

## Bio-Oil Production from Pineapple by Microwave Pyrolysis

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### Abstract

This research concerns bio-oil production from agricultural waste after the harvesting of pineapples by microwave pyrolysis. The objective of this study was to study the process of microwave pyrolysis to determine the optimum reaction temperature and bio-oil content production by bio-oil processes and compounds, including ethyl acetate (EtOAc) as a microwave absorber. From the test, comparisons could be drawn when the wattage was high compared to when the wattage was low, so for pineapple corks, pineapple leaves, and pineapple plants by wattage and time as 600W/6min, 450W/6min, and 450W/8min, the bio-oil content was 29.6%, 32%, and 36.8%, while the percentages were 14.4%, 21.6%, and 44.8% when the wattage used was 100W/16 min, 100W/20min, and 200W/16min, respectively. Bio-oil obtained from pineapple corks, pineapple leaves, and pineapple stems was obtained by using a microwave pyrolysis process, and the compounds were analyzed using the GC-MS method. Groups of compounds such as acid, ester, ketone, aldehyde, phenol, benzene, and others were found, with percentages given as follows: Ketone (85.54%), phenol (63.60%), aldehyde (60.92%), and ester (56.98%). This group of compounds was found to be useful to expand the results in the food industry, as chemicals and precursors.

**Keywords:** microwave pyrolysis, pineapple cork, pineapple leaf, pineapple stem, bio-oil composition

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## Introduction

Pineapple is an important economic crop in Thailand. It has the highest production and export volume in the world. In 2019, it was estimated that the total harvested area in the country was 480,680 rai. Last year, 14 percent gave a total yield of 1.68 million tons, while 28 percent yielded 3,455 kilograms/rai. The main pineapple products exported by Thailand include canned pineapple, pineapple juice, frozen pineapple, dried pineapple, and rambutan stuffed with pineapple. As for the production sources of pineapple, most factories, 67 percent, are in the Central region, followed by the North at 21 percent. The main production provinces are Prachuap Khiri Khan, Ratchaburi, Rayong, Phetchaburi, and Phitsanulok. Pineapple can be grown almost all year round. Therefore, pineapple harvesting can also be done almost all year round, although pineapples bear the most fruit during the annual pineapple season. The proportion of the components of the whole pineapple plant amount to 37.35 percent fruit weight, 38.78 percent leaves, 7.77 percent crown, and 12.86, 3.08, and 0.18 percent for the stem, fruit stalk, and shoot, respectively [Ban Rai District Agricultural Office, Uthai Thani Province]. The residue left over from harvesting is biomass residue, which is abundant in quantity, including the pineapple leaves, which are a part of the trunk and are cut off during harvesting. Pineapple leaves are agricultural waste that are available almost all year round and are abundant from November to April because this is the period when most farmers harvest and send the produce to factories. The use of pineapple leaves occurs in various aspects, such as innovation in oil-absorbing pineapple fiber paper, innovation in heat-resistant pineapple fiber paper, while in terms of energy it can be burned to make mixed fuel briquettes. Raw materials or waste materials such as pineapple must have the appropriate characteristics to be used to make fibers or burn, so preliminary processing is necessary. However, there is growing interest in technology for processing waste materials that is suitable for high moisture content, as is the case with pineapple leaves, pineapple crowns, and pineapple trunks. Lignocellulose is one of the potential renewable energy sources to support

