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Synthesizing the biochar from Gelam tree leaves by pyrolysis method

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KEYWORDS

Melaleuca Cajuputi leaves
Biochar
Pyrolysis
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Thermal stability

ABSTRACT

Melaleuca Cajuputi or known as Gelam Tree is widely used in the folk medicine of South-Eastern Asia. It also has medicinal and food properties. This study aims to determine the effect of pyrolysis temperature on the obtained biochar yield and the thermal stability. In synthesizing the *Cajuputi* biochar, *Melaleuca Cajuputi* leaves as the raw materials, went pyrolysis process at different temperatures; 300 °C ,400 °C and 500 °C. The maximum yield of biochar was obtained from pyrolysis at 300 °C and the least yield was exhibited from pyrolysis at 500 °C. The obtained biochars were characterized by using Fourier Transform Infrared Spectroscopy (FTIR). The spectra showed the presence of O-H, -COOH, C-O and O=C=O functional groups. The TGA analysis showed that the MCB 500 °C gave the highest thermal stability and residue obtained after the experiment was 49.96 %.

1.0 INTRODUCTION

Biopolymers are produced by living organisms. Shrimp shells, crabs and other living organisms in the sea are the most organic biodegradable chemical substances in the environment. The two main types of biopolymers are natural and synthetic biopolymers. Synthetic biopolymers are classified into two types: nondegradable and degradable. Natural polymers can be modified to satisfy specific requirements. Biopolymers are either naturally occurring substances derived from animals, plants, bacteria, and fungi, or chemically synthesized polymers containing biological elements such as sugars, amino acids, oils, or natural lipids (Tamang et al., 2022). Biopolymers such as glycolic acid, starch, lactic acid, cellulose, and others might be used to make biodegradable products (Wankhade et al., 2020). *Melaleuca Cajuputi*, a tree species belonging to the "Myrtaceae" family, is a significant natural tree on Malaysia's east coast, particularly in the state of Terengganu and is known as Gelam tree by the local people. The economic significance of *Melaleuca* forests has been extensively discussed,

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including its ability to provide timber for construction and furnishings and other traditional applications such as fuelwood, charcoal, tea tree oil, and honey (Masitah et al., 2014). *Melaleuca Cajuputi* leaves extract has also been widely utilized for decades in various uses, most notably medicinally, throughout Southeast Asia (Abdullah et al., 2018; Kueh et al., 2019). It is used to treat a variety of conditions, including coughs and colds, asthma and other respiratory infections, headaches, rheumatism, and convulsions, as well as toothache and earache relief (Desdiani, 2022). Due to its antibacterial and preservation effects, it is also used as a scent ingredient in medicinal and cosmetic goods (Wahab et al., 2022).

Biochar is a type of charcoal that is used as a soil supplement for both carbon sequestration and soil health advantages. Biochar is a stable carbon-rich substance that may endure thousands of years in the soil (Rawat, 2018). Biochar, like the majority of coal, is produced through the pyrolysis of biomass. Biochar is being studied as a possible method of carbon sequestration, as it can help reduce global warming and climate change. Biochar can improve the fertility of acidic soils (low pH soils), boost agricultural production, and provide protection against some foliar and soil-borne infections (El-Naggar, 2019). In terms of development, the International Biochar Initiatives defines biochar as the solid substance formed by the thermochemical conversion of biomass into an oxygen-limited environment (International Biochar Initiative, 2015). In the waste management industry, landfill top coverings are commonly called "bio-covers" since they improve environmental conditions for methanotrophic bacteria, increase the consumption of biotic methane, and function as huge bio-filters (Huber-Humer et al. 2009).

The current study was purposely to synthesize the *Melaleuca Cajuputi* biochars (MCB) at different temperatures. Thereafter, the biochars were characterized by FTIR to confirm the obtained compounds.

