Extraction and characterization of fulvic acid from oil palm empty fruit bunches

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Abstract

Oil palm empty fruit bunches (OPEFB) contain 27.78% lignin, which can serve as an alternative source of renewable fulvic acid, alongside soil and coal. This study aims to conduct the fulvic acid extraction process using a microwave extractor. The extraction process was carried out using H_2O_2 solvents with concentrations of 18, 21, and 24% and various sample types (OPEFB, shilajit, and commercial fulvic acid fertilizer), with three repetitions. The resulting liquid fulvic acid extract was then made into powder using freeze-drying. Quantitative testing was conducted using the spectrophotometric method, while qualitative testing employed Fourier-Transform Infrared Spectroscopy (FTIR), Proton Nuclear Magnetic Resonance (^1H NMR), spectrofluorometry, a CHN analyzer, and Thermogravimetric Analysis - Differential Scanning Calorimetry (TGA-DSC). The results showed that the best solvent concentration for the fulvic acid extraction process was H_2O_2, 21% of the total in the OPEFB sample. The highest fulvic acid content was found in the OPEFB sample at 23.59%; in the shilajit sample, it was 9.62%, and in the commercial fulvic acid fertilizer sample, it was 6.97%. The characterization results from spectrophotometric analysis, elemental analysis, and TGA-DSC analysis showed the potential of fulvic acid in the OPEFB sample, as it exhibited similarities with the analysis results of commercial fulvic acid (shilajit & commercial fulvic acid fertilizer).

[Keywords: CHN Analyzer, FTIR, HNMR, microwave extractor, TGA-DSC]

Introduction

Oil palm plantations produce a byproduct of empty oil palm bunches (EFB), reaching 3,558.9 tons per month (Januari & Agustina, 2022). EFB contains 27.78% lignin (Faramitha et al., 2024). The lignin contained in EFB can be a renewable source of fulvic acid, alongside coal, soil, and manure (Mindari et al., 2022). This aligns with the ligno-protein theory, which states that lignin plays a role in forming humic and fulvic acids (Tan, 2010).

Fulvic acid is a component of humic compounds, abundant in soil, water, coal, peat, lignite, and lignin through decomposition (Tan, 2010; Schellekens et al., 2017; Doskočil et al., 2018). Fulvic acid often occurs together with humic acid and humin in humus soil, which is known as a humic substance. Humic soil results from the decomposition of organic compounds found naturally with the help of microbes. Fulvic acid has low aromaticity, a small molecular size, numerous functional groups, and good water solubility. Among humic substances, fulvic acid has the highest chemical, physiological, and physicochemical activity.

Several studies have demonstrated the potential of fulvic acid as a growth stimulant for plants & vegetables (Zhang et al., 2021), coffee (Justi et al., 2019), and tobacco (Moradi et al., 2019). Fulvic acid application to soil can significantly increase phosphorus availability. Mao (2019) reported that the application of fulvic acid to livestock can improve growth, meat composition, and immunity in broiler chickens.

Various methods for extracting fulvic acid have been extensively studied, such as acid-base precipitation, a combination of sulfuric acid and

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ethanol/acetone, oxidative degradation, and extraction with strong acids. However, these methods are corrosive and produce small amounts of pure fulvic acid. Meanwhile, the ion-exchange resin method has low extraction efficiency and is difficult to implement (Zhang et al., 2018). This study used a microwave extraction method as a more environmentally friendly alternative. The solvent used was hydrogen peroxide, which promotes the formation of free radicals to accelerate oxygen degradation, thereby increasing extraction efficiency and shortening reaction time (Zhang et al., 2020). This combination increased the yield of fulvic acid extract in lignite by up to 4% compared to hydrogen peroxide alone, without the use of a microwave extractor.

Research on the extraction of fulvic acid from natural materials using microwave extractors has so far been limited to mineral rocks such as shilajit (Javed et al., 2013; Khan et al., 2022), lignite (Zhang et al., 2018), low-quality lignite (Gong et al., 2020; Zhang et al., 2020), hamilignite (Zhang et al., 2018), and marine sediments (Zhang et al., 2016). Meanwhile, research on the fulvic acid content in OPEFB has rarely been studied in Indonesia, especially using microwave extractors. To date, research on the extraction of fulvic acid in OPEFB has only been conducted by Dimawarnita et al. (2024) to find the optimum conditions for the fulvic acid extraction process in OPEFB using a microwave extractor. However, in this study, variations in concentration under the optimum conditions for using a microwave extractor (solvent volume, extraction time, and microwave power) have not been thoroughly investigated. Therefore, further studies on this topic are needed to strengthen the existing research findings.

