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Characterization of Liquid Smoke and Charcoal from Cocoa Pod Husks (*Theobroma cacao* L.) in North Kolaka Regency

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Abstract: This study aims to characterize the pyrolysis products of cocoa pod waste from North Kolaka Regency, Southeast Sulawesi Province. Pyrolysis takes place at various temperatures between 112 - 512 °C and produces liquid smoke and charcoal products. The highest liquid smoke yield was obtained at a temperature of 212 °C of 20.05%. The results of GC-MS analysis showed that liquid smoke contains many potential compounds consisting of acetic acid compounds with the highest concentration of 28.1%, Phenol, 2-methoxy- (CAS) Guaiacol with the highest concentration of 4.18%, 3-Hexine-2,5-diol (CAS) Hexine-3-diol-2,5 of 2.94%, and several aromatic and alcohol compound groups. FTIR analysis shows that the typical functional groups of cocoa pod charcoal consist of OH (hydroxyl) and C = C-H (aromatic) groups. XRD analysis shows that charcoal is dominated by an amorphous phase with a degree of crystallinity of 14%. Liquid smoke and cocoa pod charcoal have the potential to be used as raw materials in the chemical, health and manufacturing industries.

Keywords: Cacao pod; Characterization; Charcoal; Liquid smoke; Pyrolysis

Introduction

The cocoa plant (*Theobroma cacao* L.) is a plantation crop with significant economic value and is a potential export commodity that can generate foreign exchange for the country. In Indonesia, cocoa production ranks third in the plantation subsector, following palm oil and rubber. Cocoa pod husks contain lignocellulose, which includes active components such as the alkaloid theobromine (3,7-dimethylxanthine), flavonoids, saponins, triterpenoids, and condensed or polymerized tannins (Soares & Oliveira, 2022). The chemical components of cocoa pod husks include ammonia, hexane, alcohol, ketones, acetic acid, and phenolic compounds (Collard & Blin, 2014; Manmeen et al., 2023; Urrutia et al., 2022).

The phenolic compounds, flavonoids, tannins, and terpenoids in cocoa pod husks are known to possess antimicrobial activity. Active phenolic compounds exhibit antifungal activity by damaging fungal cell membranes, leading to changes in cell permeability, which can inhibit cell growth or result in cell death (Konuk & Ergüden, 2020; Kibet et al., 2015; Ansari et al, 2013).

Currently, cocoa pod waste is only used as animal feed, biogas and organic fertilizer, but scientific exploration of the chemical content of cocoa pod waste has not been explored further (Anoraga et al., 2024; Ouattara et al., 2021; Meza-Sepúlveda et al., 2021). One way to explore the chemical content of cocoa pod shell waste is by converting cocoa pod shell waste into liquid smoke using pyrolysis method. Pyrolysis is one of the interesting methods to recycle waste in an effort to handle organic waste. Pyrolysis of biomass at high temperatures decomposes into carbon, tar and Liquid Volatile Matter (LVM) or liquid smoke (Sari et al., 2020; Permanasari et al., 2020; Gani et al., 2024; Lu et al., 2024). Liquid smoke is the product of condensation or condensation of vapour from direct or indirect combustion of biomass containing lignin, cellulose and

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hemicellulose. The utilisation of liquid smoke in everyday life is extensive. In the food industry, liquid smoke has potential as a smoke flavour because it has functions as an antioxidant and anti-microbial (Surboyo et al., 2024; Lingbeck et al., 2014; Guillén & Ibargoitia, 1999).

