The cyclone separator application on physicochemical characterization of coconut shell-liquid smoke grade C

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ABSTRACT

The further processing of coconut shell pyrolysis into liquid smoke has been developed in recent years. However, the common technology by directly condensing the smoke has many impurity compounds which decreases the yield of liquid smoke produces. Hence, in this study the cyclone separator was applied to increase the quality of physicochemical content in coconut shell-liquid smoke (CS-LS) grade C. The physical parameters analyzed were yield, pH, density and color. The chemical parameters were analyzed by GC-MS. The result showed that CS-LS processing with cyclone separator was able to increase the total yield into 3.33%, with better color, pH and density compared to the CS-LS produced using common method (direct condensation). The application of cyclone separator was also able to increase the phenolic compounds (97%), alcoholic compounds (92%), cycloalkene compounds (91%) and to decrease the benzoic acid, carbonyl and ester up to 100% compared with common method for CS-LS grade C production.

Introduction

The high level of coconut plants production is very directly proportional to the coconut shell waste amount produced. In general, the coconut shell waste utilization is only made into handicrafts, charcoal, and some others are discarded without further processing. Dry coconut shell still contains 33.61% cellulose, 36.51% lignin, 29.27% pentose and 0.61% ash (Shelke et al., 2014), so that it can be used as raw material for liquid smoke production (Lombok et al., 2014). Liquid smoke is the condensate liquid from pyrolysis of organic compounds in an airtight state at high temperatures. Liquid smoke can be classified into 3 levels, namely, grade C, grade B, and grade A which have different functions and selling points (Kailaku et al., 2017).

The liquid smoke processing initialized with the pyrolysis process of coconut shell at the temperature range of 300-400 °C (Budaraga et al., 2016) and followed by condensation to produce liquid smoke grade C. The coconut shell-liquid smoke (CS-LS) grade C has been used for various purposes, such as wood preservatives, rubber coagulants and odour absorber (Kailaku et al., 2017). The most important point that needs to be considered in the processing of liquid smoke, is the existence of a pyrolysis smoke conditioning process that is free from tar, ash and other impurities (Shen et al., 2017). This requires special attention because the impurities will affect the yield and quality of each grade of liquid smoke produced. Therefore, it is necessary for separating tar, ash and other impurities in the pyrolysis smoke before it is condensed into CS-LS grade C. The cyclone separator technology has been reported by some researchers as an efficient technologies (70%) in the gas phase separation process when compared to other separation technology (Sakin et al., 2017; Marinuc and Rus, 2011; Chen and Shi, 2007). Cyclone separator can eliminate particulate matter from air, gas, or liquid, through a separation with the principle of centrifugal and gravitational forces. The main principle of this technology is separation based on differences in molecular weight (Taiwo et al., 2016).

In previous study, cyclone separator was able to increase the amount of CS-LS grade C, increase the phenolic functional groups and also reduce carbonyl functional groups (as impurities) when
compared with direct condensation (Putranto, et al., 2020a). However, their research suggests analyzing the CS-LS chemical compounds quantitatively to explain how much percentage of the content of each liquid smoke constituent compounds due to the existence of cyclone separator. Therefore, this study aimed to determine the physical and chemical characteristics of CS-LS grade C using cyclone separator, and compared with the common method for CS-LS grade C production.

Research Methods
The apparatus used are the processing unit of pyrolysis, cyclone separator, water pump (Nikita NS-1200), faucet, hose, buckets, glass beaker (Pyrex Iwaki TE-32), calorimeter (PCE-CSM 4), pH meter (Trans Instrument Senz pH-Scientific), pycnometer (Pyrex Iwaki PICNO-M25-C), and GC-MS (HP-6890). The materials used in this study include dried coconut shell and distilled water.

CS-LS Grade C Production
The common method for production CS-LS grade C was a direct condensation method based on Putranto et al. (2020a) study. The CS-LS grace C production starts from the pyrolysis process through pyrolysis unit designed with input capacity of 3000 kg coconut shell. Pyrolysis tank has dimensions of 2.5 m length, 1.5 m width and 1 m height. After that, the smoke was condensed directly through the condenser tank that will produce CS-LS grade C. Meanwhile, the process mechanism of CS-LS grade C production by using cyclone separator technology (Figure 1) started from pyrolysis process which then flowed into the input pipe cyclone separator and subsequently flowed into the condenser tank. The next process was condensation in the condenser which is equipped with a spiral pipe with 12 m length, made of stainless steel to expand contact with cooling water. The process output is referred to as CS-LS grade C with cyclone separator. Design of equipment for CS-LS grade C production including cyclone separator design was according to Fathussalam et al. (2019) study. The cyclone separator was also equipped with a centrifugal blower that has fluid velocity of 280 CMH.

