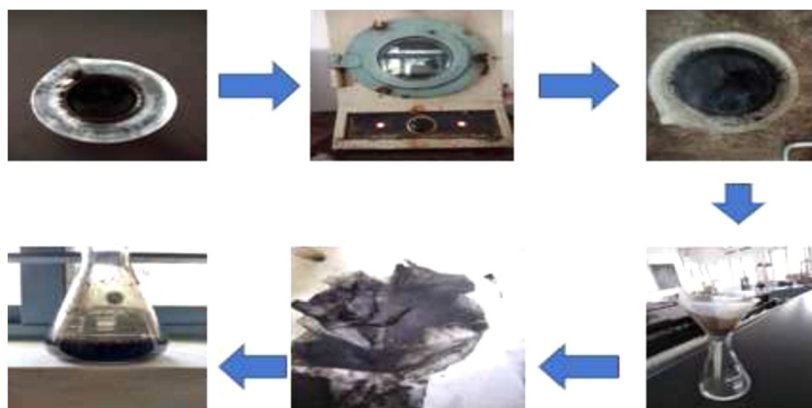


Fig. 4. Charcoal treated with sulphuric acid for 6 h in hot air oven at 130°C.

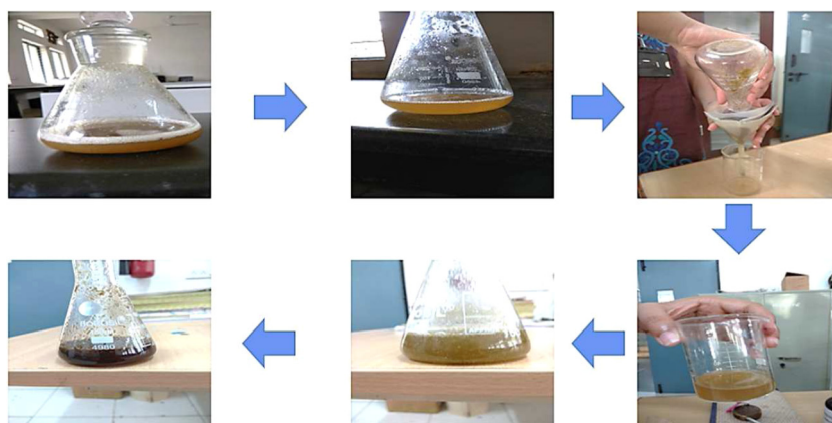


Fig. 5. Conversion of cellulose to glucose from the extract *sample 6* (10%w/w and 98 h contact time).

water. The resulting solution was filtered using whatman filter paper for products analysis. The HPLC results have been discussed in section 3.4.

2.4.1. Temperature studies on glucose conversion

In this method 5% weight ratio of the sample (concentrated glucose and DMSO from sulphonated charcoal) was taken in a 150 ml conical flask and heated to a temperature of 50°C for 98 h and then 70°C for 98 h (separately) using HCl as the catalyst, on a hot water bath, to observe the temperature effect on the glucose conversion at two different temperatures. The sample was given for FTIR-ATR.

2.4.2. Conversion of glucose to HMF

In this method, 10 ml of the extract *sample 6* (10%wt/wt and 98 h contact time) was taken in an 100 ml beaker, sulphonated charcoal catalyst was used to convert the extracted cellulose to glucose, this extraction process was carried out with constant stirring on a mechanical shaker at 120 rpm for 24 hours. This solution was filtered using a funnel with What man filter paper (grade-41). The pictorial representation is shown in Fig. 5. The filtrate was concentrated to 90% by slow heating on a hot mantle at 80°C. The concentrated sample was separated in a separating flask the organic rich layer recovered was 6 ml, it was divided into two parts, one part of 3 ml was dissolved in 10 ml DMSO without HCl catalyst and heated to 160°C for 10 min. To the other part of 3 ml organic rich layer 10 ml of DMSO was added using 1% HCl as the catalyst, and was heated to 160°C for 10 min. The FTIR-ATR, NMR and HPLC result of the sample that showed maximum HMF conversion has been explained in the results. The physical properties of this sample have been discussed in the results.

3. Results and discussion

3.1. FTIR of powdered sample

FTIR-ATR spectra of the powdered sample (Fig. 6) showed good percentage transmission at 3370 cm^{-1} linked with $-\text{OH}$ stretching vibrations attributed to the presence of phenolic, aromatics, acids, and protein impurities. Further, at peak 2968 cm^{-1} confirmed the presence of carbohydrates and lignin as ascribed with C-H bending, whereas band peak 1653 cm^{-1} inveterate presence of corroborated alkene and aromatics as qualified with C=C. Further peaks range 1320-1401 cm^{-1} endorsed C \equiv C deformation vibration, which recognized endurance of alkyne, while band peak 1019 cm^{-1} ascribed to C=O stretching, and deformation vibration recognized ether and ester in biomass. Finally, the band peaks 947-913 cm^{-1} represent C-H deformation of aromatic groups displayed low intensity due to possibly being linked with π -electron [Griffiths and De Haset, 2007].

The presence of peaks at 3370, 2700, 2900 cm^{-1} show that the sample has sucrose, cellulose, lignin, flavonoids that are essential for the synthesis of bio-resin.

3.2. Solvent extraction using alcohol, acetone, chloroform, sulphuric acid and DMSO

The extraction was clear in sulphuric acid and DMSO no extraction or poor extraction was seen in alcohol; acetone and chloroform (Fig. 7). Therefore, only sulphuric acid and DMSO were used for extraction of cellulose.

