

Experimental Investigation and Simulation of Slow Pyrolysis Process of Arabica Coffee Agroindustry Residues in a Pilot-Scale Reactor

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ABSTRACT

Coffee pulp and husk are the primary residues of the coffee agro-industry. Disposing of them into the land can bring a serious problem on the environment. Strategies are needed to convert it into more valuable products as well as reduce the risk of environmental damage. This paper reports experimental and simulation investigation on the pyrolysis of Gayo arabica coffee pulp and husk in a pilot scale reactor. The investigation included finding the chemical and physical properties of biomass under ultimate, proximate, bomb calorimeter and TGA analyses. During the pyrolysis experiments, 3 kg of dried raw material was fed into the reactor and heated from room temperature to 600 °C, then held for 2.5 h. Afterwards, the resulting biochar and pyrolytic oil were quantified for product distribution analysis. Modeling and simulation of the pyrolysis process were performed using Aspen Plus V10 software. Experimental results show that biochar is the main product giving a yield of 43.83%. The percentage of pyrolytic oil and un-condensable gas products are 25.5% and 30.67%, respectively. The thermodynamic simulation shows a good agreement with the experimental result, which helps in optimization and scaling up reactor.

Keywords: slow pyrolysis; pyrolytic oil; biochar; aspen plus; coffee agro industry residues.

INTRODUCTION

Coffee is recognized as the main commodity traded worldwide. Globally, annual coffee production increased from 140 to 158 million 60 kg bags since 2010 (International Coffee Organization, 2017). Indonesia is one of the largest coffee-producing countries in the world. Indonesia's coffee bean exports reached 433.6 thousand ton in 2010 and increased up to 467.8 thousand ton in 2017 with total value reaching 1,187.16 million US\$ (BPS-Statistics Indonesia, 2017). Bener Meriah Regency is reported as one of the main area for Arabica coffee plantation in Indonesia which has a planting area of 47,370 ha with total production reaching 11,526 tons/year (Directorate General of Estate Crops, 2017). Indeed, the processing of coffee-cherry generates significant amounts of waste depending on the type of

processing; thus utilization of this waste poses a serious challenge. Currently, the waste from coffee cherry processing has not been utilized optimally. It is mostly left on the ground or under the coffee tree as organic fertilizer or burned.

Coffee agroindustry residue is a source of biomass which has not been profitably used. A few studies have been done to investigate the potential utilization of coffee waste however profitable and technically feasible methods are still under development. Transforming this biomass into more valuable products can be carried out through a number of methods including (1) mushroom cultivation, (2) enzymes production, (3) biofuel production, (4) organic acid production, (5) bioactive compounds, (6) dietary fiber, (7) composting and vermicomposting (Janissen and Huynh, 2018). Among these options, converting the coffee waste into biofuel looks like a

more potential and applicable alternative. Pyrolysis is a low cost and effective process to convert the organics into energy-rich products (Kumar et al., 2019; Kan et al., 2016; Kumar et al., 2020). Through the pyrolysis process, biomass can be converted into bio-char as an alternative fuel to replace fossil fuels (Setiawan et al., 2019; Setiawan et al., 2020; Patel et al., 2019).

Pyrolysis is categorized into three methods i.e. slow, fast and flash pyrolysis depending on the heating rate and residence time, aiming to optimize either the bio-oil or biochar yields. Another targeted product of biomass pyrolysis is synthesis gas or hydrogen-rich gas (Kan et al., 2016). Several methods have been developed to produce bio-char, one of which is by the slow pyrolysis method. This method is suitable when biochar production is the primary target, whereas biomass is heated slowly without oxygen to a medium temperature (400 °C) for a long period of time (Basu, 2013). Indeed, biochar has become more attractive recently due to its wide-range potential applications in some areas such as wastewater treatment, soil amendment and remediation, CO₂ capture, climate change mitigation and energy storage (Leng and Huang, 2018).

A recent investigation reported that coffee pulp has been used as raw materials for bio-char production. This initial investigation result showed that the coffee pulp waste has been successfully processed into bio-char through the pyrolysis process (Setiawan et al., 2020). However, a number of aspects have not been explored yet for finding an optimum operating condition. Modeling and simulation can be a cost-effective way and save the time for understanding the operating

condition. On the basis of previous studies, simulation of the pyrolysis process has been successfully performed with the Aspen Plus software (Patel et al., 2019; Nur et al., 2019; Ward et al., 2014) for techno-economic feasibility study.

