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Determination of Optimum Condition for the Production of Rice Husk-Derived Bio-oil by Slow Pyrolysis Process

Suzana Yusup, Chung Loong Yiin, Chiang Jinn Tan, and Bawadi Abdullah

Department of Chemical Engineering, Biomass Processing Laboratory, Center of Biofuel and Biochemical, Green Technology (MOR), Universiti Teknologi PETRONAS, Tronoh, Malaysia

13.1 Introduction

The depletion of fossil fuels along with the rising demand of energy from fossil fuels has become a threat to countries which are still relying on petroleum to generate electricity and transportation fuel. Recently, studies on bio-oil have captured international interest in developing it as an alternative energy option. Every year, a large number of biomass residues are left behind after a massive number of agricultural crops have been harvested. Disposing biomass residues by open burning could contribute to global warming and environmental pollution; hence, utilizing biomass residues as raw materials to generate sustainable and renewable energy for electricity and transportation fuels offers a better alternative. Undoubtedly, sustainable and renewable energy options generated from agricultural and biomass residues have high potential to become a substitute to petroleum fuels.

Biomass can generally be defined as any hydrocarbon material which mainly consists of carbon, hydrogen, oxygen, and nitrogen, with presence of sulfur in lesser proportions. Biomass is an abundant carbon-neutral renewable resource for the production of bioenergy

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and biomaterials (Ragauskas et al., 2006). There are several methods to convert biomass into energy. Biochemical and thermochemical conversion methods can be applied to biomass to make use of its energy potential for a sustainable renewable energy option (Yaman, 2004). Biochemical conversion methods involve converting biomass into alcohols or oxygenated products by biological activity. Besides that, thermochemical conversion methods such as pyrolysis, liquefaction, gasification, and supercritical fluid extraction methods can be applied by converting biomass into energy.

Among all the thermochemical conversion methods, pyrolysis has attracted the most attention (Domínguez & Menéndez, 2006). Pyrolysis is one of the thermochemical processes that convert the solid biomass into liquid (bio-oil), gas, and char in the absence of oxygen (Salleh et al., 2011). Biomass pyrolysis converts 80–95% of the feed material to gases and bio-oil. The pyrolysis of biomass is a complex process due to differences in the chemical composition, particle structure within the biomass material, and operating condition. Pyrolysis process needs to be optimized to maximize the production of liquids (tar and bio-oil) (Natarajan & Ganapathy, 2009). Bio-oil which is also known as pyrolysis oil or bio-fuel oil was produced by pyrolysis process without any additional oxygen. Bio-oil is a complex organic mixture with high oxygen content, containing more or less solid carbon particles, and its color is usually dark brown or black, having a strong pungent smell (Xiu et al., 2012). The pyrolysis bio-oil is a complex mixture of oxygenated aliphatic and aromatic compounds (Meier & Faix, 1999).

Pyrolysis bio-oil consists of hundreds of specific organic groups which include organic acids, esters, alcohols, ketones, aldehydes, phenols, aromatic hydrocarbons, nitrogen compounds, furans, and furodrosugars (Abu Bakar & James, 2012). Bio-oil has a high potential to be the substitute for fossil fuels as it can produce heat, electricity, and chemicals. However, raw bio-oil that has not been upgraded has several undesirable characteristics such as having high water and oxygen content, high viscosity, and low calorific value and being corrosive, unstable when under storage and heating conditions, and immiscible with petroleum fuels (Iliopoulou et al., 2012). The main reason for the instability of bio-oil is the presence of oxygenated compounds in the bio-oil. They tend to further react during storage or at high-temperature conditions (Gopakumar et al., 2011). As a result of the instability, bio-oil aging occurs, causing the viscosity of the bio-oil to increase. This instability can actually be minimized or decreased through either the stabilization process after pyrolysis through upgrading methods such as emulsification with diesel oil (Jiang & Ellis, 2010) or addition of antioxidants and solvents (Wanasundara & Shahidi, 2005; Udomsap et al., 2011). The characteristics of bio-oil such as density, pH, viscosity, and heating value should be monitored. The resulting characteristics are considered as desirable for bio-oil if they increase its potential to replace fossil fuels. For instance, the bio-oil should have comparable properties with petroleum fuel such as low moisture content, viscosity, and acidity as well as high heating value as shown in Table 13.1.