In addition, in the rubber industry, liquid smoke can be used as a coagulant that produces quality rubber. In addition to having economic functions in various fields, liquid smoke from the pyrolysis of cocoa shell waste also contains lignocellulose that can be decomposed into simpler derivative compounds during the pyrolysis process (Gani et al., 2024; Sinaga et al., 2023; Gea et al., 2018). Several studies have shown that during the pyrolysis process, lignin is decomposed into phenol compounds and their derivatives, while cellulose and hemicellulose degrade into furfural, acetic acid, and various alcohol compounds, including methanol, ethanol, and propanol. In addition, alcohol-derived compounds such as ethyl acetate and glycerol were found to be formed through advanced reactions during the cooling of the liquid smoke. The derived compounds have a significant range of essential benefits in various industries, including chemical, energy, and environmental (Abdelhafez et al., 2021; Tiegam et al., 2021).

Alcohols such as methanol, ethanol, and propanol, which result from this process, are important raw materials in the chemical industry for the production of other compounds, such as formaldehyde and acetic acid, and serve as solvents in pharmaceutical and cosmetic production. Phenol compounds and their derivatives have antimicrobial properties that are important in the production of disinfectants and preservatives. Phenol also serves as a precursor in the synthesis of various industrial chemicals, such as bisphenol A, which is used in the manufacture of polycarbonate and epoxy resins. In addition, organic acids such as acetic acid, which is also formed from the decomposition process of lignocellulose, have wide applications in the food industry as preservatives and flavour enhancers, as well as in the textile and pharmaceutical industries (Faisal et al., 2024; Okiyama et al., 2021).

Studies related to the production of liquid smoke through pyrolysis have been conducted using various types of biomass, but characterisation of liquid smoke to determine derived compounds such as phenols and organic acids has not been done. Therefore, this study aims to produce liquid smoke products from cocoa pod shell waste using the pyrolysis method. Furthermore, the liquid smoke product was characterised using gas chromatography method to identify phenol-derived compounds and other organic derived compounds.

Method

Materials and Instruments

The materials used consisted of cocoa pod shell waste obtained from areas in North Kolaka Regency, Southeast Sulawesi Province and distilled water. The instruments used consisted of a pyrolysis reactor, a set of glassware, GC-MS, Infrared Spectrophotometer (FTIR), X-Ray Diffraction, and Scanning Electron Microscope (SEM). Research design and method should be clearly defined.

Sample Preparation

The cocoa pods were separated between the skin and seeds, then the skins were dried in the sun for 7-10 days and cut into ± 5 × 5 cm pieces. Dried cocoa pods are crushed with a crusher and sieved to 40-60 mesh size. Next, cocoa pod dry powder was analysed for lignocellulose content as a preliminary analysis (Tripathi et al., 2016).

Pyrolysis Process

1000 g of cocoa pods were put into a pyrolysis reactor equipped with a condenser circuit. The pyrolysis process is carried out with the combustion temperature used between 112-512 °C for 5 hours. Pyrolysis is stopped when there is no liquid smoke dripping into the collection container. Besides producing liquid smoke as a by-product, the pyrolysis process produces charcoal and tar (Tsai et al., 2018; Tegang et al., 2020; Guillén & Ibargoitia, 1999).

Characterisation of Liquid Smoke

GC-MS analysis was conducted to determine the composition and concentration of liquid smoke from cocoa pod shell waste. XRD analysis to determine the crystal structure and degree of crystallisation of cocoa pod shell charcoal. SEM analysis to determine the morphological size of the charcoal, and FT IR analysis to determine the functional groups (Oyedotun et al., 2021).

Result and Discussion

Lignocellulose Analysis

The results of lignocellulose analysis of cocoa fruit peels using the Chesson Datta method showed that the raw material contained 17.27% cellulose, 52.02% lignin and 19.56% hemicellulose, with a moisture content of 9.829%. This shows that the raw material contains high levels of hydrocarbon compounds that have the potential to produce high levels of charcoal and liquid smoke. Different research results were shown by Chen et al. (2016), that the characterisation of pine wood with pyrolysis catalyst resulted in lignin content of 28.6%, hemicellulose 30.1% and cellulose 40.8%. This indicates that the lignocellulose content depends on the type of raw material. Lignin does not have repeat units like hemicellulose and cellulose, but consists of complex phenolate units.

