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STUDY OF THERMAL TREATMENT RETENTION TIME EFFECT ON PHENOL CONTENT AND PH OF SPENT COFFEE GROUND

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Abstract: *Indonesia, as a tropical country that is ideal for growing coffee beans, produced 760.2 thousand metric tons of coffee beans in 2023. With such huge production, the sustainability of its waste, such as spent coffee grounds, becomes a concern that needs to be addressed. Thermal Treatment, a partial oxidation combustion process, becomes one of the keys to the sustainability of this byproduct. Thermal Treatment turns spent coffee grounds into more useful products, such as liquid smoke. Bio-oil, the product produced by the Thermal Treatment process, often called Liquid Smoke, is a multifunctional liquid, contains phenolic compounds with antimicrobial properties. By integrating Thermal Treatment technology into waste management, sustainable and circular bio-economy models can be promoted. This research aims to investigate and study one of the Thermal Treatment parameters, the effect of retention time. Does retention time affect the products produced in Thermal Treatment, like the phenol content and pH in liquid smoke. The Research method involves an experiment on two different retention times of 60 and 90 minutes, where spent coffee grounds are subjected to thermal treatment at different retention times to produce smoke liquid. At first, spent coffee was gathered, for pre-treatment, spent coffee grounds were baked in an oven at 100°C for 1 hour, then the spent coffee ground was divided into 500g with two samples each, as the samples would go through two different Retention times of 60 minutes and 90 minutes at a temperature of 150. Then, these samples would be put in the Thermal Treatment chamber for them to go through the Thermal Treatment process once at a time. A study with two sets of different retention times was conducted. The resulting Liquid Smokes is then analyzed for its chemical composition, the pH and phenol content. The result in phenol content is 375.7 mg/L, 364.8 mg/L, for a retention time of 60 minutes, and 412.3 mg/L, 401.6 mg/L for a retention time of 90 minutes, which shows there is a significant difference. Liquid Smoke shows 20.7% higher yield at 90 mins (198.5 mL vs. 164.5 mL), while on the other hand, biochar shows no significant difference with an average of around 70 grams across both 60 and 90 minute retention times, and pH became less acidic with longer retention from 6.85 to 7.75. In conclusion, the research on Thermal Treatment's retention time reveals that retention time durations do affect the outcomes. The result shows 90 minute retention time is more ideal since the result shows it produces higher Liquid Smoke volume (198.5 mL vs. 164.5 mL) at 60 mins, 9.8% more phenols content (412.3 mg/L vs. 375.7 mg/L).*
Keywords: *Thermal Treatment, Spent Coffee Ground, Retention Time, Liquid Smokes, Phenol Content.*

INTRODUCTION

In 2023, There are 760.2 thousand metric tons of coffee were produced in Indonesia these are according to the data of Badan Pusat Statistik, being one of the leading producers of coffee beans [1]. Because of its climate, which is ideal for growing coffee, coffee has become one of the most abundant resources in Indonesia [2]. Given the abundance, the sustainability of its waste such as Spent Ground Coffee, becomes an essential factor that needs to be looked for.

On the other hand, according to statista.com, there were approximately 8.87 thousand cafés in Indonesia as of 2022 [3]. With 1 kg of dry coffee beans, leaving 700 to 800 grams of dry spent coffee grounds [4]. the growing consumption of coffee has led to a significant increase in the generation of Spent coffee grounds, presenting both a challenge and an opportunity for sustainable waste management [5]. Traditionally, coffee waste, such as spent coffee ground, is disposed of through landfilling or incineration, just like that, contributing to environmental pollution and resource depletion [6]. However, spent coffee grounds complex chemical composition and renewable nature offer the potential for their valorization through thermochemical conversion processes, such as Thermal Treatment [6].

Thermochemical decomposition a thermal treatment process of organic material at elevated temperatures in the absence of oxygen [7], [8]. Unlike combustion or gasification, this thermal treatment does not involve oxidative reactions, which allows it to break down biomass into simpler compounds without burning it [9]. During the thermal treatment, the organic material undergoes high temperatures process, usually between 300°C to 800°C, causing it to decompose into synthetic gases, liquid bio-oil, and solid biochar [7]. This can be applied to various organic matter, such as feedstocks, wood, agricultural residues, and even waste products like spent coffee grounds [7]. This process involves the breakdown of organic materials, such as biomass, plastics, and other products, usually waste and in our case spent coffee grounds, into smaller molecular components through the use of heat [7]. This process, thermal treatment, is used for various purposes, which include the production of Liquid Smoke, biochar, and syngas, as well as, in our case, waste management and material recycling [7].

Repurposing spent coffee grounds through thermal treatment offers significant environmental, economic, and social benefits because it diverting waste, in this case spent coffee grounds, from landfills [10]. This process also mitigates greenhouse gas emissions and conserves resources by transforming spent coffee grounds into valuable bio-products like bio-oil, biochar, and syngas, that already mentioned before [10]. Not to say these products have various commercial and environmental applications, such as beneficial chemical compounds and soil amendments, creating new revenue streams and contributing to sustainable waste management solutions [10].

Bio-oil, usually called Liquid Smoke, is a complex mixture of water-soluble compounds derived from the thermal treatment of biomass. In this research case, it's from spent coffee grounds that gives beneficial components such as phenols, carbonyls, and organic acids, which contribute to its antimicrobial and preservative properties [11].

