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Clove leaf distillation using briquette fuel with starch and molasses as a binder

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ABSTRACT

Clove is a typical Indonesian plant and one of 40 essential oil export commodities. The critical oil content of cloves is distributed among flowers (15–20%), stems (5–7%), and leaves (1–4%). The simplest way to obtain clove oil is by water and steam distillation, commonly called water and steam distillation. In this research, clove leaf distillation uses briquettes made from bagasse with starch and molasses adhesive as fuel. These briquettes are made from bagasse with starch and molasses adhesive. The calorific value of briquettes prepared with starch and molasses adhesives was 28.996 MJ/kg and 27.019 MJ/kg, respectively. This calorific value is above the calorific value of SNI briquettes (20.934 MJ/kg). For comparison, the SNI for briquettes made in Japan, England, and America are 25.121 – 29.307, 30.518; 26.084 (MJ/kg) respectively. Clove leaf distillation at a temperature of 200 °C and a pressure of 1 atm for 8 h. The briquette requirements during the refining process are 20 kg and 23.5 kg, respectively, for briquettes made with starch and molasses. Clove leaf oil resulting from the distillation process is purified by an adsorption process using natural zeolite as an adsorbent. Furthermore, analysis of clove oil using GC–MS showed that the total eugenol content in the samples distilled using briquettes with starch and molasses binders was 90% and 87%, respectively. This value is greater than the total eugenol of clove leaf oil based on SNI (78%). Based on the overall analysis results, it can be concluded that the characterization of the calorific value of briquettes affects the quality of clove leaf oil.

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1. Introduction

There are many industries that use clove oil, such as the cosmetic industry [1], medical [2], food and beverage [3] and fuel oil additives [4]. Eugenol, which accounts for 87.52–96.65% of the oil's composition [5,6], is the most important constituent of clove oil [7]. Clove oil is derived by the distillation of clove flowers, stems, and leaves (Eugenia aromatic). Water distillation (hydro-distillation) or steam distillation (steam-distillation) can be used in the distillation process [8]. The yield of clove oil as a distillation product is controlled by the density of the material and the duration of distillation. The greater the density of the substance, the greater the number of possible oil sources that can be distilled. Similar to the length of refining, the longer the distillation time, the more oil that can be extracted from the material. Material density and distillation duration are critical variables that can work together to maximize yields [9,10].

Operating conditions, maximum column load, and distillation time all have an impact on the essential oil refining process. This is true for clove oil as well as other essential oils. The refining procedure frequently fails to yield the desired outcomes. Overloading the distillation column to avoid high fuel consumption and lower operating costs is one of these restrictions. Another is column under-filling. As a result of the over-refining process, the density of the material increases, making it more difficult for steam to reach all of the substance's surface. As a result, there is a decrease in oil production [9,10]. Overloading the distillation column will have no effect on the final result; it is a waste of time and money. The distillation time in the essential oil industry is based on the first distillate coming out of the condenser. This refining time can be a non-optimal refining time by considering that if the optimal load can be obtained, the optimal refining time can also be obtained. Combining material density and refining time at its

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optimum point can provide high essential oil yields and meet the density requirements [10].

Improving the quality and quantity of clove leaf oil for farmers is carried out through modification and development of the production process so that it is expected to be able to increase the competitiveness of Indonesian clove leaf oil products, which in turn will increase the income of clove farmers, increase regional income and can increase foreign exchange.

Clove leaf oil has a high selling value to increase the eugenol content and make the color bright (clear) by purification (through the adsorption process). Several refining results show that the oil can be purified by the adsorption method using complexometry [11], redistillation [12], EDTA [13], tartaric acid, and bentonite [14]. In this study, natural zeolite was used as an adsorbent.