The FTIR results of the extract samples S1, S4, S7, S8, and S11 have been shown in Fig. 8 (a), (b), (c), (d), (e).

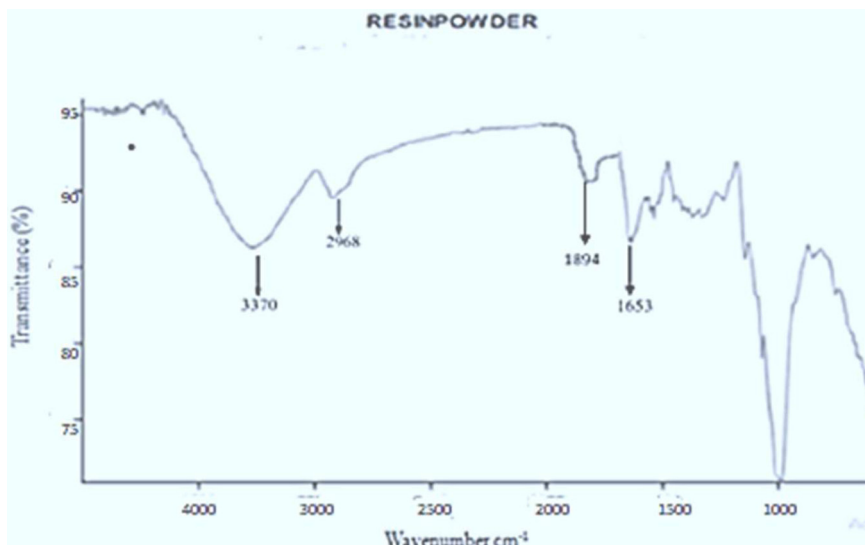


Fig. 6. FTIR of the powdered sample.



Fig. 7. Sample in Alcohol, Acetone, Chloroform.

Table 2. Phytochemical analysis of the extracts S1, S4, S7, S8 and S11.

S No.	Tests	S1	S4	S7	S8	S11
1	Carbohydrates (Molisch test)	-	-	-	-	-
a	Molisch test	-	-	-	-	-
b	Benedict's test	-	-	-	-	-
2	Alkaloids	+	-	-	-	-
3	Proteins (Biuret test)	-	-	-	+	-
4	Amino acids (Ninhydrin test)	-	-	+	-	-
5	Quinones	-	-	-	-	-
6	Flavonoids	-	-	-	-	-
a	Ferric chloride test	-	-	-	-	-
b	Alkaline reagent	-	-	-	-	-
c	Lead acetate solution	-	-	-	+	-

Fig. 8 (a), (b), (c), (d), (e), does not show specific or sharp dip at 3370 cm^{-1} but instead shows a boat like curve between range 3400 cm^{-1} to 1667 cm^{-1} , specific functional group could not be identified, therefore, phytochemical analysis was carried out to confirm the presence of compounds, such as Carbohydrates, proteins, amino acids, flavonoids. The test was carried out on the extracts S1, S4, S7, S8 and S11. The results obtained have been tabulated and presented in Table 2.

From the phytochemical analysis the extracts S1, S4, S7, S8 and S11 did not give positive results.

The observations confirm that there was no sign of the required functional groups to denote the presence of glucose and fructose in these samples. Therefore, these extracts were not used to isolate HMF.

The FTIR results of sample S2, S3, S5, S6, S9 and S10 have been presented in Fig. 9(a), (b), (c), (d), (e), (f).

The extracts S2, S3, S4, S6, S9 and S10 have shown good transmittance at 3370 cm^{-1} , this wavelength corresponds to the presence of CO bond, and OH bond, CH stretching vibrations both asymmetric, symmetric and bending was seen at 2920 cm^{-1} , and at 1850 cm^{-1} —OH stretching vibrations. The existence of these bonds, stretching vibrations and bending ascribe to the presence of moisture, phenolic, aromatics, acids, and protein impurities [Griffiths and De Haseth, 2007]. In comparison to the other extracts “sample 6” exhibits better transmittance.

3.3. Conversion of cellulose present in finely powdered sample to glucose using sulphated charcoal catalyst

The FTIR spectra provided in Fig. 10 showed good percentage transmittance at wavelength 3370 cm^{-1} , the presence of CO bond, and OH bond, CH stretching vibrations both Asymmetric, symmetric and bending was seen at 2920 cm^{-1} , and at 1850 cm^{-1} —OH stretching vibrations. These wavelengths contributed to the presence of glucose by showing the required functional groups and bonds required to form its structure.

3.3.1. Temperature studies on glucose conversion

The temperature studies at 70°C and 50°C for 98 hours showed small percentage of transmission, OH stretch is very negligible therefore this method was not considered, Fig. 11 gives the FTIR at 70°C .

HPLC results of catalyst optimization studies.

The HPLC results at 110°C , 120°C and 130°C at contact time 2 and 4 hr showed HMF conversion below 15% and HPLC results of temperatures 110°C , 120°C and 130°C at contact time 6 hr is shown in Fig. 12 a, b, c.

HPLC results from the optimisation studies have shown only HMF conversion between 24–43%. Therefore, this method was not used to develop resin.

3.3.2. Conversion of glucose to HMF

In this conversion method the FTIR spectra of the sample with HCl as the catalyst showed good results of transmittance % compared to sulphonated charcoal and temperature studies.