Biochar is an excellent soil enhancer, improving soil structure, nutrient retention, and water-holding capacity, which is especially beneficial for agricultural productivity [12]. Applying biochar in soils also sequesters carbon, helping combat climate change by locking carbon dioxide away for extended periods [12]

Many studies have shown that liquid smoke has phenol content that have antimicrobial properties that resulting in insecticidal activity against various pests, including beetles, weevils, and some moth larvae [13]. The insecticidal effect is again, attributed to a complex mixture of chemicals in liquid smoke, including phenols, carbonyls, and organic acids. These compounds have capabilities to disrupt insect development and feeding behavior and even cause mortality [13]. This is because phenol, a main component of liquid smoke/bio-oil, contain lignin that have been used in some organic pesticide formulations due to its antimicrobial and insecticidal properties [13], [14].

If we view it from an economic perspective, the conversion of spent coffee ground into products with value such as liquid Smoke, biochar, and specialty chemicals offers revenue streams and cost savings for coffee producers and waste management facilities also, by diverting spent coffee ground from landfills and incinerators, thermal treatment contributes to waste reduction and mitigates environmental pollution [15].

And thus, in light of the pressing need for sustainable waste management solutions and renewable energy sources, Repurposing spent coffee grounds with thermal treatment holds promise as a viable pathway towards achieving environmental sustainability and circular economy goals [16]. This research aims to investigate the retention time effect that one way or another lead to the feasibility, challenges, and opportunities associated with spend coffee grounds waste thermal treatment's, aiming to advance sustainable waste valorization strategies and promote the transition towards a more circular and resource-efficient society. The thermal treatment of spent ground coffee represents a promising avenue for sustainable waste valorization, resource recovery, renewable energy production, and many other possibilities [10]. If we start harnessing the chemical complexity of spent coffee ground waste through thermochemical conversion, products with value can be obtained while mitigating environmental impacts associated with traditional waste disposal methods [10]. And so, research efforts are essential to overcome technical challenges, improve process efficiency, and unlock the full potential of spent coffee grounds using thermal treatment.

The successful this thermal treatment of spent ground coffee depends on optimizing process parameters to maximize product yields and quality. Temperature, retention time, heating rate, and reactor type are critical factors influencing the thermal treatment outcomes [17]. Higher temperatures typically result in increased bio-oil yields but may also lead to more excellent thermal degradation of bio-oil components [6]. The significances of retention time affect in the extent of thermal treatment reactions will be tested in this research [7]. So, this research is done to determine one of the parameters, which is retention time or, in other words, the duration spent within the reactor at its peak temperatures.

Problem Statement

Thermal treatment is one of the promising technologies for converting organic waste into valuable products. The application of thermal treatment to spent coffee grounds presents challenges and opportunities. The complex chemical composition of spent coffee grounds requires careful consideration in optimizing thermal treatment conditions to maximize product yields and quality. Therefore, this research aims to investigate whether the duration of thermal treatment, or in other words, retention time, affects the phenol content, and pH of the resulting liquid smoke.

According to the background above, these are the problem statements:

1) Does the retention time of Thermal Treatment affect the phenol content in the Liquid Smoke?
2) What is the effect of different Thermal Treatment retention time on the pH in Liquid Smoke?

Objectives

The objectives of this final project are:

(1) To determine whether differences in retention time during the thermal treatment of spent coffee grounds affect the phenol content of the resulting product (Liquid Smoke)
(2) To determine whether differences in retention time during the thermal treatment of spent coffee grounds affect the pH of the resulting product (Liquid Smoke).

Scope and Limitations

Scope

This research focuses exclusively on the effects of retention time on phenol content, pH, and the mass/volume of the resulting products from the thermal treatment of spent coffee grounds.

Limitations

This thermal treatment is supposed to emphasizes the lack of oxygen or, in other words, an inert atmosphere in its thermal decomposition process. While it can be certain that there would be no oxygen added while undergoing the thermal treatment Process. There is not much that can be done towards oxygen that has already settled in the chambers prior to the filling of the materials. There are also limitations to the lack of standardization on the liquid smoke itself, such as which are considered averages, which are considered not ideal, etc. Also, while most research papers said the optimal temperature was 300°C, the thermal treatment chamber that I use only peaks at 150°C; thus, I chose 150°C as the temperature.

LITERATURE REVIEW

Spent Coffee Ground

According to Badan Pusat Statistik data, approximately 760.2 thousand metric tons of coffee were produced in Indonesia [18]. The sustainability of its byproducts, such as spent coffee grounds, becomes a concern we must address.

Spent coffee grounds are viewed as organic wastes that contain carbohydrates, lipids, and proteins, in addition to lignin and caffeine [4]. Complex mixtures of chemical constituents in coffee grounds make this thermal treatment an interesting and promising approach because this thermal conversion, carried out in a controlled way, is a complex process [19]. Coffee grounds can also be further converted into biochar, which is carbon-enriched material and good for soil [20]. Spent coffee grounds are a complex matrix of organic compounds, including polysaccharides, proteins, lipids, lignin, and caffeine [6], [20]. Their composition and concentration depend on the type of coffee beans, degree of roasting, and preparation mode [21]. Hence, the thermal treatment process is by which these compounds' thermal decomposition into smaller molecules is achieved under regulated conditions, giving valuable products [22].

Spent coffee grounds is full of chemical complexity, comprising carbohydrates, lipids, proteins, lignin, and caffeine, which present a unique challenge and opportunity for this thermal treatment-based valorization [23]. These organic constituents can be converted through controlled thermal degradation into a range of valuable products with potential applications in renewable energy, agriculture, and chemical synthesis [24]. One of them, a carbonaceous material called biochar that are produced during the thermal treatment, retains the carbon content of coffee grounds and possesses soil amendment properties, improving soil fertility and carbon sequestration potential, and phenol with its antimicrobial property [25].

