

FUEL PROPERTIES AND COMPOSITION ANALYSIS OF SUGARCANE BAGASSES THERMAL PYROLYTIC LIQUID

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Abstract: Sugarcane bagasse thermal pyrolytic oil is characterized in this study and compared with the other bio-waste pyrolytic oil reported in the literature. Pyrolysis experiments were performed in the temperature range between 300-500 °C at 20 °C min⁻¹ heating rate without any inert environment. The effect of temperature on the yield was studied. It was confirmed that sugarcane bagasse yield utmost 53.3 % of pyrolytic liquid at 450 °C including 16.92 % of non-condensable gas and 29.78 % of char. The pyrolytic liquid was a mixture of organic and aqueous compounds. The pyrolytic liquid was 20.26 % of water content and acidic in nature with a pH of 2.8, viscosity of 22.5 cst and pour point of -12. The NMR analysis confirmed the presence of higher 86.99 % of Phenolic or olefin type of hydrogen along with 7.66 % of CH₃γ. The GC-MS analysis confirmed that the pyrolytic liquid was a mixture of acids, esters, phenol, cresol, furan, diamine-propane, hexahydrophthalic Anhydride, Cyclopentane along with various alkane and aromatic compounds.

Key-words: Pyrolytic liquid; GC-MS; 1H-NMR; FTIR; DSC

1. Introduction

Sugarcane (*Saccharum Officinarum*) plant is a perennial grass and mainly cultivated in moderate as well as tropical regions. Sugar serves as a source of sucrose which is fermented to produce ethanol. The bagasses are the residues remain after extraction of juice from the sugarcane. India produces about 179 metric tonnes/ year of Sugarcane bagasse. Initially bagasse was burned in distinct designed furnaces for increasing the steam generated in a process. India and Brazil is the second major producers of sugarcane in the world. One ton of raw sugarcane produces about 100 kg of sugar and 270 kg of bagasse. The bagasses are used as a raw material for the production of paper, animal feed, ethanol and fuel. Sugarcane bagasses can directly burn to get energy, whereas incineration is not an efficient idea. Fermentation of Sugarcane bagasses is one of the best way to produce ethanol. The Sugar Industry itself utilises its residues for electricity production. Apart from these techniques of conversion of biomass to energy, thermochemical conversion techniques is the technique which yield solid, liquid and gaseous fuel simultaneously. However, the efficiency is quite less (10-20%) compared to thermochemical conversion techniques (67-80 %) [1]. The application of sugarcane bagasse depends on its physical properties. The fresh bagasse contains about 48 % moisture, 2 % sugar and 50 % fibre, 40-50 % cellulose 20-30% hemicellulose, 20-25% lignin and 1.5-3% ash which can be used for the production of solid fuel, liquid fuel and chemicals [2-5]. Among all the thermochemical conversion techniques, pyrolysis is only one which produces more liquid compared to solid and gaseous product. However, the product yield and it's composition varies with the pyrolysis conditions such as types of reactor, final pyrolysis temperature, sweeping gas flow rate, heat flow rate, particle size and the physical properties of the biomass [6-14]. The thermal pyrolysis of sugar cane bagasse was studied by various researchers and it was observed that the yield of pyrolytic oil, char and non-condensable gas depends on the reactor types, reaction temperature and the heating rate [5, 15-18].

In present study, sugarcane bagasse was pyrolyzed in absence of any sweeping gas flow at various temperatures. The effect of temperature on the yield of pyrolytic oil was studied. The physical properties and composition of sugarcane bagasse and its pyrolytic oil were compared with other bio-waste pyrolytic oils.

2. Materials and methods

2.1. Raw Material

The sugarcane bagasses were collected from local market, Rourkela, Odisha, India. The bagasses were oven dried and grounded into - 40 mesh size and stored in an air tight plastic container for further use.

2.2. Characterization of sugarcane bagasse

The physical properties of sugarcane bagasses were studied using proximate analysis, ultimate analysis, Thermo-gravimetric analysis and EDX analysis. Proximate analysis determined the moisture, volatile, ash and fixed carbon content in the samples using ASTM D 3172 method. The ultimate analysis (C, H, N, S and O) was conducted using FLASH 2000, US CHNS/O analyser. The calorific value was determined using LECO 350 AC Bomb calorimeter. The thermal degradation behaviour was studied using Shimadzu Differential thermogravimetric analyser (DTG60 H). The bagasse samples were put for Thermo-gravimetric studies in order to reveal its thermal degradation profile. About 6.42 mg of sugarcane bagasses sample were taken in platinum crucible and heated at a heating rate of 20 °C min⁻¹ from room temperature to 600 °C. Nitrogen gas was used at a flow rate of 30 mL min⁻¹ as carrier gas to evacuate the generated vapours from the sugarcane bagasses. The EDX (Energy dispersive X-ray Spectroscopy) analysis of the sugarcane bagasses was determined using Scanning electron microscope (Jeol, 6400, JAPAN) augmented with an X-ray detector EDX (Oxford, UK).

