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# انتاج زيت بيو نفط من شجر الأريقة باستعمال قاعدة رد فعل ثابتة

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## الخلاصة

البيو نفط ينتج من شجرة الأريقة، وهي متاحة في ولاية اسام (الهند)، وذلك باستخدام مفاعل سرير ثابت جنبا إلى جنب مع التحقق من بعض الخصائص الفيزيائية مثل القيمة الحرارية، والكثافة، اللزوجة، والمحتوى المائي، نسبة الأكسجين، نقطة الوميض، ومؤشر النار، وتحليل قيمة الرقم الهيدروجيني، ... الخ. تم دراسة تأثير المنتجات، أي السائل وشار مع درجة الحرارة. وقد تم التحقق من تباين اللزوجة والكثافة من النفط الحيوي في خمس درجات حرارة مختلفة. وتم العثور على أقصى قدر من اللزوجة الحركية والكثافة لتكون 12.15 و 1009.3 كغ / م$^3$، على التوالي. وقد اتخذ عينة مقاسها حول 2-2.8 ملم. قبل الانحلال الحراري، وقد أجريت التحاليل المباشرة والنهائية من المواد الوسيطة وجدت القيمة الحرارية من الكتلة الحيوية 13.2 إم جي / كج في حين أن القيمة الحرارية للبيونفط كما 15.2 إم جي / كغ. كما تم التحقق في التركيب الكيميائي لعينة النفط مع مساعدة من الغاز اللوني والكتلة الطيفي (GC-MS) واستعمال تقنيات التحويل بواسطة فورييه للتحليل الطيفي والأشعة تحت الحمراء.

# Production of pyrolysis oil from Areca tree using a fixed bed reactor

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## ABSTRACT

The pyrolysis oil was produced from the areca tree, available in Assam (India), using a fixed bed reactor along with the investigation of some physical properties such as calorific value, density, viscosity, water content, oxygen content, flash point, fire point, pH value etc. The effect of temperature on pyrolysis products, i.e., liquid and char was analysed and variations of viscosity and density of the bio-oil at five different temperatures were investigated. The maximum kinematic viscosity and density were found to be 12.15 cSt and 1009.3 kg/m$^3$, respectively. The particle size of biomass sample used was around 2-2.8 mm. Before pyrolysis, proximate and ultimate analyses of the feedstock were performed and the calorific value of the biomass was found to be 13.2 MJ/kg, whereas the calorific value of the pyrolysis oil was found to be 15.2 MJ/kg. The chemical composition of the oil sample was also investigated with the help of Gas Chromatography & Mass Spectroscopy (GC-MS) and Fourier Transform Infrared (FTIR) spectroscopy techniques.

**Keywords**: Areca tree; biomass; GC-MS; pyrolysis; thermogravimetric analysis.

## INTRODUCTION

As the world is facing a crisis with fossil fuel, due to day to day increase in its consumption, on the other hand due to the increasing use of fossil fuel, the pollution created by it has also become a serious problem. In order to overcome these situations, harnessing energy from renewable energy sources has been found as the only alternative. Several techniques or processes are available in existing literature for production of bio-fuel or bio-oil from biomass, out of which pyrolysis can be considered as one of the oldest and economical way (Bridgwater, 1999). In the process of pyrolysis, the biomass is heated up to around 400-700 $^0$C inside a reactor, in an oxygen free environment, where various chemical reactions take place and as a result it is converted into three main products viz. liquid, incondensable gas and char. All these products of pyrolysis have been found to have better fuel properties compared to the biomass, especially in terms of calorific value (Yin *et al*., 2013). The liquid yield from the pyrolysis of biomass is known as pyrolysis oil or bio-oil. The bio-oil has certain advantages over solid biomass, as the fuel in liquid form is easier to store and transport. Moreover, the conversion of biomass into liquid form can reduce pollution and cost of waste disposal. However, pyrolysis oil is found somewhat not suitable for direct use as a transportation fuel, because of poor quality of properties, such as water content, oxygen content, viscosity, acidity etc., which are comparatively higher than in petroleum fuel. On the other hand, the calorific value of bio-oil is lower than that of petroleum fuel. Therefore, upgrading of these properties is essential, before it is used as transportation or engine oil. Lot of research information on upgrading pyrolysis oil produced from biomass are available in literature. For example, Bridgwater (2004) and Qing *et al.* (2008) found that addition of methanol or acetone can significantly reduce viscosity of bio-oil. It was also found that due to presence of a large amount of water and oxygen content, the calorific value of bio-oil is reduced to around 45% of that of fossil fuel (Bertero *et al.,* 2012). By applying hot filter, the water content in the bio-oil was found to increase to some extent; however, the kinematic viscosity was found to decrease (Pattiya & Suttibak, 2012). Since oxygen is also an undesirable element in bio-oil, as it reduces the calorific value, Frensh & Czernik (2010) added zeolite as catalyst in the biomass and found that the oxygen content in the bio-oil is reduced, as it is converted into hydrocarbons. Qi *et al.* (2007) reported various techniques in their review work for upgrading quality of bio-oil, e.g., hydro-deoxygenetion, emulsification, steam reforming etc.

