

## CHARACTERISATION OF PYROLYSIS OIL DERIVED FROM TEAK TREE SAW DUST AND RICE HUSK

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### Abstract

The teak tree saw dust (TTSD) and rice husk (RH) were pyrolysed in a fixed bed reactor at the temperature of 450°C. The feedstock particle sizes for both the biomasses were in the range of 0.5-1.0 mm. About 33.3 and 35 wt% liquid yields were found from TTSD and RH respectively. The calorific values were recorded as 23.41 and 20.01 MJ/kg for TTSD and RH bio-oils respectively, which are slightly higher than their respective biomasses. The kinematic viscosities of the bio-oils were found to be in the range of 6.8-8.1 cSt, which are quite lower than many other bio-oils. The pH value of TTSD bio-oil (i.e., 3.4) was slightly higher than the RH bio-oil (i.e., 2.8). The bio-oils indicated acidic nature with low pH value, e.g., 2.8-3.4. The bio-oils contained mostly the carboxyl and hydroxyl functional groups to represent phenols and alcohols. Both TTSD and RH have the potential to produce pyrolysis oil; however, it requires certain upgradations to make it useful for engines.

Keywords: Pyrolysis oil, Thermogravimetric analysis, Teak tree, Rice husk, Atomic ratio.

### 1. Introduction

The crisis of petroleum fuel and environmental pollution are the major concerns in most of the countries of the world. Therefore, the uses of renewable sources to harness energy have been found increased interest in the recent years. Among all the sources of renewable energy, the biomass is one of the easily available and it can be converted into liquid (pyrolysis oil) and gaseous form, which have comparatively better fuel characteristics than the biomass [1, 2]. For conversion of biomass, pyrolysis and gasification are found to be more flexible and easier than the other techniques [3]. From the literature report, it was found that pyrolysis is one of the most economical and simple technique for thermochemical conversion of biomass [4].

**Abbreviations**

FTIR	Fourier Transform Infra-Red
IR	Infrared
RH	Rice husk
TTSD	Teak tree saw dust
TG	Thermogravimetric
TGA	Thermogravimetric analysis

Although the pyrolysis of biomass has three main products such as liquid (bio-oil), solid (biochar) and gas (incondensable gas), it is mainly optimised for bio-oil production. The bio-oil derived from biomass through pyrolysis is a dark brown free flowing liquid with a neat 'smoke' smell and its properties are quite different from the petroleum fuels [5]. The pyrolysis oil consisting of approximately 35-40 wt.% of oxygen, 55-60% wt.% of carbon, an acidic pH value, density near about of 1.2 g/cm<sup>3</sup> and 15-60 wt.% of water [6,7].

The fuel in the liquid form is easier to store and transport than the solid biomass. Moreover, by converting biomass into liquid form can reduce the pollution and cost of solid waste disposal. However, because of some poor properties, such as low calorific value, low pH, high viscosity, high water and oxygen content, certain upgradations are required to make it a suitable engine or transportation oil. Usually, the oxygen content found in the bio-oil is about 45-50 wt.%, because of which the calorific value of the oil becomes a low average value of 18 MJ/kg [8]. Qiang et al. [9] found the kinematic viscosity of the bio-oil derived from rice husk about 17cSt at 40°C and observed to be gradually decreasing with the increase of temperature. The pyrolysis oil contains substantial amount of acid contents such as carboxylic acid, formic acid and acetic acid which results low pH value, e.g., 2-3 [10].

The moisture content in the biomass feedstock plays an important role on the characteristics of the pyrolysis products. Due to the higher moisture content in the biomass, bio-oil contains more water leading to reduce the calorific value. Therefore, the recommended moisture content in the feedstock for proper pyrolysis is below 10-15 wt.% [11]. The size of the feedstocks also influences the yields and properties of the pyrolysis products. It was found that, smaller size feedstocks are mostly preferred in pyrolysis as it provides more uniform heating which results in higher liquid yields [12]. The yield of liquid product are found to be different in fixed bed and moving bed reactors although the same size (2 mm) feedstocks are used, and therefore it is very difficult to generalise the specific size of feedstock for a specific pyrolysis system [13].