Pyrolysis Process

The pyrolysis process of cocoa pod waste produces charcoal and liquid smoke. Table 1 presents the yield results for liquid smoke from North Kolaka Regency cocoa pods based on the increase in pyrolysis temperature. Table 1 shows that the largest liquid smoke product was obtained at a pyrolysis temperature of 212 °C at 20.05%, followed by 312 °C at 11.15%, then 412 °C at 10.87%, 512 °C at 5.68% and finally at 112 °C at 3.17%.

Table 1. Cocoa Pod Liquid Smoke Yield Based on Increase in Pyrolysis Temperature

Temperature (°C)	Weight (g)	Yield (%)
112	28	3.17
212	177	20.05
312	98.5	11.15
412	96	10.87
512	36	4.07

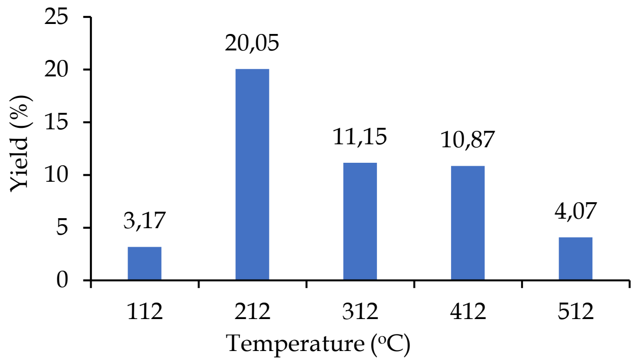

Figure 1. The effect of temperature on the yield of liquid smoke from cocoa pod skin

Pyrolysis temperature greatly affects the yield of liquid smoke. Pyrolysis temperature is a crucial factor in affecting the type and quantity of products produced, including liquid smoke yield. The thermal breakdown dynamics of the major biomass components, such as cellulose, hemicellulose, and lignin, have a significant impact on the link between pyrolysis temperature and liquid smoke production (Faisal & Gani, 2018; Guo & Bi, 2015).

Figure 1 shows the relationship between pyrolysis temperature and the amount of liquid smoke produced. An increase in temperature leads to higher pyrolysis yields, but decreases above 200 °C. The optimum temperature of the cocoa pod pyrolysis process is in the range of 212 °C which produces a yield of 20.05%. The resulting yield is almost close to the existing trend. Based on the results of previous studies (Gani et al., 2024), the yield produced in general is 31.65%. This result was obtained using coconut fibre raw materials. According to Oramahi et al. (2013), at low temperatures (<400°C), decomposition of hemicellulose and some lignin occurs, resulting in relatively high liquid smoke yields. This is due to the fact that pyrolysis at low temperatures tends to produce more volatile compounds, which are then condensed into liquid smoke. However, at higher temperatures (between 400 to 600 °C), the decomposition of cellulose and lignin increases, leading to a decrease in liquid smoke yield. At high temperatures, the volatiles produced are more likely to become gaseous due to further thermal decomposition, which results in less liquid condensate (Kan et al., 2016). Thus, as pyrolysis temperature increases, liquid smoke yield generally decreases, while gas production increases.

GC-MS Analysis of Liquid Smoke

Liquid smoke obtained from pyrolysis at a temperature of 212 °C was analyzed by GC-MS. The chromatogram peaks and compounds that make up liquid smoke with the mass fraction and type of each compound can be seen in Figure 2 and Table 2. The results of GC MS analysis for liquid smoke from cocoa fruit skin in North Kolaka Regency produced several potential chemical compounds such as the carboxylic acid group, alcohol, ester, alkane, and several aromatic compounds.

The results of GC-MS analysis showed many potential compounds in cocoa pod shell liquid smoke such as carboxylic acid compounds, alcohols, and phenols. Acetic acid compounds were found in the highest concentration between 6.74 – 28% in the retention time range of 2.9 – 5.6 minutes. This indicates that the chemical components of cocoa pod shell liquid smoke undergo significant decomposition of hemicellulose and cellulose, resulting in the formation of many acids.