2.3. Characterization of pyrolytic liquid

The fuel properties analysis, functional group and composition analysis were followed to characterize the sugarcane bagasse pyrolytic liquid. The fuel properties such as calorific value, pH, density, viscosity, flash point, pour point, cloud point and Conradson carbon residue were determined using various standard methods. The functional group present in the pyrolytic liquid was studied in the range of 400-4000 cm^{-1} using THERMO NICOLET NEXUS 470 FTIR spectrophotometer. $^1\text{H-NMR}$ analytical technique was used to estimate the total amount of hydrogen present in the pyrolytic liquid using 400 MHz (Bruker, DPX, US) gizmo analyser. Prior to the analysis, the bio-oil sample was dissolved in chloroform-d solvent. The transference in proton was determined by tetramethylsilane (a tetrahedral structure and used as standard for determining the proton transference). This analysis provided the type of molecules present in the pyrolytic oil along with the structure of the compounds present in the pyrolytic liquid. The composition of the pyrolytic liquid was determined using GC-MS analyser (Shimadzu, JAPAN) followed by NIST library.

2.4. Pyrolysis Experiment

Thermal pyrolysis of sugarcane bagasses was performed using self-fabricated a stainless steel semi-batch reactor (length: 18 cm ID: 8 cm) with one end opening, to retard the generated volatiles. About 50 g of oven dried grounded sugar cane bagasse was loaded in the reactor and heated using a cylindrical furnace. The temperature was controlled using PID controller. A series of pyrolysis experiments were performed in the temperature range of 300-500 $^{\circ}\text{C}$ at a heating rate of 25 $^{\circ}\text{C min}^{-1}$ without the flow of any inert gas. The pyrolytic vapour was condensed using water cooled glass condenser and weighted. At the end of the process, the reactor was cooled to room temperature. The remaining char in the reactor was weighted. The non-condensable pyrolytic vapour was calculated using the following equation.

Weight of non-condensable pyrolytic vapour = Initial weight of sample – (Sum of the weight of pyrolytic liquid and char)
Eq.1

3. Results and discussion

3.1. Physiochemical properties of sugar cane bagasse

Table 1 shows the physiochemical properties of sundried sugarcane bagasses. It was observed that the sugarcane bagasses contain higher amount of volatile matter content compared to fixed carbon and ash content. The ultimate analysis confirmed the presence of more carbon and oxygen content in sugarcane bagasses were observed in comparison with nitrogen and hydrogen content. The ultimate analysis also represented that sugarcane bagasses contained 2.38 % sulphur. The EDX analysis is shown Fig. 1. It was confirmed that sugarcane bagasses comprised with very less weight % of Si (0.20 %) and K (0.13 %) compared to C (58.20 %) and O (41.47 %). Table 1 also visualised the comparative study of physiochemical properties of sugarcane bagasse and other bio-wastes.

3.2. Thermo-gravimetric analysis (TGA/DTG)

The thermal degradation profile can be observed from Fig. 2. It was observed that the sugar cane bagasse is highly thermally stable up to 300 $^{\circ}\text{C}$. Very less weight loss was noticed with in the temperature 100 $^{\circ}\text{C}$ due to the evaporation of water/ moisture present in the sample and no weight loss was noticed up to 300 $^{\circ}\text{C}$. The weight loss suddenly increased with increasing in the temperature above 300 $^{\circ}\text{C}$. The sharp weight loss was noticed in the range of 300 $^{\circ}\text{C}$ - 500 $^{\circ}\text{C}$ temperature. A single weight loss peak was noticed which was clearly visualised from the DTG profile. It could be due to the degradation of cellulose. The complete degradation was not occurred within 600 $^{\circ}\text{C}$. Above 500 $^{\circ}\text{C}$ the degradation profile was a straight line. About 94 % of degradation occurred within this temperature range of 300 to 500 $^{\circ}\text{C}$. It could be concluded that about 6 % of the sugarcane bagasse has higher thermal stability. This may be due to the presence of lignin content in the sample. This analysis confirmed the active pyrolytic zone for sugar cane bagasse was in the temperature range of 300 to 500 $^{\circ}\text{C}$. Hence, the pyrolysis experiment was conducted with in this temperature range.

3.3. Effect of temperature on pyrolytic yield

The impact of temperature on pyrolytic yield of sugarcane bagasse is appeared in Fig. 3. It was observed that the yield of pyrolytic products fluctuate with increment in the temperature. The yield of pyrolytic oil expanded up to 450 $^{\circ}\text{C}$ and abatement with the bring up in the temperature. The yield of pyrolytic liquid, char and non-condensable volatile at 450 $^{\circ}\text{C}$ were 53.3 %, 29.78 % and 16.92 % by weight respectively. The yield of char was decreased from 49.54 % to 29.78 % when the temperature increased from 300 $^{\circ}\text{C}$ to 450 $^{\circ}\text{C}$ which resulted in more condensable and less non-condensable vapour. Beyond 450 $^{\circ}\text{C}$, the yield of non-condensable vapour amplified. Table 2 visualized the comparative study between sugarcane bagasse pyrolytic liquid with other bio-waste pyrolytic liquid [19-24]. It was observed that the yield of pyrolytic oil depends on the types of biomass, reactor types and the presence of carrier gas and its flow rate.