A wide range of biomasses have been found to be used as feedstock for pyrolysis, among which agricultural residues, forestry products, municipal waste are the most common [14]. Rice husk is one of the mostly abundant agricultural residues found available in many parts of the world. Several types of investigations were performed on rice husk through rapid or fast pyrolysis using fluidised bed reactors and found some suitable properties of the bio-oils derived from it [15, 16]. Feng et al. [17] used saw dust and rice husk and their mixture to produce pyrolysis oil using fluidised bed reactor in order to investigate the yields and properties of the oil and found the similar kinds of results. Recently, Kumar et al. [18] have investigated the effect of temperature and catalysts on the

behaviour of saw dust pyrolysis oil. However, the liquid yield was quite lower in both cases, i.e., with and without catalyst as compared to the present study.

The saw dust of teak tree is easily available in the village areas of Assam, India, as the wood of teak tree is mostly used for furniture making purposes. The dead inner portion of teak wood is brownish red in colour, while the living outer portion is whitish and light yellowish brown in colour. The teak tree wood is hard and ring porous. There is a strange smell in newly collected saw dust of teak tree. The rice husk is the major by-product of rice mill, which is available across the state of Assam, India. About 2 million tons of rice husk is found to be available each year in India [19]. The characteristics of pyrolysis oil derived from rice husk and saw dust are found in many previously published reports [9, 16, 17]. Feng et al. [17] showed that the volatile matter in saw dust is higher as compared to the rice husk and thus the liquid yield and calorific value. Therefore, saw dust obtained from teak tree (TTSD) is although a new biomass material, it is predicted that the bio-oil from TTSD contains higher calorific value than RH. In the present work, TTSD and RH are taken as the feedstock and a comparative study of the properties of the bio-oils is performed.

## **2. Experimental**

### **2.1. Feedstock preparation**

The teak tree saw dust and rice husk were collected from the small scale wooden mill and rice mill respectively, from the village of Assam (India). A mechanical sieve shaker (IKON<sup>TM</sup> instrument) was used along with the standard test sieve (SETHI, as per IS: 460, sieve mesh size 500 micron and 1.00 mm) to prepare the feedstocks in the size of 0.5-1.0 mm. Before feeding into the reactor, the feedstocks were dried in the sunlight for 24 h and then these were dried in the electrical heater to reduce the moisture content.

### **2.2. Proximate and elemental analysis**

In order to perform the proximate analysis of the biomass samples, ASTM D-271-48 standard was used to determine ash content, volatile matter and fixed carbon. The moisture content was determined as received basis. The elemental analysis of the biomass samples and pyrolysis oils were conducted in a CHN analyser (Perkin Elmer, series- ii). The calorific values of the biomasses and pyrolysis oils were determined with the help of the Automatic Bomb Calorimeter (Model 5E-1 AC/ML, Changha Kaiyuan Instrument Co-Ltd.).

### **2.3. Thermogravimetric analysis (TGA)**

The thermogravimetric analysis was conducted in a TGA analyser (SHIMADZU, TGA-50), in nitrogen atmosphere under the flow of N<sub>2</sub> at the rate of 30 ml/min. About 4.68 mg of biomass samples were used for the experiments. The samples were heated from the room temperature to 600°C at the rate of 10°C/min.

#### 2.4. Viscosity, density and water content

Two major properties of the bio-oil, i.e., viscosity and density, were measured with the help of a digital instrument (LEMIS Instrument, VISCODENS, VDM 300). In order to determine the water content of the bio-oil samples, the Dean and Stark Apparatus (As per IP specification) was used. For complete separation of water, the experiment was carried out for 24 h after which no more moisture was found to be received in the graduated tube.