The properties and characteristics of bio-oil depend on many factors such as the size of biomass particles, pyrolysis temperature, heating rate, vapour residence time, condensing temperature, type of biomass, moisture content in biomass, use of catalysts, sweeping gas, type of reactor etc., (Akhtar & Amin, 2012; Demiral & Sensoz, 2008; Jahirul *et al.,* 2002). The existing publications show that various types of biomasses such as wood, agricultural residues, organic wastes, aquatic plants etc., are used as feedstock for production of pyrolysis oil (Xiu & Shahbazi, 2012). Different types of reactors such as fixed bed reactors, fluidised-bed reactors, ablative reactors, auger reactors, vacuum pyrolysis reactors, rotating cone reactors, pyros reactors, plasma reactors, microwave reactors and solar reactors are used to produce pyrolysis oil (Jahirul *et al.,* 2002). However, fixed bed reactors are found to be simple and reliable and can produce same quality bio-oil as other type of reactors (Chopra & Jain, 2007; Aysu & Küçük, 2014). In this study, areca tree, a locally available biomass, is taken as feedstock for the production of pyrolysis oil using a fixed bed reactor.

## EXPERIMENTAL WORK

### Preparation of biomass sample

The biomass sample, i.e., areca tree dust, was collected from the southwest part of Assam (India), and ground in a grinding machine (BAJAJ PLATINI, Model: P x 7

MIXER GRINDER). A mechanical sieve shaker (IKON$^{TM}$ instrument) was used along with standard test sieve (SETHI, as per IS: 460) 2 mm, 2.36 mm and 2.8 mm to prepare the feedstock accordingly. Before feeding in the reactor, the sample was dried in sunlight for 24 hours to reduce moisture content.

## Proximate analysis

For proximate analysis of the biomass sample, ASTM D-271-48 standard was used to determine ash content, volatile matter, and fixed carbon. The moisture content was also determined, as received.

## Elemental analysis

Since the quality and yield of the bio-oil depends on characteristics and composition of the biomass feedstock, some preliminary analysis of the biomass sample was done, the details of which are given in Table 3. Elemental analysis of the biomass sample was carried out in a CHN analyser (Perkin Elmer, series- ii) and the heating values for both the feedstock and bio-oil were determined using the Dulong formula based on the elemental analysis, where the sulphur content was considered as negligible (Ozbay *et al.,* 2008).

## Thermogravimetric analysis

Thermogravimetric analysis (TGA) shows the degradation and volatilisation pattern of the biomass sample while heating. The experiment was carried out by using a quantity of 2.54 mg of biomass sample in a TGA analyser (SHIMADZU, TGA-50) in nitrogen atmosphere. The flow rate of $N_2$ was 30 ml/min, at a heating rate of 10 $^0$C/min.

## Production of pyrolysis oil

The production of pyrolysis oil was carried out in a lab scale pyrolysis plant. A schematic diagram of the plant is shown in Figure 2. The main components of the plant are following.- (i) A fixed bed reactor shown in Figure 1; the main body of the reactor is cylindrical and its head is conical in shape. The dimensions of the cylindrical body part are as follows-: inside diameter 88 mm, outside diameter 100 mm, cylindrical height is 150 mm, height of the conical head is 100 mm; at the top of the head there is a small opening having diameter of 27 mm to feed the biomass sample inside the reactor and the whole body of the reactor is made of stainless steel. (ii) A clamp type tubular furnace made of ceramics and stainless steel, has been used, which wraps the reactor so that the biomass gets uniform heating. (iii) A glass condenser for condensing the vapor into liquid. (iv) A $N_2$ gas cylinder to create flow of $N_2$ gas throughout the pipe connecting the reactor and condenser, and (v) a temperature controller to regulate and monitor the temperature in the reactor.