This study aimed to develop a pyrolysis model using the Aspen Plus software to explore the effect of reaction temperature and feedstock composition on pyrolysis product distribution. Validation of simulation was performed by comparing with the results of experimental investigation under a-pilot-scale slow pyrolysis set-up.

METHODOLOGY

Experimental

Gayo Arabica coffee pulp and husk were used as raw material for pyrolysis. The raw materials were collected from a coffee plantation in Bener Meriah Regency, Aceh Province. Coffee pulp and husks are the first by-products of the coffee cherry processing. Coffee pulp is the outer skin of coffee cherry which is peeled-off first. In turn, coffee husk is the second layer of coffee cherry (Janissen and Huynh, 2018). Prior to analysis, the raw materials were dried by natural drying under the sun for five days. The schematic of the experimental set up is shown in Figure 1. This set-up consists of a pyrolysis reactor, condenser, separator, gas burner and furnace. At any experiment, the amount of raw material fed into the reactor was 3 kg (Setiawan et al., 2020).

In order to understand the thermophysical properties of coffee pulp and husk, a number of

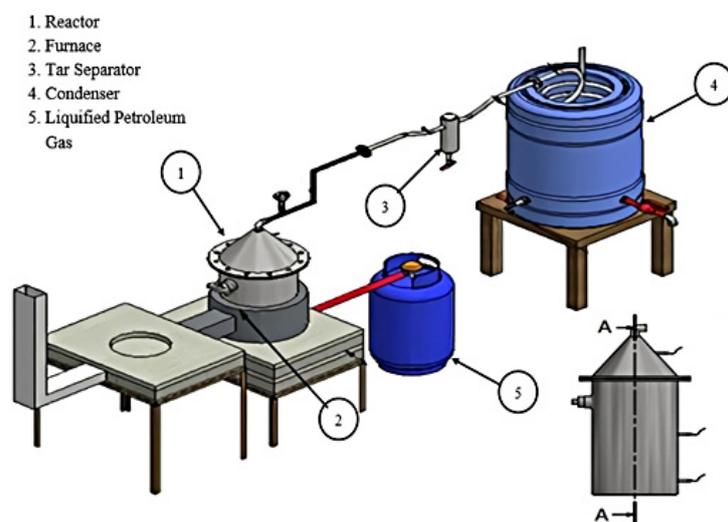


Figure 1. Slow-pyrolysis experimental set-up (Setiawan et al., 2020)

analyses were performed, including thermogravimetric analysis (TGA), proximate analysis, ultimate analysis, differential calorimetry analysis (DSC) and bomb calorimeter. The analysis of the content of moisture, ash, volatile matter and fixed carbon, proximate analysis was carried out following the ASTM D1762-84 procedure. Ultimate analysis was performed based on the procedure of ASTM D5373-16. The TGA and DSC analyses were assessed under Mettler Toledo TGA/DSC machine with high temperature furnace by purging with nitrogen at a flow rate of 20 ml/min and heating rate of 10 °C/min. The samples were placed in an alumina crucible *c.a.* 10 mg, then heated from an ambient temperature up to 1000 °C. Koehler K88990 Bomb-type Calorimeter was used to analyze the caloric value of coffee pulp and husk.

Simulation

The Aspen Plus software was used to simulate the pyrolysis process of Arabica Gayo coffee pulp and husks. The Aspen Plus is software that can be used to simulate chemical-thermodynamic processes based on mass and energy balance, vapor-liquid equilibrium, mass transfer, heat transfer and kinetic chemistry. Initially, the

properties of raw materials should be defined based on the data from coffee pulp and husk characterization. It is known that these raw materials are categorized as non-conventional component. Then, the stream class was set as MIXCINC as the components involved in this simulation are mixing of conventional, non-conventional and solids components. In this analysis, the particle size distribution (PSD) of raw materials was neglected. The RK-SOAVE property method was chosen to calculate the physical properties of conventional mixed components and CISOLID components during analysis. The HCOALGEN and DCOALIGT models were used to calculate the enthalpy and density of coffee pulp and husk. The description of the Aspen Plus operating unit model used in this simulation is shown in Table 1, while the process flow diagram of the simulation is shown in Figure 2.