2.0 EXPERIMENTAL PROCEDURE

Melaleuca Cajuputi leaves were washed using tap water and were dried in the oven at 75 °C. It was grounded and sieved with 5 mm in size. Furthermore, the *Melaleuca Cajuputi* powder was placed in the oven for pyrolysis at temperatures of 300 ºC, 400 ºC and 500 ºC for 2 hrs. The schematic figure in biochar preparation is shown in Figure 1. The produced sample was weighed to determine the solid yield of MCB. The % of yield was calculated by using Equation 1. Fourier-Transform Infrared Spectroscopy (FTIR) was utilized to determine the MCB functional group. The KBr disc was prepared with each containing 1.5 mg of sample and 200 mg of KBr with an IR range of 4000–400 cm^{-1}. Thermogravimetric analysis (TGA) was carried out using a TGA/SDTA851e/LF1100/MT1/698/ (METTLER). The scan range was from 40 °C to 550 °C at a constant heating rate of 10 K/min and continuous nitrogen flow as dry gas at 100 ml/min.

$$\frac{W_f}{W_i} \times 100\% \qquad \text{Equation 1}$$

Where; Wf = final weight, Wi = initial weight

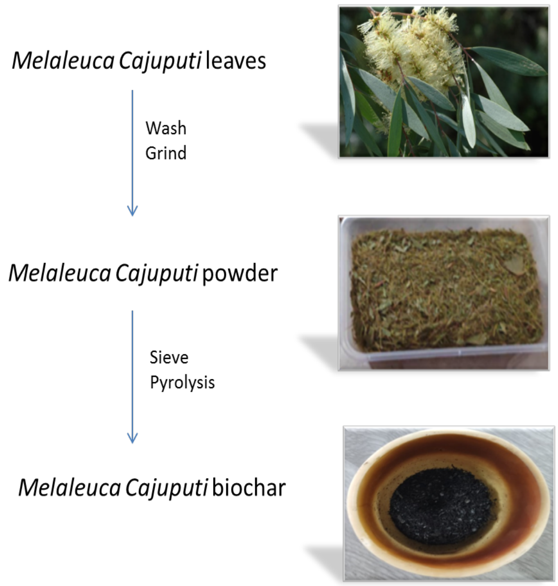

Figure 1: Schematic route of MCB preparation.

3.0 RESULTS AND DISCUSSION

All MCB synthesized were recorded in Table 1. The same weight of samples was used, 128g. The pyrolysis time for each temperature was the same. The lowest yield of biochar was shown by pyrolysis sample at 500 ^{o}C after hrs, which is 10 g, contributing to 7.8 % of yield. This is due to the loss of high water content from leaves at higher temperatures (Justina et al., 2017). The pyrolysis at 300 ^{o}C and 400 ^{o}C showed a biochar mass of 42 g (32.8 % of yield) and 24 g (18.8 % of yield), respectively. For feedstock to be converted into biochar of consistent quality and output, knowledge of the feedstock is necessary. This is because depending on the kind of feedstock made of multiple components having a distinct physicochemical characteristic, the pyrolytic product will be affected by the energy conversion efficiency of pyrolysis (Weldekidan et al. 2019).

Pyrolysis is the thermal decomposition in a limited oxygen environment, of biomass into a carbon-rich solid residue (char, gases and liquids). The thermochemical conversion of biomass by pyrolysis is a well-established method for producing high-value-added materials including biochar, bio-oil, and syngas. Depending on the target substance, pyrolysis can be divided into distinct categories such as torrefaction, hydrothermal carbonization (HTC), slow pyrolysis, quick pyrolysis, and flash carbonization with varying reaction conditions (Pandey et al. 2020). The most common thermochemical conversion to produce high-yield biochar as the primary product is slow pyrolysis. Long reaction times are a drawback of slow pyrolysis, though. Reports of torrefaction processes that operate at lower operating temperatures than traditional pyrolysis have been on the rise recently, and attention is now being drawn to a technique for creating biochar from HTC utilizing wet feedstock (Chun et al., 2021).

In the pyrolysis process, several steps are reported. Carbonization emphasizes carbon enrichment, as opposed to breakdown, and is often used interchangeably with pyrolysis. At 110-180 ^{o}C, the biomass starts to soften and chemically-bound water starts to be driven off. Torrefaction is a chemical process that takes place at a temperature of approximately 180-300 ^{o}C which produces a more energy-dense, stable, sterile feedstock or soil amendment. The activation process refers to further enhancement of charcoal via chemical processes and/or higher temperature oxidation to produce activated carbon with high microporosity and surface area.