Characterization and Analysis Method
In this study, there were CS-LS grade C produced both by common processes (directly condensation) and using cyclone separator which were tested for their physical and chemical characteristics. The physical parameters including yield, pH, colour (Hunter method) and density (pycnometer method). The chemical parameters were characterized by GC-MS to determine the types of compounds contained in CS-LS. In addition, CS-LS was also compared with Standard Quality of Wood Vinegar Japan.

Results and Discussion
Physical Characteristics of CS-LS Grade C
The physical characterization of CS-LS grade C by using a common method and cyclone separator method can be seen in Table 1. Around 3 tons of coconut shell is used as the input process of CS-LS production. On the direct condensation method, the yield of CS-LS grade C was 1.67%, while the yield of CS-LS by using cyclone separator was 3.33%. This shows that the cyclone separator technology application can increase the yield twice when compared with the common method. The filtering process of smoke from pyrolysis tank by cyclone separator can produce cleaner smoke which indirectly makes the condensation process more optimal and increases the yield of liquid smoke (Shen et al., 2017).

Figure 1. Illustration of CS-LS grade C production by using cyclone separator
Table 1. Physical parameters of CS-LS grade C

| Physical Parameters | Units | Kailaku et al. (2017) | Budaraga et al. (2016) | Direct Condensation Method (Putranto et al., 2020b) | Cyclone Separator Method | Quality Standard of Wood Vinegar Japan (Yatagai, 2002) |
|---------------------|-------|-----------------------|-----------------------|---------------------------------------------------|------------------------|-----------------------------------------------|
| Yields              | %     | -                     | 34.42                 | 0.83                                              | 3.33                   | -                                             |
| pH                  |       | 2.76                  | 3.50                  | 4.40                                              | 3.16                   | 1.50 - 3.70                                   |
| Density             | kg/L  | -                     | 1.027                 | 1.021                                             | 1.019                  | >1.005                                        |
| Color L             |       | 29.87                 | 2.58±0.01            | 14.50±0.01                                       | 14.73±0.0             | 4                                             |
| a                   |       | 39.37                 | 0.42±0.02            | 2.86±0.06                                        | 4.33±0.28             |                                              |
| b                   |       | 47.92                 | 1.67±0.02            | -0.21±0.04                                       | 0.06±0.08             |                                              |
| c                   |       | -                     | -                    | 2.87±0.06                                        | 4.26±0.19             |                                              |
| h                   |       | -                     | -                    | 355.84±0.8                                       | 0.81±0.97             |                                              |
| Colour quality      |       | Dark and cloudy       | Light-brown          | Dark-brown                                       | Reddish yellow-brown  | Reddish yellow-brown to reddish pale-brown   |

Based on Table 1, the pH value of CS-LS with cyclone separator (3.16) is in line with the quality standard of wood vinegar Japan. The use of cyclone separator can reduce the pH value of liquid smoke grade C. If the pH value is low, the liquid smoke has high quality. The lower pH value also increases antibacterial properties. The lower pH values also affect the organoleptic characteristics, durability, and product storability (Budaraga et al., 2016). The density values of the direct condensation method and cyclone separator method did not show a significant difference (Table 1). However, the density values are also in accordance with the quality standard of Japanese wood vinegar (Yatagai, 2002). The density value of CS-LS can be influenced by the presence of tar compounds in liquid smoke that cannot dissolve and also the presence of other heavier compounds.

Furthermore, the direct condensation of CS-LS grade C has a dark-brown colour which is characterized by the hue value of 355.84±0.89. Meanwhile, the using cyclone separator has a hue value of 0.81±0.97, which indicates a reddish yellow brown colour. The output of CS-LS production using cyclone separator was also in-line with quality standard of wood vinegar from Japan. The colour difference is influenced by the tar and carbonyl compound presence which affects the appearance of CS-LS becoming darker (Budaraga et al., 2016). The brighter colour on CS-LS using cyclone separator, indicates that tar and carbonyl have been filtered. The quantitative value of the reduction in those compounds is explained in the chemical characteristic section. The brighter colour in CS-LS shows a better quality of CS-LS. On the other hand, the liquid smoke colour can be influenced by temperature and pyrolysis technology which causes degradation of cellulose, hemicellulose, and lignin.