Figure 2 shows that the raw material for coffee pulp and husks with a flowrate of 3 kg/h is fed into the RYIELD reactor to be converted from unconventional components into conventional components based on ultimate analysis. Then the elements coming out of the RYIELD reactor enter the RGIBBS reactor, where the formation of syngas is based on thermodynamic equilibrium

Table 1. ASPEN Plus unit operations model description

Aspen plus ID	Block ID	Description
RYIELD	DECOMP	Models the conversion of non-conventional materials to conventional simulation components
RGIBBS	REACTOR	Calculates the pyrolysis products distribution via the minimization of Gibbs free energy method
SSPLIT	FILTER	Separates the char from the product vapor by specified split ratio
PUMP	PUMP	Pump
HEATX	CONDEN	Two stream heat exchangers
FLASH2	SEP	Separates the pyrolytic oil from un-condensable gases
CALCULATOR	DEC	Specification of the RYIELD mass yield fractions of cellulose, hemicelluloses and lignin

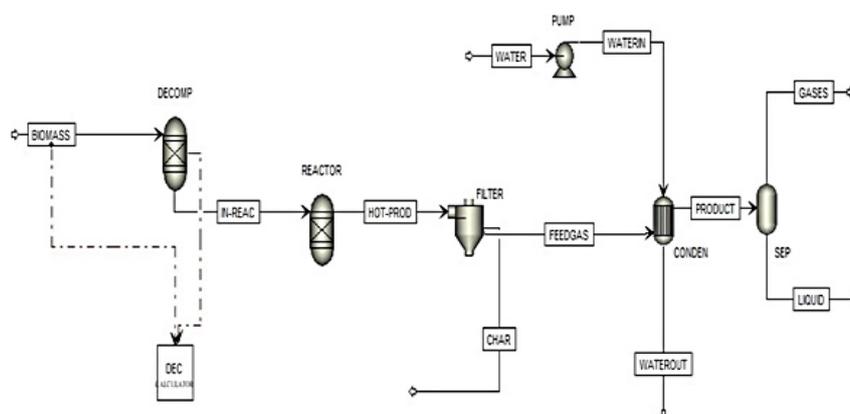


Figure 2. Process flow diagram of simulation

according to the temperature and pressure applied. SSPLIT is used to separate bio-char from vapor products. Later, the vapor product will be cooled before it is separated into the pyrolytic oil product and the un-condensable gas. In this simulation, the pyrolysis temperature was varied from 300 to 500 °C based on the TGA analysis data where the decomposition temperature of raw materials is within this range. The composition of raw material was simulated by 100% coffee pulp, 100% coffee-husks, 25% coffee pulp 75% coffee husks, 25% coffee husks 75% coffee pulp, 50% coffee pulp 50% coffee husks in order to observed the effect of different characteristic of raw material on the product yield.

RESULTS AND DISCUSSION

Characteristics of raw materials

The physical properties of coffee pulp and husk were assessed initially to find out the content of water, ash, volatile matter and fixed carbon following the ASTM procedure D1762-84 (Aller et al., 2017). Chemical composition is necessary to be analyzed in order to set the properties during the simulation process. These analyses involve air-dried basis. The results of the proximate and ultimate analysis of the Arabica coffee pulp and husk are shown in Table 2.

In general, coffee pulp and husk contain more volatile matter than fixed carbon. Table 2 indicates that coffee husk has higher fixed carbon content and quite lower ash content compared to coffee pulp. In terms of the volatile matter content, coffee husk is

also higher, contributing to 68.6% of total sample mass. Ultimate analysis results also indicate that the amount of carbon on coffee husk sample is slightly higher. This suggests that the bio-char produced from the coffee pulp and husk is feasible.