#### 2.5. Flash point, fire point and pH value

An open cup digital tester (REICO EQUIPMENT AND INSTRUMENT PVT. LTD, model RFFP) was used to determine the flash point and fire point of the pyrolysis oils. The pH values of the bio-oils were determined with help of a pH meter (SYSTRONICS, Digital pH Meter 802). The FTIR analysis of the bio-oil samples was performed using a FTIR instrument (NICOLET, Impact 410) with a resolution of  $4\text{ cm}^{-1}$ .

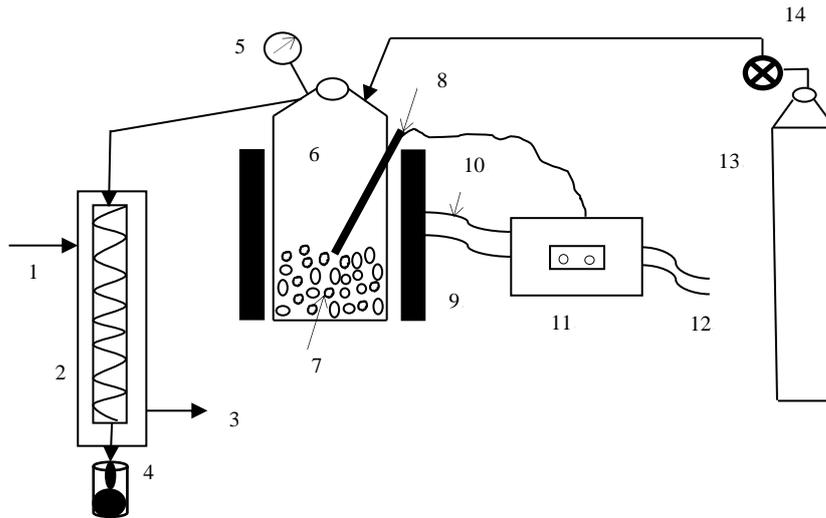
All the above properties were evaluated three times and the average values are reported in this article. The standard deviations of various tests values ranged in 0.5-1.5.

#### 2.6. Bio-oil production

A lab scale pyrolysis plant was developed in Tezpur University, Assam, India, in order to produce the pyrolysis oil from the local biomasses as shown in Fig. 1. A fixed bed reactor made of stainless steel is used for the pyrolysis, the main body of which is cylindrical in shape with a conical head. The inside and outside diameters of the cylindrical part of the reactor are 88 mm and 100 mm respectively. The length of the cylindrical part of the reactor is 150 mm, while the length of conical part is 100 mm. At the top of the reactor, a small opening with a diameter of 25 mm is made to feed the biomass sample inside the reactor. The major part of the reactor is surrounded by a clamp type tubular furnace (2 kW) made of ceramic and stainless steel. In order to condense the pyrolysis vapour, a glass condenser is used. A nitrogen gas cylinder is connected to the reactor to provide a flow of  $\text{N}_2$  (99.9% purity) throughout the pipe connecting the reactor and condenser. The continuous monitoring and regulation of the temperature and heating rate was performed with the help of a digital temperature controller.

About 80 g of biomass sample was placed gently in the reactor through the opening at the top of the reactor and it was tightly closed. The temperature was programmed at  $10^\circ\text{C}$  for 1.0 min with  $10^\circ\text{C}/\text{min}$  increment and held at  $450^\circ\text{C}$ , for at least 1 h. The literature study reveals that the optimum liquid yield in pyrolysis is found at around  $450^\circ\text{C}$ , such as the liquid yield of rice husk pyrolysis was maximum, e.g., 56 wt.% at  $450^\circ\text{C}$  and  $480^\circ\text{C}$  [16], while the optimum temperature for liquid yield in pyrolysis of rice straw ranged in  $440\text{-}500^\circ\text{C}$  and the properties were evaluated for the bio-oil produced at  $445^\circ\text{C}$  [20]. Similarly, the optimum liquid was obtained in between  $400\text{-}500^\circ\text{C}$  during the pyrolysis of waste bamboo [21]. Further, the highest bio-oil was obtained from saw dust at  $450^\circ\text{C}$  (26 wt.%) without catalyst, however, it reduces while catalyst is used [18].