**Fig. 1.** The fixed bed reactor

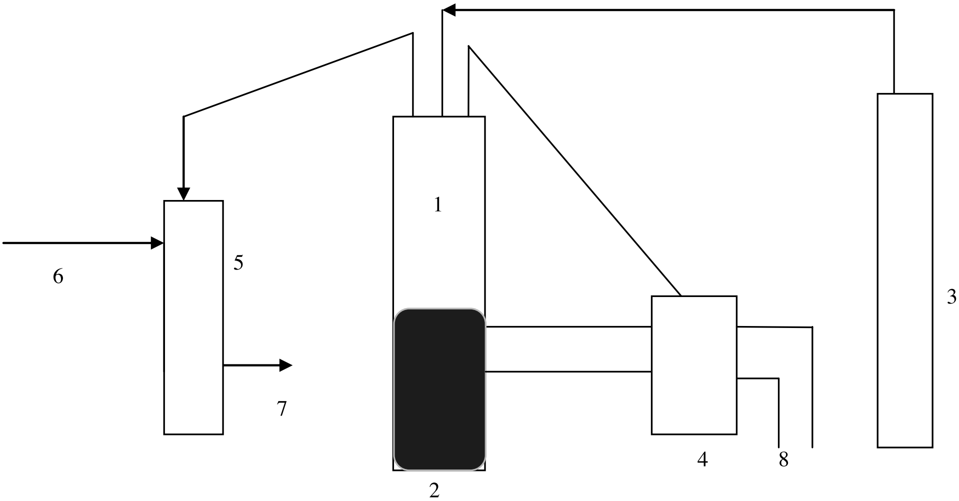

**Fig. 2.** Block diagram of the experimental device

1: Reactor, 2: Tube furnace, 3: $N_2$ cylinder, 4: Temperature controller, 5: Glass condenser, 6: Cold water inlet, 7: Hot water out let, 8: To power supply.

Areca tree dust was used as feedstock for bio-oil production and placed gently in the reactor, using different amounts at different runs. Heating in the reactor was applied mainly by the help of a tube furnace that wraps the reactor. The average heating rate monitored was about 50 $^0$C/min, in order to have the temperature reach up to 400-700 $^0$C. During heating, nitrogen gas was supplied intermittently at a flow rate of 100 ml/m$^3$, to enhance the flow of vapor produced inside the reactor. This vapor was then allowed to pass through a condenser, where tap water was flowing at a temperature of 15 $^0$C; the condensed liquid was then collected in a container. The experiment was carried out for a period of 1 hr to 1.5 hr for each run to obtain the complete liquid product. The average amount of liquid yield was found to be around 35 mass % and the color of the liquid was found to be dark brown.

### Kinematic viscosity and density

The kinematic viscosity and density are two major properties of bio-oil, which determine the flow characteristics of the fuel. Both the properties were measured with the help of a digital instrument (LEMIS Instrument, VISCODENS, VDM 300) at various temperatures.

### Water content

Water content, which determines the calorific value and flow characteristics, is one of the major components of any bio-oil. The presence of higher amount of water content reduces the heating value of the bio-oil. For determining the water content, a Dean and Stark Apparatus (as per IP specification) was used. For complete separation of water, the experiment was run for around 24 hours.

### pH Value

The pH value determines the acidic nature of the fuel. The highly acidic oil can create wear and tear in the apparatus and container. It was determined by the help of a pH probe with digital meter (SYSTRONICS, Digital pH Meter 802).

### Flash point and fire point

In order to identify the ignition behaviour of the liquid, flash point and fire point are very important properties, which were measured by the help of an open cup digital tester (REICO EQUIPMENT AND INSTRUMENT PVT. LTD, model RFFP).

### GC-MS and FTIR

For evaluating and identifying the chemical composition of pyrolysis oil, a coupled GC-MS instrument (Perkin Elmer, GC-Clarus 600, MS-Clarus 600 C) was used. The analysis parameter of GC-MS is shown in Table 1. The identification of the peaks is based on the mass spectra with the NIST 2004 library.

FTIR analysis was carried out using a FTIR instrument (NICOLET, Impact 410) with a resolution of 4 cm$^{-1}$.

**Table 1.** Analysis parameter of GC-MS

| Parameter | Specification |
|---|---|
| GC Line temperature | 250 $^0$C |
| Source temperature | 150 $^0$C |
| Oven temperature | 120-270 $^0$C, 20 $^0$C/min |
| Carrier gas | Helium |
| Carrier gas flow rate | 1 ml/min |
| Split ratio | 50:1 |
| Acquisition mode | Threshold |
| Capillary column | 30 m x 0.25 mm, and 0.25µm film thickness (Elite-5 MS) |
| Electron energy | 70 eV |

## RESULTS AND DISCUSSIONS

### Proximate and elemental analysis of biomass

Information obtained from proximate and elemental analyses of the biomass sample is shown in Table 2 and Table 3.

**Table 2**. Proximate analysis of the biomass (wet basis)

| Moisture (mass %) | Ash (mass %) | Volatile matter (mass %) | Fixed carbon (mass %) |
|---|---|---|---|
| 13.13 | 20.38 | 47.7 | 18.79 |

**Table 3.** Elemental analysis of the biomass

| | |
|---|---|
| Carbon (mass %) | 40.93 |
| Hydrogen (mass %) | 5.96 |
| Nitrogen (mass %) | 0 |
| Oxygen by difference (mass %) | 53.11 |
| Calorific value (MJ/kg) | 13.2 |

In most of the biomasses such as rice straw, bamboo sawdust, rapeseed cake etc., the gross calorific value lies between 16 and 24.5 MJ/kg (Jung *et al.,* 2008; Sundaram & Natarajan, 2009; Culcouglu *et al*., 2005). However, in areca tree dust, because of high water and oxygen content, the calorific value was found to be quite less. No nitrogen content was found in the biomass sample and sulphur content was considered to be negligible.