Commercial products, namely shilajit and fulvic acid fertilizer, were used as comparisons. Shilajit is a mineral known as a natural source of fulvic acid, found only in specific mountain ranges. In contrast, fulvic acid fertilizer offers several advantages in agriculture, as reported by He et al. (2022). In this study, fulvic acid will be extracted from OPEFB using a microwave extraction method with H_2O_2 as the solvent, following the optimum conditions for microwave extraction reported by Dimawarnita et al. (2024).

The obtained data will be analyzed using analysis of variance (ANOVA) to determine the effect of each factor and its interactions. Further testing using the Tukey test will then be conducted to determine the best treatment. The characterization of fulvic acid will be referenced to Gong et al. (2020) and Zhang et al. (2020), utilizing a spectrophotometer, FTIR, spectrofluorometer, HNMR, CHN analyzer, and TGA-DSC.

Materials and Methods

Materials

The tools used in this study were a microwave extractor, condenser, vacuum pump, separatory funnel, glass beaker, Erlenmeyer flask, and measuring cylinder. The materials used were 1-2 cm dry empty oil palm bunches containing 27.78% lignin and technical hydrogen peroxide (H_2O_2) with a purity of 95%.

Extraction of fulvic acid from OPEFB

Fulvic acid extraction was performed using the method described by Dimawarnita et al. (2024), with variations in H_2O_2 concentration and sample type. Each combination of variations will be repeated three times, as shown in Table 1. Samples (OPEFB, shilajit, and commercial fulvic acid fertilizer) that were dried at 80°C for 24 hours were extracted using optimum conditions (Figure 1). The extracted results were then collected in a 50 mL Falcon tube, up to a volume of ±25 mL, and stored in a freezer at -80°C. Next, the samples were dried using the freeze-dry method at -80°C using a Labconco FreeZone Benchtop freeze dryer for 48-72 hours. The freeze-dried fulvic acid results were then stored in an airtight container at -20°C.

Table 1. Experimental design for fulvic acid extraction using a microwave extractor

Sample	H_2O_2 concentration (%)		
	18 (a)	21 (b)	24 (c)
OPEFB (T)	Ta	Tb	Tc
Shilajit (S)	Sa	Sb	Sc
Fulvic acid fertilizer (P)	Pa	Pb	Pc

OPEFB= Oil palm empty fruit bunches

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┌─────────────────────────────────────────────────────────┐
│   1 g of sample (OPEFB with a size 1-2 cm, Shilajit,     │
│          commercial fulvic acid fertilizer)              │
└─────────────────────────────────────────────────────────┘
                            ⬇
┌─────────────────────────────────────────────────────────┐
│              Add 35 mL of 18-24% H₂O₂                    │
└─────────────────────────────────────────────────────────┘
                            ⬇    Constant stirring at microwave extractor with
                                 300W microwave power for 10 minutes
┌─────────────────────────────────────────────────────────┐
│            Mixture of crude FA and sample solution       │
└─────────────────────────────────────────────────────────┘
                            ⬇    Separated residue from mixture solution
┌─────────────────────────────────────────────────────────┐
│            FA solution colored yellow to orange          │
└─────────────────────────────────────────────────────────┘
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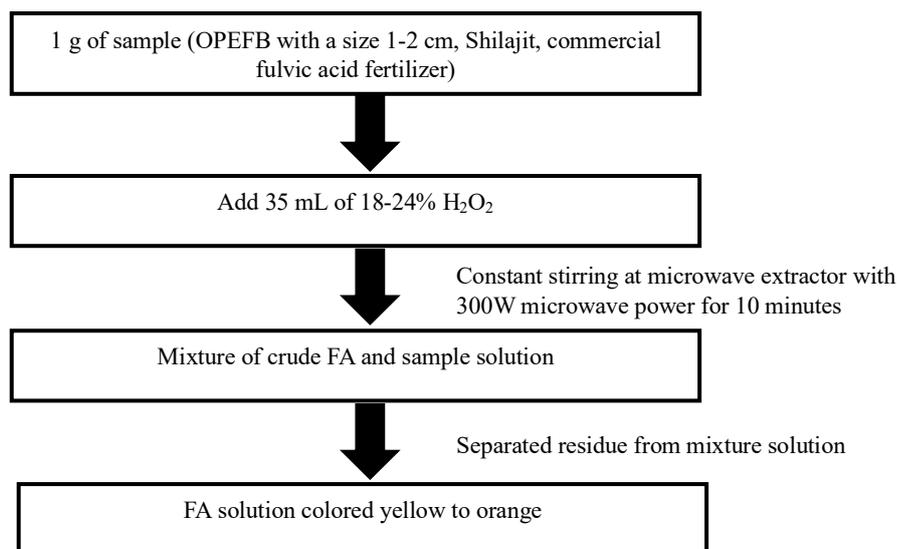
Figure 1. Flowchart of fulvic acid extraction stages using a microwave extractor

Spectrophotometry

Fulvic acid levels were analyzed using a spectrophotometric method, as described by Gan et al. (2007). HCl was added to the sample until the pH was below two, and humic acid precipitate formed. Then, the supernatant was separated and pipetted into a 100 mL volumetric flask, where it was dissolved in distilled water and calibrated. The samples were measured using a spectrophotometer at wavelengths of 350, 370, 400, 450, and 500 nm, respectively. The measurement results were calculated to obtain the percentage unit levels by averaging the levels from 5 wavelengths.