The functional group present in MCB was determined by Fourier Transform Infrared Spectroscopy (FTIR) analysis. Surface functional groups on biochars are one of the key features which determine biochar properties and their potential applications. Figure 2 shows the FTIR spectra for *Melaleuca Cajuputi* after the pyrolysis process, thereby revealing the function of the group's presence.

Based on the FTIR spectrum, carboxyl group presence in three different temperatures of MCB with the wavenumber of 1205–1124 cm^{-1} (C–O). Carboxyl functional groups are known to be effective in adsorbing/stabilizing several contaminants, such as heavy metals (Uchimiya et al., 2012), pharmaceuticals and others (Guedidi et al., 2017).

The alcohol presence decreased when the pyrolysis temperature was increased due to the thermal destruction of cellulose, hydroxyl group and aliphatic groups. There is a sharp intensity absorption in the absorption areas at the wave number of 3700–3584 cm^{-1} (O–H) will allow the compound to contain oxygen-related groups, such as alcohol or phenol.

A functional group for carboxylic acid also has been found at the wavenumber of 1440–1395 cm^{-1} (–COOH). Carboxylic acids can functionalize the biochar surface (Lonappan et al., 2020; Sun et al., 2015). Moreover, apart from adsorption, biochar with the copious presence of carboxylic groups on the surface can be effectively used for various other applications including catalysis, biochar-supported nanostructures, and energy storage, among others (Liu et al., 2015). Stretching vibration of O=C=O (CO_2) with wavelength 2400–2300 cm^{-1} was observed.

Table 1: Yields obtained after the pyrolysis process at different temperatures.

Temperature (℃)	**Weight of MC leaves before pyrolysis (g)**	**Average weight of MCB after pyrolysis (g)**	**Yield (%)**
300	128	42.0 g	32.8
400	128	24.0 g	18.8
500	128	10.0 g	7.8

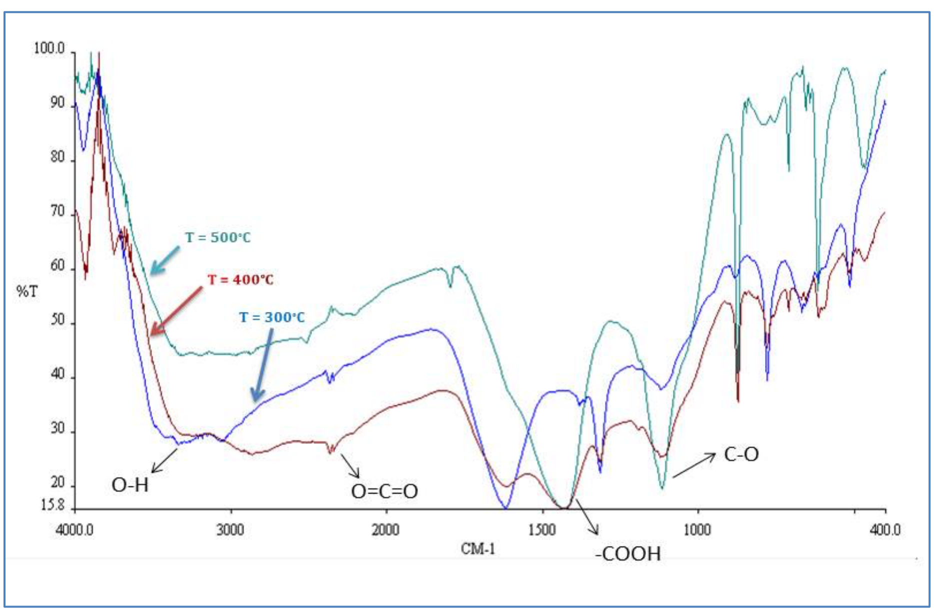

Figure 2: FT-IR spectra of *Melaleuca Cajuputi* biochar synthesized at different temperatures.

