

Rubber Wood as a Potential Biomass Feedstock for Biochar via Slow Pyrolysis

Adilah Shariff, Radin Hakim, Nurhayati Abdullah

Abstract—Utilisation of biomass feedstock for biochar has received increasing attention because of their potential for carbon sequestration and soil amendment. The aim of this study is to investigate the characteristics of rubber wood as a biomass feedstock for biochar via slow pyrolysis process. This was achieved by using proximate, ultimate, and thermogravimetric analysis (TGA) as well as heating value, pH and lignocellulosic determination. Rubber wood contains 4.13 mf wt.% moisture, 86.30 mf wt.% volatile matter, 0.60 mf wt.% ash content, and 13.10 mf wt.% fixed carbon. The ultimate analysis shows that rubber wood consists of 44.33 mf wt.% carbon, 6.26 mf wt.% hydrogen, 19.31 mf wt.% nitrogen, 0.31 mf wt.% sulphur, and 29.79 mf wt.% oxygen. The higher heating value of rubber wood is 22.5 MJ/kg, and its lower heating value is 21.2 MJ/kg. At 27 °C, the pH value of rubber wood is 6.83 which is acidic. The lignocellulosic analysis revealed that rubber wood composition consists of 2.63 mf wt.% lignin, 20.13 mf wt.% cellulose, and 65.04 mf wt.% hemicellulose. The volatile matter to fixed carbon ratio is 6.58. This led to a biochar yield of 25.14 wt.% at 500 °C. Rubber wood is an environmental friendly feedstock due to its low sulphur content. Rubber wood therefore is a suitable and a potential feedstock for biochar production via slow pyrolysis.

Keywords—Biochar, biomass, rubber wood, slow pyrolysis.

I. INTRODUCTION

THE utilisation of fossil fuels has been steadily increasing each year and became the primary sources of carbon dioxide (CO₂) emission into the atmosphere [1]. CO₂ is the main source of greenhouse gases (GHG) and led to the world's global warming and climate change [2]. The renewable energy strategies can cut down the emissions of CO₂ into the atmosphere, but they are unable to reduce the CO₂ that is already been released into atmosphere, and reverse the climate change. One promising approach to lower CO₂ in the atmosphere is the utilisation of the biomass.

Lignocellulosic biomass such as wood wastes, waste paper, saw-dust, grass and the biomass-derived char (biochar), is non-food biomass and therefore viewed as a promising feedstock for renewable and alternative fuels [3], [4]. Lignocellulosic biomass is considered as a carbon-neutral resources that does not increase the CO₂ concentration in atmosphere, since the plants absorb CO₂ from the atmosphere in their growing process. It also has the advantages of high volatile matter content, low ash, sulphur and nitrogen contents,

and therefore has low pollution to the environment when burned [5].

Various forms of biomass are consumed all over the world for energy production. Energy generated from biomass produces less GHG and improves the environment's ecosystem. One of the indigenous biomass resources, especially in Southeast Asia is produced from the rubber plantation wastes such as rubber wood (RW) [6]. Rubber tree is one of the main plantations in Malaysia with an estimated plantation area of 1.82 million ha, which accounts for 20% global plantations [7]. The rubber trees are cut after their latex yielding period of around 25 years, and the wood could be utilized for many downstream processes [8]. RW is estimated to gross yield 180 m³ per ha, and in the process of converting the raw logs into sawn timber, the yield was estimated to be only 20%, and the remainder stays as residual biomass [7].

Biomass can be converted into biochar via slow pyrolysis process. Pyrolysis can be defined as the direct thermal decomposition of the organic materials in the absence of oxygen to obtain solid, liquid, and gas products [7]. Pyrolysis is divided into slow pyrolysis, fast pyrolysis, and flash pyrolysis. For slow pyrolysis, the temperature is <650 °C with heating rate <30 °C/min for a long residence time up to >1h. For fast pyrolysis, the temperature is >650 °C with heating rate >50 °C/min for short residence time <1h. The condition of flash pyrolysis is same as fast, only the temperature for flash pyrolysis is <650 °C [9]. According to Park et al., the slow pyrolysis is the most suitable pyrolysis process to produce biochar [10]. By using various types of reactor configuration for slow pyrolysis under complete or partial exclusion of oxygen [11], the biomass is heated at temperature between 300 °C to 650 °C [12] with low heating rate which is below 30 °C/min [13] and longer residence time about 1h and above [14]. The different parameters of the pyrolysis process such as temperature, heating rate and residence time influence the yield percentage and the properties of the product. The feedstock characterisation affects the pyrolysis percentage yield and properties of the product [15].