Fourier-Transform Infrared Spectroscopy (FTIR)

A 0.5 g powder sample was prepared in an airtight container. The sample was then analyzed using a Thermo Scientific Nicolet iS 5 FTIR-ATR instrument with a wavelength range of 4000-400 cm^{-1}. The reading results were presented in the form of an FTIR spectrum, expressed in units of wavelength (cm^{-1}).

Proton Nuclear Magnetic Resonance (HNMR)

A 0.5 g powder sample was prepared in an airtight container. The sample was then dissolved in DMSO-d6 solvent and analyzed using an Agilent HNMR instrument with a DD2 console system operating at 500 MHz (^1H) and 125 MHz (^{13}C). The reading results are in the form of an HNMR spectrum in ppm (parts per million) for protons, and their energy axes are called δ (delta) or chemical shifts. Generally, signals for organic compounds range from 0 to 12 ppm.

CHN *Analyzer*

A 0.001 g sample of powder was weighed in a tin foil. The sample was then formed into tablets using a tablet press. The sample was then measured using a Vario El Elementar brand CHN analyzer. Acetanilide was used as the standard for the CHN test. The measurement results included data on %C, %H, %N, area C, area H, area N, C/N ratio, and C/H ratio.

Thermogravimetric Analysis - Differential Scanning Calorimetry (TGA-DSC)

A 0.005-0.010 g sample of powder was weighed and placed in a plate furnace. The sample was then measured using a Linseis Simultaneous Thermal Analyzer. The measurement results were in the form of a graph plotting the relative mass change (%) and heat flow (mW) against temperature (°C). The sample was measured at a temperature range of 1000 °C, using nitrogen gas flow and a heating rate of 10 °C/minute.

Results and Discussion

Fulvic acid content

Fulvic acid extract liquid samples were quantitatively analyzed using a spectrophotometer. Fulvic acid extraction was performed using optimum microwave reactor conditions for the OPEFB sample, as described by Dimawarnita et al. (2024), namely, 300W microwave power, 10 minutes extraction time, and 35 mL reagent volume. Fulvic acid extraction used two variables: sample type and hydrogen peroxide solvent concentration. Fulvic acid extraction was repeated

3 times for each variation. The results of the fulvic acid content measurements are shown in Figure 2.

Based on the graph in Figure 2, the OPEFB sample with 21% H_2O_2 had the highest fulvic acid content, reaching 23.59%. Meanwhile, the lowest fulvic acid content was 6.20% for the commercial fulvic acid fertilizer sample with 24% hydrogen peroxide (H_2O_2). According to Gong et al. (2020), the most influential factors in the microwave extractor extraction process are H_2O_2 concentration, microwave power, and reaction time. When the H_2O_2 concentration is too high, the resulting •HO and O^{2-} will oxidize and decompose the resulting fulvic acid, decreasing yield.

Fulvic acid levels of various sample types and H_2O_2 solvent concentrations, as tested by ANOVA and Tukey's follow-up test, are presented in Table 2. Based on the results of this analysis, the Tb treatment (23.59%) was significantly different from Ta and Tc (6.43% and 6.56%). In the shilajit sample, the Sa treatment (9.62%) was not significantly different from Sb and Sc (9.00% and 8.15%). In the

commercial fulvic acid fertilizer sample, the Pa treatment (6.97%) was not significantly different from Pb and Pc (6.72% and 6.20%). Overall, the 21% H_2O_2 concentration in OPEFB produced the highest fulvic acid levels, while shilajit produced higher fulvic acid levels than commercial fulvic acid fertilizer at all H_2O_2 concentration treatments. Fulvic acid levels in the Tb treatment were higher than in Ta and Tc. This may be due to the influence of oxidation mechanisms and free radicals produced by H_2O_2, namely hydroxyl radicals (•OH), under certain conditions. The selective nature of H_2O_2 in degrading lignin in a sample should indicate a correlation between H_2O_2 concentration and sample type. Increasing H_2O_2 concentration will further reduce the lignin content in the sample (Zuidar et al., 2014). At too low a concentration, the number of radicals produced is insufficient to oxidize lignin, which is the source of fulvic acid. At high concentrations, the abundance of free radicals can lead to nonspecific degradation, thereby reducing oxidation efficiency in the sample (Ristiawan & Syafila, 2015).
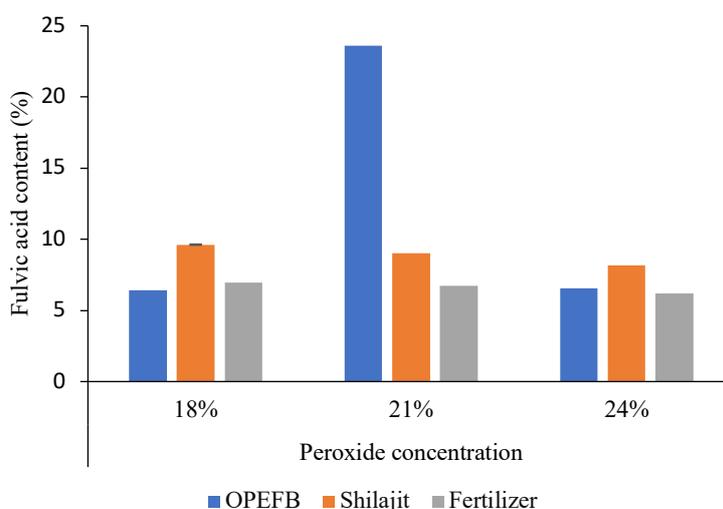