Repurposing spent coffee grounds through thermal treatment offers several environmental, economic, and social benefits [26]. By diverting spent coffee ground from landfills to be used on thermal treatment-based valorization that are contributes to waste reduction, greenhouse gas emissions mitigation, and resource conservation makes repurposing spent coffee grounds through thermal treatment addresses critical environmental and economic challenges by transforming waste into valuable bio-products [10].

Thermal Treatment

This thermal treatment is a thermochemical decomposition process that does transformation of organic materials into a mixture of solid char, liquid bio-oil, and gases [7]. It has gained attention for its ability to convert various organic materials into valuable products [27]. The thermal treatment of spent ground coffee offers opportunities for waste valorization, resource recovery, and sustainable production of bioenergy and bio-based chemicals [28].

The thermal treatment of spent ground coffee yields many different valuable products, including Bio-oil, usually called Liquid Smoke, Biochar, Syngas, and volatile organic compounds (VOCs) [16]. Liquid smoke, a dark, viscous liquid, contains a mixture of oxygenated hydrocarbons, phenolic compounds, and organic acids, which can be upgraded into biofuels or used as chemical synthesis [10]. Biochar, a carbonaceous material, have carbon content that retained out of the original coffee grounds and possesses adsorption properties beneficial for soil remediation and carbon sequestration [16]. Syngas, a synthetic gas one of byproduct of thermal treatment composed mainly of hydrogen, carbon monoxide, and methane able to be combusted for heat and power generation or further processed into synthetic fuels and chemicals [29]. VOCs that are emitted during thermal treatment which require downstream treatment to minimize environmental impacts [30].

This thermal treatment involves heating organic materials to high temperatures, typically ranging from 300°C to 800°C, without presence of oxygen [7]. This was to prevent combustion and allow the material to decompose into solid, liquid, and gaseous products. The absence of oxygen is one of crucial elements which needed to prevent the materials from burning, to ensure they break down into other chemical compounds rather than fully oxidizing them into carbon dioxide and water [31].

Its potential to mitigate environmental pollution and reduce greenhouse gas emissions becomes one of the primary motivations for studying thermal treatment, lies in [36]. By converting organic waste materials such as agricultural residues, municipal solid waste, and spent coffee grounds into valuable products, this thermal treatment offers a sustainable alternative to conventional waste disposal methods like landfilling and incineration [31].

And so this thermal treatment was chosen for this study because it maximizes bio-oil production from spent coffee grounds (SCGs), the key objective of this research. This method's rapid heating rates and short vapor residence times preferentially break down spent coffee ground’s lignin and cellulose into condensable vapors rich in phenolic compounds, rather than forming excess biochar or syngas. While there are challenges such as our equipment limited temperatures to 150°C (below ideal for this type of thermal treatment range) and we can’t make sure the chamber environment is 100% absence of oxygen we still maintained short retention times (60-90 min) to mimic fast thermoclysis conditions and optimize liquid smoke yield. This technique is particularly suitable for spent coffee grounds, which then decompose into phenols under this type of thermal treatment. This approach directly supports our goal of investigating phenol yield variations under different processing times.

Liquid Smoke

Bio-oil or liquid smoke is a liquid product derived from biomass thermal treatment and, in our case, right now, Spent Coffee Ground. This thermal treatment itself, as already described before, is a thermal decomposition process conducted in the absence of oxygen, which breaks down organic materials into bio-oil, syngas, and bio-char [31]. Complex mixture of water and organic compounds with various applications in energy production, chemicals, and materials is a byproduct of thermal treatment called Liquid smoke represents a versatile and renewable source of energy and chemicals [32]. Its production from biomass offers environmental benefits and economic opportunities, contributing to sustainable energy solutions.

Liquid Smoke derived from spent coffee ground’s thermal treatment contains a mixture of oxygenated hydrocarbons, phenolic compounds, and organic acids, which can be refined into biofuels or used as precursors for specialty chemicals such as Organic pesticides [33]. The process which produces liquid smoke that captures a complex mixture of organic compounds, including phenols, carbonyls, and acids. Research has recently shown that these compounds can have pesticide properties, making liquid smoke a potential with its antimicrobial properties [34]. Not

Stemming from its rich composition of bioactive compounds, liquid smoke shows its pesticidal effectiveness. Phenols, for instance, are known for their antimicrobial and insecticidal properties [35]. These compounds have capability to disrupt the bacteria and fungi cell membranes, succumbing to their death. Additionally, carbonyls and organic acids found in liquid smoke work as insect repellents and growth inhibitors. the combination of these compounds creates a multiple approach to pest control, targeting a wide range of agricultural pests [36].

Antimicrobial Potential

Phenol, one of the compounds inside liquid smoke have been used in some organic pesticide formulations due to its antimicrobial and insecticidal properties [37]. Repelling pests, inhibiting their growth, or causing lethal effects are known mechanisms the substances have, thereby protecting crops effectively also organic pesticides are increasingly favored in sustainable agriculture due to their lower environmental impact and compatibility with organic farming practices. These compounds work through various mechanisms, including repelling pests, inhibiting their growth, or causing lethal effects, thereby protecting crops effectively [38].