The phenol compound (Phenol, 2-methoxy-(CAS) Guaiacol) was identified at a retention time of 13.425 minutes with a concentration of 4.18%. Other phenol compounds identified were 2-Methoxy-4-methylphenol and Phenol, 2,6-dimethoxy-(CAS) 2,6-Dimethoxyphenol with concentrations of 2.50% and 2.44%. Phenol is one of the important compounds that has many benefits. The benefits of phenol are mainly related to its unique chemical properties, namely as an aromatic compound containing a hydroxyl group (-OH) bound to a benzene ring (Manmeen et al., 2023). One of its main applications is in the manufacture of plastics, resins, and other industrial chemicals. Phenol is used as a raw material in the synthesis of polycarbonate, nylon, and various phenol-formaldehyde resins that are widely used in the automotive and construction industries. In the agricultural sector, phenol serves as a precursor to many pesticides and herbicides, which are essential for protecting crops from pests. Meanwhile, phenol-derived compounds, such as bisphenol A (BPA), play a role in the production of heat-resistant polymers used in various consumer products such as electronic appliances and food containers (Urrutia et al., 2022).

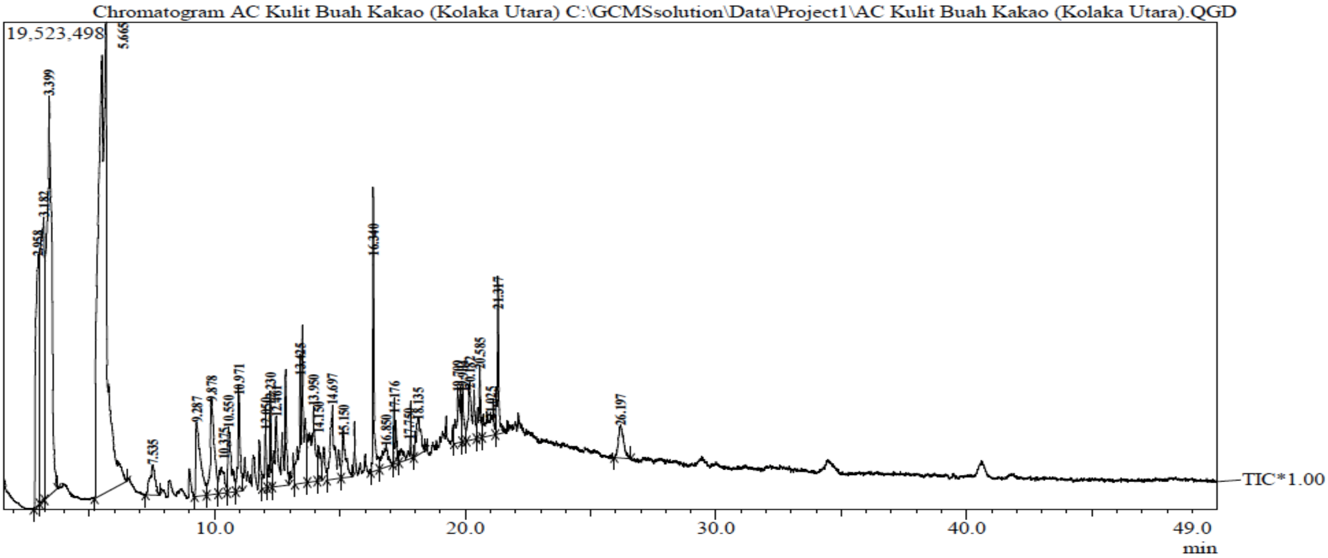

Figure 2. Chromatogram of liquid smoke

Table 2. Composition of Liquid Smoke Compounds at Pyrolysis Temperature of 212 °C