Fig. 13 shows clear wavelength range $2880\text{--}2860/2970\text{--}2950\text{ cm}^{-1}$ of Methyl CH stretch and $1470\text{--}1430/1380\text{--}1370\text{ cm}^{-1}$ of Methyl CH bend, $3350\text{--}3500\text{ cm}^{-1}$ shows OH stretch C=C is seen in the range $1680\text{--}1620\text{ cm}^{-1}$. The FTIR shows the important basic bonds and functional group present in HMF, but the aldehyde group and presence

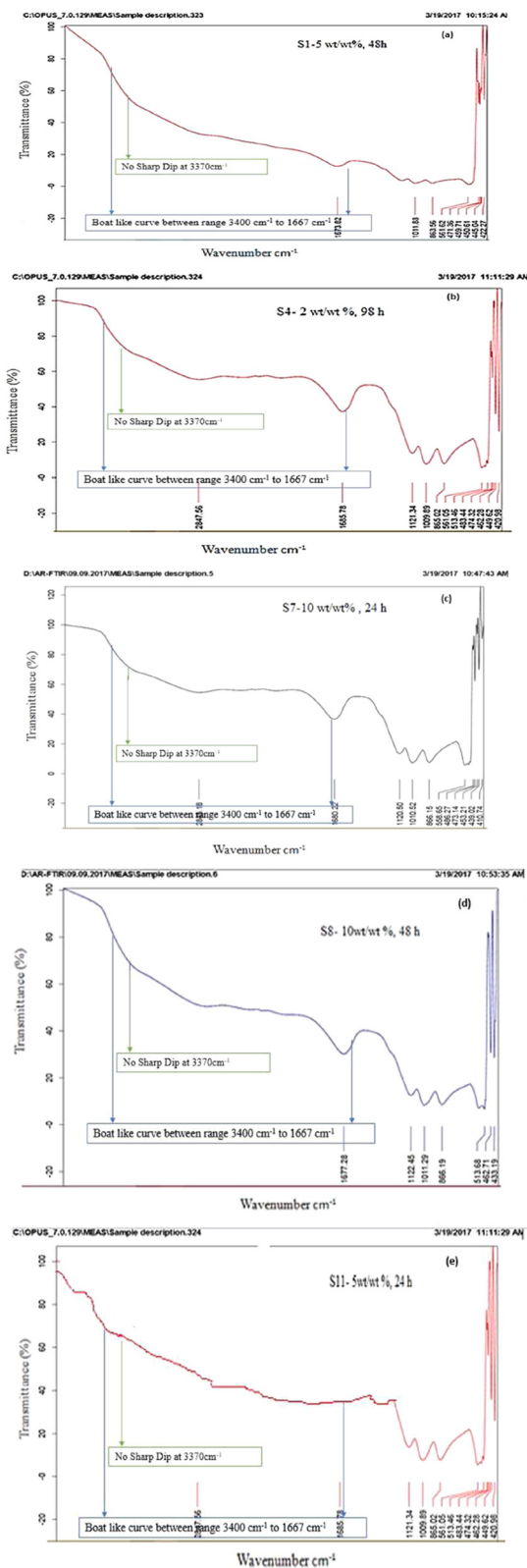


Fig. 8. (a), (b), (c), (d), (e). FTIR results show Boat like curve for Sample S1, S4, S7, S8, and S11.

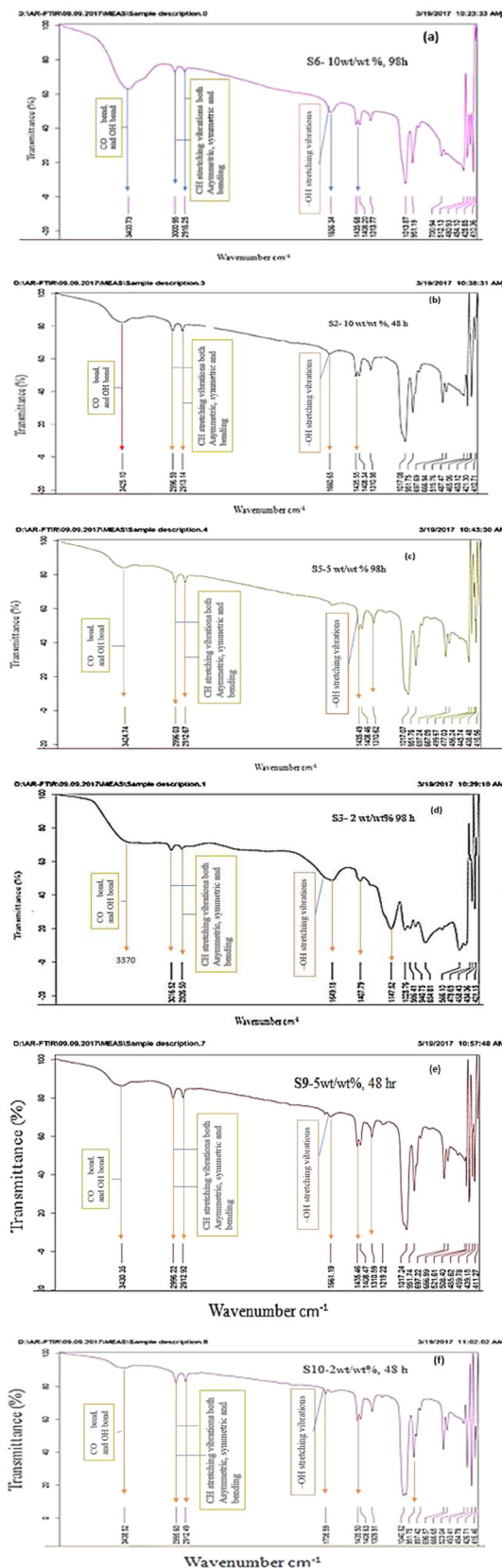


Fig. 9. (a), (b), (c), (d), (e), (f). FTIR of the Extracts- Sample S2, S3, S5, S6, S9 and S10 that showed the presence of phenolic, aromatics, acids, and protein impurities.