METHOD

Research Framework

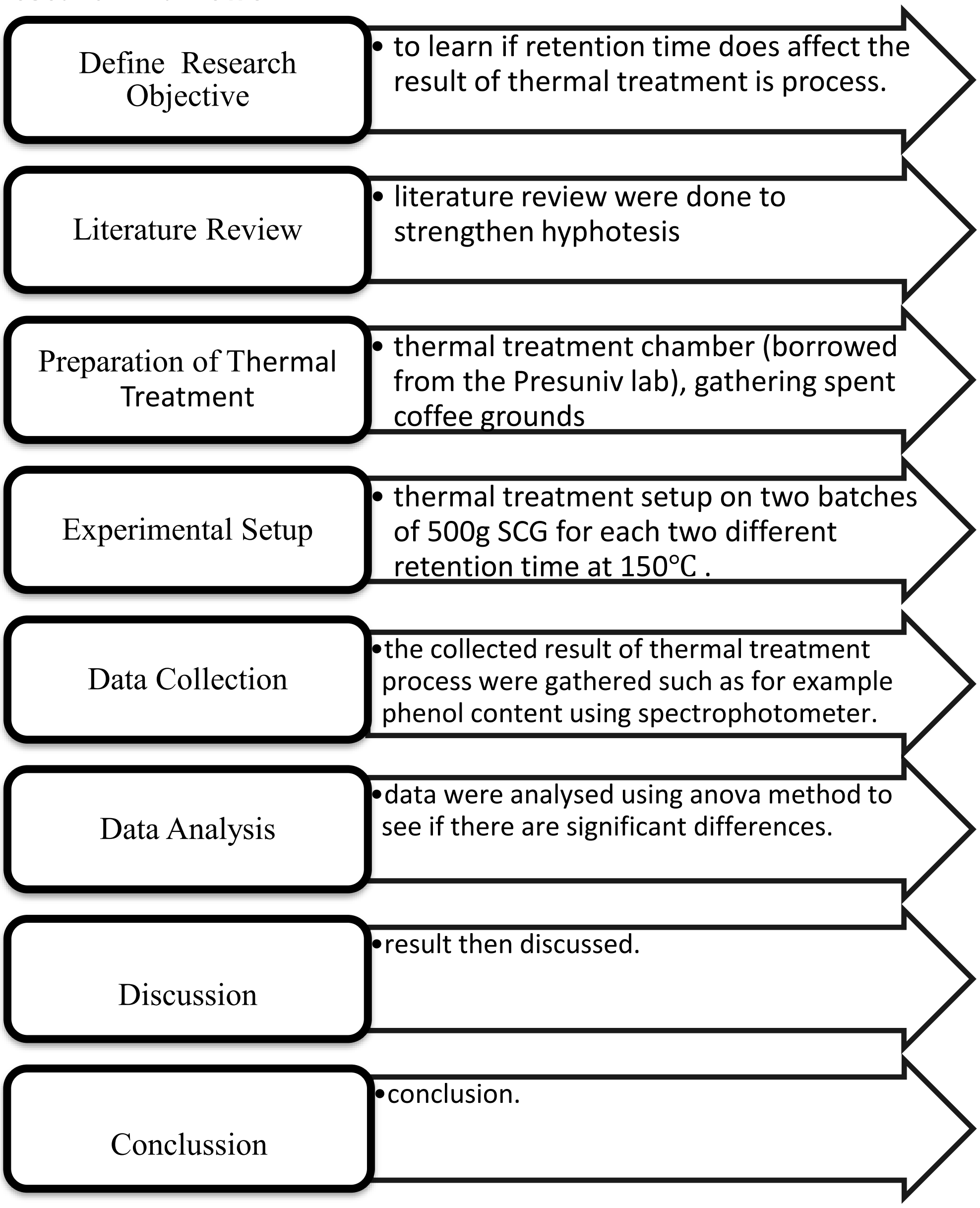

Fig 1. Research's Flowchart

This research began by identifying the problem that is Spent Coffee Grounds, and proposing thermal treatment as a solution. The study then focused on optimizing the thermal treatment process, specifically examining how retention time affects the results. Which, one way or another, would lead to the optimization of the thermal treatment process. Literature reviews were done to strengthen the hypothesis of determining whether retention time affects the result of the thermal treatment process. As preparation, a thermal treatment chamber was borrowed from the President University lab, and then gathering spent coffee grounds for it to be dried and baked to remove any dampness that might hinder the thermal treatment process. After the preparation and pre-treatment were done, the experiment started by setting up two batches of 500g spent coffee ground for each of the two different retention times, 60 minutes and 90 minutes, that would go through thermal treatment at a temperature of 150°C. data were analyzed using the ANOVA method to see if there were significant differences.

RESULTS AND DISCUSSIONS

First and foremost, there is a problem with the initial premise of the temperature the plant was for the temperature at 300°C for the retention time, but the thermal treatment chamber only peaks at 300°F or, in other words, 150°C which is less ideal as the thermal treatment temperature.

Fig 9. Thermal Treatment Temperature

The temperature of thermal treatment, which only peak at 150 °C which is not ideal for thermal treatment. This is also most likely the reason some tar-like substance is produced along with the liquid smoke that might impact phenol measurement, which is then why it was filtered using 0.45 μm PTFE syringe filters.

Fig 10. Tar-like substance on the surface of Liquid smoke.