Among various biomass species, rice husk is a potential source of energy and a value-added by-product of the rice milling industry (Sharma & Rao, 1999). In recent years, utilization of agriculture waste and residues (for instance, rice husk) for energy generation has gained a lot of attention. Rice is one of the major food crops in the world. The production of rice generates a great amount of waste in the world, namely, rice husk. Rice husk is the outer covering of the paddy and accounts for 20–25% of its weight (Jenkins, 1989) which stood at 672 million tons in the year 2010 (GeoHive, 2011). Rice husk is produced in the first step of the milling process

Table 13.1 *Properties of petroleum fuel*

Properties	Value
Elemental composition (wt%)	85.00
Carbon	11.00
Hydrogen	1.00
Oxygen	0.30
Nitrogen	
Moisture content (wt%)	0.10
Heating value (MJ/kg)	40.00
Viscosity at 40°C (cP)	18.00

when the husk is removed from the grain in the husking stage of the rice mill. Rice husk usually ends up being burned in open air, therefore causing environmental pollution and wastes of various biomass resources (Ezzat et al., 2012).

Generally, rice husk is a cellulosic material, consisting of about 20–35% cellulose, 15–30% hemicelluloses, 5–10% lignin, and some materials. Rice husk is a renewable source of energy, with a high calorific value of about 4260 kcal/kg on dry basis, and is characterized by low bulk density and high ash content (18–22% by weight). The large amount of ash generated during combustion has to be continuously removed for a smooth operation of the system. Rice husk may be treated by pyrolysis methods to obtain fuels as discussed in previous papers (Islam & Ani, 2000; Williams & Nugranad, 2000). Biomass pyrolysis essentially converts 80–95% of the feed material to gases and bio-oil. The pyrolysis process converts biomass into high-energy-content biofuels, in the absence of oxygen/air, leading to the formation of solid (charcoal), liquid (tar and other organics), and gaseous products.

Pyrolysis can be divided into two types: fast pyrolysis and slow pyrolysis. These reactions are different mainly in terms of heating rates and maximum reaction temperatures (Brown et al., 2011). Heating rates for slow pyrolysis are typically below 100 K/min, whereas fast pyrolysis can achieve heating rates exceeding 1000 K/min. Reaction temperatures are about 300°C and 500°C for slow and fast pyrolysis, respectively. Slow pyrolysis requires several minutes or even hours, while fast pyrolysis is completed within 2 s. This difference in time results in dramatic differences in product distributions.

In this study, an attempt was given to produce bio-oil through slow pyrolysis of rice husk (RH) at different heating rates in order to determine the optimum reaction condition that will give maximum liquid yield. The characteristics of bio-oil produced at different heating rates are then analyzed. Apart from that, the properties of bio-oil that was produced at optimum operating condition are compared with those in literature.

13.2 Experimental Study

13.2.1 Biomass Preparation and Characterization

Raw rice husks were taken from local suppliers. The rice husk is then ground to smaller particle sizes using FRITSCH Cutting Mill. After the grinding process, the rice husk is reduced into the desired feedstock size within the range of 0.25–0.50 mm. The ground rice husk is sieved using CISA BA 500 N sieve shaker to obtain the desired feedstock size of 0.25–0.50 mm. Sieved rice husk is then dried in the oven at 100°C to remove the moisture content. Ultimate analysis of the RH is carried out using LECO 932 CHNS Analyzer and the higher heating value (HHV) is measured using IKA C5000 bomb calorimeter.