Figure 3 displays the TG and DTG profiles of coffee pulp decomposition for a temperature range up to 1000 °C where a significant weight of sample was lost during heating in nitrogen. From TG plot it can be found that the initial sample weight was 10.52 mg and the final weight of sample after heating was 2.06 mg. This suggests that there was 19.58% of total sample mass which is mostly ash, was left on crucible after the experiment. This is nearly similar to the ash content reported in Table 2. From DTG curve, it is shown that the rate of thermal decomposition of coffee pulp mainly consists of three phases, i.e. dehydration, devolatilization, and lignin decomposition (Alias et al., 2014). Within the dehydration phase, *ca.* 12% of sample mass is lost starting from room temperature to 180 °C. This weight loss is due to removal of water molecules retained in the pores of biomass and removal of some very light volatiles. The second stage of coffee-pulp decomposition takes place within the temperature range of 180 to 400 °C which is generally due to the devolatilization process of volatile matter. It is suggested that the carbohydrate rich volatile fraction of organic matter decomposes within the range of 190 to 300 °C followed by organic polymer fractions rich in lipids up to 350 °C (Patel et al., 2018). Most of the volatile fraction is decomposed at 400 °C and the devolatilization rate decreases rapidly (Zhang et al., 2017). Within the third stage, the decomposition

Table 2. Proximate and ultimate analysis results

Analysis parameters	Sample		Basis
	Coffee pulp	Coffee husk	
Total moisture (%)	12.11	12.61	As received
Proximate analysis	-	-	-
Moisture in air dried (%)	10.54	10.44	Air dried basis
Ash (%)	16.98	3.34	Air dried basis
Volatile matter (%)	55.58	68.58	Air dried basis
Fixed carbon (%)	16.90	17.64	Air dried basis
Ultimate analysis	-	-	-
Total sulfur (%)	0.26	0.28	Air dried basis
Carbon (%)	36.88	44.00	Air dried basis
Hydrogen (%)	5.36	6.19	Air dried basis
Nitrogen (%)	2.10	1.04	Air dried basis
Oxygen (%)	38.42	45.15	Air dried basis
Chlorine (%)	0.054	0.031	Air dried basis

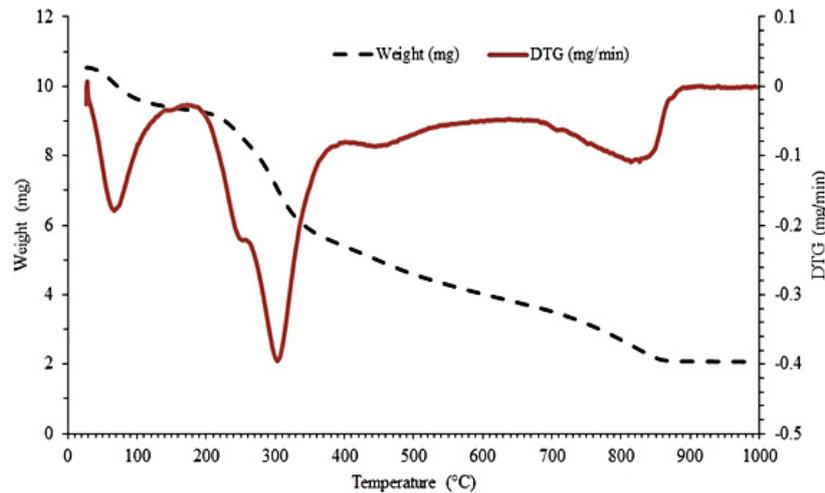


Figure 3. TGA and DTG of Coffee Pulp

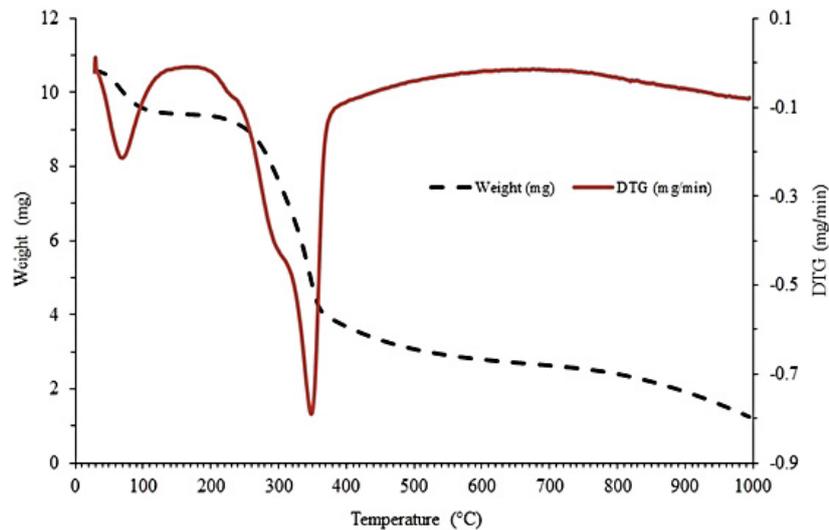


Figure 4. TGA and DTG Coffee Husk

of less biodegradable protein, lignin and synthetic organic polymers taken place until 900 °C.