During the experiment, nitrogen gas was supplied at the flow rate of 25 ml/min intermittently to enhance the flow of vapour, produced inside the reactor. This vapour was passed through a condenser with the cold-water circulation at the temperature of 10°C. The operating pressure in the reactor during the experiment was maintained at 0.5 MPa. The condensed liquid was collected in a container at the end of the experiment. As the reactor cooled down to the room temperature, the conical head of the reactor was opened for collecting the char stored inside the reactor and the inner surface of the reactor was cleaned. The liquid yields of TTSD and RH pyrolysis were about 33.3 and 35 wt.% respectively. These values are found to be relatively higher than few recently published articles [18, 22].



**Fig. 1. Schematic diagram of lab scale pyrolysis plant.**

1: Cold water in, 2: Condenser, 3: Hot water out, 4: Bio-oil container, 5: Pressure gauge, 6: Reactor, 7: Biomass feedstock, 8: Temperature sensor, 9: Furnace, 10: Power supply to the furnace, 11: Temperature controller, 12: Input power, 13: N<sub>2</sub> cylinder, 14: Gas flowmeter.

### 3. Results and Discussion

#### 3.1. Biomass characterisation

The results obtained from proximate and elemental analysis of TTSD and RH are reported in Table 1 along with some other biomasses. The volatile matter in TTSD has been found to be higher than RH. The higher volatile matter is an indication of higher amount of cellulose and hemicellulose content in the biomass. Further, the amounts of volatile matters found in TTSD and RH are quite higher than the other biomasses as shown in Table 1. The fixed carbon amounts in both TTSD and RH are quite lower as compared to all the biomasses. This result confirms the presence of lower amount of lignin in TTSD and RH. The ash contents of TTSD and RH are found to be within the range of other biomasses, and thus these biomasses can be considered as the suitable feedstocks for bio-oil production. The calorific values of TTSD and RH are found to be

similar to many other biomasses such as rice straw, bamboo sawdust, rapeseed cake, etc. [1, 5, 15, 20, 23-25]. The variation of calorific values in the biomasses is due to the type of biomass, amount of moisture, oxygen, carbon and hydrogen contents as seen in Table 1.

**Table 1. Proximate and elemental analysis of TTSD and RH.**

	Rice husk [15]	Rice straw [15]	Sweet sorghum bagasse [1]	RH (this study)	Soybean [23]	Pine saw dust [5]	TTSD (this study)	Mesquite saw dust [5]	Wheat shell [5]
Moisture (wt%)	1.3	4.1	4.17	9.34	9.5	10.2	10.05	12.5	18.8
Volatile matter (wt%) <sup>db</sup>	69.3	74.3	76.33	78.59	69.28 <sup>wb</sup>	--	94.92	--	--
Fixed carbon (wt%) <sup>db</sup>	16.8	16	15.98	8.71	14.92 <sup>wb</sup>	--	2.98	--	--
Ash (wt%) <sup>db</sup>	13.9	9.8	3.53	12.7	6.30 <sup>wb</sup>	0.3	2.10	0.6	5.4
Calorific value (MJ/kg)	16.8	18.6	18.57	17.05	18.17	15.4	16.45	15.4	14.1
Elemental analysis (wt%)									
C	50.4	54.3	45.71	39.19	41.7	51.6	51.76	53.0	46.8
H	6	5.7	5.80	5.98	6.17	5.2	7.55	4.8	6.0
N	0.9	1.6	0.33	0.97	7.14	--	0.87	0.6	3.4
O (by difference)	42.6	38.4	48.16	53.86	44.99	43.2	39.82	41.6	43.8
H/C	--	--	0.13	1.83	1.78	--	1.75	--	--
O/C	--	--	1.05	1.03	0.81	--	0.58	--	--