Figure 2. Fulvic acid level (%)

Table 2. Fulvic acid levels from ANOVA analysis, followed by the Tukey test

Sample	Fulvic acid content (%)		
	H_2O_2 18% (a)	H_2O_2 21% (b)	H_2O_2 24% (c)
OPEFB (T)	6.43[cd]	23.59[a*)]	6.56[cd]
Shilajit (S)	9.62[b]	9.00[b]	8.15[bc]
Fulvic acid fertilizer (P)	6.97[cd]	6.72[cd]	6.20[d]

[*)]Note: Numbers followed by the same letter indicate no significant difference in the Tukey test at the 0.05%.

OPEFB= Oil palm empty fruit bunches

Different sample types can also affect the levels of fulvic acid produced. The OPEFB sample, known for its high lignin content, produced the highest fulvic acid content at 23.59%. This result is still very far when compared with the source of fulvic acid originating from lignite, namely 60.97% (Zhang et al., 2020) and 30.55% (Gong et al., 2020). Meanwhile, the highest fulvic acid levels in shilajit samples and commercial fulvic acid fertilizers were only 9.62% and 6.97%. Lignin is a compound that forms fulvic acid based on the ligno-protein theory (Tan, 2010), so that with the use of selective oxidants such as hydrogen peroxide and optimum extraction conditions using a microwave extractor, the fulvic acid levels produced by OPEFB samples may be much higher than commercial products such as shilajit and commercial fulvic acid fertilizers.

FTIR Analysis

The results of the FTIR analysis in the form of a comparison graph between % transmittance and wavelength (cm^{-1}) for the fulvic acid extract of OPEFB, shilajit, and commercial fulvic acid fertilizer samples can be seen in Figure 3. The wave numbers used are 4000–400 cm^{-1} (fingerprint area). The three samples exhibit the five strongest peaks in the wavenumber region of 3200 cm^{-1}, 2800 cm^{-1}, 1600 cm^{-1}, 1380 cm^{-1}, and 1100 cm^{-1}. A strong vibration in the wave number range of 3500 cm^{-1} in each sample indicates the presence of the O-H group. According to Zhang et al. (2020), there is also a strong peak from the O-H group at the wave number of 3423 cm^{-1}, and there is also a strong peak at (Joordan, 2019Gong et al., 2020;) at the wave number of 3400-3220 cm^{-1}. This occurs because H$_2$O$_2$ was used as a solvent to reduce the noise generated by the sample solution.

According to Gong et al. (2020), vibrations in the 2800 cm^{-1} wavenumber region are attributed to alkyl (C-H) stretching from aliphatic hydrocarbons (open chains). This is supported by Jordaan (2019), who states that fulvic acid exhibits vibrations at wavenumbers 2940–2840 cm^{-1}, originating from the C–H bonds of the aliphatic hydrocarbon chain. Meanwhile, at wavenumbers 2900 cm^{-1}, a peak appears in the OPEFB and shilajit samples, which can originate from the alkene group (C=H) or the aldehyde group (H-C=O) (Gong et al., 2020). The next peak is at 1600 cm^{-1}, found in all three samples, which may originate from the vibration of the C=C bond in the aromatic group (Asemani, 2020). This vibration also appeared in Jordaan's (2019) research, where a peak was observed at wave numbers 1620-1600 cm^{-1}, originating from the aromatic ring of the C=C bond or the H-C=O bond found in conjugated ketones.