Decomposition profile of coffee husk is shown in Figure 4. On the basis of this figure, the process of evaporation of water content takes place at a temperature range of 38–105 °C. The decomposition or devolatilization process occurs at temperatures of 200–1000 °C. From the DTG curve it was shown that the maximum rate of dehydration process occurred at a temperature of 66 °C, while the highest devolatilization rate of coffee husk takes place at a temperature of 347 °C.

The comparison of calorific value of the Gayo Arabica coffee pulp and husk are shown in Table 3. This data indicates that the coffee husk has a higher heating value compared to the coffee pulp. This is supported by the data from ultimate analysis in Table 2, where coffee husk contains higher C and H elements compared to coffee pulp. In addition,

the ash content in the coffee husk is much lower than the coffee pulp.

Temperature profile and product distribution of pyrolysis experiments

The pyrolysis experiment was performed in a purposely built reactor as shown in Figure 1. There were three measurements points of temperature *i.e.* at the bottom of vessel (T_1), the middle (T_2) and the top parts (T_3). Figure 5 displays the temperature changes as a function of reaction time during the pyrolysis experiment at a temperature below 600 °C. To heat up the reactor, an LPG burner was ignited from the bottom of reactor and produced a rate of heating of *c.a.* 3 °C/min during the first 100 minutes run. The maximum pyrolysis temperature of T_1 was reached after 160 minutes operation. The experiment was stopped

Table 3. The calorific value of the Gayo Arabica coffee pulp and husk

No	Sample name	Specification	Calorific value (J/g)
1	Arabica gayo coffee pulp	Sundried	13,837
2	Arabica gayo coffee husk	Coffee Husks	16,868

after four hours run where no pressure was built-up suggesting a completed pyrolysis process of coffee-pulp. To ensure the consistency of measurement, pyrolysis test was repeated three times.

Observation of the temperature changes at the middle section of reactor (T_2) suggests a slow increase of temperature. After 150 minutes of operation, the temperature just reached 350 °C. Obviously, a significant low heating ramp was observed at the top section of reactor reaching a maximum temperature of 316 °C after 2-hour run. It is noteworthy that at 50 minutes run, the temperature of T_2 and T_3 were dropped due to the opening of valves for collecting the liquid products. After 100 minutes in operation, all temperatures are slightly decreasing which is mostly due to the decrease in the pressure of LPG tank. When the reactor has reached the room temperature, biochar product was collected and weighted carefully. Product distribution based on pyrolysis experiment of coffee-pulp is tabulated in Table 4 suggesting biochar as the main product.

Validation of the thermodynamic model

The pyrolysis process of the Gayo Arabica coffee peels performed in the Laboratory was compared with the simulations performed using the Aspen Plus V10 software. The comparison data between the experimental and simulation results are shown in Table 4. This table

demonstrates that the differences between the simulation results and those obtained from experimental work are relatively small. This indicates that the simulation model that has been developed in this investigation is approaching the real process. A slightly higher difference was found in un-condensable gas yield which most likely due to gas leakage during experiments.

Simulation results

The yields of pyrolysis products (bio-char, pyrolytic oil and un-condensable gas) are respectively shown in Figures 6, 7, and 8. In these figures, the effects of raw material combinations on the yield of pyrolysis product are explored at various temperatures. The purpose of this simulation is to find the optimum condition for pyrolysis process of coffee pulp and husk. Figure 6 shows the yield of bio-char obtained from the simulation of a mixture of coffee pulp and husk with variations in the composition of mass and temperature ratio. It can be seen that the bio-char products produced decreases with increasing temperature. This is due to higher reactor temperature which makes the devolatilization process faster and carbon levels begin to decrease (Basu, 2013). The higher the portion of the coffee pulp in the raw material mixture, the more bio-char is produced. This has correlation with proximate analysis result where coffee pulp has much higher ash content but low