3.4. Fuel properties of pyrolytic liquid

The fuel properties of sugarcane bagasses pyrolytic liquid are shown in Table 3, which also visualised the fuel properties of other bio-wastes pyrolytic oil reported elsewhere [19-24]. This analysis predicted that the pyrolytic oil cannot be used as a fuel directly because of various drawbacks. The pyrolytic oil should be upgraded before considering as fuel. The fuel properties confirmed that the calorific value of sugarcane bagasse pyrolytic liquid was found to be 32 MJ kg^{-1} , which was much less in comparison with diesel. In contrast, the pyrolytic liquid was acidic (pH 2.8) and contained 20.26 % of water. This may the cause which resulted in low viscous pyrolytic liquid (22.5 Cst. @ 40 $^{\circ}\text{C}$). However, the pour point lies in the range of petroleum fuels. This study confirmed that sugar cane bagasse pyrolytic oil can be used as fuel after proper treatment.

3.5. DSC analysis of pyrolytic liquid

Differential scanning calorimetric (DSC) analysis determined the thermal property of sugarcane bagasses pyrolytic liquid and is shown in Fig. 4. The analysis was performed in the temperature range of -50 $^{\circ}\text{C}$ to 100 $^{\circ}\text{C}$. The analysis signified two exothermic peaks which showed the crystallization behaviour of the pyrolytic liquid. One peak was in higher temperature region (0 to 65 $^{\circ}\text{C}$) and another was in the lower temperature region (0 to -50 $^{\circ}\text{C}$). The liquidity of the pyrolytic liquid was affected by the temperature.

The flow ability of liquid was decreased with the decrease in the temperature due to the formation of crystals. The composition of the liquid such as unsaturated fatty acids and saturated fatty acids determines the low temperature flow ability of the pyrolytic oil. It was observed that the peak noticed in the range of (0 to -50 °C) due to the formation of low-temperature freezing crystals in the pyrolytic liquid usually associated with unsaturated fatty acids. However, the peak obtained in the high temperature region (0 to 65 °C) was due to the formation of high temperature freezing crystals and mainly consists of saturated fatty acids. The saturated compounds present in the pyrolytic liquid are responsible for the poor cold flow properties and the unsaturated esters are mainly blameable for the reduced oxidation stability which reduces the thermal stability.

3.6. FTIR analysis of pyrolytic liquid

The FTIR spectrum of sugar cane bagasse pyrolytic oil is shown in Fig. 5. This analysis confirmed that sugarcane bagasse pyrolytic oil included with various group hydrocarbons along with other compounds. The O-H absorptions are generally quite intense and smoothly curved which was observed at 3412.57 cm^{-1} might be due to the presence of water and alcohol in the pyrolytic oil. A very small peak observed around 2800 cm^{-1} indicated the presence (C-H stretch) aldehyde group in the pyrolytic liquid. The peak observed at 1711 cm^{-1} might be due to the presence of carboxylic acid (RCO_2H) or Ketone (RCOR') group of compounds. The absorption peak noticed at 1659 cm^{-1} might be due to the presence of alkenyl ($\text{C}=\text{C}$ stretch) group or primary and secondary amine (N-H bend). The conjugated ketone group compounds noticed at around 1690 cm^{-1} and carboxylate (carboxylic acid salt) at around 1391 cm^{-1} . The absorption peak noticed at 1274 cm^{-1} showed the presence of primary or secondary bend OH group, 2105 cm^{-1} showed the presence of mono-substituted alkyne group ($\text{C}\equiv\text{C}$). The presence of disulfides (C-S stretch) and aryl disulfides (S-S stretch) were noticed with the absorption peak at 665 and 444 cm^{-1} respectively. It was also noticed that the pyrolytic oil contained some inorganic compounds such as phosphate ion and detected at 1052 cm^{-1} .

3.7. ^1H -NMR of pyrolytic liquid

Fig. 6 shows the ^1H -NMR analysis of sugarcane bagasse pyrolytic liquid. This analysis computes the occurrence of various protons due to chemical shift. The total hydrogen percentage in the aliphatic, olefinic and aromatic resonances at different chemical shift regions is given in Table 4. It was observed that the phenolic or olefin proton shift took place in 6.5–5.0 ppm region containing higher 86.99 % of total hydrogen compared to aromatic and naphthenic hydrocarbons. The chemical shift of proton noticed in the region 6.5–8.5 ppm quantified the presence of alkanes, alkenes, aromatics and other chemical compounds such as phenols, alcohols and acids. The existence of such chemical compounds was also depicted from FTIR analysis and NMR analysis.