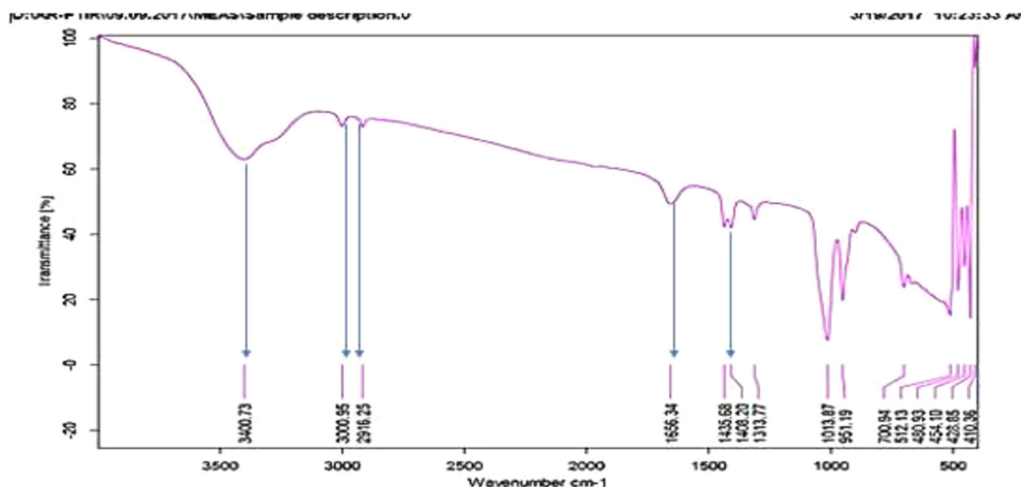


Fig. 10. FTIR of Conversion of Cellulose present in Finely powdered Sample to Glucose Using Sulphated Charcoal Catalyst.

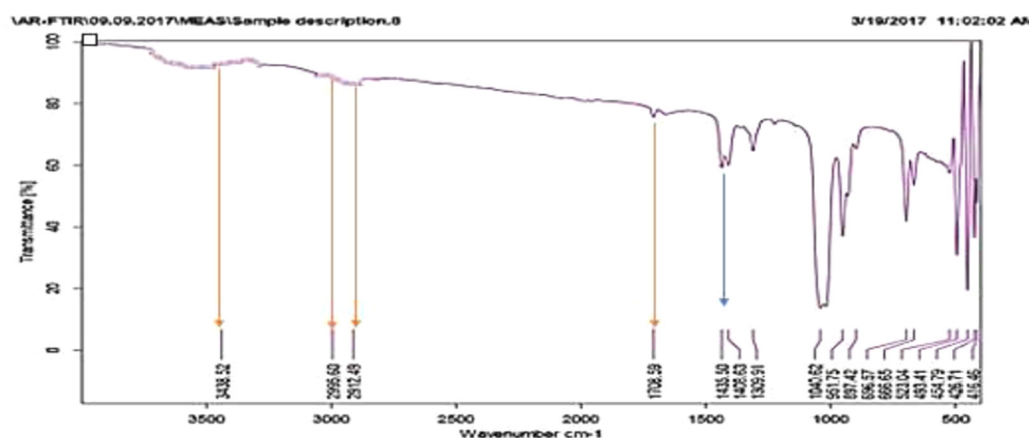


Fig. 11. FTIR at 70°C from Temperature Studies.

of furfural ring cannot be predicted. The Nuclear Magnetic Resonance (NMR) results show more specific functional groups and benzene rings.

Nuclear Magnetic Resonance (NMR)

¹H NMR study of purified HMF was performed in a nuclear magnetic resonance (NMR) spectrometer (Model No: ASCEND 400, Bruker). The purified HMF was mixed with Deuterated chloroform (CDCl₃) and filtered with a 0.2- μ m sterilized filter disc (cellulose nitrate) to remove the solid particles. Further, Topspin 2.1 with a Gaussian function software was used to acquire the H NMR spectra. H NMR was conducted at IISC Bengaluru. The Glucose to HMF conversion method showed the presence of chemical compounds and specific bonds, between d and c (6.5 and 7.5 ppm) as seen in Fig. 14, the chemical nuclei shift of organic compound six carbon that is heterocyclic with a furan ring was seen. The peak (Beyond 9 ppm) gives an aldehyde group. The peak b depicted hydroxyl group, methyl group was noticed at peak a. The structure of HMF was confirmed.