the rapidly growing energy demand. Abundant biomass is an attractive and viable method for energy production through a carbon-neutral cycle [Raheem, Abdul, et al., 2015] for the conversion of biomass into bio-oil, charcoal, and gas [Yaman, Serdar., 2004]. Through pyrolysis, bio-oil can be upgraded to transport fuel. It is obtained through hydro-deoxidation and rapid pyrolysis techniques. It has been proven to produce high yields of bio-oil. Fluidized bed reactors are suitable for rapid pyrolysis conditions causing particles to disperse quickly along with good heat transfer [Bridgwater, Anthony V., 2012] The microwave pyrolysis process has caught the attention of researchers because of its rapid and uniform volumetric heat distribution [Miura, Masakatsu, et al., 2004]. The low dielectric energy conversion microwave process requires an adsorbent to convert energy to biomass [Ren, Shoujie, et al., 2012]. The production of high-yield bio-oil from biomass is achieved via microwave pyrolysis. The chemical nature, quantity, size, and shape of the adsorbent are important factors that control the heating rate, the pyrolysis temperature, the product distribution, and micro plasma dot formation throughout the reaction mixture. A very slow or fast heating rate leads to biochar formation. Gaseous products and bio-oils that contain fine chemical compounds with high added value, such as phenol, aromatic hydrocarbons, and furans, etc., have received great attention from researchers. The current study focuses on upgrading lignocellulose from pineapple agricultural waste from Thailand's major cash crop, which accounts for 37.35 percent of the total fruit weight, and 38.78 percent of the composition of the whole pineapple plant. Cork comprises 7.77 percent, while the stem, stalks, and shoots were 12.86 percent, 3.08 percent, and 0.18 percent, respectively [Borges, Fernanda Cabral, et al., 2014]. The biomass residues from harvesting are abundant. From all of the above, it can be concluded that microwave pyrolysis is interesting because it is suitable for the conditions of agricultural waste which results after harvesting pineapple fields. It has high humidity and a wide variety of product lines can be developed into value-added products with zero waste. This research therefore tested pineapple waste in Thailand to obtain

a product line. The focus is on the production of bio-based oils and the development of processes that can be extended. Also, it covers the analysis and evaluation of other product developments derived from the process to add value for maximum benefit.

## Materials and Methods

### 1. Raw materials

Biomass samples were taken for extensive research from pineapple plantations in Uthai Thani Province, Thailand. In this study, the pineapple agricultural waste was divided into pineapple corks, pineapple leaves, and pineapple stems for investigation. Samples were initially chopped to a size of 2-3 cm, were annealed at 105 °C in a hot air oven for 24 h, and were then reduced to a smaller size of 150 µm for chemical composition analysis. Proximate analysis examined moisture, volatility, carbon and ash content. \*Test program 1) Heat from 25.00 °C to 107.00 °C; Nitrogen 2) Hold at 107.00 °C until constant mass; Nitrogen 3) Heat from 107.00 °C to 950.00 °C at 50.00 °C/min; Nitrogen 4) Hold for 7.00 min at 950.00 °C; Nitrogen 5) Heat from 600.00 °C to 750.00 °C at 3.00 °C/min; Oxygen 6) Hold at 650.00 °C until constant mass; Oxygen \*as test sample received basis. Each biomass sample has a different reaction point based on wattages of 100W, 200W, 300W, 450W, 600W, 750W and times of 4, 6, 8, 12, 16, and 20 minutes. These settings are used to study the amount of bio-oil obtained from the next sequence of microwave polarization.

### 2. Microwave process

The microwave pyrolysis oven experiment uses a multi-mode on-off type (Samsung Microwave: ME711K/XST) operating at various power levels such as 100W, 200W, 300W, 450W, 600W, and 750W with frequency 230 V / 50 Hz at 4 min, 8 min, 12 min, 16 min, 20 min. The microwave oven is hexagonal at 45 °C for good dispersion in the reaction with raw materials such as pineapple corks, pineapple leaves and pineapple plants as shown in Fig. 1. The reaction was carried out in a 250 mL round bottom quartz flask with intra-process agitator at 15 r/s, and the pyrolysis vapors were transferred through a modulator, connecting the borosilicate to the condensation system. The quartz water was placed in a cylindrical glass container with glass wool insulation to avoid heat loss caused by electric current to the condenser oven chamber, and the 150 ml collection flask was maintained at a low temperature (10 °C) by circulating coolant continuously. Teflon tape was used to seal all joints to prevent microwave leakage and to stop vapor escaping into the atmosphere. Thermocouple-based temperature measurement of the reaction mixture was carried out inside the microwave oven. The thermocouple used in this work consisted of four layers of insulation: alumina beads, Al foil, glass tube, and Al foil. Microwave interference was minimized by temperature sensors. The ungrounded configuration ensured good electricity and microwave insulation. Therefore, the temperature delivered was only for the reaction mixture during the experiment; the thermocouple was inserted into the reactor and positioned to accurately measure the temperature of the mixing reaction.