According to Gong et al. (2020), the wavenumber range of 1900-1000 cm^{-1} corresponds to the vibration of oxygen-containing functional groups. Zhang et al. (2020) also observed a vibration at a wavenumber of 1646 cm^{-1}, originating from the C=C bond. Then, a peak at wave number 1380 cm^{-1} was found in all three samples, which likely originates from the vibration of methyl or methylene groups (Zhang et al., 2020). Additionally, a peak is observed at a wavenumber of 600 cm^{-1}, which is exclusive to commercial fulvic acid fertilizer samples. According to Jozanikohan & Abarghooei (2022), the peak at this wavenumber may originate from Si-O stretching, which may be present in the fulvic acid fertilizer samples. Meanwhile, this peak does not appear in the OPEFB and Shilajit samples. This indicates the presence of silicate content, which is commonly found in fertilizer products. All three samples exhibited similar peaks. There was only a slight difference between the fertilizer and other samples, namely a peak at 600 cm^{-1}, indicating Si-O stretching. The similarity of the peaks at these wavelengths suggests that the extract from the OPEFB sample does indeed contain fulvic acid, as do the other commercial products.

HNMR Analysis

HNMR analysis of fulvic acid extract samples from OPEFB, shilajit, and commercial fulvic acid fertilizers used DMSO as a solvent in the analysis process. In the resulting spectrum, the DMSO and water peaks appeared as chemical shift peaks at 2.5 ppm and 3.4-3.5 ppm, respectively, as shown in Figure 4. The 3.3-4.5 ppm peak arises from hydrogen bonds to heteroatoms such as O, N, and S (Zhang et al., 2020). Additionally, a chemical shift peak is observed at 10.19-10.22 ppm in all three samples. This signal originates from protons from carboxylic acid groups (-COOH) detected at 10-13 ppm (Gunawan & Nandiyanto, 2020). This signal arises due to the abundance of carboxylic acid groups (-COOH) in fulvic acid, which is relatively abundant, as indicated by elemental analyzer characterization (Zhang et al., 2020).

In contrast to the OPEFB samples and commercial fulvic acid fertilizer, the spectrum produced by the shilajit sample (Figure 4b) exhibited various chemical shift peaks, including those in the ranges of 4.57-5.52 ppm, 7.40-7.92 ppm, and 10.21 ppm. The chemical shift peak in the 4.57-5.52 ppm range could originate from the chemical shift produced by the -OH group. This broad chemical shift can be attributed to the induction effect of electronegative atoms, such as oxygen, a phenomenon known as deshielding (Gunawan & Nandiyanto, 2021). Furthermore, a peak with low intensity appears at the chemical shift range of 7.40-7.92 ppm, representing the aromatic group contained in the fulvic acid compound (Gunawan & Nandiyanto, 2021).
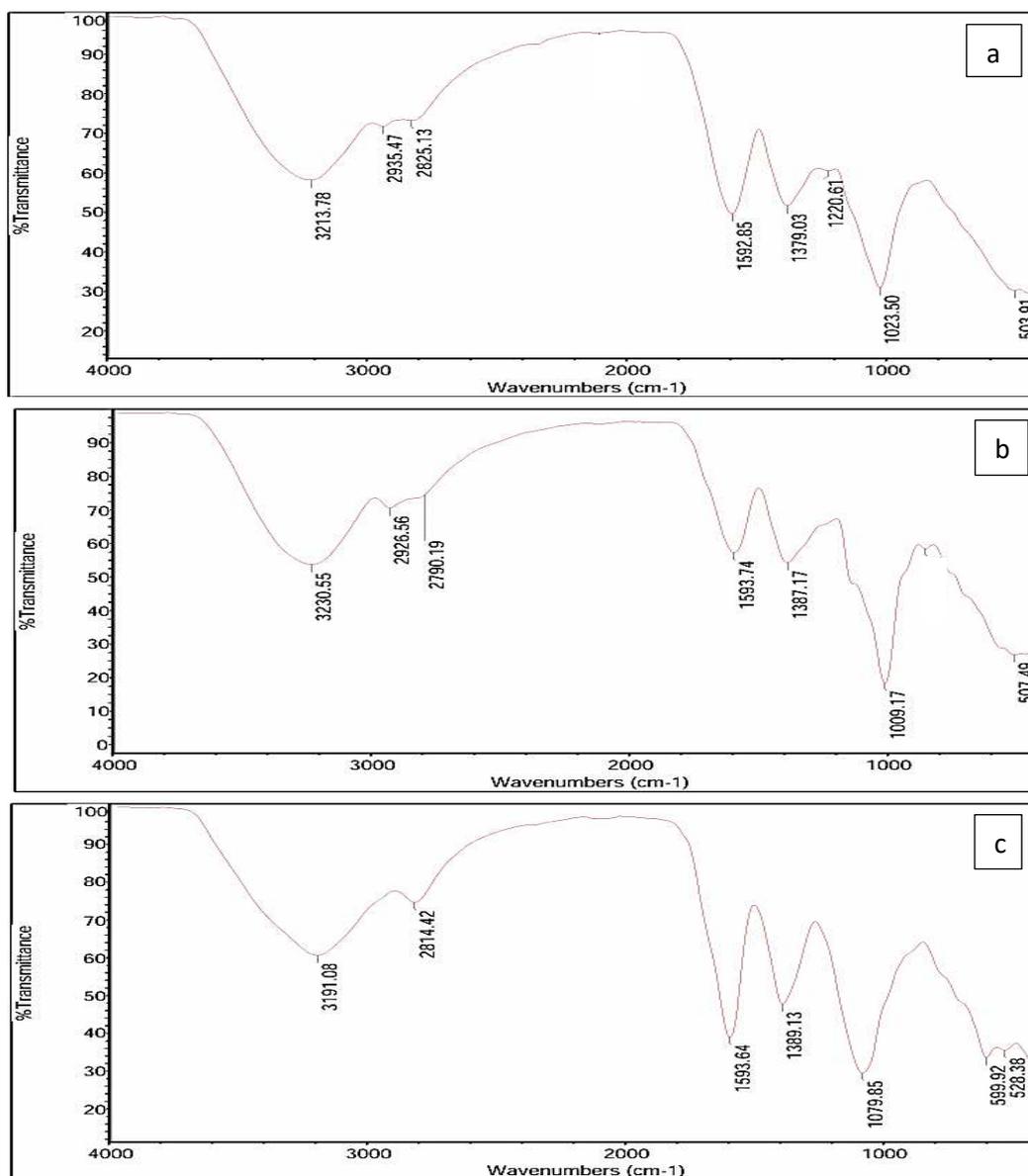
Figure 3. FTIR analysis results on fulvic acid extract powder from samples: (a) OPEFB; (b) commercial shilajit; (c) fertilizer