13.2.2 Experimental Procedure

The rice husk sample is placed in a borosilicate tube. Approximately 0.6 g of glass wool is placed above the rice husk sample to avoid the rice husk from being disseminated from the tube during pyrolysis reaction inside the furnace. The experiment is set up and nitrogen gas is purged for 5 min at a rate of 500 ml/min before the experiment. Based on the literature review, the pyrolysis temperature is set at 500°C and the nitrogen gas flow rate is at 100 ml/min in order to have the optimum production of bio-oil (Sukiran et al., 2009). The temperature of the heater and thermocouple is recorded every 2 min. The time at which the first drop of bio-oil is formed is recorded. When the temperature of the heater and thermocouple reaches a stable condition, the experiment is stopped. The reactor is left to cool down. The condenser is weighed to calculate the liquid yield. The bio-oil is collected for characterization analysis. A total of 16 runs were carried out at four different heating rates with four runs at 5°C/min, 10°C/min, 15°C/min, and 20°C/min, respectively. The liquid yield is calculated using Equation 13.1:

$$\text{Yield}_{\text{bio-oil}} = \frac{m_{\text{bio-oil}}}{m_{\text{biomass}}} \times 100\% \tag{13.1}$$

Meanwhile, the overall conversion of biomass into gas and oil products can be determined by using Equation 13.2:

$$\text{Conversion}\,\% = \frac{\text{decrease in weight of reaction mixture}}{m_{\text{biomass}}} \times 100\% \tag{13.2}$$

Apart from that, the gas yield and residue yield can be calculated using Equation 13.3 and Equation 13.4, respectively:

$$\text{Yield}_{\text{gas}} = \text{conversion} - \text{yield}_{\text{bio-oil}} \tag{13.3}$$

$$\text{Yield}_{\text{residue}} = 100\% - \text{conversion} \tag{13.4}$$

13.2.3 Equipment

The reactor used in this study is a semibatch reactor as shown in Figure 13.1.

In every run, about 15 g of dried RH is placed into the borosilicate glass tube and heated in a vertical furnace. Before the reaction starts, nitrogen gas is allowed to flow through the tube for a few minutes to drive out all the oxygen. The desired temperature of the reaction, heating rate of 20°C/min, and the flow rate of nitrogen gas are set. The pyrolysis vapor is carried out by the nitrogen gas and passes through an ice bath condenser, where the condensable vapor will condense to form bio-oil.

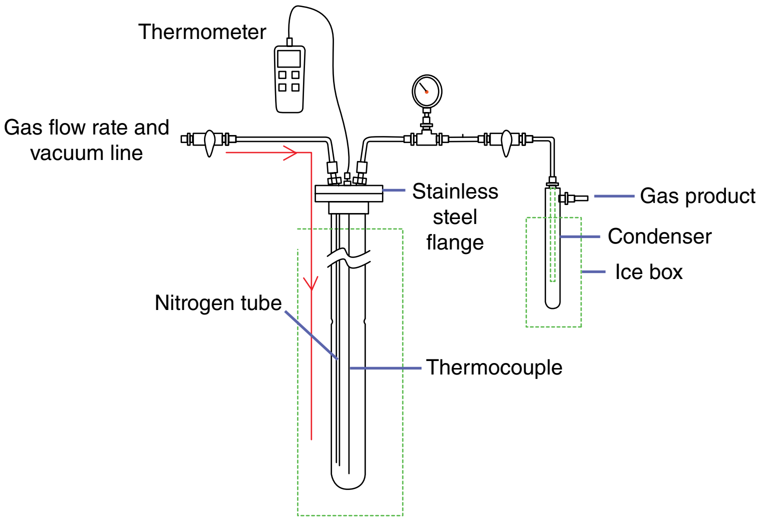

Figure 13.1 *Semibatch reactor*

13.2.4 Characterization of Bio-oil

The bio-oil produced at different heating rates is characterized. The physical properties of bio-oil such as density, pH, viscosity, and heating value are measured using Anton Paar DMA 4500 M density meter, EUTECH pH 510 pH/mV/°C meter, Brookfield CAP 2000+ viscometer, and IKA C5000 bomb calorimeter, respectively. Ultimate analysis using LECO CHNS Analyzer 932 is carried out to determine the elemental content of bio-oil. Besides that, field emission scanning electron microscope (FESEM) is used to study the morphology of raw RH and residue after pyrolysis. The chemical composition of the bio-oil produced is tested using Agilent GC/MS 5975 C.