## Thermal analysis

The degradation behaviour of the biomass, i.e. weight loss with increase in temperature was investigated by the help of a TGA analyser. The TGA profile as per the experiment is shown in Figure 3. It was observed (Figure 3) that the maximum rate of weight loss of the biomass occurred at temperatures ranging between 254.87 $^0$C and 354.32 $^0$C. Beyond this temperature range, due to volatilisation, the rate of weight loss of biomass becomes insignificant.

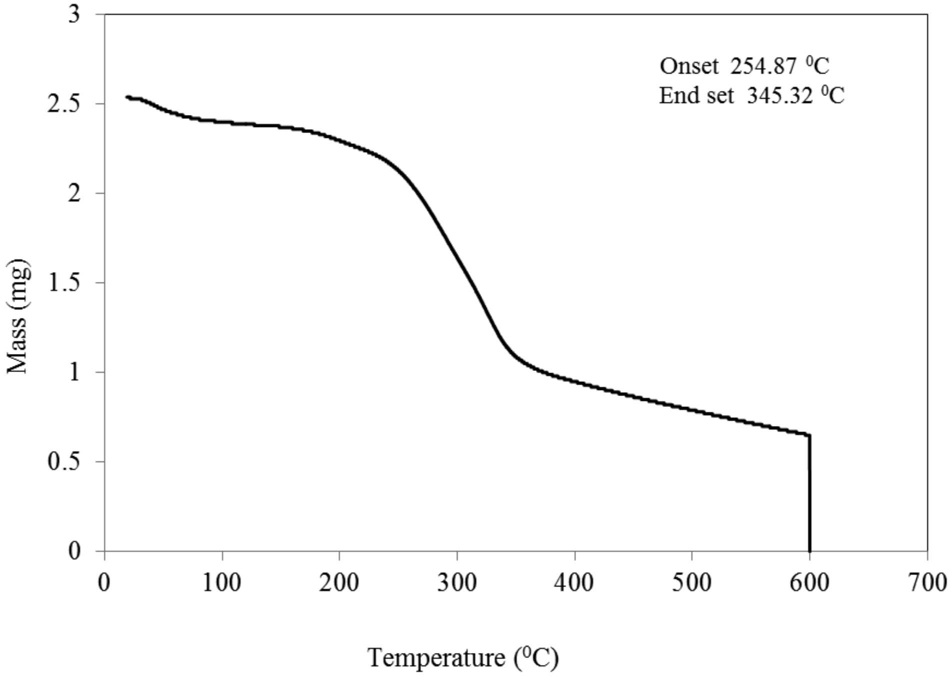

**Fig. 3.** TGA profile of areca tree dust.

## Influence of temperature

The variations in yield of pyrolysis liquid and char with temperature can be seen from the curves shown in Figure 4. The liquid yield was found to be increasing linearly with increase in temperature up to 700 $^0$C, due to increase in conversion of biomass into condensable gas, when it reached the maximum (37%); afterwards, the liquid yield started decreasing. This could be due to secondary cracking of the biomass particles, leading to reduction in liquid yield.