Elemental analysis

Fulvic acid extracts were quantitatively analyzed for elemental content in each sample using a CHN analyzer. This was done to determine the C, H, and N content of each sample, allowing for the calculation of molar ratios for C/N and C/H. The results of the elemental content measurements in the samples are shown in Table 3. The data used for comparison included elemental analysis and molar ratios in OPEFB, shilajit, and commercial fulvic acid fertilizer samples. OPEFB and commercial fulvic acid fertilizer had higher C and H contents than shilajit. Meanwhile, the N content in OPEFB and commercial fulvic acid fertilizer was higher than in shilajit. According to Motojima et al. (2009), the

high H content is due to their greater aliphatic characteristics, and the higher N content is related to their low humification rate. In comparison, OPEFB and shilajit samples showed a lower C content. This could be due to carbon mineralization in the OPEFB samples. The smaller the amount of inorganic carbon contained, the higher the C content in the elemental analysis of a sample.

Gong et al. (2020) and Zhang et al. (2020) utilized lignite samples as a source of fulvic acid, reporting C content elemental analysis results of 40.22% and 45.69%, respectively. Meanwhile, the H element content in the sample was 3.81% and 3.64% and the N content in the sample was 1.82% and 1.92%.
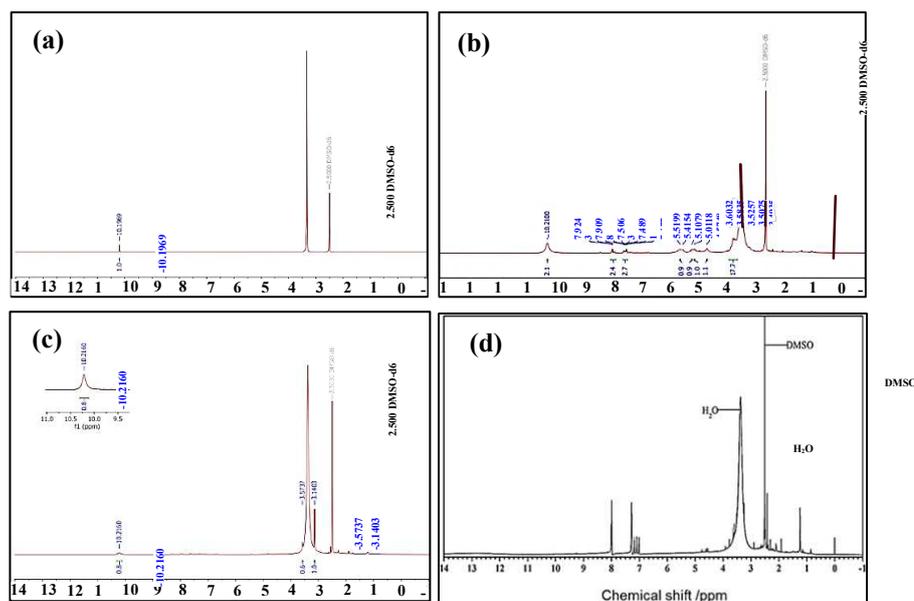
Figure 4. HNMR spectra of powdered fulvic acid extracts from samples: (a) OPEFB; (b) shilajit; (c) commercial fulvic acid fertilizer, and (d) fulvic acid extract Zhang et al. (2020)