13.3 Results and Discussion

13.3.1 Characterization of RH

The properties of raw RH sample are summarized in Table 13.2.

13.3.2 Characterization of Bio-oil

Table 13.3 shows the properties of the bio-oil produced at different heating rates. Normally, bio-oil is highly acidic and the pH of bio-oil is reported between the range of 2 and 3. The most acidic pH value obtained is 2.68 at a heating rate of 20°C/min. In this work, the pH value decreased with the increase in heating rates, as shown in Table 13.4. Hence, the bio-oil produced is more acidic at higher heating rates. Most of the acidity may arise from the presence of acetic acid, but other carboxylic acids, phenols, and other acidic compounds will also have a significant contribution. The viscosity of bio-oil produced increased with an increment of the heating rates. The viscosity values of bio-oil with the heating

Table 13.2 *Properties of RH (wt%)*

Properties	Measured value
Ultimate analysis (wt%)	41.34
Carbon	3.56
Hydrogen	0.37
Nitrogen	0.15
Sulfur	54.59
Oxygen (by difference)	
Proximate analysis (wt%)	
Volatiles	77.13
Fixed carbon	2.12
Ash	12.25
Moisture content	8.50
HHV (MJ/kg)	16.72

Table 13.3 *Properties of bio-oil produced at different heating rates*

Properties	Heating rate (°C/min)			
	5	10	15	20
pH	2.83	2.76	2.71	2.68
Viscosity at 40°C (cP)	42.35	44.56	45.21	47.83
Density at 20°C (kg/m^3)	1060.14	1064.85	1073.80	1082.69
Ultimate analysis (wt%)				
Carbon	20.45	22.13	23.87	26.76
Hydrogen	8.53	8.98	9.03	9.22
Nitrogen	0.18	0.22	0.25	0.31
Sulfur	0.27	0.35	0.41	0.44
Oxygen	70.75	68.32	66.44	63.27
HHV (MJ/kg)	9.89	11.26	12.13	13.69

Table 13.4 *Comparison of bio-oil properties with literatures*

Characterization parameters	This work	Abu Bakar and James (2012)	Zheng (2007)	Guo et al. (2011)
pH	2.68	3	2.8	3.36
Viscosity (cP)	47.83	1.79	152.32	82.43
Density (kg/m^3) at 20°C	1082.69	1065	1190	1210
Ultimate analysis (wt%)				
Carbon	26.76	23.38	41.7	35.63
Hydrogen	9.22	10.39	7.7	7.00
Nitrogen	0.31	0.51	0.3	—
Sulfur	0.44	0.09	0.2	—
Oxygen	63.27	65.63	50.3	57.37
HHV (MJ/kg)	13.69	13.61	17.42	13.36

rates of 5°C/min, 10°C/min, 15°C/min, and 20°C/min are 42.35 cP, 44.56 cP, 45.21 cP, and 47.83 cP, respectively. On the other hand, the measured density of bio-oil is increasing as the heating rates are elevated.

The highest density value of 1082.69°kg/m^3 measured at 20°C is obtained at a heating rate of 20°C/min. Higher density value will attribute to an increase in the water content which does not favor the production of bio-oil. Apart from that, it can be seen from Table 13.2 that the carbon, hydrogen, nitrogen, and sulfur contents are increasing as the heating rates are increased. The nitrogen and sulfur contents were still considered low in all of the bio-oils, showing their potential as a clean fuel when used for combustion purposes. The elemental carbon, hydrogen, nitrogen, sulfur, and oxygen contents of bio-oil basically have low carbon content and high oxygen content. High oxygen content is not favored because it will lead to higher instability of bio-oil.