**Chemical Characteristics of CS-LS Grade C**

The GC-MS analysis (Table 2) showed that phenols are the most dominant compounds in both direct condensation method and cyclone separator method. The phenolic compounds contain high antimicrobial to prevent spoilage microbes. This character made CS-LS grade C widely used as a wood preservative. In addition, the CS-LS grade C using cyclone separator also contained several compounds including alcoholic, alkoxy, and cycloalkene compounds, whereas direct condensation method contained acid, alkoxy, ester, carbonyl and cycloalkene compounds.

Based on Table 2, there were significant differences in chemical compounds of CS-LS produced by using direct condensation and using cyclone separator method. The phenolic compound detected in CS-LS with direct condensation was 40.73%, whereas with CS-LS with cyclone separator it was 80.39% (increased 97%). The alcoholic compound was also increased around 92%, where by using the common method of CS-LS the percentage was 0.51% and with cyclone separator the percentage was 6.29%. The cycloalkene compounds in CS-LS using cyclone separator technology increased by around 91% when compared with common method of CS-LS.
Furthermore, there were 17.55% alkoxy compounds in the product from common method of CS-LS and 5.33% in the liquid smoke produce using the cyclone separator method (decreased around 70%).

Table 2. Chemical characteristics of CS-LS grade C

| Chemical Compounds | Direct Condensation (Putranto et al., 2020b) | Cyclone Separator (This study) |
|-------------------|---------------------------------------------|--------------------------------|
|                   | Retention Time | Percent Area | Retention Time | Percent Area |
| Phenolic Compounds |                |              |                |              |
| Phenol            | 2.922          | 11.45        | 2.910          | 21.26        |
| Phenol, 2-methyl  | 3.688          | 1.25         | 3.670          | 2.61         |
| Phenol, 4-methyl  | 3.905          | 1.72         | 3.882          | 2.28         |
| Phenol, 2-methoxy | 4.185          | 6.32         | 4.179          | 21.21        |
| Phenol, 2,3-dimethyl | 4.842    | 0.40         | -              | -            |
| Phenol, 3-ethyl   | 5.076          | 0.69         | 5.059          | 0.42         |
| 2,5-Xylenol       | 5.425          | 0.24         | -              | -            |
| Phenol, 2-methoxy-4-methyl | 5.499 | 2.75 | 5.482 | 8.80 |
| Phenol, 4-ethyl-2-methoxy | 6.660 | 0.67 | 6.642 | 5.67 |
| Phenol, 2,6-dimethoxy | 7.608 | 12.90 | 7.591 | 14.15 |
| Phenol, 2,6-dimethoxy-4-(2-propenyl) | 11.209 | 0.92 | 11.175 | 0.47 |
|                  |                |              |                |              |
| 2-Ethylidiphenylmethane | 11.335 | 1.42 | -              | -            |
| Phenol, 2,6-dimethyl | -            | -           | 4.362          | 0.40         |
| Phenol, 2,4-dimethyl | -            | -           | 4.813          | 1.13         |
| Phenol, 2-methoxy-3-methyl | -          | -           | 5.305          | 0.61         |
| Phenol, 4-methoxy-3-methyl | -          | -           | 5.402          | 0.84         |
| Phenol, 2-methoxy-4-(1-propenyl) | -          | -           | 8.888          | 0.54         |
|                  |                |              |                |              |
| TOTAL             | 40.73          | 80.39        |                |              |
| Alkoxy Compounds  |                |              |                |              |
| 2,3,5-Trimethoxytoluene | 9.940  | 8.20        | -              | -            |
| 2,3,5-Trimethoxytoluene Benzene | -       | -           | 9.912          | 4.75         |
| 1,2,5-trimethoxy-3-methyl | -       | -           | -              | -            |
| 2-Methoxy-4-propyl-phenol | -       | -           | 10.020         | 0.58         |
|                  |                |              |                |              |
| TOTAL             | 8.20           | 5.33         |                |              |
| Benzoic Acid Compounds |            |              |                |              |
| Benzoic acid, 2,5 | 8.557          | 6.47         | -              | -            |
| Benzoic acid, 2,4 | 8.671          | 7.25         | -              | -            |
| 3-Hydroxy-4-methoxybenzoic acid | 8.854 | 9.35 | -              | -            |
| Benzoic acid, 4-hydroxy-3-methoxy | 9.832 | 0.52 | -              | -            |