The international Bio-char Initiative (IBI) defines bio-char as “the solid material derived from the carbonization of biomass that may be added to soils with the intention to improve soil functions and to reduce emissions from biomass that would otherwise naturally degrade to GHG” [16]. Biochar is a promising material for climate change mitigation and adaptation, as it can sequester atmospheric C while improving the quality of soil where it is stored [17], [18]. Woolf et al. analysed the global potential for sustainable global biochar

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development, with results showing that net of GHGs could be reduced as much as 1.8 Gt CO₂ [11].

The aim of this study is to characterise the biomass feedstock in order to identify their suitability to undergo the thermochemical conversion process. Analysis methods to identify the characteristic of feedstock are proximate, ultimate, lignocellulosic, calorific value, pH, and thermogravimetric. Proximate analysis was performed to identify the moisture content (MC), volatile matter (VM), ash content (AC), and fixed carbon content (FC) of feedstock [19]. Feedstock with high VM and low AC satisfies some of the main criteria for biochar production via slow pyrolysis. High VM indicates that the feedstock is easy to decompose by heat [20], and low ash indicates that the feedstock sample will produce less slag during pyrolysis process [21]. Ultimate analysis was performed to determine the elemental composition of carbon (C), hydrogen (H), nitrogen (N), sulphur (S) and oxygen (O). C, H and O are the main components of the carbohydrates chains in an organic structure, which are the elements that make up the main hydrocarbon chains in biochar [22]. Feedstock with low S and low N will produce low non-environmental gases during thermochemical process [23]. The percentage of C, H and O also affects the calorific value of the feedstock [24]. The heating value of a biomass fuel may be reported on two properties; namely, the higher heating value (HHV) and lower heating value (LHV). The HHV refers to the heat released from the fuel combustion with the original and generated water in a condensed state, while the LHV is based on gaseous water as the product [25].

II. MATERIALS AND METHODS

A. Feedstock Preparation

The RWs were collected from Bukit Jambul Rubber Plantation, Gelugor, Pulau Pinang, Malaysia. RWs were clean by applying air pressure to remove excess dirt and sand. RW were cut into a pellet size about 2-4 cm length for storage purpose. To avoid the growth of fungus, the RW were dried in a conventional oven (Venticell 222-Standard) at a temperature 105 °C until the MC is less than 5 mf wt.% [28]. 200 g of RW was grinded to powder form using conventional grinder.

B. Proximate Analysis

Proximate analysis was performed to determine the MC, VM, AC, and FC of RW. Analyses of MC, VM and AC were performed according to ASTM E871, ASTM E830, and ASTM E872, respectively.

The average results from the proximate analysis were presented in weight percentage of moisture free basis (mf wt. %). The FC was calculated from (1):

$$FC (mf wt. \%) = 100 - [VM + AC](mf wt. \%) \quad (1)$$

C. Ultimate Analysis

Ultimate analysis was performed to determine the elemental composition of C, H, N, S, and O in RW. Powdered RW was analysed by using Perkin Elmer 2400 Analyzer to determine

the percentage of C, H, N, and S. The percentage of O was obtained from (2):

$$O (mf wt. \%) = 100 - [C + H + N + S](mf wt. \%) \quad (2)$$

D. Heating Value

The HHV of RW was analysed and obtained by using the Parr 6200 Isoperibol Calorimeter. Approximately 1 g of powdered RW was put in the water jacket oxygen bomb and undergo combustion inside the bomb calorimeter. The LHV of RW was calculated from the HHV and H content according to (3) [26]:

$$LHV = HHV - 2.442(8.936H/100)MJ/kg \quad (3)$$

E. Lignocellulosic

The extractives of alcohol-benzene solubility, extractives of hot-water and cold-water solubility were determined from ASTM D1107-96 and D1110-84, respectively. The percentages of lignin, holocellulose, and alpha-cellulose were determined by using standard methods ASTM D1106-96, D1104-56, and D1103-60, respectively. The percentage of hemicellulose was determined by the difference of holocellulose and alpha-cellulose. Alpha-cellulose is also known as cellulose [27].

F. Thermogravimetric Analysis

The thermal and kinetic behaviour of the RW was evaluated by using TGA and differential thermal analysis (DTA). The analysis was performed by using Perkin Elmer STA 6000 computerized thermogravimetric analyser. Powdered RW was heated from 27 °C to 900 °C at 5 °C/min in the presence of 30 mL/min N₂ gas.