**Table 1.** Proximate analysis

Pineapple	Moisture (wt.%)	Volatility (wt.%)	Fixed carbon (wt.%)	Ash (wt.%)
Pineapple cork	2.93	75.07	16.23	5.77
Pineapple leaves	3.75	73.72	15.01	7.53
Pineapple stalk	7.15	72.67	15.03	5.15

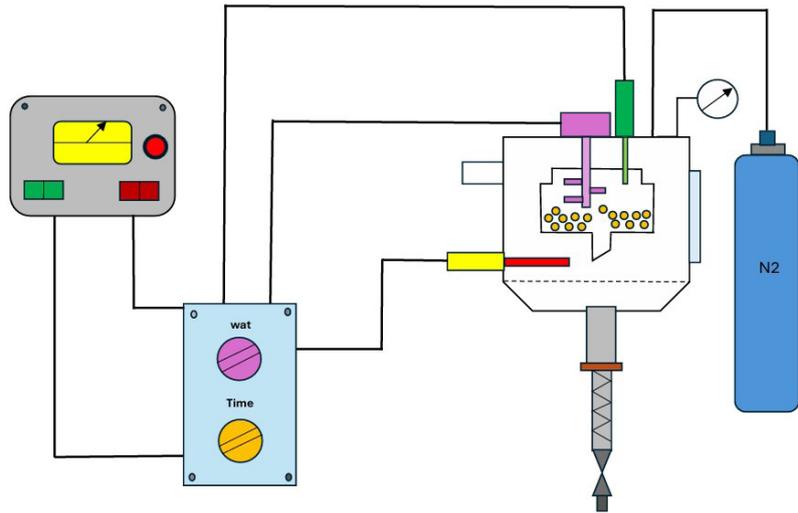


Figure 1. Microwave pyrolysis process

**3. Pyrolysis experiments**

In all experiments, the substrate was dried at 105 °C and centrifuged to 150 μm. The prepared raw material was mixed with ethyl acetate (EtOAc) to allow infiltration into the biomass to form a homogeneous mass. The moisture produced by the mixing is the waveguide of the microwave. In a 4:1 reaction, the required mass and the substance are mixed to be homogeneous. Before the start of the experiment, all systems were cleared with N<sub>2</sub> at a flow rate of 1 mL STP min<sup>-1</sup> for 10 min. The inert gas environment inside the microwave power reactor was required, and the time was set using the control panel of the pyrolysis microwave oven. The mass of charcoal and bio-oil and the mass of non-condensing gas was calculated by mass equilibrium. Bio-oil, gas, and dry charcoal yields were calculated as (mass of product) × 100/ (initial mass of dried pineapple).

**4. Selection of microwave oven walls for electromagnetic radiation**

Microwave oven walls were designed using austenitic stainless steel chemical composition. Austenitic stainless steel contains at least 10.5 percent nickel and 8 to 12 percent nitrogen, carbon, and many other elements in solution. Chromium gives the steel high corrosion resistance, while nitrogen is a hardening agent. Microwave reflection including scattering throughout the oven was assessed along with Magnetic Fields, Electrical Fields, and RF Strength using the RD-630 magnetic field leakage test instrument. This measures EMF as Electric