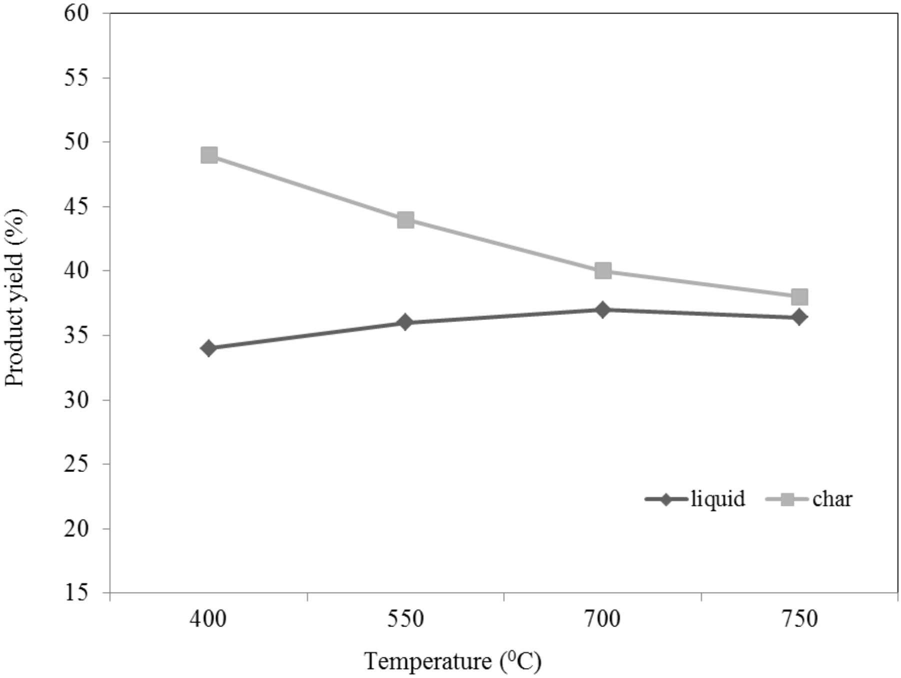

**Fig. 4.** Variation of pyrolysis product yield with temperature

## Influence of feedstock amount

Few experiments were carried out using different amounts of feedstock, arbitrarily in an increment of 5 g in each run, at 700 $^0$C and the respective bio-oil and char yields were recorded as shown in Table 4. It has been observed that the rate of increase of liquid yield with increase of feedstock amount was less, as compared to the rate of increase of char yield. It is possibly due to the larger particle size of the biomass used. In a fixed bed reactor with medium feed size of 0.6-0.85 mm, rapeseed gives maximum oil yield. Feed size below or above this range gives lower yield of bio-oil and as the amount of feed stock increases, it gradually starts resisting uniform heat transfer to all the particles and hence a fraction of the sample remains in the form of char, without converting to condensable gas (Onay & Mete Koc-kar, 2004). However, the oil yield depends upon the type of biomasses and therefore, specific data about particle size of feedstock for different biomasses is not available. Moreover, reducing the size of biomass particles also lead to increasing the cost of grinding (Akhtar & Amin, 2012). Park *et al.* (2014) used a particle size of 1-3 mm in production of pyrolysis oil from wild reed and found an appreciable amount of oil yield (around 50 mass%). It can be noted that yield of pyrolysis also depend on some other factors such as pyrolysis temperature, condensing temperature, rate of heating and flow of sweeping gas.

**Table 4**.Yield of bio-oil and char produced at different pyrolysis temperatures.

| Run | Biomass (g) | Bio-oil yield (g) | Char (g) | Incondensable gas, by difference (g) |
|---|---|---|---|---|
| 1 | 60 | 22 | 24.5 | 13.5 |
| 2 | 65 | 24.2 | 26.1 | 14.7 |
| 3 | 70 | 25.7 | 28 | 16.3 |
| 4 | 75 | 26.5 | 31 | 17.5 |
| 5 | 80 | 27.4 | 34.6 | 18 |
| 6 | 85 | 28 | 37.5 | 19.5 |

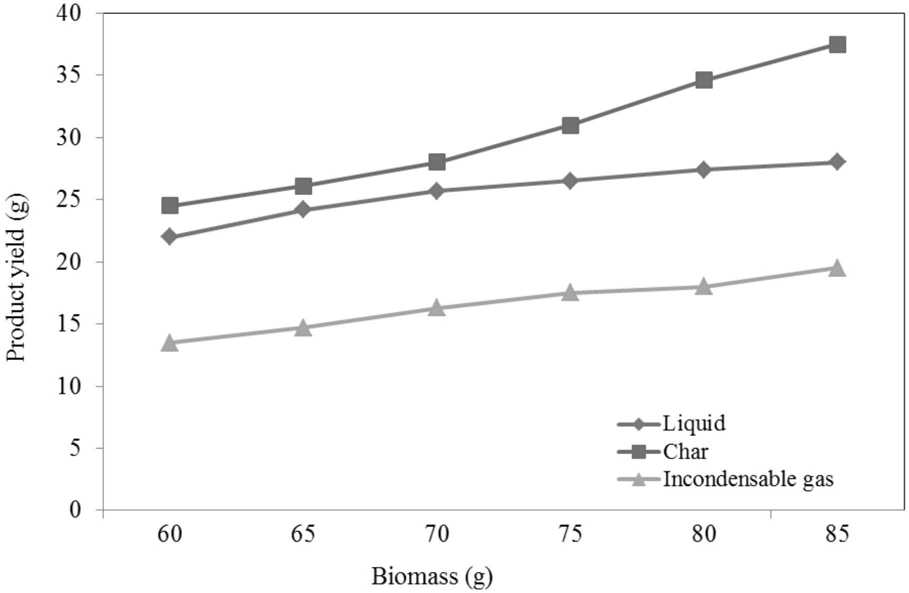

**Fig. 5.** Variation of liquid and char yield

## Variation of kinematic viscosity and density of the liquid

Kinematic viscosity and density are some other important properties of the liquid yield produced during the pyrolysis process, which play a major role in the quality of pyrolysis oil, such as atomization and combustion, on which the design and operation of fuel injection system depends (Qiang *et al.,* 2009).