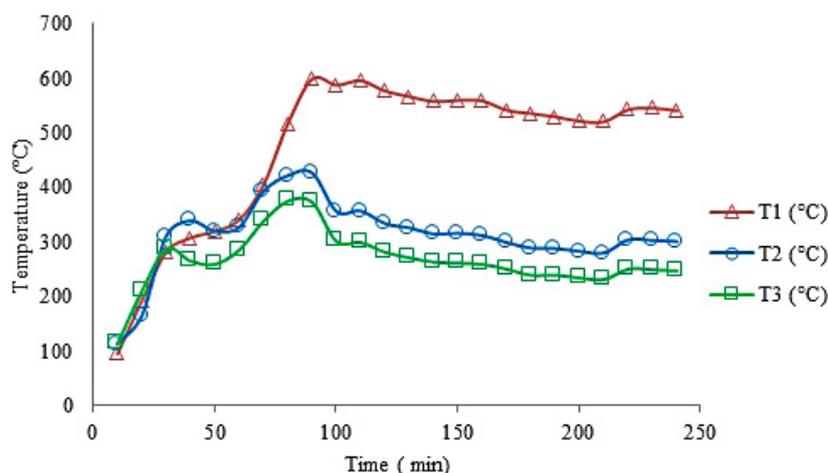
**Figure 5.** Temperature changes versus time recorded during pyrolysis experiment

Table 4. Validation of simulation results

Parameters	Experimental results	Simulation results	% error
Bio-char	42.87	40.91	4.57
Pyrolytic oil	26.47	24.43	7.70
Un-condensable gas	30.66	34.66	13.04

volatile matter content, compared to coffee husk. Thus, the mixture with more coffee pulp mass fraction will produce a higher bio-char mass.

The yield of pyrolytic oil in pyrolysis simulation of a mixture of coffee pulp and husk with variations in the composition of mass and temperature ratio is shown in Figure 7. In general, the yield of pyrolytic oil decreases along with increases in temperature. This is similar to what has been reported in the literature where further increase in temperature can reduce the amount of pyrolytic oil production (Seri and Putri, 2017). On the other hand, the pyrolysis process with 100% coffee husk produces more pyrolytic oil compared to those process by mixing with coffee pulp. As can be seen

from proximate analysis data in Table 2, coffee husk has higher volatile matter content. Therefore feedstock mixture with larger fraction of coffee husk mass leads to higher pyrolytic oil yield. This is in line with the information reported in the literature where raw materials with a high content of volatile matter produce more pyrolytic oil (Ighalo and Adeniyi, 2019; Syamsudin et al., 2016). It is noteworthy that modeling of the pyrolysis process applies the chemical reaction of compounds contained in the biomass (cellulose, hemicellulose and lignin). Indeed, pyrolytic oil consists of hundreds of organic compounds therefore proposing a model for all compounds involved in this reaction is not practicable (Lestinsky and Palit, 2016).