G. Ph Analysis

The pH value of RW was measured by using Jenway 3015 pH meter at 27 °C. 1 g of powdered RW was dissolved in 100 ml of de-ionized water in a conical flask. The mixture was stirred with a magnetic stirrer for 30h occasionally [28] before measurements were taken.

H. Slow Pyrolysis and Biochar Yield

Fig. 1 shows a schematic diagram of the bench-scale fixed bed reactor used for slow pyrolysis. The reactor was made of stainless steel with a diameter of 10 cm and a height of 30 cm, and placed inside an electrically-heated furnace model F62700. In each test, 100 - 400 g of RW was heated from room temperature to 500 °C at approximately 5 °C/min, and maintained for at least 2h to allow sufficient time for complete pyrolysis. Note that a pyrolysis temperature of 500 °C was selected as an indicative temperature for comparison of biochar properties.

The pyrolysis vapour purged by nitrogen (0.2 L/min) passes through a series of condensers for collection of condensable products (bio-oil including water). The non-condensable gases were then allowed to escape out from the laboratory through the fume cupboard. The quantity of biochar produced was determined by the weighing the pyrolyser. The biochar yield (solid residue) of the RW was calculated from (4):

$$\text{Biochar Yield} = \frac{\text{mass of residue (g)}}{\text{mass of sample (g)}} \times 100\% \quad (4)$$

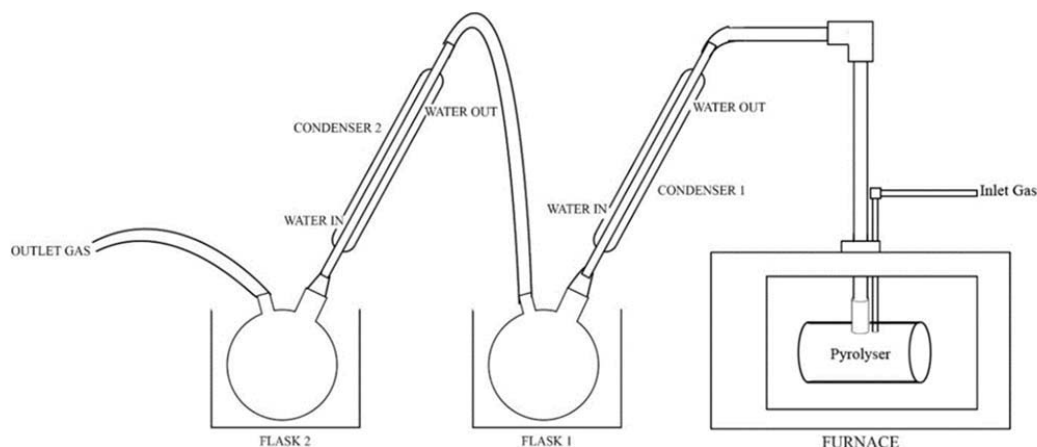


Fig. 1 Experimental set up for slow pyrolysis process

III. RESULTS AND DISCUSSION

A. Biomass Characterisation

From the Table I, the RW contained 4.13 mf wt.% MC, 86.30 mf wt.% VM, 0.60 mf wt.% AC, 13.10 mf wt.% FC and 6.59 VM/FC ratio. The RW showed high VM content, according to Ronsse, high VM made a biomass feedstock a highly reactive fuel with a faster combustion rate during devolatilisation phase [14]. A study by Kaewluan and Pipatmanomai [29] stated that high VM produced high percentage of vapour (composition of liquid and gases) at high temperature. RW revealed low AC, according to Dall’Ora et al. [30], ash could affect the biomass devolatilisation rate and oxidation rate during combustion and pyrolysis process. Ash were composed of minerals (silicon, magnesium and etc), the minerals contain in AC will react with the oxygen to produce mineral gases and vapour. Lee et al. (2013) study that a feedstock with high FC is able to produce high percentage of biochar during pyrolysis process [31]. Lee et al. also stated a feedstock of VM/FC ratio affect the biochar production during pyrolysis process. According to Lee et al., a lower VM/FC will produce more biochar.

Table I also shows RW contained 44.33 mf wt.% C, 6.26 mf wt.% H, 19.31 mf wt.% N, 0.31 mf wt.% S and 29.79 mf wt.% O. RW contained a high percentage of N. A high nitrogen concentration will reduce the hydrocarbon yields during thermochemical conversion and replace by the production of a non-environmental gases NO_x gases. High N in RW could be contributed by the nitrogen-rich fertilizer applied to the soil at the rubber plantation. RW has low percentage of S and produce low amount of sulphur oxide SO_x gases which are non-environmental.