3.8. GC-MS analysis of pyrolytic liquid

The GC-MS analysis of sugarcane bagasse pyrolytic liquid is indicated in Table 4. The sugarcane bagasse pyrolytic oil is compared with other bio-waste pyrolytic oil [23, 25, 26]. The presence of the chemical compounds in the pyrolytic oil characterized using FTIR and NMR analysis was also established from GC-MS analysis. It was observed that the pyrolytic liquid consisted with various oxygenated hydrocarbon compounds. The occurrence of oxygenated compounds in the pyrolytic oil resulted in less calorific value. The existence of various chemicals compounds were observed from this analysis. The major chemicals were included with 5.06 % of ester compounds, 5.24 % of Benzoic acid, 6.84 % of hexahydrophthalic Anhydride, 4.32% of cyclopentane dione, 11.68 % of phenolic compounds, 9.2 % of furan and its associated compounds, 3.07 % of Creosol and 1.90 % of p-cresol. The amount of phenolic and furans were higher compared to others. Along with these chemicals a little amount of hexane, aldehyde, aromatics and alkenes were exist in the pyrolytic oil. It was observed that most of the chemicals present in the pyrolytic oil were due to the degradation of cellulose. Fewer amounts of chemicals were noticed in the pyrolytic liquid which may be formed due to the degradation of lignin. Benzene, phenols, cresols and various other aromatic compounds produced from pyrolysis of lignin, due to the aromatization reaction. From Table 4, it was also observed that the chemical compounds formed by the pyrolysis of biomass not only varies with biomass types but also depends on the pyrolysis conditions. Qiang et al., and Tsai et al., studied the pyrolysis of rice husk [25, 26] and reported similar observations. The comparison between the GC-MS analysis of pyrolytic oils confirmed that the composition of pyrolytic oil varies with the pyrolysis conditions even the raw materials are indifferent.

4. Conclusion

From this study, it was observed that sugarcane bagasses can be a potential source for the production of biochemical and bio-oil. It was also confirmed that the without the flow rate of inert gas during pyrolysis biomass resulted in aqueous rich pyrolytic liquid. However, the characterization study visualised the existence of various valuable chemical compounds in the pyrolytic liquid which can be distilled or extracted for further use. The chemical compounds such as Furans can be used as a liquid fuel directly. The acid compounds can be esterified to produce ester and purified form of aromatic compounds can be used as fuel. Hence, further study is required to observe the effect of various upgrading techniques on the fuel properties and composition of sugar cane bagasse pyrolytic liquid which are the future studies.

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Figure captions

- Fig. 1.** EDX analysis of Sugar cane bagasse
Fig. 2. TGA/DTG analysis of Sugarcane bagasses
Fig. 3. Effect of temperature on the yield of sugarcane bagasse pyrolysis
Fig. 4. DSC analysis of Sugar cane bagasse pyrolytic liquid
Fig. 5. FTIR analysis of sugarcane bagasse pyrolytic liquid
Fig. 6. ¹H-NMR analysis of sugarcane bagasse pyrolytic liquid

Tables

Table 1. Comparative study between the physiochemical properties of sugarcane bagasse with other bio-waste

Proximate analysis					
Moisture content (%)	Volatile matter content (%)	Fixed carbon content (%)	Ash content (%)	HHV (MJ Kg ⁻¹)	Raw materials
1.94	71.48	15.57	11.01	19.56	Sugarcane Bagasse (present study)
3.3	76.83	18.20	1.17	18.34	Mesua Ferrea [20]
5.34	72.11	12.90	9.65	15.72	Jute Dust [21]
8.4	84.2	11.9	3.9	19.6	Switch Grass [22]
-	75.85	20.1	4.14	16.81	Sugarcane Bagasse [23]
10.1	75.5	11.2	3.2	20.15	Coconut Shell [24]
6.8	67.2	21.6	4.4	20	Olive Bagasse [25]

Ultimate analysis					Raw materials
Carbon (%)	Hydrogen (%)	Nitrogen (%)	Sulphur (%)	Oxygen (%)	
56.16	3.51	6.21	2.38	31.73	Sugarcane Bagasse (present study)
47.5	5.43	1.15	-	45.9	Mesua Ferrea [20]
43.71	6.18	1.38	-	48.75	Jute Dust [21]
42	6.1	0.4	0.1	47.4	Switch Grass [22]
48.67	6.70	0.45	0.08	44.10	Sugarcane Bagasse [23]
64.23	6.89	0.77	0.50	27.61	Coconut Shell [24]
53.4	7.5	1.7	-	37.4	Olive Bagasse [25]

Table 2. Comparative study between the pyrolysis yields of sugarcane bagasse pyrolytic liquid with other bio-waste

Raw material	Optimum Temperature (°C)	wt. % yield	Reactor type	Carrier gas	Carrier gas flow rate (mL min ⁻¹)
Sugarcane bagasse (present study)	450	52.00	Semi-batch	--	--
Mesua ferrea [20]	550	29.6	Fixed bed tubular	N ₂	100
Jute dust (21)	500	31.11	Fixed bed	N ₂	--
Switch grass [22]	600	37	Pressure reactor	N ₂	--
Sugarcane Bagasse [23]	475	56	Fixed-bed tube	fire	--
Coconut Shell [24]	575	49.5	Semi-batch	--	--
Olive bagasse[25]	500	37.7	Tubular	--	150

Table 3. Comparative study between the fuel properties of sugarcane bagasse pyrolytic liquid with other bio-waste

Fuel properties	Density (kg m ⁻³)	Kinematic viscosity (Cst.)	Carbon residue (wt. %)	Flash point (°C)	Pour point (°C)	Gross calorific value (MJ kg ⁻¹)	Water content (wt. %)
Present study	1052	22.5	2.25	52	-10	32	10.58
Mesua ferrea [20]	0.972	--	--	50	7	32.63	--
Jute dust (21)	0.9582	48	--	65	--	26.71	--
Switch grass [22]	920	10	--	-	-	36.3	--
Sugarcane Bagasse [23]	1150	21.50	--	>72	-12	23.50	11.60
Coconut Shell [24]	1053.6	1.47	5.56	--	--	19.75	--
Olive bagasse [25]	1070	--	--	77	--	31.8	--