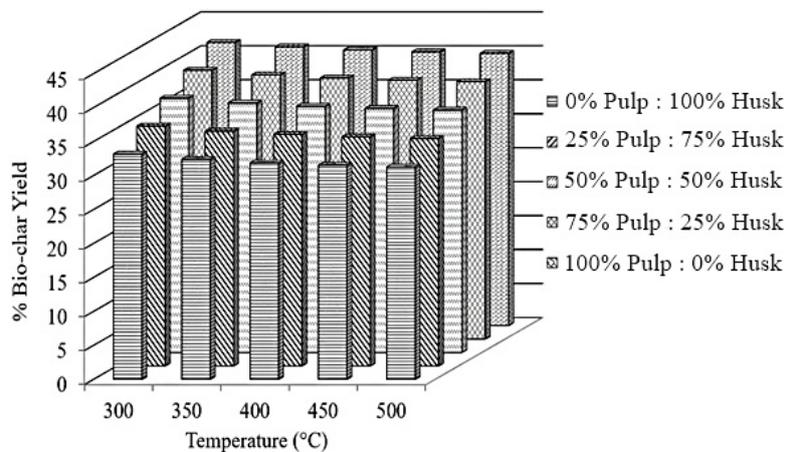


Figure 6. Bio-char yield

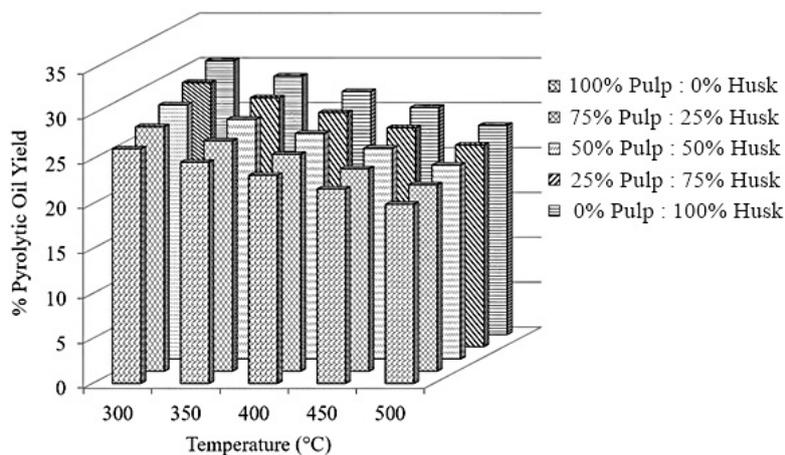


Figure 7. Pyrolytic oil yield

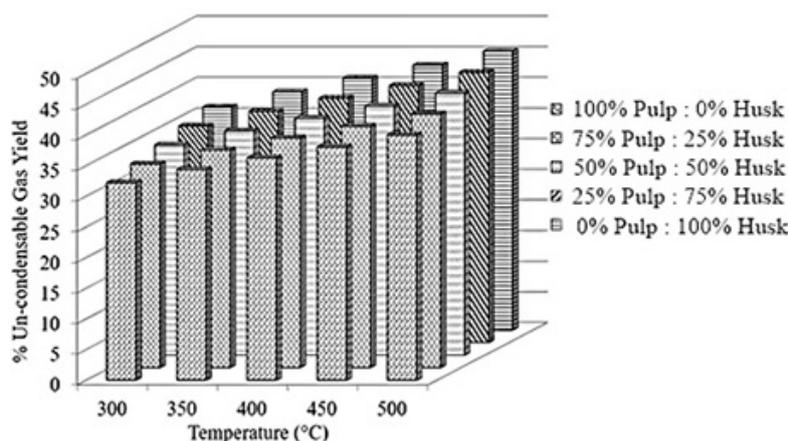


Figure 8. Un-condensable gas yield

The yield of un-condensable gas calculated from pyrolysis simulation of coffee pulp and husks at various pulp to husk ratios and reaction temperature is shown in Figure 8. This graph shows that the highest un-condensable gas yield is obtained at a temperature of 500 °C with 100% of coffee husk as raw material, which accounted for 45.47% of total product. In turn, the lowest un-condensable gas yield is obtained at a temperature of 300 °C by feeding the reactor with 100% coffee pulp, i.e. 32.31% of total product. The yield of un-condensable gas increased along with reaction temperature (Adeniyi et al., 2019; Ighalo and Adeniyi, 2019; Xianjun et al., 2015). In addition, the yield of un-condensable gas increases along with the increase in the amount of the coffee husk in feed. This is mostly because the content of volatile matter in coffee husks is higher compared to coffee pulp. The greater the volatile matter content of the raw material, the higher the fluid phase products (pyrolytic oil and un-condensable gas) produced (Ighalo and Adeniyi, 2019).

CONCLUSIONS

Pyrolysis of Arabica coffee agroindustry residues has been experimentally performed and simulated using the Aspen Plus V10 software with a steady state thermodynamic model. This investigation uses coffee pulp and husk at different mass fractions within a temperature range of 300 °C to 500 °C. The results show that the higher the coffee husk content in feed, the more yield of pyrolytic oil and un-condensable gas in the product. This is because coffee husk has a higher volatile matter content compared to coffee pulp. Meanwhile, the higher the coffee pulp mass fraction in

the feed, the more bio-char in the product. The yield of bio-char and pyrolytic oil decreases with increasing temperature. In contrast, the yield of un-condensable gas increases along with temperature. Eventually, the yield of bio-char produced from coffee husk is higher compared to that produced from coffee pulp. This is most-likely due to higher fixed carbon content and lower ash content of coffee husk compared to coffee pulp.

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