db: dry basis, wb: wet basis

The TGA profiles of TTSD and RH are shown in Fig. 2. In both the samples, the moisture removal from the biomasses can be seen within 100°C. However, minor weight losses can be observed up to about 250°C in the RH profile, which is assumed to be due to the evaporation of moisture and volatile matters as well as the decomposition of hemicellulose. In TTSD, no significant weight loss is observed up to 200°C. The major weight losses, i.e., approximately 50 wt% TTSD and 44 wt% RH are observed within the temperature of 250-350°C. This can be observed by the sharp decrease of the TG curves.

The patterns of the TG profiles found in this study are quite similar to the TG profiles of bamboo saw dust and rice straw [20]. These major weight losses indicate the decomposition of hemicellulose and cellulose. The weight loss up to 250°C as shown by TGA curves (Fig. 2.) indicates the decomposition of hemicellulose and the weight loss between 250-350°C is due to decomposition of cellulose. The further weight loss after 350°C as seen in the TG profiles indicates the decomposition of lignin content in the biomass [20]. The cellulose is a polysaccharide in which D-glucose is uniformly linked with  $\beta$ -glycosidic bonds. It has the crystalline structure and offers great resistance to acids and alkalis. The hemicellulose is also a polysaccharide whose degree of polymerization is higher than cellulose and smaller in size and therefore, it decomposes at low temperature such as 250°C as compared to cellulose.

Since lignin consists of benzene rings, it is more aromatic as compared to cellulose and hemicellulose, and therefore, it requires more thermal energy in order to break the bonds and found to be decomposed in a broad higher temperature region [26, 27]. This is an agreement of the result of proximate analysis with higher volatile matters, as the volatile matters are derived from cellulose and hemicellulose which are likely to convert into bio-oil [14]. The remaining solid products of TTSD and RH are about 21 wt% and 27 wt% respectively and thus it can be concluded that pyrolysis was almost completed at 450°C [21]. TTSD and RH can be considered as softwood biomasses, as the maximum thermal decomposition in both the biomasses occurs at low temperature [28]. From the TG analysis, the minimum pyrolysis temperature requirement for TTSD and RH can be considered as 350°C, as about 70% of the biomass weight was found to be lost within this temperature.

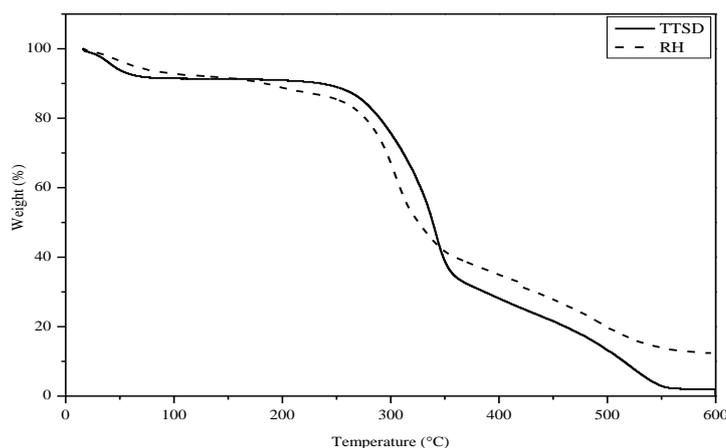


Fig. 2. TGA of TTSD and RH.