Table 3. Elemental analysis results

Sample	Elemental analysis (%W)			Molar ratio (mmol.g^{-1})	
	C	H	N	C/N	C/H
OPEFB	29.55	5.43	3.52	8.39	5.44
Shilajit	33.36	5.72	1.94	17.2	5.83
Fulvic acid fertilizer	21.13	4.43	3.14	6.72	4.77
Gong *et al.* (2020)	40.22	3.81	1.82	22.1	10.56
Zhang *et al.* (2020).	45.69	3.64	1.92	23.8	12.55

OPEFB= Oil palm empty fruit bunches

Compared to the three samples, the results of the C element analysis in the two references were greater. The C and N element analyses were smaller than those of the three samples. This difference in elemental quantities affects the C/N and C/H ratio values of the samples, where the greater the C content and the smaller the N or H content, the greater the C/N or C/H ratio value produced. In the lignite fulvic acid sample (Zhang et al., 2020), the resulting C/N ratio reached 23.80, and the C/H ratio reached 12.55. Meanwhile, in the three samples, the C/N ratio and C/H ratio, from highest to lowest, were EFB fulvic acid, shilajit fulvic acid, and fertilizer fulvic acid, with values of 8.39, 17.20, and 6.72 for C/N ratios and 5.44, 5.83, and 4.77 for C/H ratios.

The molar ratio is calculated to obtain detailed information regarding the fulvic acid content in a sample based on the results of its elemental analysis. The C/H ratio decreases with increasing aliphatic content, and the C/H ratio is negatively correlated with the level of humification. Humification refers to the formation and accumulation of dark-colored organic matter from humic compounds, serving as an important indicator of the level of aromatic condensation. These data indicate that EFB and shilajit have a high level of low humification and a lower aromatic carbon ratio than commercial fulvic acid fertilizers (Motojima et al., 2009).

Javed et al. (2013) reported that microwave extraction did not significantly alter the molecular structure of fulvic acid in terms of elemental compositions, such as C/N and C/H ratios. This may be attributed to the fact that the microwave extractor allows the use of a closed container during the irradiation process, reducing the oxidative environment of the reaction. Based on the characterization results, using elemental analysis, the compound likely containing the highest fulvic acid content is commercial fulvic acid fertilizer, as evidenced by its low C/H ratio.

TGA – DSC

TGA analysis was performed to observe the decomposition of a compound by monitoring the change in sample mass as the temperature was controlled to change in a controlled manner, either at a constant temperature or held at a constant temperature over time. Fulvic acid pyrolysis was observed at 0-1000°C, as shown in Figure 5.
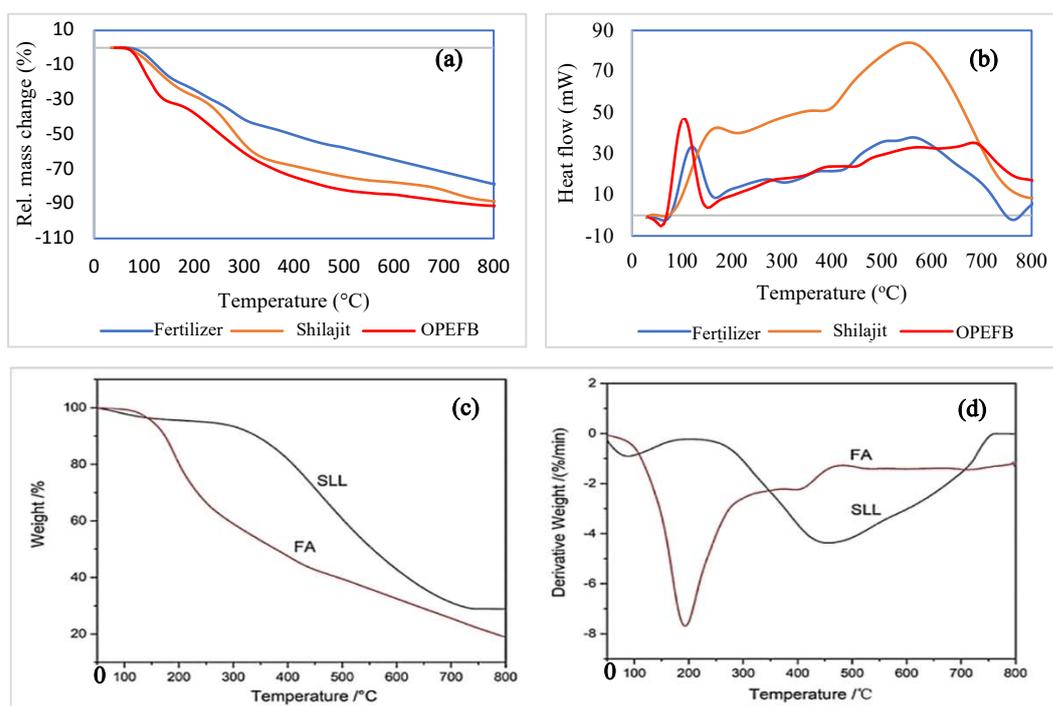
Figure 5. TGA-DSC results; (a) TGA of OPEFB, shilajit and fertilizer; (b) DSC of OPEFB, shilajit and fertilizer; (c) TGA of lignite fulvic acid (Gong et al., 2020); (d) DSC of lignite fulvic acid (Gong et al., 2020)