Field (EF), Magnetic Field (MF), and Radio Frequency (RF) Field via the 3-in-1 Multimeter Electromagnetic Radiation Sensor. Sensitivity EF: 1 V/m, MF: 0.1 mG or 0.01 uT, RF: 0.01 mW/m<sup>2</sup>. Measuring range EF: 1-1999 V/m, MF: 0.1-999.9 mG or 0.01-99.99 uT, RF: 0.01-19.99 mW/m<sup>2</sup>. Alarm threshold, Safe EF: <40 V/m, MF: < 4 mG, RF: <5 mW/m<sup>2</sup>. Alert, EF: 40~80 V/m, MF: 4~8 mG, RF: 5~10 mW/m<sup>2</sup>. Avoid, EF: > 80 V/m, MF: > 8 mG, RF: > 10 mW/m<sup>2</sup>. Measurement accuracy ± 5, bandwidth 50 Hz – 3500 MHz, Operating temperature and humidity temperature: -10 ~ +60 °C: humidity: <80% RH. The test results can display the LCD screen’s radiation value after processing by the micro-controller chip, effectively resisting electromagnetic radiation.

**5. Microwave velocity to pineapple cork, pineapple leaves, and pineapple stalk with ethyl acetate adsorbent**

To find the speed of the waves from high density to low density:

$$v = f \times \lambda \tag{1}$$

*v* is wave speed in m/s

*f* is wave frequency Hz

*λ* is wavelength m

Calculating the angle of refraction of light without specifying the medium requires the use of Snell’s law, which states that :

$$n_1 \sin(\theta_1) = n_2 \sin(\theta_2) \tag{2}$$

where: ( *n*<sub>1</sub> ) and ( *n*<sub>2</sub> ) are the refractive indices of the medium through which light passes.

**Table 2.** Measurement of microwave radiation flowing through the oven wall of austenitic stainless steel

Measurement distance 50 cm	Magnetic Fields	Electrical Fields	RF Strength
Measurement through a glass window	37.7 mG	1925 V/m	14.25 mW/m <sup>2</sup>
Measurement without passing through a glass window	7.5 mG	0.-650 V/m	13.09 mW/m <sup>2</sup>

$\theta_1$  is the angle of incidence.

$\theta_2$  is the angle of refraction.

## 6. Product characterization of bio-oil via gas chromatography-mass spectrometry (GC-MS)

Examples of bio-oil, pineapple corks, pineapple leaves, and pineapple plants obtained by microwave glorification were analyzed for bio-oil compounds using the Auto sampler models: GC Sampler 80, Agilent Technologies, USA Gas chromatograph (GC) model: 7890B, Agilent Technologies, USA Mass spectrometer (MS) model: 5977A MSD, Agilent Technologies, USA NIST/EPA/NIH mass spectral library version: 2.2 (2014). Autosampler: Mode: Liquid Injection volume: 0.2  $\mu$ L GC: Column: HP-5MS (Agilent Technologies, USA, 30 m, 0.25 mm ID., 0.25  $\mu$ m df). Injection mode: Split (split ratio 50:1). Carrier gas: He. Injection temperature: 250 °C. Flow rate: 1 mL/min. Temperature program: start 40 °C (hold 3 min) to 230 °C (hold 3 min) at rate 5 °C/min, to 300 °C (hold 5 min) at rate 10 °C/min. MS: Ion source: EI (70 eV). Mass filter: Quadrupole Mode: Full scan Mass range: 33 – 500 m/z. Interface temperature: 300 °C. Ion source temperature: 230 °C. Solvent cut time: 2.5 min. Scan speed: 1,562 u/s.

## Results and Discussion

### 1. Efficiency of microwave oven walls, austenitic stainless steel

Austenitic stainless steel was selected for the design of the microwave oven body. This type of material, austenitic stainless steel, has corrosion and high-temperature resistance. It was also found that it reflects microwave radiation uniformly along the structure under heat. As for the reflection of microwave radiation, austenitic stainless steel efficiently allows the production of high-quality bio-oil extracts when using pineapple cork, pineapple leaves, and pineapple stalk. In addition, the microwave leakage test was also per-