Table 4. NMR analysis of sugarcane bagasse pyrolytic liquid

Types of hydrogen	Chemical shift	Percentage of total hydrogen
Phenolic or olefin Proton	6.5– 5.0	86.99
Hydroxyl groups or ring-join methylene(Ar–CH ₂ –Ar), aliphatic alcohols or amine group	4.5– 3.3	0.82
CH ₃ CH ₂ and CH to an aromatic ring	3.3-2.0	1.63
CH ₂ and CH β to an aromatic ring (naphthenic)	2.0-1.6	1.99
β-CH ₃ , CH ₂ and CH γ to an aromatic ring	1.6-1.0	0.91
CH ₃ γ or further from an aromatic ring	1.0-0.5	7.66

Table 5. GC-MS analysis of sugarcane bagasse pyrolytic liquid and other bio-waste pyrolytic oil

Compounds	Sugarcane bagasse	Rice husk [26]	Rice husk [27]	Coconut Shell [24]
Furan ,tetrahydro-2,5-dimethoxy-	✓	--	✓	--
1,2-Hexanediol	✓	--	✓	--
1,2-Cyclopenten-2-methyl-	✓	--	✓	--
2(5H)-Furan one	✓	--	✓	--
3-Methoxy-2,2-Dimethyl Oxirane	✓	--	--	--
1,2-Cyclopentane dione-	✓	--	✓	--
2(5H)-Furan one ,5-Methyl	✓	✓	--	--
4-Amino-6-hydroxy pyrimidine	✓	--	--	--
3-Hexane-2-one,3-Methyl-	✓	--	--	--
2-Furancarboxyldehyde,5-methyl	✓	--	--	--
Phenol	✓	✓	✓	✓
Pentatonic acid,4-oxo-methylester	✓	--	--	--
Oxazolidine,2-butyl,2-ethyl,3-methyl	✓	--	--	--
Cyclohexane,3-Methyl-	✓	--	--	--
1-Hydroxy-2-pentanone-	✓	--	--	--
2-Hydroxy-3-methyl-	✓	--	--	--
1H-Pyrazole,1,3,5-trimethyl-	✓	--	--	--
1-Butene,3-3 Di-methyl-	✓	--	--	--
Phenol,3-Methyl-	✓	--	--	--
1,3-Dioxirane-2-Propanol,2-Methyl	✓	--	--	--
1H-Pyrrole,1-Methyl	✓	--	--	--
p-cresol	✓	✓	--	✓
4-Hydroxy-2,5-dimethyl,3(2H)-furan one	✓	--	--	--
N-Methyl-1,3-diaminepropane-	✓	--	--	--
Maltol	✓	--	✓	--
2-Cyclohexanone-1-one,3-ethyl-2-hydroxy	✓	--	✓	--
Phenol, 4ethyl-	✓	✓	✓	--
2,6-Dimethyl- 2,6-Octadine	✓	--	--	--
Creosol	✓	--	--	✓
3-Aminopiperidin-2-one	✓	--	--	--
Catecholborane	✓	✓	--	--
3-Hexanal	✓	--	--	--
Phenol,4-ethyl-2-Methoxy	✓	--	✓	--
2-Methyl cyclohexanol	✓	--	--	--
Hexahydrophthalic Anhydride	✓	--	--	--
Vanillin	✓	✓	--	✓
3-(Methyl Sulfonyl)-Benzoic acid	✓	--	--	--
Benzoic acid,2,6 Dimethoxy	✓	--	--	✓
Ethyl 4- Methyl Hexanoate	✓	--	--	--
Benzoic acid,4-Hydroxy-Butyl Ester-	✓	✓	--	--
Methyl 3,5-Dimethoxy benzoate	✓	--	--	--
3-4 Dimethoxy Benzoic acid	✓	✓	--	✓
Furfural	✓	✓	--	--
2-Hexanone, 3-Methyl-	✓	--	--	--
Phenol,2,6-Dimethoxy-	✓	--	--	--
Phenol, 2-Methoxy	✓	--	--	✓
Cyclohexanone, 3-Methyl-	✓	--	--	--
Benzoic acid, 4-Hydroxy-3-Methoxy-	✓	--	--	✓