According to Gong et al. (2020), generally, at temperatures of 50-100°C, drying and dehydration processes occur, as can be seen in Figure 5a. Specifically, the OPEFB, shilajit, and fertilizer samples lost less than 13.5%, 6.5%, and 3.5% of their weight, respectively.

This weight loss can be attributed to water adsorbing onto the fulvic acid surface and evaporating as the temperature increases. The temperature at which the rate of weight loss accelerates varies for each sample type. The OPEFB sample begins to lose weight at 52°C, the shilajit sample at 32°C, and the fertilizer sample at 49°C. This indicates that the loss of small molecular substances present in its structure begins to occur as moisture is removed. According to Gong et al. (2020), the weight loss in fulvic acid samples from lignite can exceed 40%. As shown in Figure 5b, between 100–300°C, the weight loss in the fertilizer sample is approximately 41.2%, while the weight losses in the shilajit and OPEFB samples are 55.4% and 60.5%, respectively.

DSC analysis was performed to observe changes in heat flow from the samples when the temperature was controlled. The DSC thermograms of the three fulvic acid samples are shown in Figure 5b, which displays one endothermic peak at approximately 0-200°C in the OPEFB, shilajit, and fertilizer samples,

respectively: 104.4°C, 171.0°C, and 120.2°C. The broad peaks occurring in the fulvic acid and shilajit samples suggest the decomposition of simple organic structures (Gnananath et al., 2020). The differences in endothermic peak temperatures among the three samples may be attributed to variations in the abundance of volatile, small-molecule compounds with poor stability.

According to Gong et al. (2020), several stages of decomposition occurred in the samples when read using thermal analysis. At around 400°C, weight loss in all three samples began to appear in the fulvic acid DSC curves, likely due to the fracture of bridge bonds and the decomposition of most oxygen-containing functional groups. At 500°C, the weight loss rate of the shilajit sample was significantly higher than that of OPEFB and commercial fulvic acid fertilizer. This may be due to the higher content of more easily pyrolyzable substances in the shilajit sample than in the OPEFB and fertilizer samples. When the temperature exceeded 600°C, the pyrolysis reaction in the shilajit sample and commercial fulvic acid fertilizer entered the final stage, marked by a decrease in the weight loss rate. In contrast, the final stage of pyrolysis in the OPEFB sample only began to occur at 700°C. Table 4 shows the DSC peaks that appeared in various studies other than this study.

Table 4. Differential Scanning Calorimetry (DSC) peaks appeared in various studies

No	Study	Source	DSC findings	Thermal behavior
1	Gnananath et al. (2020)	Shilajit, Peat, dissolved organic matters, etc.	Broad endothermic peaks at 229°C	Indicate decomposition of simple and labile organic structures
2	Gong et al. (2020)	Fulvic acid from organic waste	Multiple decomposition stages up to 600°C	Major weight loss occurs at 400–500°C due to the fracture of oxygen-containing functional groups, while weight loss up to 600°C indicates a pyrolysis reaction.
3	This study	Fulvic acid from OPEFB, shilajit, and fertilizer	Endothermic peaks at 104.4°C (OPEFB), 171.0°C (shilajit), and 120.2°C (fertilizer)	Indicates variable composition and stability among samples, such as volatile, small-molecule compounds with poor stability

Conclusion

Fulvic acid extraction using a microwave extractor (microwave power 300 W; extraction time 10 minutes; solvent volume 35 mL) with variations in H_2O_2 solvent concentration and sample type produced significantly different results. The best H_2O_2 solvent concentration was 21%, and the best sample type was OPEFB. The results of the fulvic acid content produced the highest value of 23.59% for the OPEFB sample, 9.62% for the shilajit sample, and 6.97% for the commercial fulvic acid fertilizer sample. Based on previous quantitative tests, the fulvic acid extract powder sample was characterized as having the highest yield of fulvic acid. The characterizations were FTIR analysis, HNMR, spectrophotometry, spectrofluorometry, elemental analysis, and TGA-DSC analysis. Several characterizations revealed that the optimal results of fulvic acid from the samples varied according to each type of characterization; this could be attributed to the lower purity of the fulvic acid powder produced. Based on various characterizations, the tested OPEFB samples contain fulvic acid, just like other commercial products. This adds value to palm oil waste, offering potential future applications in various fields and paving the way for continued innovations in palm oil waste.

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