High Performance Liquid Chromatography (HPLC) Analysis

A Zorbax SB C-18 reverse phase column (Agilent) was used, Agilent 1100 HPLC device, which comprises a degasser (G1379A), quaternary pump (G1311A), autosampler (G1313A) and diode array detector (DAD) model G1315. Separations were carried out in an ACE C18 column is considered better for separating compounds, 250 x 4.6 mm x 5 μ m particle sized. The mobile phase used was methanol: water (85: 15, v/v); the prepared mobile phase was placed into the HPLC device

and passed through the column at a flow rate of 1 mL/min to condition the column. The sample injected into the HPLC system. Flow rate: 5 μ L/min. Injection time: 10 min. The temperature of the column compartment was 25°C. The detector was set at 283 nm.

The percentage conversion of HMF was carried out at Chemistry Department, BMS College of Engineering, Bengaluru. The amount of HMF present in the organic sample with HCl as the catalyst was 51% while the rest was unconverted glucose has been presented in Fig. 15. The sample without catalyst showed only 35% conversion therefore, it was rejected.

The percent conversion from Fig. 14

$$\text{Hydroxymethyl Furfural conversion} = \frac{295.10311}{(272.46146+295.10311)} \times 100 = 51.9099\%$$

$$\text{Unconverted Glucose conversion} = \frac{272.46146}{(272.46146+295.10311)} \times 100 = 48.0901\%.$$

3.4. Physical properties

The physical property was studied at room temperature 25°C, pH meter Beckman model 72 was used to determine the pH of the resins. Density was determined by specific gravity method. The solid content % was determined by petri dish method. Initially the weight of the empty petri dish was determined, later, the thin film of resin was applied on petri dish the readings before and after heating and drying were noted. Table 3 shows the important comparative studies carried out on developed bioresin, synthetic resin and the sap-based resins

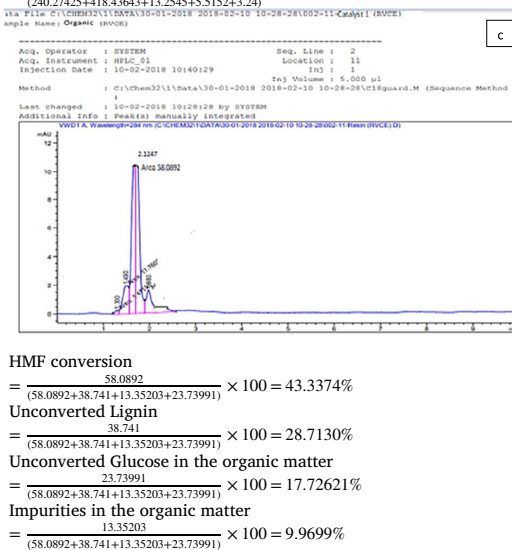
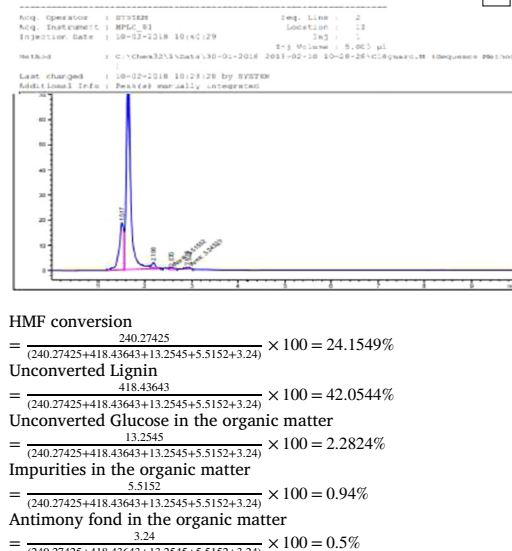
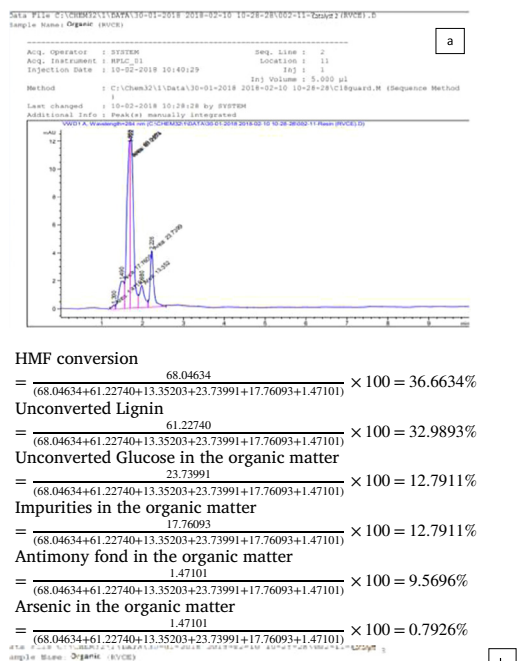


Fig. 12. HPLC Results of the HMF Conversion by the optimisation studies of $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ for 6 hr contact at (a) 110°C (b) 120°C and (c) 130°C .