The kinematic viscosity of the pyrolysis oil produced from areca tree dust was found to be 12.15 cSt at a temperature of 40 $^0$C, which is quite lower than the kinematic viscosity of many other bio-oils measured at 40 $^0$C, such as kinematic viscosity of bio-oil produced from sugarcane bagasse, where it was reported to be 26.7 cSt (Garcìa-Pèrez *et al*., 2002). Boucher *et al.* (2000) found the kinematic viscosity of pyrolysis oil from soft wood to be 38 cSt at 40 $^0$C, and Qiang *et al.* (2008) observed that the kinematic viscosity of bio-oil at 40 $^0$C varied from 13-25 cSt at different storage time. Again, Qiang *et al.* (2009) has mentioned in the review study that kinematic viscosity of the bio-oil varies in a wide range of 10-100 cP at 40 $^0$C.

The density of the bio-oil has been measured at various temperatures and these values are plotted against the temperatures as shown in Figure 6. The density of the bio-oil was found to be normally as high as 1300 kg/m$^3$ (Feng *et al.* 2006), and as low as 964 kg/m$^3$ (Culcuoglu *et al*. 2005). However, for the areca tree dust pyrolysis oil, the density was recorded as 1009.3 kg/m$^3$at 30 $^0$C, which gradually decreased to around 987 kg/m$^3$ as the temperature reached 60 $^0$C. The density of the bio-oil from different residual sources: pine wood, mesquite and wheat shell was found as 1060-1070 kg/m$^3$ (Bertero *et al.* 2012) and these values can be compared with the density found in the present work.

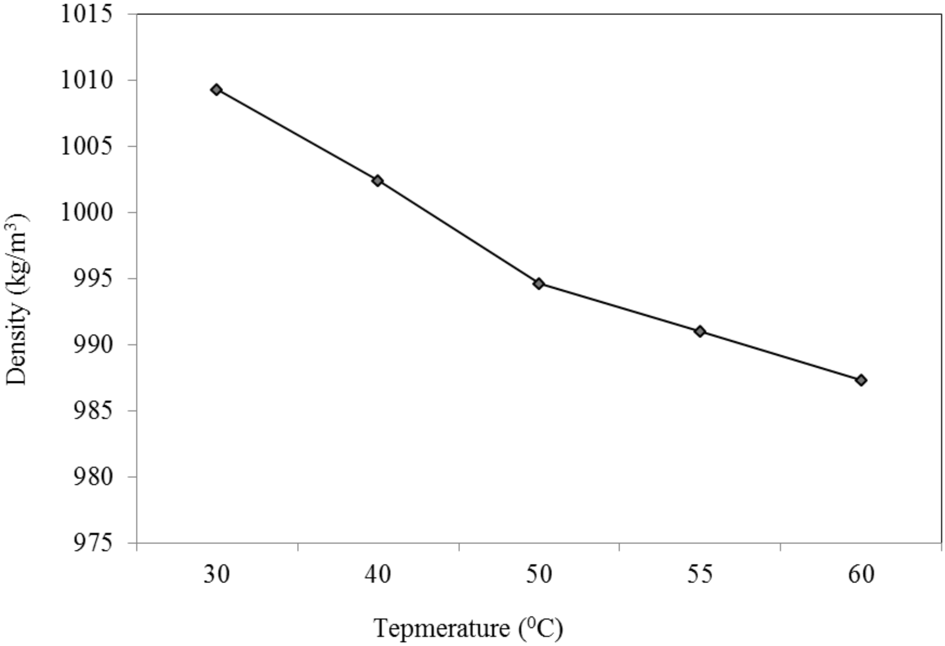

**Fig. 6.** Variation of density of areca tree dust pyrolytic oil with temperature.

## Some physical properties of the pyrolysis oil

Results of the ultimate analysis of physical properties of the pyrolysis oil obtained from areca tree dust are listed in Table 5. It can be observed from the data reported in Table 5 that the calorific value of the liquid is lower due to the higher amounts of oxygen and water contents in the bio-oil. The pH value of the liquid shows that the bio-oil is acidic in nature, which is similar to other pyrolysis oil with pH value 2-4 (Luo *et al.,* 2004) derived from three wood feedstocks: *Pterocarpus indicus, Cunninghamia lanceolata and Fraxinus mandshurica*.

The flash point and fire point of the pyrolysis oil obtained from the areca tree dust, as shown in Table 5, can be compared with some other bio-oil, as in most of the bio-oils, the flash point is found to be within 40-70 $^0$C, and sometimes above 100 $^0$C (Qiang *et al*. 2009).