|                  |                |              |                |              |
| TOTAL             | 23.85          | 0            |                |              |
| Ester Compounds   |                |              |                |              |
| Vanillin          | 8.283          | 0.60         | -              | -            |
|                  |                |              |                |              |
| TOTAL             | 0.60           | 0            |                |              |
| Alcoholic Compounds|               |              |                |              |
| Ethanone, 1-(4-hydroxy-3-methoxyphenyl) | 9.426 | 0.51 | -              | -            |
| 2H-Pyran-2,4(3H)-dione, 3-acetyl-6-methyl | -       | -           | 8.831          | 6.29         |
|                  |                |              |                |              |
| TOTAL             | 0.51           | 6.29         |                |              |
| Carbonyl Compounds|                |              |                |              |
| 3-Hexene, 3,4-dimethyl | 3.465      | 0.24         | -              | -            |
|                  |                |              |                |              |
| TOTAL             | 0.24           | 0            |                |              |
| Cycloalkene Compounds|             |              |                |              |
| 2,3-Dimethyl-2-cyclopenten-1-one | 4.053 | 0.21 | -              | -            |
| 2-Cyclopenten-1-one, 2,3-dimethyl | -         | -            | 3.636          | 0.40         |
| 2-Cyclopenten-1-one, 3-ethyl-2-hydroxy | -         | -            | 4.522          | 0.41         |
|                  |                |              |                |              |
| TOTAL             | 0.21           | 2.26         |                |              |
Table 2 also showed that the cyclone separator application eliminated the benzoic acid, carbonyl and ester compounds up to 100% in the CS-LS grade C produced. The compound that was removed during the separation process in the cyclone separator is mostly semi-solid liquids in the form of tar and benzo(a)pyrene which are indicated as CS-LS grade C impurities (Budaraga et al., 2017). The compounds included in benzo(a)pyrene in this study was benzoic acid functional group such as 3-hydroxy-4-methoxy benzoic acid, benzoic acid 2,5, benzoic acid 2,4 and benzoic acid 4-hydroxy-3-methoxy. Benzo(a)pyrene is one of the Polycyclic Aromatic Hydrocarbons (PAH) which can be formed in CS-LS and is well known as a carcinogenic compound (Budijanto et al., 2008). The factor that greatly influences the formation of PAH compounds is the pyrolysis temperature. Budijanto et al. (2008) reported that PAH compounds including benzo(a)pyrene were not found in CS-LS on pyrolysis temperature under 400 °C. Even though this study used a pyrolysis temperature of 350 °C, the benzoic acid functional group (indicated as one of benzo(a)pyrene compound) was detected, and even reached 23.85% (Table 2). Therefore the cyclone separator is one of the most important components that must be considered to reduce carcinogenic compounds such as benzo(a)pyrene which can still be formed in the CS-LS produced by pyrolysis temperature under 400 °C.

A significant reduction in benzoic acid compounds through the application of cyclone separator technology indicates that this technology can be applied and developed to produce high quality of CS-LS grade C and at the same time, it is potential to further processes like re-distillation and filtration to produce high quality of CS-LS grade A (as a food preservative). The presence of benzo(a)pyrene in liquid smoke used as food preservatives must be reduced since it very dangerous for humans due to its carcinogenic nature (Budaraga et al., 2016).

To get quality of CS-LS grade A as a natural preservative of food, the phenol content in liquid smoke has a very important role. According to Girard (1992), the quantity and quality of phenolic compounds contained in liquid smoke are directly related to lignin content and pyrolysis temperature. The pyrolysis temperature differences may affect the lignin degradation process, which directly affects the content of phenolic compounds in the liquid smoke (Budaraga et al., 2016). The different temperatures (between 181-500 °C) used in pyrolysis coconut shell may produce different pH value and total phenols, as summarized in Table 3.