Figures

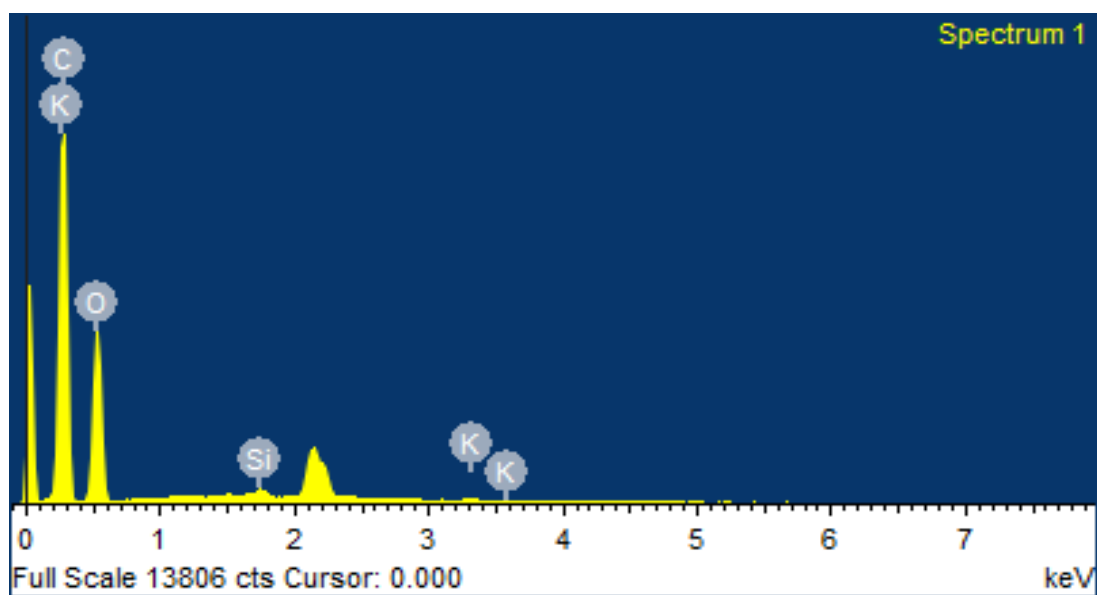


Fig. 1.

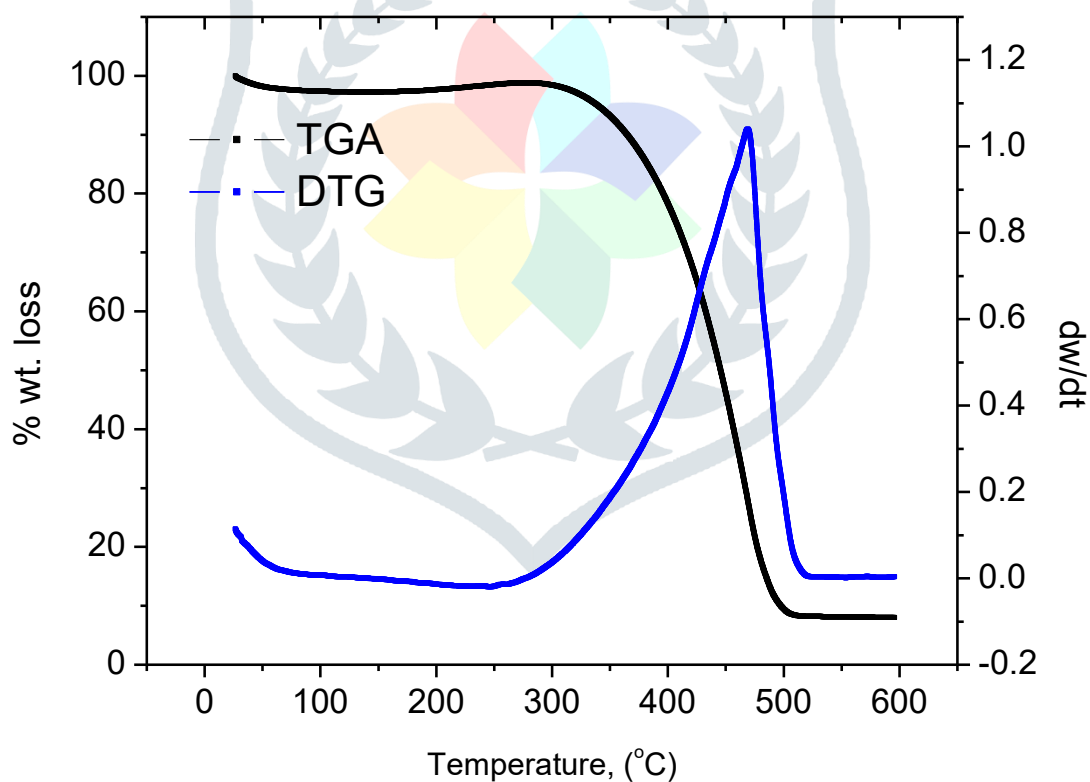


Fig. 2.