Table I also shows RW contained 9.91 mf wt.% of extractives, 2.63 mf wt.% lignin, 97.17 mf wt.% holocellulose, 20.13 mf wt.% cellulose, and 77.04 mf wt.% hemicellulose. According to Yang et al. [32], hemicellulose mostly will decompose to liquid and gas at range 200 °C - 350 °C. Lignin and cellulose only will start to decompose at temperature higher than 300 °C. The feedstock with high hemicellulose

and cellulose content could produce higher char yield during the pyrolysis process [31]. Extractives contribute to liquid and gas products either through simple volatilisation or decomposition. Minerals in general remained in the char where they are turned to ash [33].

TABLE I
CHARACTERISATION OF RAW RW

Analysis		RW
Proximate analysis (mf wt.%)	Moistures	4.13
	VM	86.30
	Ash	0.60
	Fixed Carbon ^a	13.10
	VM/FC	6.59
Chemical analysis (mf wt.%)	C	44.33
	H	6.26
	N	19.31
	S	0.31
	O ^a	29.79
	H/C	0.14
Calorific Value (MJ/kg)	HHV	22.5
	LHV	21.2
Lignocellulosic analysis	Extractive	9.91
	Lignin	2.63
	Holocellulose	97.17
	Cellulose ^{a,b}	20.13
	Hemicellulose	77.04
	pH	6.83

^a Value is calculated from the differences.

^b Cellulose is difference of holocellulose and hemicellulose

The pH value obtained for RW is 6.83, and the RW raw samples show a slightly acidic characteristic. Table I reveals that the RW HHV is 22.54 MJ/kg and LHV is 21.17 MJ/kg. The HHV and LHV could be affected by the percentages of C, H and O. According to Channiwala and Parikh (2002), C and H contributes positively to the HHV, while the content of O influences negatively [24].

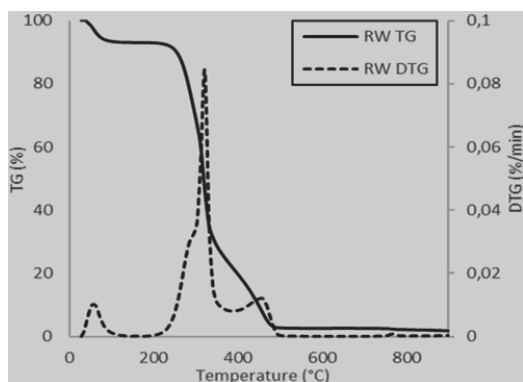


Fig. 2 TGA results of RW

B. TGA Analysis

Fig. 2 shows the thermal degradation of the RW which is determined by the thermogravimetric (TG) and derivative thermogravimetric (DTG) curves. TG curve represent the fractional weight loss of the feedstock as a function of temperature, while the DTG curve is the plot of the rate of mass change, dM/dt versus temperature. The weight loss of the RW rapid weight loss occurred in the temperature range 220 °C - 340 °C. The weight of RW decreased from 93% to 46% in this range. Between 340 °C - 490 °C, the weight further decreased from 46% to 7%. Beyond 490 °C, the weight of RW continues to decrease at a less rapid rate.

From the DTG curve in Fig. 2, a small peak was observed below 100 °C. The formation of this small peak is due to loss of moisture. In temperature range of 200 °C - 490 °C, a high peak and a small hump could be observed. The high peak occurred in the range of 200 °C - 370 °C, with the maximum weight loss rate 0.08404 %/min at 320 °C. The small hump appeared in the range of 370 °C - 490 °C, with the maximum weight loss rate 0.01192 %/min at 423 °C. These peaks and humps represent the degradation of lignocellulosic component. The formation of high peak due to degradation of hemicellulose while the appearance of small hump represents the degradation of cellulose. The degradation of lignin could not be observed clearly in the DTG curve as its degradation occurs over a wide range of temperature. Yang et al. [32] studied the decomposition of hemicellulose, cellulose and lignin separately. According to Yang et al., the decomposition of hemicellulose occurs in between 220 °C - 320 °C, cellulose decomposes in between 320 °C - 450 °C, while lignin decomposes in between 27 °C - 900 °C.