According to Budaraga et al. (2016), the temperature of 400 °C is the best temperature for the pyrolysis process of coconut shell, because at this temperature, the cellulose component in the raw material could produce organic acids and higher phenolic compounds. On the other hand, pyrolysis temperature of 350-420 °C was chosen because in that temperature range, it can produce CS-LS with a high phenolic content (90.75%) and has carcinogenic or polyaromatic hydrocarbon (PAH) compounds which are smaller than at temperatures below 350 °C and above 450 °C (Hadanu and Apituley, 2016). However, in this study, the pyrolysis temperature was set at 350 °C which is intended to reduce the energy needed during the pyrolysis process. When compared with the pyrolysis temperature of 400 °C, total phenol produced from several studies was 1.73% (Budaraga et al., 2016), 4.63% (Yuningsih and Anggarini, 2013), and 43.6% (Surboyo et al., 2019). Meanwhile, at pyrolysis temperature of 450 °C, total phenolic content 38.37% (Swastawati, 2008). At pyrolysis temperature of 500 °C, the total phenolic produced was 4.62% (Yuningsih and Anggarini, 2013). With the pyrolysis temperature used in this study, coupled with the cyclone separator, the high total phenolic content of CS-LS (80.69%) can be achieved at a low pyrolysis temperature. This result is in agreement with the study by Rizal et al. (2020) which shows that lignin, which is one of the largest components of coconut shells, was decomposed and produced phenol at a pyrolysis temperature of 300-500 °C. The high phenolic content in CS-LS also has the potential to be continued into a CS-LS nanoparticle solution by adding chitosan and maltodextrin (Ali et al., 2014).

Furthermore, nanoparticles which also contain phenolic compounds can be used as a anti-biofouling agent on the mixed matrix membrane (Wibisono et al., 2020a). The mixed matrix membrane enhanced with phenolic compounds was able to inhibit bacterial growth and potentially used for a safe and non-toxic tool for food processing (Wibisono et al., 2020b).

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In addition, some pyrolysis temperatures show that the pH value of CS-LS is not a significant difference and tends to be acidic. The pyrolysis temperature above 100 °C triggers the decomposition of raw material components and, at higher pyrolysis temperature, the pH value may decrease (Budaraga et al., 2016). The lower pH value would lead to increasing antimicrobial properties of liquid smoke. In acidic conditions, microbial and bacterial growth will be inhibited, coupled with the high phenolic content as an antimicrobial, the CS-LS preservative function becomes more effective. Based on Table 3, the use of cyclone separator as a smoke separator before the condensation process proved to be quite effective and efficient to produce CS-LS grade C with a low pH value, low pyrolysis temperature and a high total phenolic compound when compared with the other studies.

**Conclusions**

The application of cyclone separator can affect the physical (i.e. yield, pH, density, colour) and chemical characteristics of CS-LS grade C. The yield of CS-LS grade C using cyclone separator method increased twice with better colour, yield, and density compared with the common method (direct condensation). The application of cyclone separator has a significant difference in the chemical content of CS-LS grade C. A significant reduction in impurity compounds (benzoic acid and carbonyl), make it suitable for further processing into CS-LS grade A as a natural food preservative. In addition, the better yield and quality of the CS-LS grade C by using cyclone separator as described in this study, also make it potential to be applied to the industrial liquid smoke to improve the quantity and quality of liquid smoke in Indonesia.

**Conflict of interest**

The author declares that there is no conflict of interest in this publication.

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| Pyrolysis temperature (°C) | pH  | Total Phenol(%)* | References |
|---------------------------|-----|-------------------|------------|
| 400                       | 3.5 | 1.73              | Budaraga et al. (2016) |
| 350                       | 2.13| 2.92              | Yuninginsih and Anggarini (2013) |
| 300                       | 1.64| 3.48              | Yuninginsih and Anggarini (2013) |
| 450                       | 1.48| 3.67              | Yuninginsih and Anggarini (2013) |
| 181-200                   | 4.1 | 3.85              | Lombok et al. (2014) |
| 500                       | 1.27| 4.62              | Yuninginsih and Anggarini (2013) |
| 400                       | 1.23| 4.63              | Yuninginsih and Anggarini (2013) |
| 250                       | 3   | 15.16             | Rizal et al. (2020) |
| 450                       | 2.5 | 38.37             | Swastawati, (2008) |
| 400                       | -   | 43.6              | Surboyo et al. (2019) |
| 233                       | 2.71| 74.96             | Sarwendah et al. (2019) |
| 350-420                   | -   | 90.75             | Hadanu and Apituley (2016) |
| 350                       | 3.16| 80.39             | This study |

Note: *Total phenol and its derivative compounds
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