formed from the selection of this type of wall material, austenitic stainless steel. Table 2 shows the findings, indicating that the emission of magnetic fields reacted with three types of materials, namely pineapple cork, pineapple leaves, and pineapple stalk, showing that the Magnetic Fields were 7.5 mg, the Electrical Fields were 0 - 650 V/m, and the RF Strength was 13.09 mW/m<sup>2</sup>. The selection of austenitic stainless steel as a material for constructing the oven wall was developed by using a microwave process combined with pyrolysis. It was found that the measurement of the microwave emission emitted from the magnetic field was within the safe range. Very high RF radiation was measured with peak levels (highest measured peak values) of 354,000, 1690,000, and >2,500,000  $\mu$ W/m<sup>2</sup> compared to a peak value of 9,000  $\mu$ W/m<sup>2</sup> in a small office environment with a peak (maximum) RF radiation of 3,500  $\mu$ W/m<sup>2</sup> [Hardell, L., & Nilsson, M.,2023]. Microwave oven safety standards from the US CDRH/FDA/DHHS are presented in conjunction with consensus safety standards such as the IEEE ICES C95.1-2005 safety standard, which measures maximum allowable exposure levels for industrial applications [Barron, Dave.,2020].

### 2. The effect of the ratio on the reaction between microwave and ethyl acetate

The bio-oil production from pineapples was conducted by microwave pyrolysis at the optimum mixing ratio of 4:1 where this optimum affects the selection of the austenitic stainless steel material by calculating the refraction of the reacting microwave waves as:

( $n_1$ ) Refractive index of the first medium (In this case, it is the air ( $n_1 = 1.00$ ))

( $\theta_1$ ) The angle of incidence (90°)

( $n_2$ ) The refractive index of the second medium (ethyl acetate ( $n_2 = 1.33$ ))

( $\theta_2$ ) The angle of refraction we want to find

$$1.00 \sin 45^\circ = 1.33 \sin (\theta_2)$$

Then we can solve the equation to find ( $\theta_2$ ):

$$\sin (\theta_2) = \frac{1.00 \sin 45^\circ}{1.33}$$

as ( $\sin(45^\circ) = \frac{\sqrt{2}}{2}$ ):

$$\sin (\theta_2) = \frac{1.00 \frac{\sqrt{2}}{2}}{1.33} = \frac{\sqrt{2}}{2.133} \approx 0.53$$

$$\theta_2 = \sin^{-1}(0.53) \approx 32.1^\circ$$

Therefore, the angle of refraction of light upon entering ethyl acetate is approximately  $32.1^\circ$

### 3. Effect of ethyl acetate (EtOAc) absorption

The absorption of microwaves converting energy into heat is suitable for the example of pineapples affecting bio-oil production, bio-charcoal derived from pineapple corks, pineapple leaves, and pineapple stalks. In general, carbon-based materials such as silicon carbide (SiC), activated carbon (AC), and biochar are good microwave absorbents [Chen, Paul, et al., 2015, Merckel, Ryan David.,2015]. These absorbents are widely used in microwave pyrolysis [Shokrzadeh, Shahab, and Eric Bibeau., 2016]. The effects of using activated carbon and biochar as microwave adsorbents in biomass pyrolysis for bio-oil production were compared [Dinc, Gamze, and Esra Yel.,2018]. The activated carbon yields more bio-oil than biochar. This can be attributed to the high dielectric tangent loss in activated carbon [Chen, Paul, et al.,2015, Merckel, Ryan David.,2015]. In the case of pineapple corks, pineapple leaves, and pineapple plants in this study, the use of ethyl acetate (EtOAc) at a ratio of 4:1 as an adsorbent in place of the carbon material was compared by comparing wattage and time: 600W/6min, 450W/6min, and 450W/8min had bio-oil content of 29.6%, 32 %, and 36.8% and 14.4%, 21.6%, and 44.8% with the wattage used at 100W/16 min, 100W/20min, 200W/16min, respectively. Water was used as an adsorbent in biomass and water was used to enable the pyrolysis process to begin at temperatures below  $200^\circ\text{C}$ . Bio-oil at a feedstock-to-adsorption ratio of 10:1 was found to occur at 48% but was also presented in terms of cost of production [Mutsengerere, S., et al.,2019, Matovic, Miodrag Darko, ed.,2013]. However, the chemical ethyl acetate (EtOAc) was added to

the samples of pineapple corks, pineapple leaves, and pineapple plants. The drying process yielded a high bio-oil content to a certain extent compared to the use of carbon-containing materials and the oil composition of the group of phenolic compounds used as substrates.