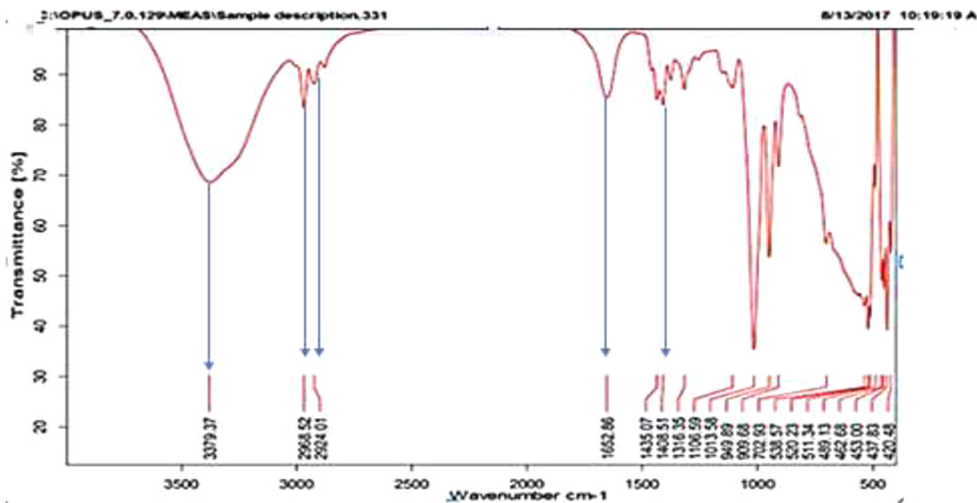


Fig. 13. FTIR of HMF from Glucose to HMF.

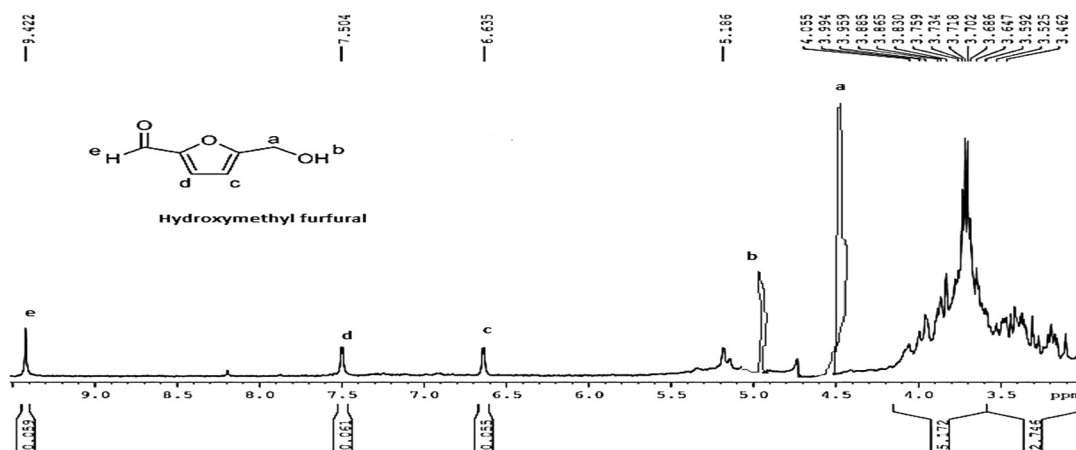


Fig. 14. Proton NMR of HMF from Glucose to HMF.

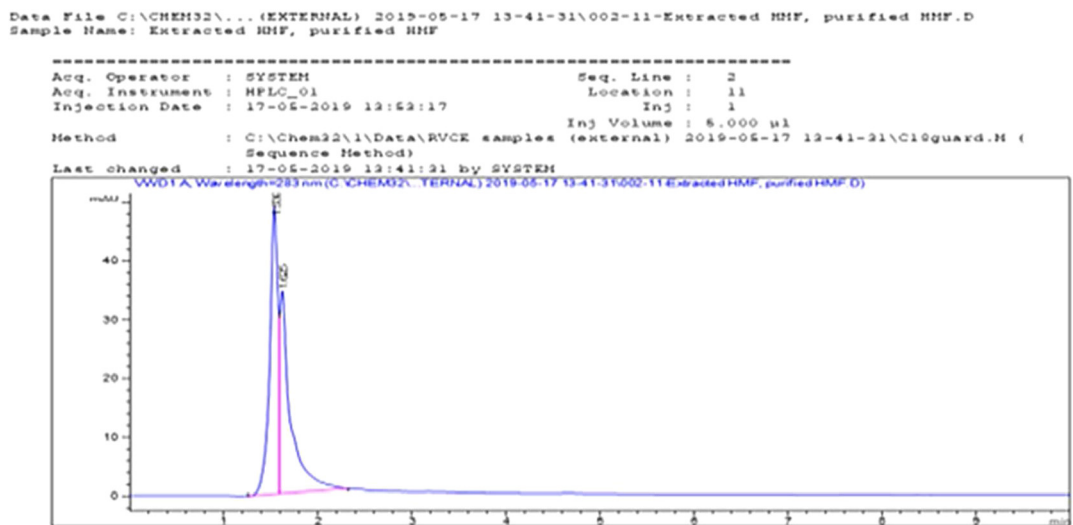


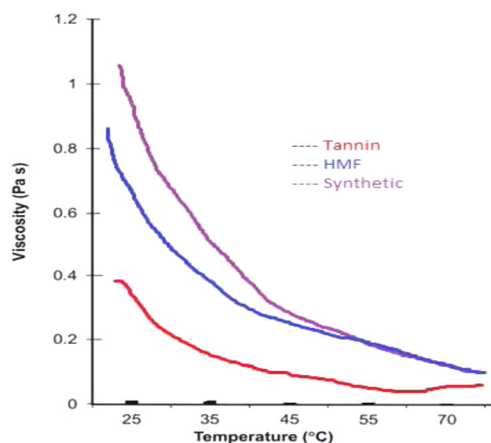
Fig. 15. HPLC analysis of HMF obtained from Glucose to HMF Conversion with HCl catalyst.

Table 3. Physical Properties of the Resins developed, available from literature and commercially available.