Name of Compound	Conc. (%)	Retention Time (minute)
Acetic acid (CAS) Ethylic acid	6.74	2.958
Acetic acid (CAS) Ethylic acid	7.47	3.182
Acetic Acid, Anhydride With Formic Acid	15.08	3.399
Acetic acid (CAS) Ethylic acid	28.11	5.665
Pyridine (CAS) Azine	1.12	7.535
3-Hexyne-2,5-diol (CAS) Hexyne-3-diol-2,5	2.5	9.287
3-Hexyne-2,5-diol (CAS) Hexyne-3-diol-2,5	2.94	9.878
Pentane, 2-bromo- (CAS) 2-Bromopentane	0.92	10.375
2(3H)-Furanone, dihydro- (CAS) Butyrolactone	1.59	10.550
2(3H)-Furanone, dihydro- (CAS) Butyrolactone	1.68	10.971
Butanoic acid, 2-propenyl ester (CAS) Allyl N-Butanoate	0.91	12.05
2-Furanmethanol, tetrahydro- (CAS) Tetrahydrofurfuryl alcohol	1.02	12.23
Benzenesulfonic acid, 4-hydroxy- (CAS) Benzenesulfonic acid, p-hydroxy-	3.62	12.461
Phenol, 2-methoxy- (CAS) Guaiacol	4.18	13.425
1-Tetracosanol (CAS) Tetracosanol	2.61	13.95
Nona-3,5-Dien-2-ol	0.76	14.15
2-Methoxy-4-methylphenol	2.50	14.697
Oxirane, 2-butyl-3-methyl- (CAS) 2,3-Epoxyheptane	1.18	15.15
Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol	2.44	16.34
Benzene, 1,2,3-trimethoxy- (CAS)	0.75	17.17
1,6-Anhydro-Beta-D-Glucopyranose (Levoglucosan)	1.17	18.135
2-Propenal, 3-(1-aziridinyl)-3-(dimethylamino)- (CAS) 3-Aziridino-3-Dimethylamino	1.59	19.70
2-Octene, 3,7-Dimethyl-, Cis/Trans	0.63	19.90
1,4-diaza-2,5-dioxobicyclo[4.3.0]nonane	1.74	20.18
1,4-diaza-2,5-dioxo-3-isobutyl bicyclo[4.3.0]nonane	0.92	20.585
5,10-Diethoxy-2,3,7,8-Tetrahydro-1h,6h-Dipyrrolo[1,2-A;1’,2’-D]P	1.72	21.317
Stigmasta-3,5-Dien-7-One (Cas) .Delta.-3,5-Sitostadiene-7-One	1.19	26.197

Benzenesulfonic acid, 4-hydroxy- (CAS) aromatic compounds were also found in high concentrations of 3.62% at a retention time of 12. 46 minutes, and complex alcohol compounds such as 3-Hexyne-2,5-diol (CAS)

Hexyne-3-diol-2,5; 1-Tetracosanol (CAS) Tetracosanol; and 2-Furanmethanol, tetrahydro-(CAS) Tetrahydrofurfuryl alcohol were found in concentrations of 2.94, 2.61, and 1.02%, respectively. The large number of potential compounds found in cocoa waste liquid smoke indicates that cocoa pod shell waste has the potential to be further explored.

Characterisation of Cocoa Pod Shell Waste Charcoal

FTIR Analysis

Figure 3 shows the FTIR Analysis spectrum for cocoa pod husk charcoal from North Kolaka Regency. The spectrum shows absorption at 873.75-617.22 cm^{-1} which is the peak of C=C-H (aromatic H). At wave number 1029.99 cm^{-1} is the C-O absorption peak which indicates dehydration and depolymerization for cellulose and hemicellulose content (Kafouris et al., 2020).