The heating value of bio-oil gives information related to the energy content of bio-oil, which has the potential of being upgraded to transportation fuels. The HHV of bio-oil can be estimated using the following formula with the unit of MJ/kg (Parikh et al., 2005):

$$\mathrm{HHV} = 0.3491\mathrm{C} + 1.1783\mathrm{H} + 0.1005\mathrm{S} - 0.1034\mathrm{O} - 0.0151\mathrm{N} - 0.0211\mathrm{A}$$

where A represents ash; C, carbon; H, hydrogen; N, nitrogen; O, oxygen; and S, sulfur.

The result shows that the estimated HHV for the bio-oil produced is slightly lower compared to the estimated HHV of raw rice husk. The HHV rose with increase in the heating rates. The highest HHV of 13.69 MJ/kg is obtained at slow pyrolysis condition of heating rate at 20°C/min.

As shown in Table 13.4, the pH value and density of bio-oil in this work are comparable with other literatures. In addition, the bio-oil produced has moderate value of viscosity which favored the range of bio-oil application. Based on the ultimate analysis, the oxygen content measured in this work is almost the same in other findings which resulted in proportionate HHV. Hence, the slow pyrolysis bio-oil produced from this work has a high potential to substitute conventional fossil fuel produced from fast pyrolysis process.

13.3.3 Parametric Analysis

Table 13.5 shows the result of conversion, gas yield, and residue yield calculated for 16 runs.

To study the trend of the results, the average values of liquid yield, gas yield, residue yield, and conversion are calculated and plotted as shown in Figure 13.2.

Figure 13.2 shows that the liquid yield and conversion increased with elevation of heating rates. As the heating rate increases, the gas yield is slightly increased. However, the gas yield decreased at a heating rate of 20°C/min due to higher liquid yield and conversion produced. Besides, the residue yield decreases as the heating rates increase. The rate of mass or heat transfer in the complex matrix of biomass at low heating rates allows intraparticle cracking. Extended exposure times resulting from low heating rates favor secondary reactions such as cracking, repolymerization, and recondensation of radical components leading to the formation of char with limited oil and gaseous products (Gerçel, 2004). Thus, maximum liquid yield of 35.38 wt% and highest conversion value of 63.82 wt% are obtained at a heating rate of 20°C/min.

Table 13.5 *Conversion, gas yield, and residue yield*

Run	Heating rate (°C/min)	Decrease in weight of reaction mixture (g)	Conversion (wt%)	Gas yield (wt%)	Residue yield (wt%)
1	5	9.07	60.43	32.78	39.57
2	5	8.84	58.93	31.80	41.07
3	5	8.85	59.00	28.73	41.00
4	5	8.91	59.36	29.31	40.64
5	10	9.21	61.28	30.07	38.72
6	10	9.14	60.81	30.67	39.19
7	10	9.45	62.92	31.96	37.08
8	10	9.15	60.88	30.14	39.12
9	15	9.26	61.65	28.63	38.35
10	15	9.52	63.34	34.07	36.66
11	15	9.33	62.20	29.67	37.80
12	15	9.43	62.74	31.60	37.26
13	20	9.54	63.43	27.39	36.57
14	20	9.49	63.18	28.16	36.82
15	20	9.66	64.36	29.45	35.64
16	20	9.66	64.31	28.76	35.69