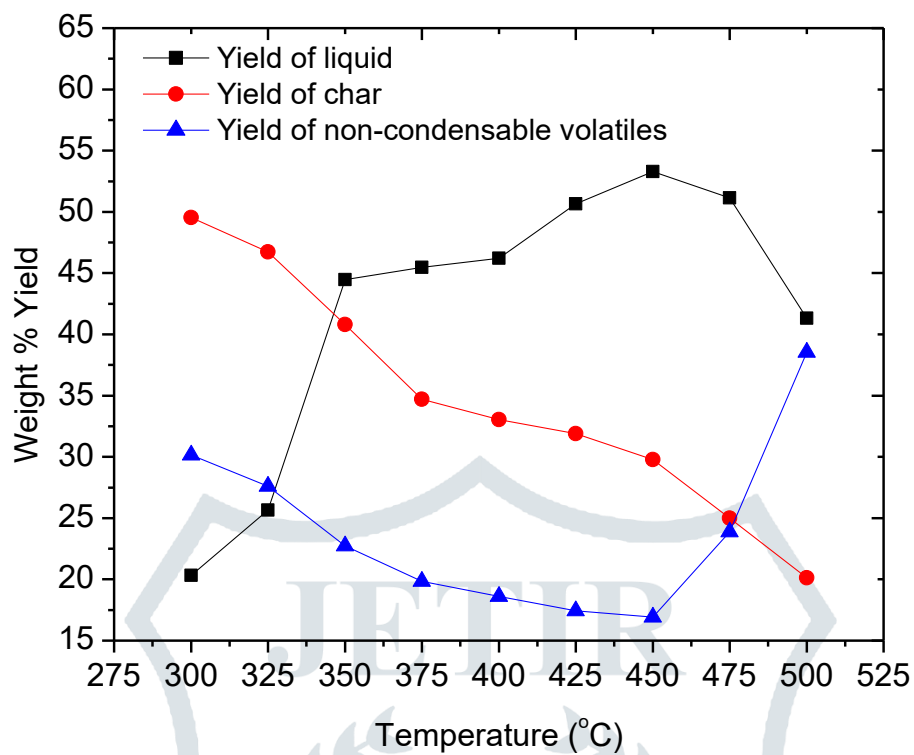


Fig. 3.

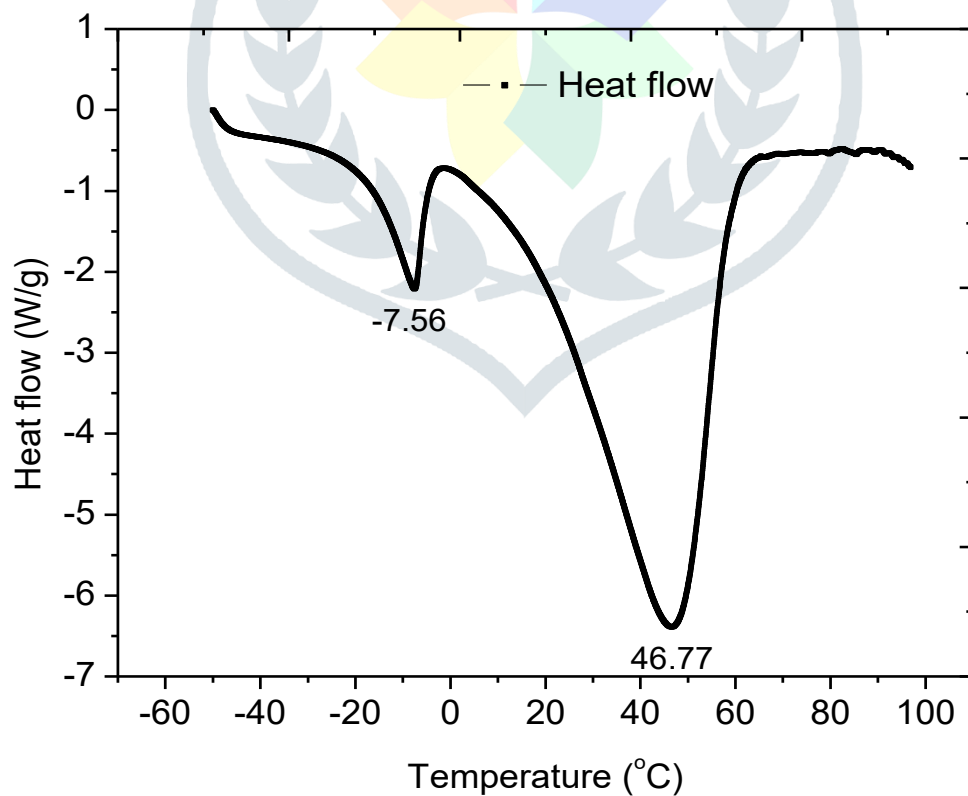


Fig. 4.