Changes in the aromatic peak at 1589.34 cm^{-1} indicate the presence of C-H from lignin and the peak at wave number 3408.22 cm^{-1} indicates the presence of hydroxyl groups (O-H). The results of this study are supported by Hu et al. (2021) that the FTIR analysis for Coir Pith Black Liquor (CBL) showed 3420 cm^{-1} indicating OH, absorption of 1610 cm^{-1} indicating the presence of lignin C-H groups, absorption of 1247 cm^{-1} indicating the presence of C-O groups and 586-891 cm^{-1} indicating the presence of C=C-H groups (aromatic H).

SEM Analysis

Figure 3 shows SEM micrographs of the surface morphology of charcoal samples from cocoa pod skin. The image shows a porous structure with several large voids in the micropore scale of 20 µm. This porosity is one of the common characteristics of charcoal material, which is produced during the pyrolysis process. The voids are probably formed by volatile compounds that are released from the biomass during the pyrolysis process, leaving behind a lighter, porous structure (Hu et al., 2021; Qiao et al., 2024).

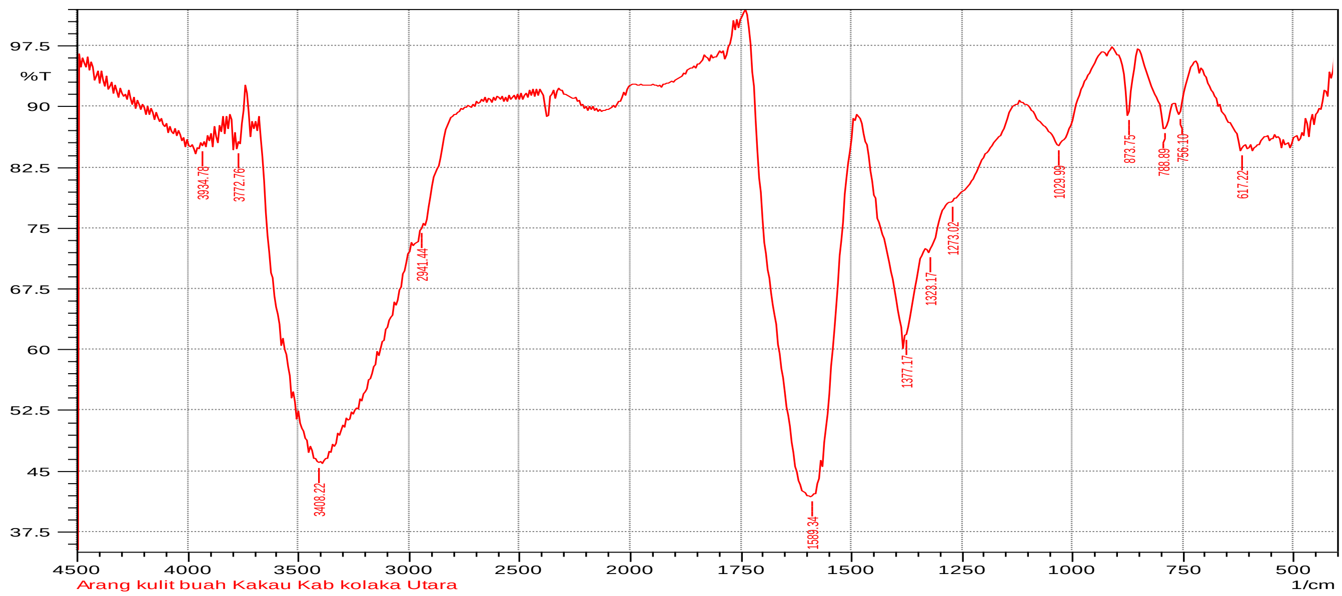

Figure 3. FTIR spectra of cocoa pod shell waste charcoal

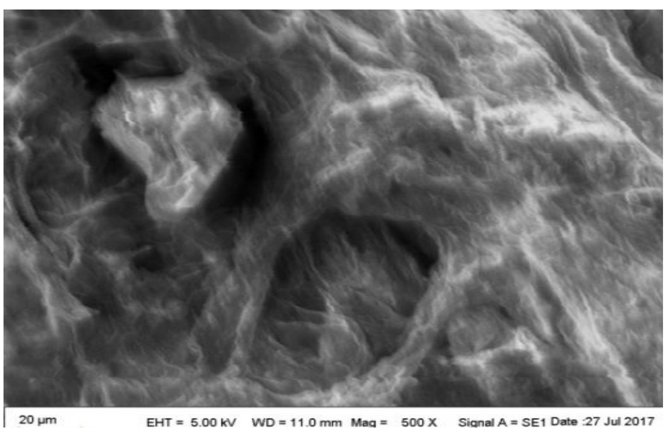

Figure 4. SEM micrograph of cocoa pod shell charcoal

More specifically, the charcoal surface in these micrographs is uneven, with indentation patches and non-uniform layers. The rough surface shows that the biomass was severely thermally damaged, resulting in micro-fragments with a wrinkled structure (Su et al., 2024). This porous structure is crucial due to the influences of charcoal adsorption capacity, particularly in applications such as water filtration or pollutant absorption. These pores of varying sizes can improve the specific surface area of porous carbon materials, which is generally seen as a positive feature (Kafouris et al., 2020).

XRD Analysis

Figure 5 shows the results of XRD analysis for cocoa fruit skins of North Kolaka Regency. The sharp peaks observed around 22–23° and 43–44° (2θ) indicate the presence of certain crystalline phases in the char. These peaks can be attributed to the graphitic structure (sp^2 carbon) formed during the high-temperature decomposition of biomass during pyrolysis (Elyounssi & Halim, 2014). This phase is generally produced by the high-temperature pyrolysis of biomass, where some carbon is organized into a graphite-like structure.