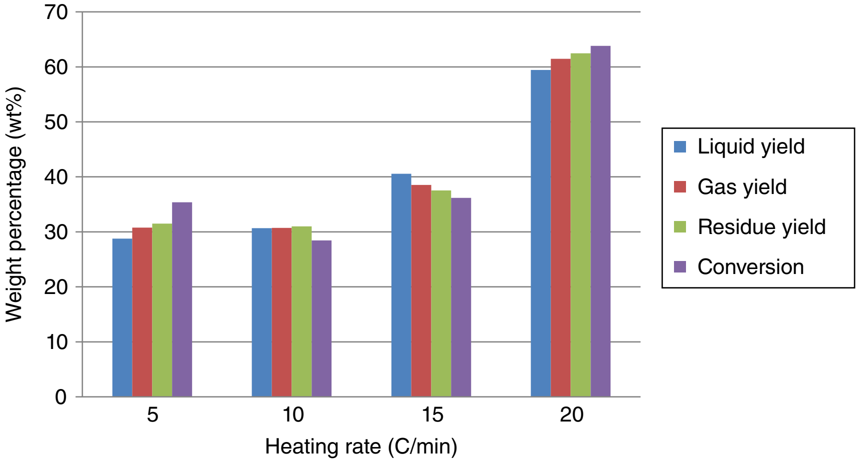

Figure 13.2 *Average values of liquid, gas, residue yield, and conversion*

13.3.4 Field Emission Scanning Electron Microscope

To gain insight into a particle’s structure, the morphologies of raw rice husk and the residue left after slow pyrolysis at a heating rate of 20°C/min are examined by FESEM. FESEM micrographs of raw rice husk are presented in Figure 13.3.

FESEM micrographs of residue after slow pyrolysis in nitrogen atmosphere at a heating rate of 20°C/min are presented in Figure 13.4.

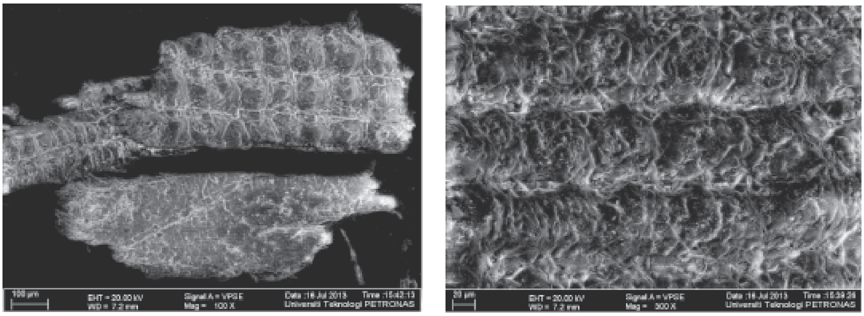

Figure 13.3 *Field emission scanning electron microscope of raw rice husk*

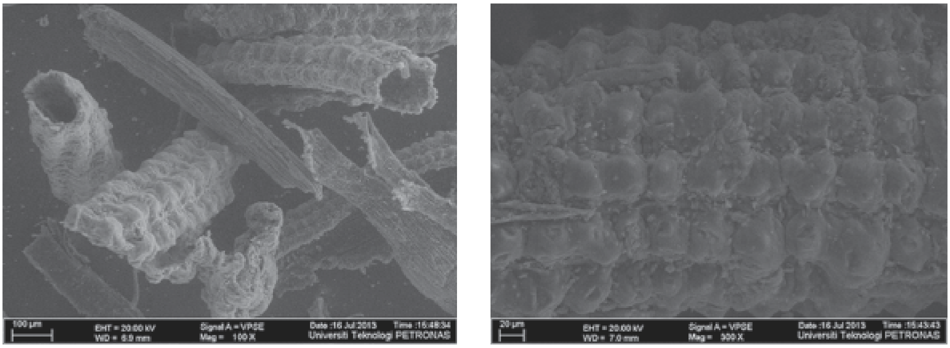

Figure 13.4 *Field emission scanning electron microscope of residue*

As shown in Figure 13.3, the structure of raw rice husk was well organized and originally in globular shape. The transverse section is dense, with no existence of pores. However, after slow pyrolysis in nitrogen atmosphere at a heating rate of 20°C/min as shown in Figure 13.4, the surface texture of rice husk residue changed. The globules shrunk and were densified due to the release of volatile products (Vlaev et al., 2003). Evaporation of volatile materials creates a large number of button-like structures or bumps interspaced with small pores formed on the particles with rough surface. The pores appear as channels from where the cellulose material was preferentially removed during pyrolysis (Jenkins, 1989).