### 3.2. Bio-oil characterisation

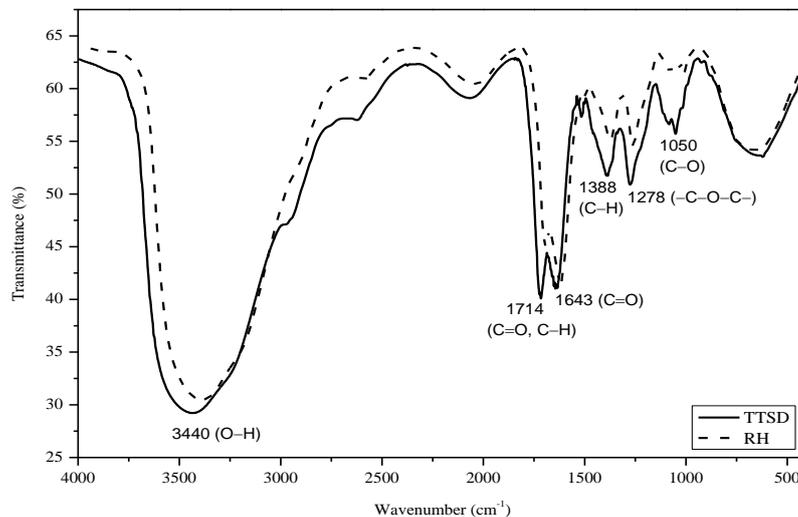
The results of elemental analysis and the physical properties of TTSD and RH pyrolysis oils are shown in Table 2. The calorific value of the rice husk (RH) bio-oil in the present work has been found to be slightly better than the previously reported value by Pattiya and Suttiback [15]. The calorific value of bio-oil of TTSD is higher than RH as well as some other bio-oils as seen in Table 2. However, TTSD and RH bio-oils have lower calorific values than the rapeseed cake and soybean bio-oils (Table 2). This is possibly due to high water content in the raw biomass and bio-oil, high oxygen content and low carbon content in the bio-oil. In general, as the oxygen and water content in the bio-oil increases, the calorific value decreases as seen clearly in Table 2. However, the water content reduces the viscosity of the bio-oil and helps in atomization and combustion [10].

The atomic ratios, i.e., H/C and O/C, in TTSD and RH bio-oils are found to be within the range of other bio-oils as shown in Table 2. It can be observed that the H/C and O/C ratios in both TTSD and RH have decreased from the feedstock to bio-oil, (see Tables 1 and 2) and this indicates the production of less oxygenated and more carbonaceous liquid fuel. The flash points of TTSD and RH bio-oils ranged between 63-90°C, which is a good agreement with some previous publications [23, 25, 29].

The pH value of the bio-oils shown in Table 2 indicates that the bio-oil of RH is less acidic than TTSD. However, the pyrolysis oils derived from lignocellulosic biomasses are generally acidic in nature [15]. This acidic nature of the pyrolysis oils with low pH values indicates the presence of significant amount of acid contents such as carboxylic acids. Further, the pH value of RH and TTSD bio-oils are quite comparable with all other bio-oils, except wheat shell, as shown in Table 2. The pH values of the bio-oils found in the present work are slightly higher than the bio-oil obtained from Pine saw dust, e.g., 2.5 [30].

The kinematic viscosities of the RH and TTSD pyrolysis oils measured at 40°C and 60°C are reported in Table 2. The decrease of viscosity in RH and TTSD bio-oils with increase of temperature followed the similar pattern, as observed by Qiang et al. [9]. The viscosity of the bio-oils in the present study was found to be lower than most of the previously produced bio-oils as reported in Table 2. This lower viscosity is the result of higher amount of water content in the bio-oil. From Table 2, it is seen that, the density of the TTSD and RH bio-oils are consistent with the range of other bio-oils, e.g., 964-1300 kg/m<sup>3</sup> as found elsewhere [16, 17, 25, 31].

The shape, position and intensity of the peaks found in FTIR spectrum, reveal the functional group and structure of the compounds present in the pyrolysis oil. The IR spectrum of TTSD and RH pyrolysis oils are plotted in Fig. 3. The broad band in the frequency range 3600-3200 cm<sup>-1</sup> (about 3440 cm<sup>-1</sup>), in both TTSD and RH bio-oils represents the polymeric O-H, water, phenol and alcohol with O-H stretching vibration [9, 32]. The peak found within 1750-1650 cm<sup>-1</sup> indicates the presence of carboxylic acid (C=O stretching vibration together with C-H stretching vibrations), which contains carboxyl and hydroxyl functional groups [33]. The presence of olefinic compounds such as aromatic hydrocarbons, phenols and esters can be identified with C=O stretching vibration absorption at 1643 cm<sup>-1</sup> [1]. The C-H bending vibrations within 1465-1380 cm<sup>-1</sup> (at 1388 cm<sup>-1</sup>) indicate the presence of compounds with alkane groups in the pyrolysis oils [34].