Clove oil distillation takes around 7–8 h at a stable temperature of 300–500°Celsius, whereas clove leaf distillation can be completed in less than an hour at a stable temperature over 200°Celsius [15]. Because the amount of energy required for clove leaf distillation is not prohibitively high, it is more likely to be used by local firms. It is important to investigate alternatives to fossil fuels, such as firewood, in order to cut operating costs in the home business. In this study, briquettes from bagasse were utilized as a substitute for fossil fuels in the distillation process, with starch and molasses serving as binders. Briquettes from bagasse were created with starch and molasses as binders. Briquettes can be used to replace fossil fuels, which can save operating expenses.

Based on the description and previous research, it is necessary to study the replacement of fossil fuels with briquettes. Heating value, one of the factors that affect the combustion process, is studied by comparing the effect of briquette binders (starch and molasses) on the clove leaf distillation process.

2. Materials and method

2.1. Materials

As fuel in the clove leaf refining process, briquettes are produced from sugarcane bagasse from the rest of the manufacture of PG sugar. Malang Grand Garden. Briquettes are made with starch and molasses as glues.

The clove leaves used in this study were obtained from Kediri, East Java, Indonesia. Clove leaves were conventionally dried for four days and then dried in the sun until the moisture content was less than 10%. Clove leaves are cut into 2 cm pieces to increase the surface area.

The resulting clove leaf oil is purified using a natural adsorbent, namely natural zeolite, obtained from mountainous areas in South Malang. This zeolite is reduced in size to 60 mesh. Because of its porous characteristics, zeolite is used as a natural adsorbent. The reduction in size to 60 mesh is intended to increase the surface area so that the adsorption process can occur properly. Before use, the zeolite was heated in an oven for 1 h at 100 °C. Heating is done to open the zeolite pores and minimize the occurrence of diffusion barriers in the adsorption process.

2.2. Method

2.2.1. The briquette making process

The process of making briquettes follows previous research [16,17]. Making briquettes consists of four stages: size reduction, composting, size separation, mixing, and printing. In stage 1, the bagasse is reduced by chopping the bagasse to a uniform size of 60 mesh in a chopping machine. In the second stage, the bagasse was put into the machine with an operating condition of 230C for 120 min. The resulting charcoal is cooled and crushed. The third step is to put the crushed charcoal into a glass beaker filled with water. Let it stand for 24 h to separate into two layers, namely top and bottom. The bottom layer is used as raw material for making briquettes. The mixing stage is carried out by mixing bagasse charcoal, starch, and molasses in a ratio of 1–5% of the weight of the charcoal, respectively. The starch was first dissolved in hot water (70C) according to the variation of the addition ratio, constantly stirring until homogeneous. After the mixing stage is completed, the next step is the briquette printing process, using a briquette printing machine. The pressure given is 8 kg/cm2. The briquettes are dried using an oven for 24 h at a temperature of 120C. Briquettes prepared with starch adhesive are denoted as B1, and briquettes prepared with molasses adhesive are denoted as B2. Bomb Calorimeter analyzes the calorific value of briquettes.

Furthermore, B1 and B2 are used as fuel for the clove leaf distillation process. These briquettes are placed in a furnace; above this furnace, a distillation column is placed, where the process of making clove leaf oil takes place.

2.2.2. The clove leaf oil making process

Clove leaf oil is made using a steam-distillation process. A total of 1500 g of dried clove leaves were put into a distillation column measuring 25 cm in diameter and 75 cm in height. Water is added to the column at a rate of 20% of the column height (15 cm, calculated from the top of the column). The distillation process lasts for 8 h.

Clove leaf oil is produced from the distillation process, cooled to room temperature, and allowed to stand for 24 h to form a layer of oil and water. Furthermore, clove leaf oil is passed through an adsorber column measuring 20 cm in diameter and 80 cm in height. In the middle of the adsorber column, there is a container for placing the zeolite with a height of 40 cm, precisely 20 cm from the bottom and 20 cm from the top of the adsorber column. Clove leaf oil was passed through zeolite and then analyzed for its eugenol content.

The process of making clove leaf oil is shown in the process diagram in Fig. 1.