### 4. Comparison of bio-oil content effects of high wattage and low wattage

Bio-oil production from agricultural residues such as pineapple corks, pineapple leaves, and pineapple plants, was found at a higher wattage range compared to reaction time. From the experiment, it can be seen that when the wattage increases, pineapple corks, pineapple leaves, and pineapple plants produce bio-oil content of 29.6%, 20.8%, and 36.8%, respectively. The bio-transfer content is 5.1%, 5.5%, and 2.8%, respectively, at 600W/6min, 600W/8min, and 450W/8min, respectively, corresponding to the reaction time of the microwave pyrolysis process. When the wattage was 750W/4min for pineapple cork and pineapple leaves there was a 17.6% and 12.8% reduction in bio-oil content, but the pineapple plants that had the same wattage difference at the reaction time showed bio-oil content at 32.8%. From the genetic structure, it was found that the emergence of the bio-oil of the pineapple stem was better because the wattage and the time used were suitable for the biofuel content. The study of low wattage compared to the bio-oil content revealed that pineapple corks, pineapple leaves, and pineapple plants produced bio-oil content via the microwave purification process of 14.4%, 24.0%, and 44.8%. Each phase of the reaction took 100W, 300W, and 200W at a reaction time of 16 min in sequence. When comparing between high and low wattage for the reaction of the microwave pyrolysis process, using a long time makes the molecules volatile, leading to better dissociation or hydrocarbon compounds as shown in Figure 2, revealing the amount of bio-oil produced in the microwave pyrolysis process.

### 5. Comparison of bio-oil content compared to volatile compounds of pineapples such as pineapple corks, pineapple leaves, and pineapple plants

Figure 3. shows the bio-oil content comparison from pineapple samples based on the experiment to

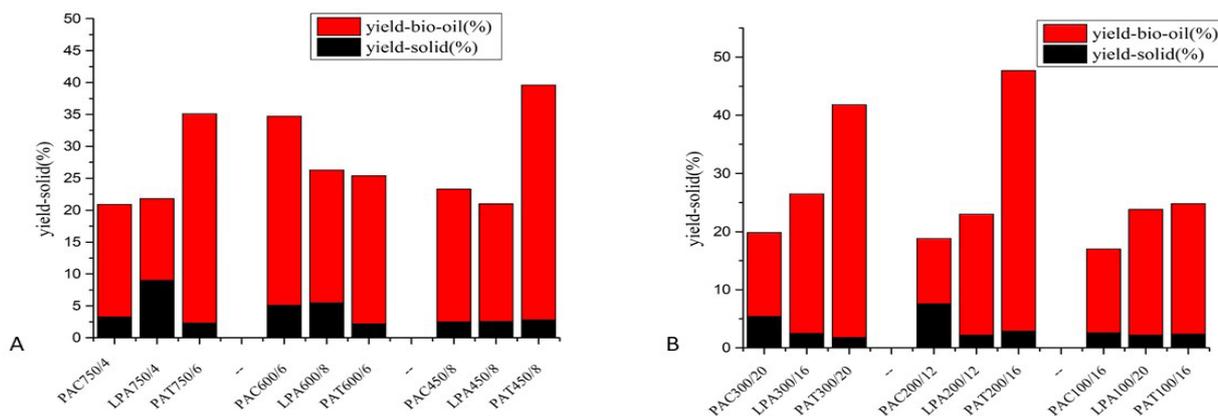


Figure 2. Comparison of wattage per bio-oil and biochar from pineapple cork, pineapple leaves, and pineapple plants (A high wattage, B low wattage)