13.3.5 Chemical Composition (GC–MS) Analysis

The chemical compound of bio-oil product was analyzed by using gas chromatography–mass spectroscopy (GC–MS). The bio-oil produced at different heating rates is analyzed for its chemical composition. The components of the bio-oil can be classified into aldehydes, acids, alcohols, ketones, phenols, furans, and sugars (Diebold, 1999). In this work, the area % of the GC–MS chromatogram was used to indicate the amount of the various chemical compounds in the bio-oil. Figure 13.5 summarizes the components contained in the bio-oil produced at different heating rates which are extracted from GC–MS chromatograms with the area % readings.

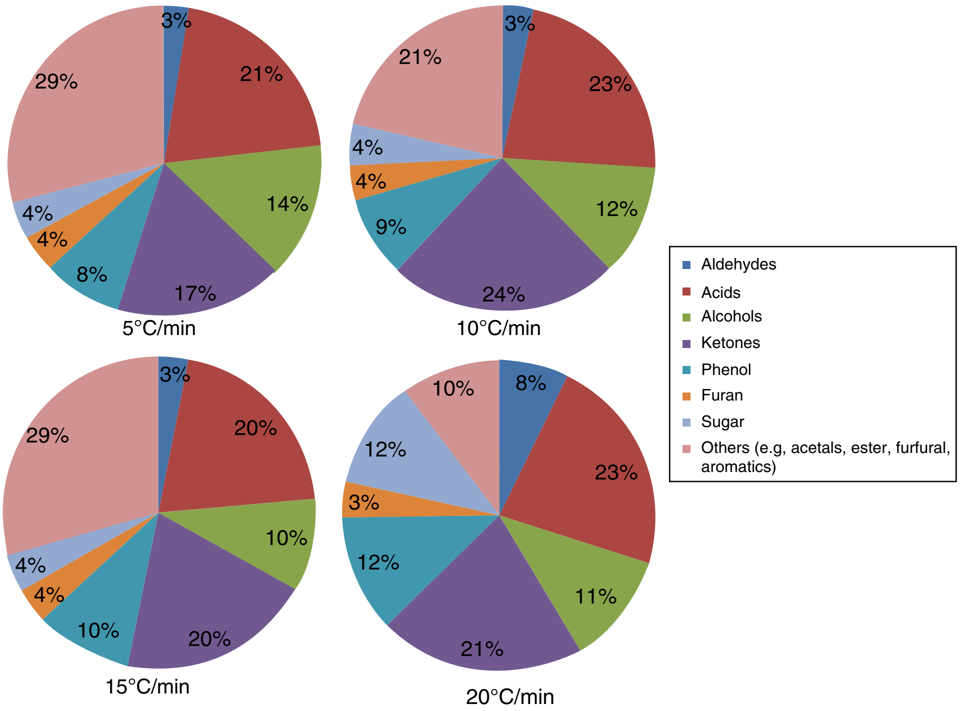

Figure 13.5 *Chemical composition of bio-oil at different heating rates*

As shown in Figure 13.5, the bio-oil produced at a heating rate of 20°C/min has more aldehydes, acids, ketones, phenol compounds, and more compounds of heavy molecular weight which cause high-viscosity reading. The higher number of phenol and acid compounds contributes to the higher pH number of bio-oil that was produced at a heating rate of 20°C/min.

13.4 Conclusion

In this study, bio-oil is produced from slow pyrolysis of RH. The optimum reaction condition result in maximum liquid yield is determined at a heating rate of 20°C/min, a temperature of 500°C, and a nitrogen flow rate of 100 ml/min. The maximum average liquid yield of bio-oil obtained is 35.38 wt% with conversion value of 63.82 wt%. The physical properties of bio-oil produced at the optimum condition are as follows: pH value of 2.68; viscosity value of 47.83 cP; elemental composition of 26.76 wt% carbon, 9.22 wt% hydrogen, 0.31 wt% nitrogen, 0.44 wt% sulfur, and 63.27 wt% oxygen; density value of 1082.69 kg/m^3 at 20°C; and HHV of 13.69 MJ/kg. The aforementioned desirable properties of bio-oil produced from slow pyrolysis in this show that bio-oil has a high potential as a substitute for conventional fossil fuel.

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