Experiment Result

Mass and Volume

Table 2. Resulting Volume of Liquid Smoke from thermal treatment

No	Retention Time (minute)	Initial Mass (g)	Temperature (°C)	Liquid Smokes Volume (ml)
1	60	500	150°C	165
2	60	500	150°C	164
3	90	500	150°C	204
4	90	500	150°C	193

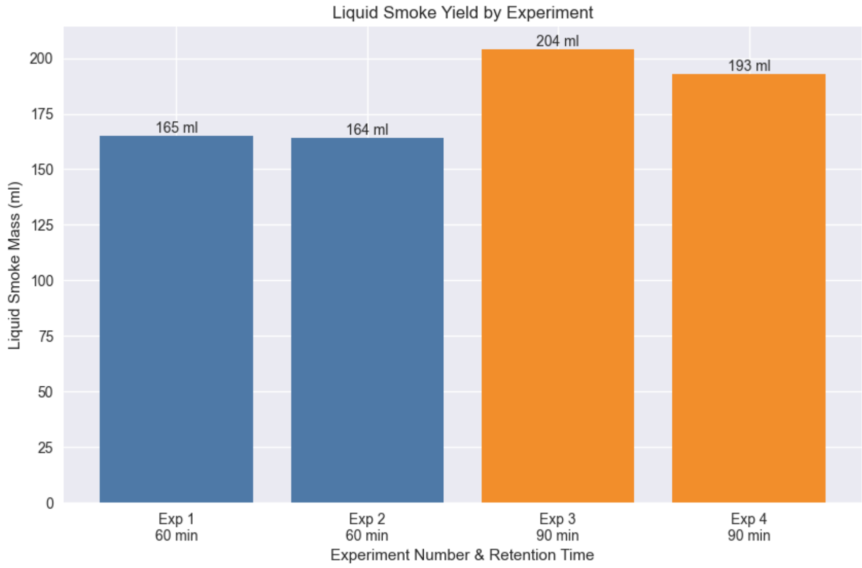

Fig 11. Bar chart of Resulting Liquid Smoke.

The chart shows that samples of retention time 90 minutes yields more Liquid Smoke than samples of retention time 60 minutes. This suggests that increasing the retention time might allow for more complete thermal degradation of the mass coffee grounds, leading to greater yields of liquid smoke with 20.7% higher yield at 90 mins.

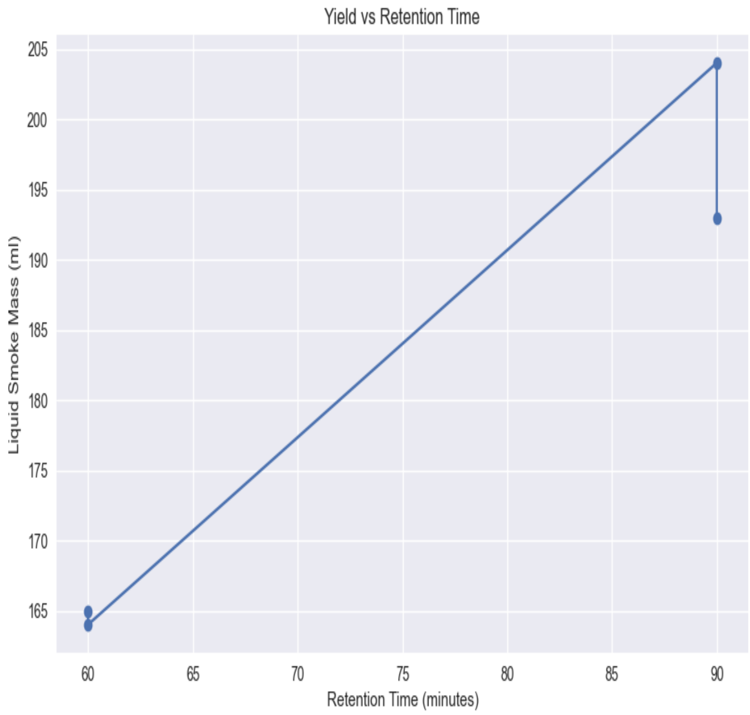

Fig 12. Trend graph of Resulting Liquid Smoke.

The graph shows that the yield of Liquid Smoke are increasing when the retention time is longer at 90 minutes than the 60 minutes.

Table 3. Resulting Mass of Biochar from thermal treatment

No	Retention Time (minute)	Initial Mass (g)	Temperature (°C)	Bio Char Mass (g)
1	60	500	150°C	69
2	60	500	150°C	74
3	90	500	150°C	67
4	90	500	150°C	64

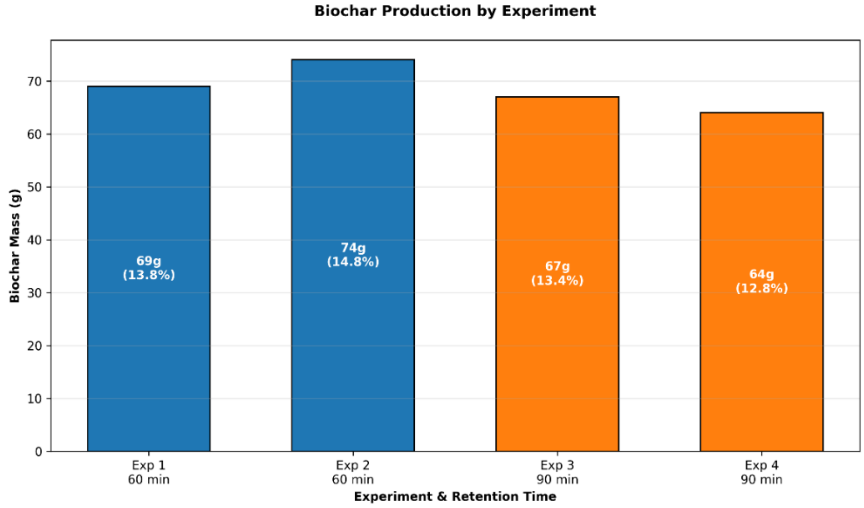

Fig 13. Bar chart of Resulting Bio Char.