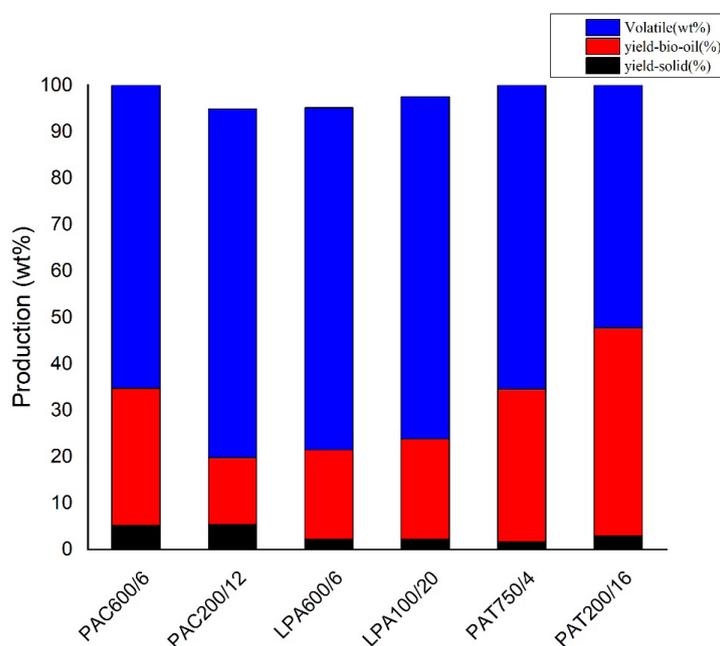


Figure 3. Comparison of bio-oil content with volatile compounds from pineapple corks, pineapple leaves, and pineapple plants

produce bio-oil from pineapple agricultural waste such as pineapple corks, pineapple stems, and pineapple leaves according to the wattage of the microwave pyrolysis machines at 100, 200, 300, 450, and 600 watts at working intervals of 16, 12, 8, 6, and 4 minutes at a ratio of 4:1 using the chemical ethyl acetate (EtOAc) as a microwave absorbent. The amount of bio-oil obtained was compared to the volatile substance of the seedling biomass of pineapple cork, pineapple leaves, and pineapple plants. It was found that the range of wattage and time of effect was as follows. In terms of low wattage and high wattage that yielded the highest bio-oil

content, pineapple cork (PAC) produced bio-oil content of 14.4% (200W/12min) and 29.6% (600W/6min). Pineapple leaves (LPA) contained 21.6% (100W/20min) and 19.2% (600W/6min) bio-oil content, but it was found that the bio-oil obtained from pineapple plants was the highest at 44.8% (200w/16min) and 32.8% (750W/4min) compared to biomass as shown in Table 1 as a volatile substance. The content of pineapple corks, pineapple leaves, and pineapple plants was 75.07% wt, 73.72% wt, and 72.67% wt, respectively, compared to the microwave pyrolysis process used in bio-oil production. The use of chemicals as a heat wave absorber of mi-

crowaves in the reaction causes molecules to dissociate as well as altering the percentage of evaporation. The amount of biochar that was obtained was proportional to the amount of bio-oil produced in the ratio of 4:1 for the next microwave planification process.

#### 6. Properties of bio-oil from pineapple corks, pineapple leaves, and pineapple plants by GC-MS

Properties of bio-oil from pineapple corks, pineapple leaves, and pineapple plants underwent GC-MS analysis by analyzing bio-oil samples. The researchers used a range of high wattages for high oil content including a low-temperature range used for the analysis of oil compounds. The main group of compounds found included acid, ester, ketone, aldehyde, phenol, benzene, and others, which are hydrocarbon compounds. Biomass-found compounds were analyzed as follows. Pineapple cork (100W/16min) contained 6 others (27.92%), acid (2.61%), ester (3.42%), ketone (25.8%), aldehyde (14.08%), and phenol (10.16%), for the microwave reaction. The most complete pyrolysis occurred in the case of pineapple leaves (600W/8min). The five main compounds were found including others (47.51%), acid (3.53%), ester (4.56%), ketone (18.32%), and phenol (9.24%), while pineapple leaves (100W/20min) formed

the same composition but differed in the number of compounds including the pineapple tree (200W/16min) and the reaction range of 750W/6min. Five compounds were formed but differed from acid, benzene, and ketone which were not found in bio-oil processed by microwave pyrolysis. However, it was also found that at high wattage, some compounds in the bio-oil were not found at 600W/16min. Compounds were found as 4 others (42%), acid (18.34%), phenol (9.65%), and benzene (6.37%), respectively, in comparison with the others. It was the least found as shown in Figure 4. A total of seven main compounds were found in pineapple corks, pineapple leaves, and pineapple plants. These were phenol and other compounds, followed by ketones, respectively.