**Table 5.** Some physical properties of the bio-oil

| Elements | Amount/value |
|---|---|
| Carbon (mass %) | 25.84 |
| Hydrogen (mass %) | 12.25 |
| Nitrogen (mass %) | 0 |
| Oxygen by difference (mass %) | 61.91 |
| Calorific value (MJ/kg) | 15.2 |
| pH value | 2.3 |
| Flash point ($^0$C) | 58 |
| Fire point ($^0$C) | 63 |
| Water content (mass %) | 32 |

## GC-MS and FTIR analysis

The GC-MS analysis was applied to identify the main products and their chemical structure present in the pyrolysis oil. Many small peaks are observed in the GC/MS chromatogram (Figure 7), out of which main compounds separated are listed in Table 6, along with their mass percentages.

The shape, position and intensity of the peak in the FTIR spectrum reveal the functional group and structure of the compounds present in the liquid sample. The IR spectrum of the pyrolysis oil is shown in the Figure 8. Presence of –OH group is detected at 3418.65 cm$^{-1}$ within the range of 3600–3200 cm$^{-1}$. Phenol is identified at 1388.60 cm$^{-1}$ with C–O stretching vibration. Primary and secondary alcohols are identified by the peaks within 1350–1260 cm$^{-1}$ with C–O stretching vibration; again tertiary alcohol is detected by the peak within 1400–1300 cm$^{-1}$ with C–O stretching vibration. At 1712.58 cm$^{-1}$with C=O stretching vibration indicates the presence of ketone. Presence of aldehyde can be identified at 1638.69 cm$^{-1}$ with C–H stretching vibration.