**Fig. 3. FTIR of TTSD and RH pyrolysis oil.**

**Table 2. Physical properties of TTSD and RH pyrolysis oil.**

Properties	Wheat shell [5]	Sweet sorghum bagasse [1]	Rice husk [15]	RH (this study)	Rice straw [15]	Sugarcane bagasse [29]	TTSD (this study)	Rapeseed cake [24]	Soybean [23]
Higher calorific value, as produced basis (MJ/kg)	6.02	12.39	19.4	20.01	22.1	22.4	23.41	26.7	33.6
Elemental analysis (wt%)									
C	69.9	22.08	58.9	61.05	64.1	54.6	65.08	63.56	67.89
H	9.7	6.52	7.0	5.97	7.6	6.45	7.19	8.89	7.77
N	11.5	0.21	0.9	0.91	0.8	0.73	0.89	3.6	10.84
O (by difference)	8.8	71.2	33	32.07	27.3	38.07	26.84	23.74	13.5
H/C	1.90	0.3	1.2	1.37	1.4	--	1.46	1.68	1.37
O/C	0.07	3.22	0.5	0.59	0.3	--	0.42	0.28	
Flash point	--	--	--	72	--	<90	70	83	63
pH	6	2.84	2.9	3.4	3.4	2.7	2.8		
Kinematic viscosity (cSt)									
at 40°C		1.89	10.3	8.1	13.3	26.7	7.6		
at 60°C		1.23		6.8		11.2	6.9		
Density (kg/m <sup>3</sup> )									
at 40°C				1180		1195	1200		
at 60°C				1168		1180	1190		
Water content (wt%)	84.4	56.29	23.46	23.22	22.14	13.8	24.45	0.001	0

The absorptions occurred between 1300-900 cm<sup>-1</sup> in both the pyrolysis oils are possibly due to C–O vibrations of carbonyl components such as alcohols, esters, carboxylic acids or ethers, whereas absorption at 1278 cm<sup>-1</sup> can be assigned to –C–O–C– for ethers [35]. The peaks found within the band of 1500-1000 cm<sup>-1</sup> (centred at 1050 cm<sup>-1</sup>) in TTSD confirm the presence of esters, primary, secondary and tertiary alcohol with C–O stretching vibration. However, no such peak was observed in that region in case of RH bio-oil.

#### 4. Conclusions

Teak tree saw dust (TTSD) is the new biomass which was taken in this study for production of bio-oil and the characteristics of the bio-oil are consistent with and in some cases better than the bio-oils produced from other biomasses. Some characteristics of the RH bio-oil in this study are better than the previously produced RH bio-oil such as calorific value, pH and viscosity. Further, the study reveals that the TTSD and RH biomasses are mainly composed by cellulose and hemicellulose as well as small amount of lignin. The decomposition of the biomasses almost completed at around 450-500°C and thus pyrolysis temperature for the experiment was set at 450°C. The calorific values of TTSD and RH bio-oils were higher than some other bio-oils, although it is not enough for direct use as engine oil, and therefore certain necessary upgradations are required. Due to higher amount of oxygen and water contents, the calorific values of the bio-oils were found to be lower. Both the bio-oils were acidic nature due to low pH value (i.e., 2.8 and

3.4). Similar to other pyrolysis oils, the TTSD and RH bio-oils also contain carboxylic acids and alcohols due to pyrolysis of cellulose and hemicellulose and less aromatic hydrocarbons due to less pyrolysis of lignin.

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