Resin	Synthetic resin	Developed Bioresin	Tannin Bioresin [Paridah et al., 2019]
Colour	Yellow	Brown	Reddish brown
pH, 25°C	7.66	6.99	6.1
Density g/ml at 25°C	0.5-096	1.156	2.34
Solid Content%	25	24	21

Table 4. Comparative Results of Resin Viscosity and Curing Time.

Resin	Viscosity (Pa.s) at 25°C	Viscosity Fall	Curing Time and temperature
HMF	0.39	Falls for every temperature > +20°C	4 hours for temperature 25°C 5.5 hours for temperature 35°C
Synthetic	0.46	Falls for every temperature > +35°C	3 hours for temperature 25°C 5 hours for temperature 35°C
Tannin [Paridah et al., 2019]	0.18	Falls for every temperature > +10°C	6 hours for temperature 25°C 7 hours for temperature 35°C

**Fig. 16.** Viscosities of different Resins at different temperature.

From literature the specific gravity of synthetic HMF is 1.161 of 98% purity [Zhou et al., 2017].

The viscosity of the three resins at different temperatures was studied using Brookfield Viscometer model RVF available at RVCE, the resin viscosity decreased with increase in temperature (25°C, 35°C, 45°C, 70°C), seen in the graph presented in Fig. 16. The comparative studies of resin viscosity and curing time was recorded in Table 4.

With increase in temperature the viscosity falls for thermosetting polymers because the atoms in the polymer chain increase its motion and become more fluid. Table 4 gives the results of comparative study on viscosity, viscosity fall and curing time with temperature. The curing and gel time was carried out on wood veneers of size 5 cm x 5 cm about 0.2 cm thick, the resin was applied on every single veneer, and 8 veneers were sandwiched together using hot press at 45°C to form a wood composite. The composite was heated in an oven at different temperature and the gel/curing time was studied. It was found that beyond the curing capacity it would break or crack at higher temperature.

3.5. Tensile properties

The stiffness and tensile strength are the important mechanical properties of any resin system. Tensile tests were performed at room temperature (25°C) for 3 days and for 4 hours at 35°C using EMIC 2000 (India) universal testing machine test executed at RV College of Engineering, Bengaluru. The measurements followed were in accordance to ASTM D 7031-04 standard. The graphs of the tensile modulus and strength of the pure synthetic resin, HMF and Tannin resins cured at 25°C and 35°C was furnished in Fig. 17(a) and (b)

The post cure beneficial effect at 35°C for 4 hours is predominantly seen in synthetic and HMF resin. It is also seen that after cure for 3 days at 25°C room temperature synthetic resin has good strength and stiffness, HMF has good strength but lesser than synthetic resin because the purity is 51%.

The shrinkage occurs in the resins during curing because of reorientation and rearranging of the resin molecules in the gelled and liquid phase. To reach the cured state tannin and HMF require considerable molecular arrangement, HMF showed 8% shrinkage and tannin 28% shrinkage. The synthetic resin showed 2% shrinkage with no volatile products.

Based on the standard guide to evaluate tensile strength mechanical properties of wood polymer composites (WPC) ASTM D 7031-04 was used. A 2 mm/min cross head speed on universal testing machine (EMIC 2000) at room temperature was used to study flexural and tensile strength on wood composite. Three sets of specimens were used for the testing. The dimensions of the specimens were 5 cm x 5 cm about 0.2 cm thick, the resin was applied on every single veneer and 8 veneers of 2.4 cm thickness. Fig. 18 a & b represents the tensile and flexural mode graph, that indicates HMF can be in power with the synthetic resins if the percentage conversion was increased. The results of the tensile strength have been recorded in Table 5.

The HMF wood composite showed no fracture or delamination for tensile strength 18.5 MPA a little less than synthetic resin because of 51% purity. The stress on synthetic and HMF wood composite with respect to strain % the curves lie very close to each other the performance is little less in HMF because of its purity.

3.6. SEM analysis

The SEM analysis of the developed bioresin and the existing synthetic resin available in the market was studied at the IDRC research Unit, R V College of Engineering. Two separate veneers of size 2 cm x 2 cm were taken, HMF resin was applied to one and the synthetic resin commercially available in the market was applied to the other and was dried for an hour and then the SEM analysis was carried out.

The SEM images of 10 μm magnification showed large pockets of the veneer with the synthetic resin presented in Fig. 19a, but no pockets but present of some impurities was seen in Fig. 19b.

4. Conclusion and future work

This research aimed to convert specific biomass waste to value, the FTIR-ATR spectroscopy results showed the presence of moisture, phenolic, aromatics, acids, and protein impurities, carbohydrates and lignin which would only be biodegrade or used as fertilizers or energy. The ex-

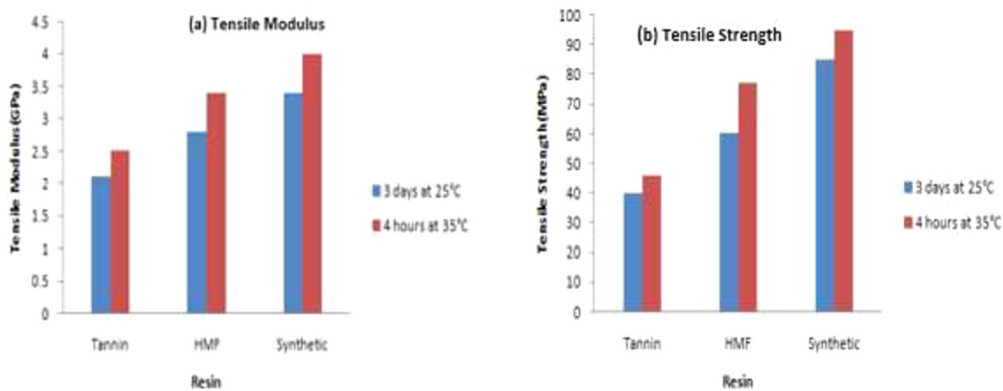


Fig. 17. (a) and (b) Tensile Modulus and Strength of different Resins cured at 25°C and 35°C.