Figure 3 shows the degradation behavior of the biomass. There are two stages present, which are the first stage due to the limitation of moisture content and the small amount of volatile matter compound (Mansor, 2017). The second stage is a sign to the plant biomass of the cellulose, hemicellulose and lignin degrade. At the first stage of analysis, MCB 300 °C shows a fast degradation as early as at a temperature of 46.4 °C. Meanwhile, MCB 400 °C exhibits degradation at 96.0 °C. As expected, MCB 500 °C presents the first degradation at a temperature of 128.0 °C. The trend is due to the temperature used for the sample preparation. The higher temperature used means more moisture content is already lost during pyrolysis. The mass keeps decreasing when the second degradation stage starts, at 300 ^{o}C for MCB 300 °C and at 350 ^{o}C for MCB 400 °C, respectively. MCB 500 °C shows the late second degradation which is at 370 ^{o}C. The removal of extractives and lignin compounds affects the thermal stability of the materials, probably the increasing porosity (Maia et al., 2014).

Furthermore, after the experiment temperature of 350 °C, all of the biochar show the mass amount of residue. The residue obtained for MCB 300 °C, MCB 400 °C and MCB 500 °C is 6.92 %, 26.89 % and 49.96 %, respectively. The TGA results confirmed the high stability of the MCB 500 °C sample.

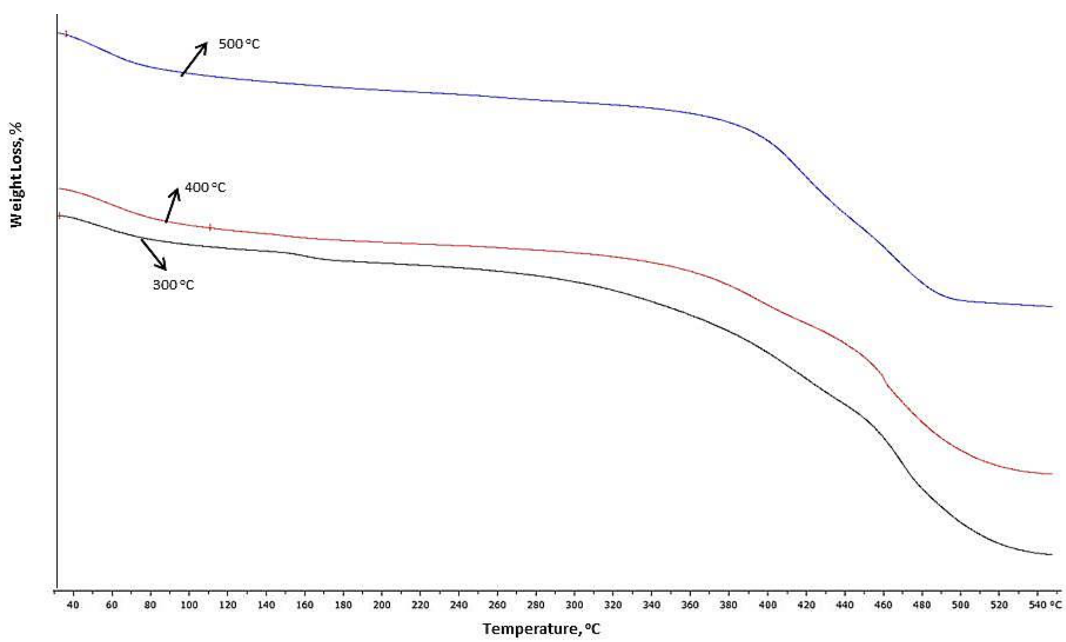

Figure 3. TGA analysis of MCB at different temperatures

4.0 CONCLUSION

The *Melaleuca Cajuputi* biochar was successfully synthesized via pyrolysis process at three temperatures; 300 °C , 400 °C and 500 °C. The minimum yield of the biochar, 7.8 % was obtained from the maximum temperature used, 500 °C; due to the higher moisture loss. The maximum yield of 32.8 % was found from the 300 °C MCB. The functional groups of C-O, O-H, -COOH and O=C=O were examined from the biochars. The thermal analyses proved that the MCB 500 °C gave the highest thermal stability which the first degradation taking place at 128 °C while the second degradation was at 370 °C. The residue obtained after the experiment was 49.96 %.

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