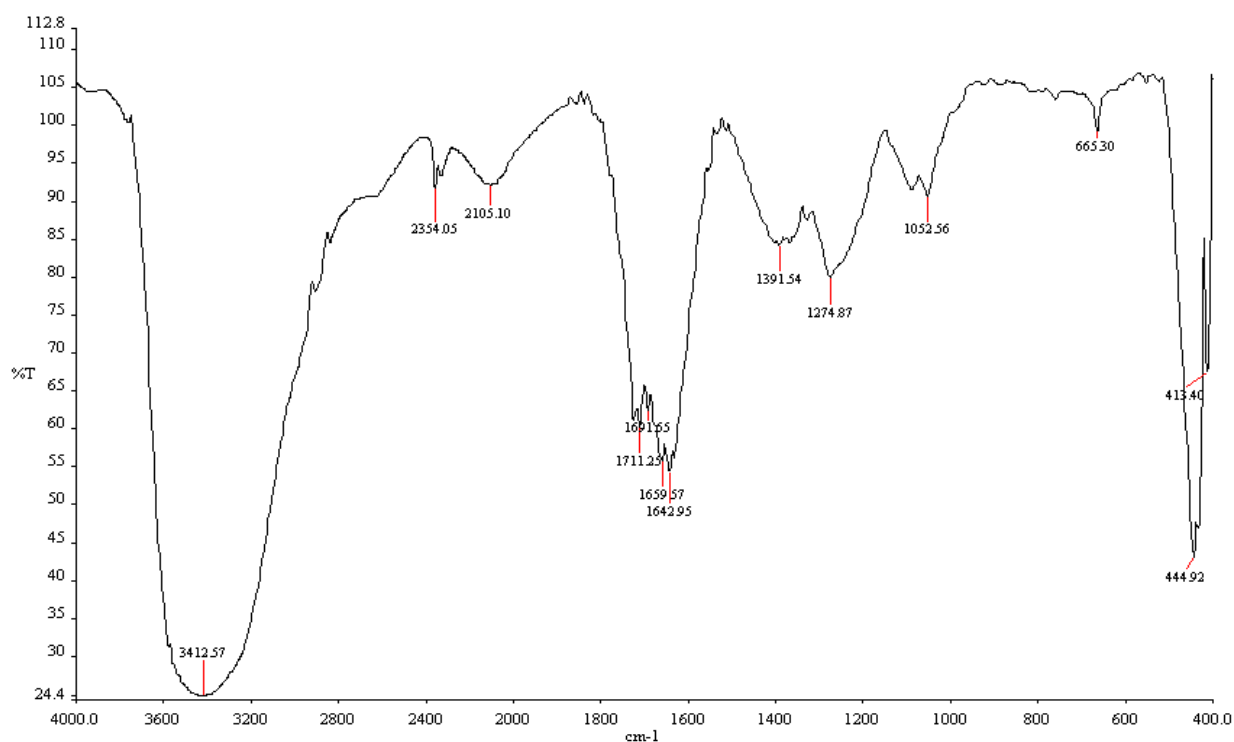


Fig. 5.

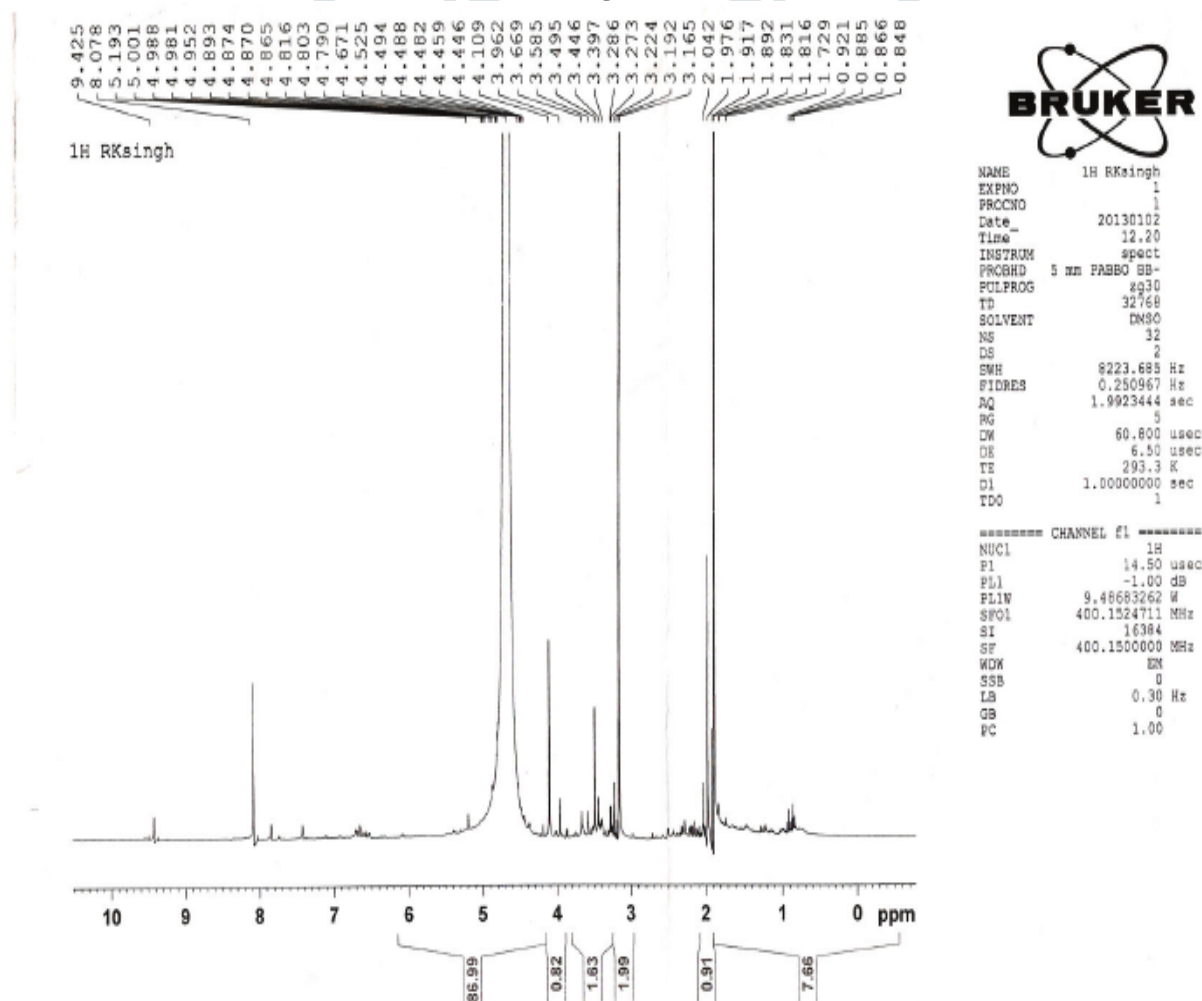


Fig. 6.