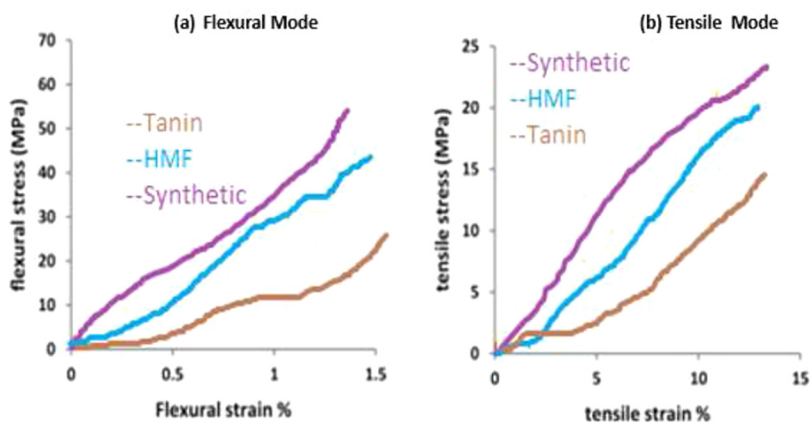


Fig. 18. (a) Flexural Mode, (b) Tensile Mode of resin wood composites.

Table 5. Results of tensile mode and flexural mode carried out on wood composites.

Material	Tensile Mode			Flexural Mode		Remarks
	Tensile Strength (MPa)	Elongation (%)	Tensile Toughness (Nm/m ³)	Flexural Strength (MPa)	Elongation %	
Resin/Wood Composite (WC)						Tensile/Flexural
HMF/WC	18.5	1.05	138.5	58.5	0.5	Strong and not very tough compared to synthetic because purity is less
Synthetic/WC	22	1.5	142	61	0.3	Strong and tough
Tannin/WC	14.5	0.97	124	39	0.7	Not Strong and not tough

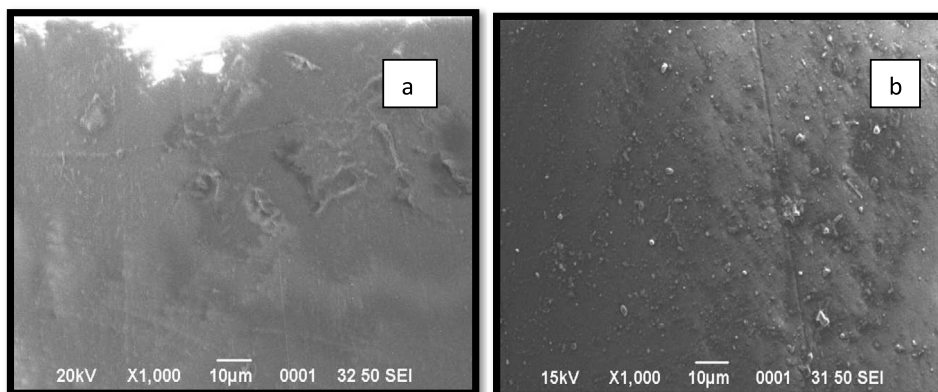


Fig. 19. (a) Synthetic Resin. (b) Developed bioresin.

traction of glucose from biomass using DMSO solvent and then convert it to a biopolymer (HMF). DMSO proved to be a very good solvent.

The FTIR-ATR results proved sample 6 (10% wt/wt of sample in DMSO for 98 hours at 35°C) was a good extract that contained glucose.

Glucose to HMF conversion method proved to be the best as maximum percentage transmittance was observed in FTIR-ATR. The Proton NMR of glucose to HMF conversion method defined the structure of HMF.

The HPLC analysis displayed 51% of HMF conversion. The physical property comparative studies such as density, pH, gel time and solid content the HMF resin comprehended the values of synthetic resin.

The cure time of HMF for 3 days at 25°C room temperature showed good strength and stiffness. The Tensile strength on HMF composites showed predominant strength of 18.5 MPa but lesser than synthetic resin because of 51% purity.

The physical property of HMF lies close to the synthetic resin available in market.

The SEM analysis revealed no pockets but some impurities in bioresin.

The extraction of Hydroxy methyl furfural from biomass can replace synthetic HMF that is very expensive.

Future work

Methods to increase the purity of HMF are necessary for better application of HMF as a bioresin. The advantages and disadvantages in purity

Declarations

Author contribution statement

Vinutha Moses: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper. Archana Narula: Performed the experiments. Chetan N: Contributed reagents, materials, analysis tools or data. Ranjeet Mishra: Analyzed and interpreted the data.

Funding statement

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Data availability statement

Data included in article/supp. material/referenced in article.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

Acknowledgements

I thank R V College of Engineering, Bangalore, India and MS Ramaiah Institute of Technology, Bangalore, India for providing lab facilities and characterization services.

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