2.2.3. Gcms analysis

To determine eugenol compounds in clove leaf oil using GCMS (**Gas chromatography–mass spectrometry**). GCMS is a tool used to analyze compounds in samples. The separation mechanism

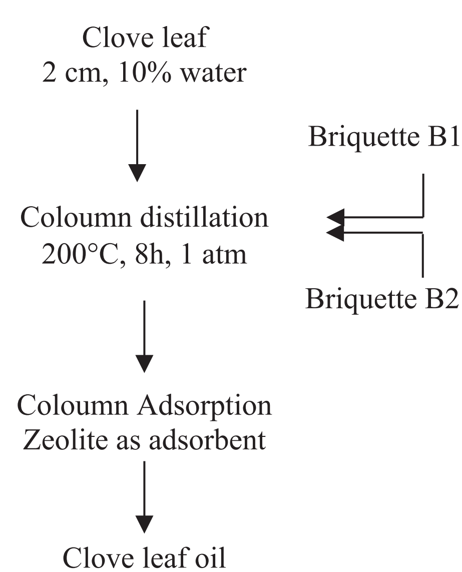

Fig. 1. The process of making clove leaf oil.

between several compounds occurs due to differences in the solubility of each in the moving solvent and in the absorption of each compound into the stationary phase. In gas chromatography (GC), the mobile phase is helium gas. Mass spectroscopy (MS) is a method for analyzing pure compounds that have been separated from GC. By using GCMS, we can identify any compounds contained in the sample, determine the presence or absence of certain compounds in a sample, and determine the level of concentration of compounds in a sample.

2.2.4. Ftir analysis

Analysis FTIR (Fourier Transform Infrared Spectroscopy) is an analytical technique used to identify organic, polymeric, and, in some cases, inorganic materials. The FTIR analysis method uses infrared light to scan test samples and observe chemical properties. This analysis was conducted to determine the presence of compounds contained in clove leaf oil. The specific compound that must be present in clove leaf oil is eugenol.

3. Result and discussion

Clove leaf oil is generally made by refining using wood fuel or LPG (Liquid Petroleum Gas). The availability of wood is running low, and the procurement of wood as fuel can endanger environmental conditions, resulting in flooding. Meanwhile, the use of LPG is not economical because the price is getting more expensive. To overcome problems related to the procurement of refining energy, briquettes made from bagasse are used.

Briquettes from bagasse are prepared with starch and molasses adhesive. This is done to get the best performance from briquettes, following SNI Briquettes. Studies on the effects of adding starch and molasses have been carried out in previous studies [16,17]. The heat test analysis of briquettes (B1 and B2) is presented in Table 1.

Table 1 shows that the briquettes prepared with starch and molasses binders have a higher heating value than the SNI briquettes (20.934 MJ/kg). Henceforth, high-value briquettes fuel clove leaf distillation (28,996 MJ/kg and 27,019 MJ/kg).

Clove leaf distillation for eight hours using briquettes from bagasse with starch and molasses binder to replace wood. The use of briquettes as fuel can reduce operating costs because they are made from biomass.

Clove leaf oil produced from the refining process is further purified by an adsorption process using natural zeolite as an adsorbent. Furthermore, clove leaf oil was analyzed using GCMS. The results of the GCMS analysis can be seen in Table 2.

The results of the GC–MS analysis on clove leaf oil samples produced using briquettes with starch and molasses binder are shown in Table 2.

The total concentration of eugenol in both samples showed a value that exceeded the SNI for clove oil. It can be explained that the resulting clove leaf oil is purified by the adsorption process on the adsorber column. Natural zeolite as an adsorbent with a height of 30 cm was placed in a column with a diameter of 15 cm and a height of 50 cm. The process of refining clove leaf oil with natural zeolite can produce clove leaf oil with a total eugenol of 90% for samples prepared with starch, while samples using briquettes with molasses binder have a total eugenol of 87%.

The use of briquettes prepared with different binders had a significant effect on the eugenol content produced. The eugenol content in the clove leaf oil produced in this study exceeded the SNI for clove leaf oil, as shown in Table 3.

FTIR analysis was carried out to determine the presence of compounds contained in clove leaf oil. The specific compound that must be present in clove leaf oil is eugenol, as presented in Fig. 1.