#### Conclusion

This research concerns the design and construction of a microwave pyrolysis procedure for processing pineapple agricultural waste in bio-oil production, and testing to find the most suitable conditions for that production.

1. The microwave pyrolysis process at the optimum conditions for bio-oil production at the best

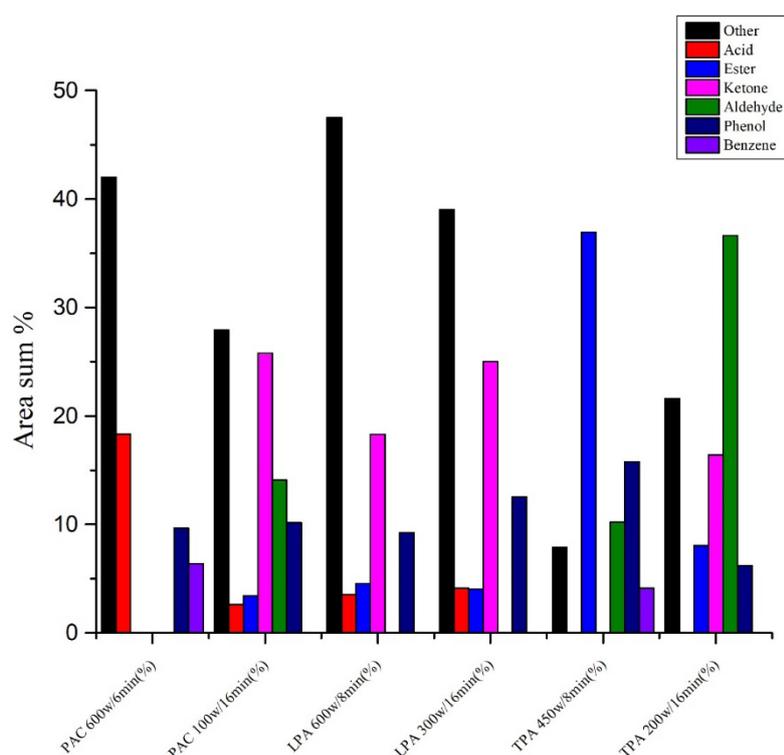


Figure 4. Bio-oil compounds by GC-MS method

high-low wattage conditions, for pineapple cork at 600W/6min and 100W/16min revealed that the total oil content was 29.6% and 14.4%. Ketone bio-oil composition of 25.8% and acid at 18.34% were found in large quantities in the bio-oil.

2. The microwave pyrolysis process at the optimum conditions for bio-oil production at the best high-low wattage conditions, for pineapple leaves at 450W/6min and 100W/20min showed that the oil content was 32.00% and 21.60%. Ketone groups in the bio-oil composition at 25.01% and 18.32% were found in large quantities in the bio-oil.

3. The microwave pyrolysis process at the optimum conditions for bio-oil production at the best high-low wattage conditions, for pineapple stems at 450W/8min and 200W/16min revealed the percentage of oil content at 36.80% and 44.80%. Aldehyde bio-oil composition of 36.62% and ester of 36.92% were found in large quantities in the bio-oil.

4. Wattage and reaction time affect the microwave pyrolysis process. The effect on the composition of the bio water produced was compared to the initial biomass of volatile matter to the experimental ratio of 4:1 and the use of ethyl acetate (EtOAc) as a microwave absorber. This causes a reaction involving heat which takes place with the biomass.

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