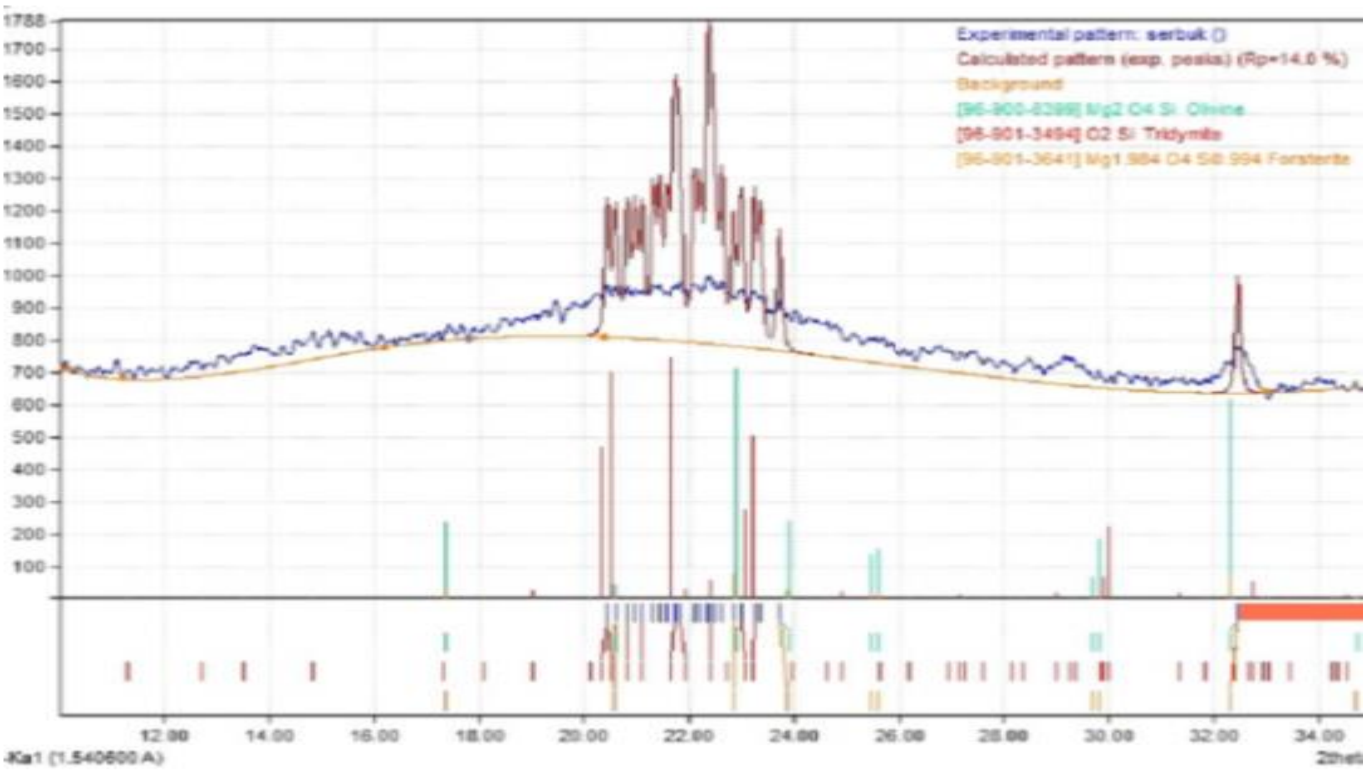

Figure 5. XRD diffractogram pattern of cocoa pod shell charcoal

The XRD pattern also shows low grass-like peaks, particularly in the low-angle region, indicating the presence of substantial amorphous phases. In the case of charcoal, this amorphous phase results from the unstructured carbon structure that is common in biomass-based pyrolysis products. This is prevalent in charcoal products because not all carbon is properly organized into a crystalline structure, particularly at moderate pyrolysis temperatures (Pijarn et al., 2021). This is consistent with the study's findings, which show that the described charcoal samples were produced through pyrolysis at a relatively low temperature of 212 ^oC.

Based on the results of the interpretation of XRD data using the Reitica program, it was obtained olivine type of 77.0%, with the formula form Mg_2O_4Si, orthorhombic crystal system, and density of 3.217 g/cm^3. In addition, 23.0% trydynite was also obtained, with the formula form SiO_2, orthorhombic crystal system, density of 2.297 g/cm^3, and degree of crystallinity of 14.0%. Overall, XRD analysis indicates that cocoa pod shell charcoal consists of a combination of amorphous and crystalline phases, with dominant amorphous carbon and a small number of crystalline phases identified as graphite and possibly other compounds. This structure indicates that pyrolysis produces charcoal products with physical properties that allow applications in adsorption or catalysis, considering the presence of porous structures and amorphous phases that play a role in the adsorbent properties of charcoal.