Fig. 2 shows the FTIR analysis for clove leaf oil samples produced using briquette fuel with starch (B1) and molasses (B2) adhesives. We have seen the peak in the wave 1700 – 1650 cm^{-1}, which shows the spectrum wavelength C = O. This identification indicates the vibrational mode of terpenoids such as eugenol.

Table 1
Heating Value of Briquette.

No	Ratio of Binder	Heating Value (MJ/kg)	
		(B1)[17]	(B2) [18]
1	1%	26.964	27.019
2	2%	21.277	23.300
3	3%	23.313	24.280
4	4%	28.996	24.141
5	5%	28.184	25.113

Table 2
Aromatic compound identified in clove leaf oil.

Sampel	Compound	%
A	Eugenol	40
	Eugenol acetate	32
	Terpenoid	18
B	Eugenol	40
	Eugenol acetate	29
	Terpenoid	18

Table 3
SNI of Clove Oil (*SNI 06–2387-2006).*

No	Parameters	Units	Condition
1	Colour	–	tawny
2	Density	g/mL	1.0250 – 1.0609
3	Refractive index at 28 °C	–	1.52 – 1.54
4	Solubility in ethanol	–	1: 2 (Clear)
5	Total eugenol	%, v/v	Min 78

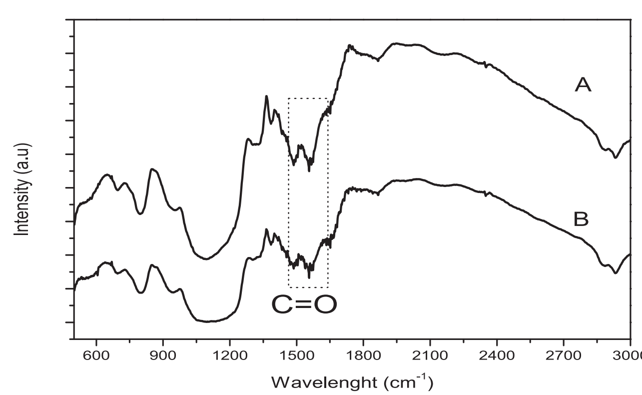

Fig. 2. Spectrum FTIR of clove leaf oil.

Table 4
Effect binder on the burning of briquette.

Parameter	Binder	
	Starch (sampel B1)	Molasse (B2)
Firing up time	13: 09 (min: sec)	15:32 (min: sec)
The Maximum temperature	305 °C	287 °C
Burn-up factor	98%	93%
Amount	20 kg (in 8hr)	23.5 kg (in 8 hr)

In general, the spectrum wavelength for clove oil is 1000 – 1600 cm^{-1}. Fig. 2 also shows the stretching vibration of the aromatic C = C bond at 1514 cm-1 in eugenol, eugenol acetate, and methyl 2-hydroxybenzoate [18–39] and the asymmetrical strain COC bond of the ether functional group (1100–1210 cm^{-1}) in eugenol [38,41].

The GCMS and FTIR analysis results showed the presence of eugenol in clove leaf oil, which was distilled using bagasse briquettes. Starch and molasses influence the quality of clove leaf oil, and its eugenol content exceeds the SNI for clove leaf oil.

The observations in Table 4 can explain the need for briquettes as fuel.

From the analysis results shown in Table 4, it can be seen that the combustion characterization of sample B1 is superior to that of B2. It can also be confirmed from observational data in the field when briquettes are used as fuel in the clove leaf oil distillation process, which takes 8 h. The need for briquettes (B1) is less than that of B2, where the calorific value of B1 is higher than that of B2.

4. Conclusion

Clove leaf oil refining using briquettes as fuel has been successfully carried out. The analysis of the eugenol content with GCMS showed that the number exceeded the SNI for clove leaf oil. Moreover, the FTIR spectrum confirmed the presence of eugenol. Briquettes from bagasse prepared with starch as a binder can be used as a substitute for fossil fuels.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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