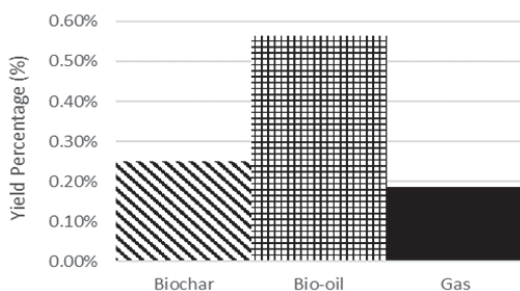


Fig. 3 Product yield of RW pyrolysis at 500°C

C. Products Yield from Slow Pyrolysis

Fig. 3 showed the mass yield of pyrolysis products produced at 500°C using the lab-scale reactor. The biochar yield for RW were 25%. The biochar yields were in reasonable agreement with the relative weight at 500 °C in the TGA curves (Fig. 2). The bio-oil yields (including the water condensed) were over 50%. The high amount of bio-oil yields was caused by the inorganic and water in raw samples, which were added to the biochar and bio-oil, respectively. According to Lee et al. [31], a high VM feedstock would produce high vapour, while high fixed carbon will produce high char yield.

The biochar yields from the organic portion of RW which is typical for lignocellulosic biomass. This sample is taken from the storage tissue of biomass that contains a larger proportion of cellulose and hemicellulose. Yang et al. [32] stated that, the cellulose and hemicellulose contribute to the formation of char during pyrolysis.

TABLE II
CHARACTERISATION OF RW BIOCHAR AT 500 °C

Analysis		RW
Proximate analysis (mf wt.%)	Moistures	0.74
	VM	20.23
	Ash	2.45
Chemical analysis (mf wt.%)	Fixed Carbon ^a	77.32
	VM/FC	0.26
	C	62.40
	H	2.24
	N	5.22
	S	0
	O ^a	30.14
	H/C	0.04
	O/C	0.48
	pH	9.3

^a Value is calculated from the differences.

D. Biochar Characterisation

Table II summarises the properties of biochar produced at 500 °C. The VM/FC ratio decreased to 0.26 as a result of pyrolysis. Therefore, the biochar products became highly resistant to further biological or thermal decomposition. The pyrolysis process increased the AC of the biochar.

Table II also lists the result of ultimate analysis. The biochar became highly carbonaceous, with a carbon content 62%. The amount of H, N and S contained in the raw sample decreased with pyrolysis, and very low or insignificant amount of S was observed. This is due to oxidation at higher temperature, and it reflects the conclusion drawn by Schmidt and Noack [34] on the characteristics of black carbon. Schmidt et al. claimed that biochar sample represents a continuum from partially charred organic material to graphite/soot particles, with no clear boundary.

The elemental ratios of H/C and O/C explain the degree of aromaticity, maturation, and bonding arrangement of the biochar. Results in Table II show that both H/C and O/C ratios in the experimentally produced biochar were decreased. These ratios in the raw material are consistent with C atoms that are essentially saturated due to lignocellulosic material [7]. The

very low H/C and O/C ratios obtained for the biochar indicated that carbon in biochar is predominantly unsaturated, and C atoms are bonded directly with other carbons.

The pH is an important property of the soil, and it influences the types of plants and microbes to thrive, and the availability of nutrients to be absorbed [31]. Soil acidification is the result of nitric acid and sulfuric acid from fossil-fuel combustion [35] and the long-term application of nitrogen compounds fertilizers [36]. Neutralizing acid soils by applying biochar can improve the soil quality, and increase the productivity of crops. As also listed in Table II, the biochar tended to be highly alkaline (9.3). The alkalinity of biochar is influenced by three factors: (i) organic functional groups, (ii) carbonates, and (iii) inorganic alkalis [37], [38]. The contribution of organic functional groups such as $-\text{COOH}$ and $-\text{OH}$ is known to decrease with increasing pyrolysis temperature by progress of thermal decomposition. In contrast, the contribution of carbonates formation (such as CaCO_3 and MgCO_3) and inorganic alkalis (such as Na and K) becomes important above $500\text{ }^\circ\text{C}$ [37]. As a result, higher pyrolysis temperatures significantly increased the pH of biochar [39]–[41]. The organic functional groups in the biochar sample were not analysed in this study.

IV. CONCLUSION

In this study, biochar was produced by using waste RW in a fixed-bed reactor under $500\text{ }^\circ\text{C}$ with $5\text{ }^\circ\text{C}/\text{min}$ for at least 2h with a $0.2\text{ L}/\text{min}$ N_2 gas purges. The physicochemical properties of RW and RW biochar were investigated. Pyrolysis was found to greatly influence chemical properties of the derived biochar. The FC increased due to pyrolysis, and showed an inverse effect in the VM/FC ratio. Thus, the biochar products became highly resistant to further biological or thermal decomposition. The carbon content of RW increased due to pyrolysis, and decreased the elemental ratios of H/C and O/C. Therefore, suggest that more carbon can be locked-up in the soil from biochar produced via slow pyrolysis.

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