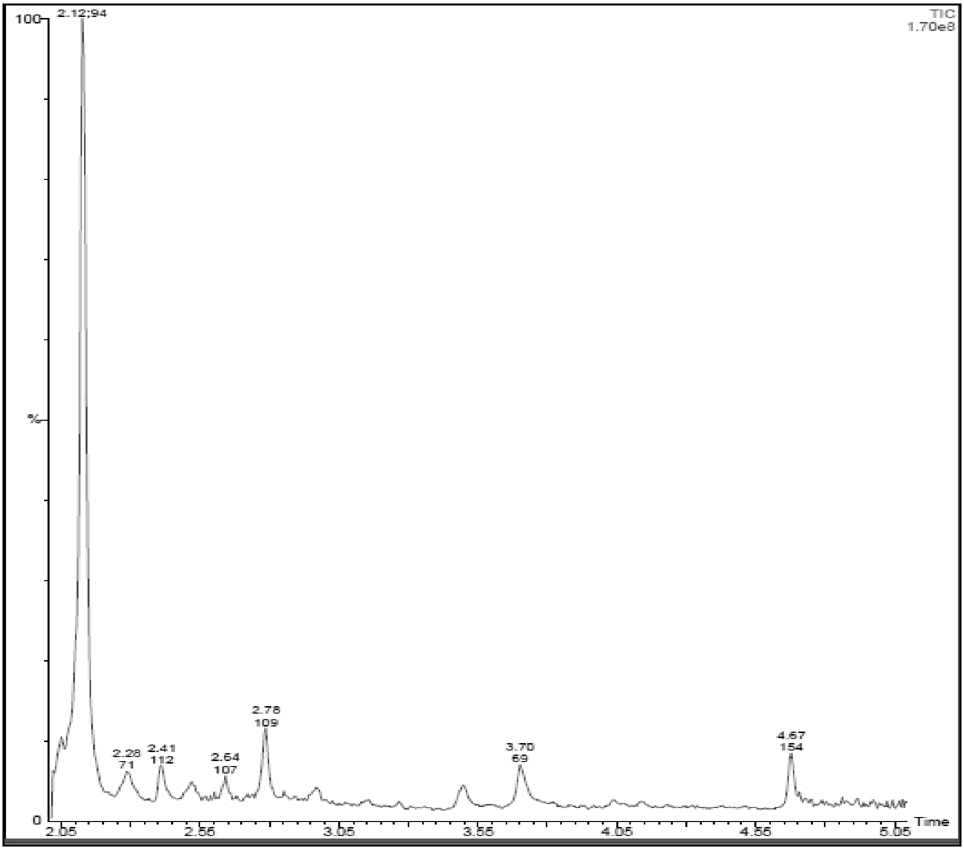

**Fig. 7.** GC-MS profile of areca tree dust pyrolysis liquid

**Table 6.** Compounds of areca tree dust pyrolysis liquid in GC/MS analysis

| Sl no | Compound name | Relative mass content (%) | Retention time (min) | Molecular Weight | Formula |
|---|---|---|---|---|---|
| 1. | Phosphonic acid (P-hydroxyphenyl) | 71.6 | 2.213 | 174 | $C_6H_7O_4P$ |
| 2. | Phenol, 2-methoxy | 5.99 | 2.784 | 124 | $C_7H_8O_2$ |
| 3. | Di(1-methylcyclobutyl) ether | 5.9 | 3.699 | 154 | $C_{10}H_{18}O$ |
| 4. | Phenol, 2,6-dimethoxy | 4.1 | 4.674 | 154 | $C_8H_{10}O_3$ |
| 5. | 4-methoxy-6-methyl-6,7-dihydro-4H-furo(3,2-C) pyran | 3.2 | 2.289 | 168 | $C_9H_{12}0_3$ |
| 6. | 2,7-dihydroxy-2,4,6-cycloheptatrien-1-one | 2.9 | 3.494 | 138 | $C_7H_6O_3$ |
| 7. | 2-ethyl-5-propylcyclopentanone | 2.5 | 2.409 | 154 | $C_{10}H_{18}O$ |
| 8. | 2-cyclopenten-1-one,3-ethyl-2-hydroxy | 2.3 | 2.96 | 126 | $C_7H_{10}O_2$ |
| 9. | Phenol,4-methyl | 1.6 | 2.639 | 108 | $C_7H_8O$ |

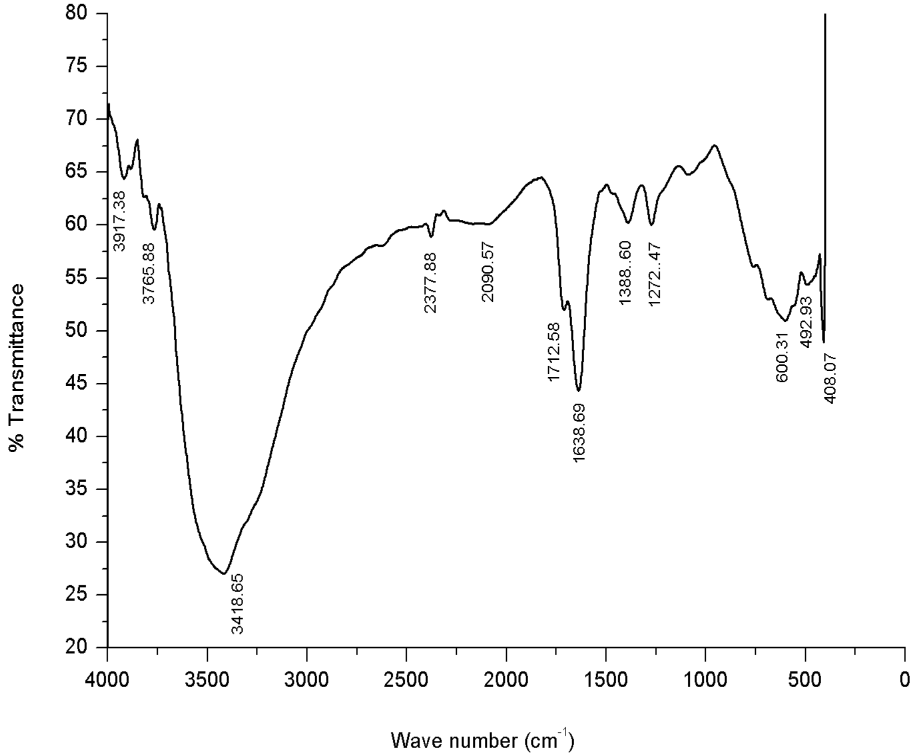

**Fig. 8.** FTIR of areca tree dust pyrolysis liquid

## CONCLUSIONS

Oil was produced by pyrolysis from areca tree using a lab scale fixed bed reactor. The yields of pyrolysis products were found to be influenced by temperature. The liquid yield increased with increase in temperature and reached the maximum at 700 $^0$C and then it started decreasing slowly; whereas the char yield was found decreasing continuously with increase in temperature. The amount of feedstock also affects the product yield, as both liquid and char yields increase as the feedstock amount was increased. However, the rate of increase of liquid yield is lower than the char yield. The bio-oil was found to have maximum viscosity at 40 $^0$C, which gradually decreased with increase in temperature. The density of the bio-oil was also found to be decreasing with increase in temperature. The calorific value of the pyrolysis oil was found to be lower, when compared to petroleum fuel; possibly due to higher water and oxygen content. The pyrolysis oil consists of several complex chemical compounds such as phenol, phosphonic acid, ether, 2-ethyl-5-propylcyclopentanone, 4-methoxy-6-methyl-6, 7-dihydro-4H-furo (3,2-C) pyran etc. The presence of these compounds and different functional groups in these compounds were detected at a corresponding range of wave numbers by the help of FTIR analysis. From the analysis of properties of pyrolysis oil derived from areca tree, it can be concluded that this will be suitable for use in engines and as transportation fuel, after upgrading.

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