The chart shows biochar yield was relatively the same, with an average of around 70 grams across both 60 minute and 90 minute retention times. This result might means that that within the range of temperatures and retention times used in this study, the amount of biochar produced after thermal treatment is not significantly affected. There's also a possibility that the lack of significant difference biochar mass could be due to the lower temperature of thermal treatment, which may not have been high enough to influence the rate of biochar formation significantly.

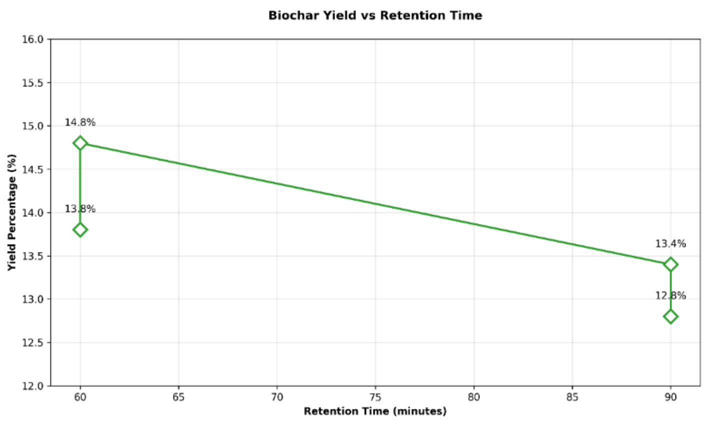

Fig 14. Trend graph of Resulting Biochar.

The graph shows that the biochar yield of 90 minutes retention time is lower than the yield of 60 minutes.

Table 4. ANOVA result of Liquid Smoke volume

Source of Variation	SS (Sum of Squares)	df (Degrees of Freedom)	MS (Mean Square)	F-value	F-critical	P-value
Between Groups	1156.0	1	1156.0	37.902	18.513	0.025 (< 0.05)
Within Groups	61.0	2	30.5			
Total	1217.0	3				

Since the F-value (37.902) is greater than the F-critical value (18.513), the null hypothesis is rejected, suggesting a significant difference in Liquid Smoke volume between the two retention times.

Since the p-value is less than 0.05, the null hypothesis is rejected. This suggests that there is a significant difference in liquid smoke volume between the 60-minute and 90-minute retention times.

Table 5. ANOVA result of Bio Char mass

Source of Variation	SS (Sum of Squares)	df (Degrees of Freedom)	MS (Mean Square)	F-value	F-critical	P-value
Between Groups	36.0	1	36.0	4.235	18.513	0.12 (≥ 0.05)
Within Groups	17.0	2	8.5			
Total	53.0	3				

The F-value (4.235) is less than the F-critical value (18.513), meaning the null hypothesis can't be rejected. This suggests there is no significant difference in Bio Char Mass between the two retention times.

P-value = 0.1, Since the p-value is greater than 0.05, can't reject the null hypothesis.

The conclusion is that there is not enough evidence to conclude that there is a significant difference in Bio Char Mass between the 60-minute and 90-minute retention times.

pH

Table 6. pH of the liquid smoke

No	Retention Time (minute)	Initial Mass (g)	Temperature (°C)	Liquid Smokes Mass pH
1	60	500	150°C	7.0
.2	60	500	150°C	6.7
3	90	500	150°C	7.7
4	90	500	150°C	7.8

Indonesian National Standard (SNI) 6989.11-2019 was used to measure the pH of liquid smoke. pH meter was used to obtain the pH values. For accurate measurement, the meter was calibrated beforehand using buffer solutions of pH 4 and 7. The pH measurement was repeated a few times for better reliability.

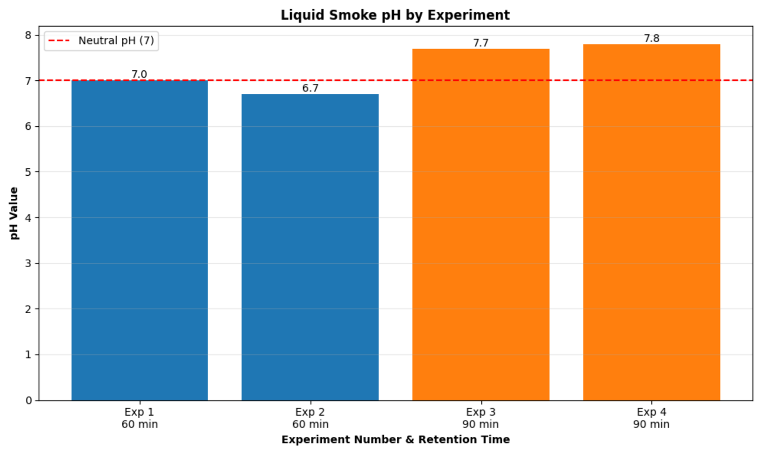

Fig 15. Bar chart of Resulting pH.

pH increases in 90-minute experiments compared to 60-minute runs, which is different than the typical liquid smoke result. Probably because of the temperature of thermal treatment, which unideal the acidity of liquid smoke is not as acidic as most research that has already been conducted shows.