Conclusion

Pyrolysis of cocoa pod waste from the North Kolaka Regency has been carried out. Pyrolysis of cocoa pods at various temperatures produces liquid smoke and charcoal products. The highest yield of liquid smoke was obtained at the temperature of 212 ^oC of 20.05% that contains acetic acid compounds with the highest concentration of 28.1%, Phenol, 2-methoxy- (CAS) Guaiacol with the highest concentration of 4.18%, 3-Hexyne-2,5-diol (CAS) Hexyne-3-diol-2,5 of 2.94%, and several groups of potential aromatic and alcohol compounds. FTIR analysis shows that the typical functional groups of cocoa pod charcoal consist of OH (hydroxyl) and C=C-H (aromatic) groups. XRD analysis shows that charcoal is dominated by amorphous forms with a degree of crystallinity of 14%. The results of the analysis of liquid smoke and charcoal show that the pyrolysis product of cocoa pods can be used as a raw material in producing potential compounds such as carboxylic acids and phenols through liquid smoke, as well as an adsorbent and catalyst for the charcoal product.

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Author Contributions

The main authors Mochammad Wijaya: designing research, conducting research, collecting data, and writing research articles. The authors Muhammad Nur Alam and Muhammad Wiharto, helped prepare the report and research instruments and conducted data analysis.

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Conflicts of Interest

The authors declare no conflict of interest.

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