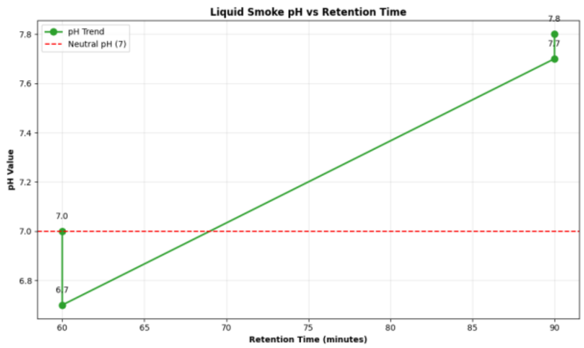

Fig 16. Trend graph of Resulting pH.

The trend graph shows that the pH are increasing when the retention time is longer at 90 minutes than the 60 minutes.

Table 7. ANOVA result of pH

Source of Variation	SS (Sum of Squares)	df (Degrees of Freedom)	MS (Mean Square)	F-value	F-critical	P-value
Between Groups	0.81	1	0.81	32.4	18.51	0.030 (< 0.05)
Within Groups	0.05	2	0.025			
Total	0.86	3				

Since the F-value (32.4) is greater than the F-critical value (18.51), the null hypothesis is rejected. This means that there is a statistically significant difference in the pH values based on the retention time.

Since the p-value is less than 0.05, the null hypothesis was rejected. This suggests that there is a significant difference in pH between the 60-minute and 90-minute retention times.

Phenol Content

Table 8. Phenol Content

No	Retention Time (minute)	Initial Mass (g)	Temperature (°C)	Absorbance	Phenol Content mg/L
1	60	500	150°C	1.109	375.7
.2	60	500	150°C	1.077	364.8
3	90	500	150°C	1.217	412.3
4	90	500	150°C	1.185	401.6

Absorbance, determined using a spectrophotometer, measures the amount of light absorbed using distilled water as blanco. The absorbances taken from the spectrophotometer are 1.109, 1.077 at 60-minute retention time and 1.217, 1.185 at 90-minute retention time [41] [44].

$$y = m \times x + b$$

$$x = \frac{y - b}{m}$$

where:

y = Absorbance = 1.109, 1077 for 60 minute retention and 1.217, 1.185 for 90-minute retention time.

m = Slope of the curve = 0.002939 L/mg (slope from calibration)

b = Intercept = 0 = 0,005, even after zeroing, blanks may have slight absorbance
x = Concentration = ?
. [40], [43], [49].

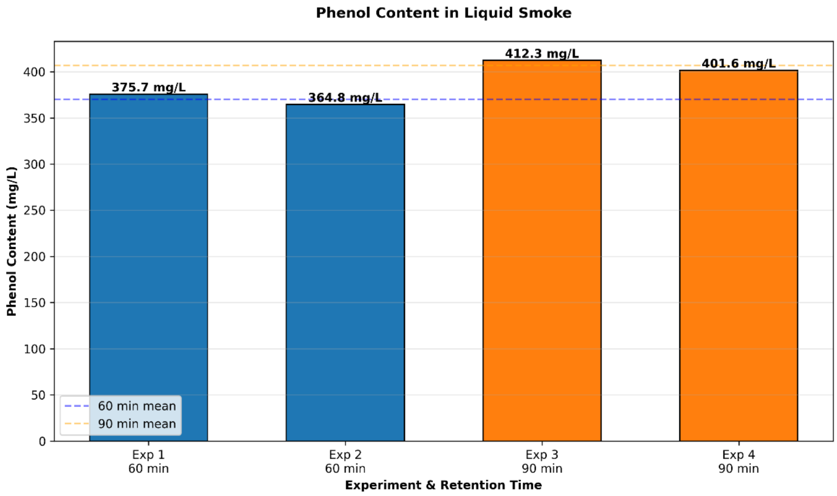

Fig 17. Bar chart of Resulting Phenol Content.

60-minute retention time produces an average of approximately 370 mg/L, while the 90-minute samples resulted in a higher average of 407 mg/L, which might be because of prolonged thermal degradation of lignin, low temperature also may have favoured incomplete reactions, artificially inflating phenol yields. .

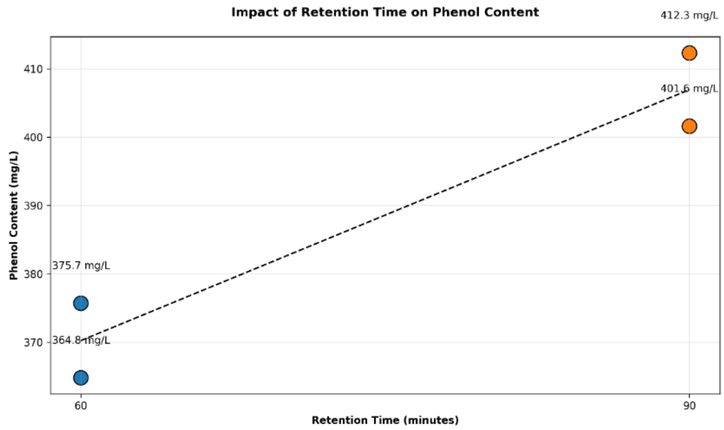

Fig 18. Trend graph of Resulting Phenol Content.

The graph shows that the phenol content of liquid smoke are increasing when the retention time is longer at 90 minutes than the 60 minutes.

Table 9. ANOVA result of Phenol Content

Source of Variation	SS (Sum of Squares)	df (Degrees of Freedom)	MS (Mean Square)	F-value	F-critical	P-value
Between Groups	1346.89	1	1346.89	23.09	18.51	0.041 (< 0.05)
Within Groups	116.65	2	58.33			
Total	1463.54	3				

Since the F-value (23.09) is greater than the F-critical value (18.51), the result is significant, and the null hypothesis is rejected, indicating a difference in phenol content based on retention time.

Since the p-value is less than 0.05, the null hypothesis was rejected. This suggests that there is a significant difference in phenol Content between the 60-minute and 90-minute retention times.

Discussion

Thermal Treatment Temperature and Process Outcomes

The temperature of this thermal treatment, which is one of the keys that need to be observed in the experiment, only reached 150°C, a lower temperature than typically used for optimal this thermal treatment reactions, ranging from 300°C to 800°C. This suboptimal temperature most likely impacted the efficiency of the process, which also means the quality of the end products. Higher temperatures generally result in greater biomass breakdown into , syngas, and biochar. The lower temperature may have contributed to the formation of a tar-like substance observed in the liquid smoke that was filtered using 0.45 μm PTFE syringe filters, which indicates incomplete thermal degradation of the organic materials in the spent coffee grounds.

Despite this limitation, the experiment yielded significant data on liquid smoke production and its phenolic content. The retention time, or the duration that the coffee grounds spent in the reactor, varied between 60 and 90 minutes to determine how this factor influenced the composition and quantity of the products.

Volume and Mass of Liquid Smoke and Biochar

The results showed there is are significant difference in the mass of liquid smoke produced between the two retention times. In the 60-minute retention time, an average of approximately 164.5 mL of liquid smoke was produced, while the 90-minute retention time resulted in a higher average of 198.5 mL. These findings show that longer thermal treatment times allow for more Liquid Smoke gathered. using ANOVA analysis as a tool to confirm that this difference was statistically significant, with a p-value below 0.05. This suggests that increasing the retention time allows for more complete thermal degradation of the coffee grounds, leading to greater yields of liquid smoke.

On the other hand, the mass of biochar did not show a significant difference between the two retention times. The biochar yield was relatively consistent, with an average of around 70 grams across both 60- and 90-minute retention times. This might suggest that within the range of temperatures and retention times used in this study, the amount of solid residue remaining after thermal treatment is not significantly affected by the duration of the process. There's also possibilities that the lack of significant variation in biochar mass could be due to the lower temperature of this thermal treatment, which may not have been high enough to influence the rate of biochar formation significantly.

pH and Phenol Content of Liquid Smoke.

Another important aspect of the research on the effect of retention time was on the chemical composition of the liquid smoke, particularly its pH and phenol content. Phenol content is one of the components to measure the quality of liquid smoke, as phenols contribute to its preservative and antimicrobial properties. The result also shows that retention time are significantly influenced the phenol content of the liquid smoke, with the 60-minute samples shows an average phenol content of around 370 mg/L, while the 90-minute samples averaged around 407 mg/L. This might be caused by prolonged thermal degradation of lignin. Low temperature may also have favoured incomplete reactions, artificially inflating phenol yields. This finding also shows that longer thermal treatment times allow for more complete thermal degradation of the organic materials, leading to higher concentrations of phenolic compounds in the resulting liquid smoke.

The result shows an increase on the pH of the liquid smoke the longer retention times, with the 60-minute samples averaging around 6.85 and the 90-minute samples averaging around 7.75. This shows the difference was statistically significant, indicating that longer retention times produce less acidic liquid smoke. This may have resulted due to a more thorough breakdown of acidic compounds in the spent coffee grounds during prolonged exposure to heat. Regardless, ANOVA shows the results that there's a significant difference in pH and phenol content between the two retention times, with a p-value of less than 0.05.

Implications for Thermal Treatment Optimization.

This experiment is highlighting the retention time in the thermal treatment of spent coffee grounds. While higher temperatures have the possibility to yield even more significant results, this study demonstrates that extending the retention time can increase the yield and quality of liquid smoke even at lower temperatures. the experiment result, which is the increased phenol content and less acidic pH of the liquid smoke produced with a 90-minute retention time, suggests that longer thermal treatment durations could improve the product's potential for use as an organic pesticide or preservative.

In terms of waste management and resource recovery, this research supports the viability of this thermal treatment as a method for valorising spent coffee grounds. By converting spent coffee grounds into bio-oil (liquid smoke) and biochar, this process not only reduces waste sent to landfills but also generates valuable products with commercial and environmental applications.

CONCLUSIONS

The results of this thermal treatment at different retention times (60 minutes and 90 minutes) reveal statistically significant differences ($p < 0.05$) in phenol content, with measured values of 375.7 mg/L and 364.8 mg/L for the 60-minute treatment, and 412.3 mg/L and 401.6 mg/L for the 90-minute treatment. This demonstrates that retention time directly affects phenol production in liquid smoke.

Different thermal treatment retention times significantly affected the pH of liquid smoke. Increasing the retention time from 60 minutes with an average pH of 6.85 to 90 minutes with an average pH of 7.75 resulted in a statistically significant ($p < 0.05$) alkaline shift.

Recommendation

The study considered retention time as the variable, but other factors, such as temperature, feedstock moisture content, and particle size, may also influence the thermal treatment process. The consideration of these variables in future work could fill in more of the blanks regarding the capacity that spent coffee grounds thermal treatment might have in waste management and bio-product

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