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Treatment of Ginger Industrial Waste Using the Carbon Dioxide Supercritical Extraction (CDSE) Method

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Abstract: Ginger industrial waste is available in abundance, unprocessed and harmful to the environment because of its smells. Researchers propose that ginger industrial waste be processed using water distillation and Carbon Dioxide Supercritical Extraction (CDSE) methods. The purpose of this study was to utilize ginger industrial waste, which is currently discarded. Ginger industrial waste was collected from the ginger beverage industry. Essential oil was extracted from the waste using two methods: water distillation and CDSE. Both essential oils were characterized to determine their chemical components and composition, as well as their physical and chemical properties in accordance with the Indonesian National Standard for ginger essential oil (SNI 1312:2021). The characteristics analyzed included specific gravity, refractive index, optical rotation, and chemical compound profile, as determinated using GC-MS. The yields of essential oil obtained from the water distillation and CDSE methods were 0.18% and 0.36%, respectively. The results of the specific gravity, refractive index, and optical rotation tests for the two methods did not differ significantly. The chromatogram profiles of the two methods also did not differ significantly. The study concluded that the CDSE method was superior to the water distillation method. The CDSE method is faster, requires no fuel, produces higher yields, and is environmentally friendly.

Keywords: ginger, waste, distillation, extraction, CDSE.

Introduction

Ginger (Zingiber officinale Roscoe) is a rhizome widely used as a culinary spice worldwide due to its sharp, spicy flavor, primarily due to gingerols and shogaols, and its distinctive aroma associated with volatile components (sesquiterpenes and monoterpenes) [1]. A total of 3.5 million tons of raw ginger were produced globally in 2017, increasing to 4.3 million tons in 2020, with the majority, at 77%, cultivated in Asia [2]. Developing countries, including Indonesia, exported 152,000 tons of dried ginger to Europe in 2020. Typically, fresh ginger rhizomes contain water, oleoresin, essential oil, and non-essential oils [3]. Ginger rhizomes contain 4-10% essential oil, resulting in significant amounts of ginger industrial waste. Ginger waste, which can include stems and leaves, is usually sent directly to landfills, burned, or used as animal feed [4,5,6].

Depending on the ginger source, ginger peel, stems, and leaves can also be waste in industrial processing. However, ginger stems and leaves are typically post-harvest waste and have little or no commercial value, but contain proteins and polysaccharides, such as sugars, starch, cellulose, hemicellulose, and lignin [7,8]. The ginger waste generated from the herbal medicine and beverage industry is enormous [5]. This waste is usually sent to landfills, incinerated [6], or used as low-value animal feed (ginger pulp flour) [9]. Various methods of utilizing ginger industrial waste include adsorbents for wastewater treatment [10] and raw materials for biodiesel production [11]. Ginger industrial waste produced by each industry is approximately 10 kg every day. If in one area there are 10 ginger drink industries, 100 kg of waste is produced per day.

Due to these problems, researchers have proposed a new method for utilizing ginger industrial waste, namely Carbon Dioxide Supercritical Extraction (CDSE), also known as Supercritical Fluid Extraction (CDSE). This method has become the most widely used method for extracting and isolating essential oils from aromatic plants [12,13,14,15,16,17]. In general, the efficiency of CDSE depends on several parameters, including flow rate, pressure, temperature, and extraction time. The CDSE method has been successfully used for the extraction of essential ginger oil [18,19]. Essential oil extraction using distillation has several drawbacks, such as long extraction times, high fuel consumption, air pollution, and high costs. CDSE is a new, more efficient, and lower-cost extraction method. CO_2 gas operates in a supercritical state, which is affected by temperature and pressure. A supercritical fluid is a fluid with a temperature higher than the critical temperature and a pressure higher than the critical pressure [20].

CO_2 gas, as an extractant, is in a critical state at 31.1 °C and 7.38 MPa. These operating conditions are inexpensive and suitable for materials with high boiling points and heat sensitivity. CO_2 gas is highly stable, non-toxic, and does not cause oxidation of products. Extraction of CO_2 gas does not contain nitrates, hazardous heavy metals, and is free from harmful solvent residues. CO_2 gas is easily recovered in its pure state by varying the temperature and pressure. CO_2 gas can be reused for further processes, making supercritical CO_2 extraction more efficient. CDSE with CO_2 gas is an environmentally friendly extraction model [21,22,23]. Several researchers have used the CDSE method for the extraction of essential oils, such as the extraction of turmeric [24], cumin (Carum carvi L.) [25], sucupira branca seeds [26], Algerian rosemary [27], lippia thymoides Mart. and Schauer (Verbenaceae) [28], leptocarpha rivularis [29], dried ginger [30], candeia wood (Eremanthus erythropappus) [31], clove leaves (Syzygium aromaticum) [32], rosehip seeds [33], santolina chamaecyparissus [34], perovskia atriplicifolia benth [35], and chamaecyparis obtuse [36].

Materials and Methods

Equipment and Materials

The equipment used included a set of boiling distillation apparatus, a set of CDSE apparatus, a Shimadzu QP 2010 SE Gas Chromatography-Mass Spectrometry (GC-MS), an Abbe refractometer, a Po-lax-2L polarimeter, and a pycnometer. The research materials were ginger industrial waste obtained from the ginger extract beverage industry in Sleman Regency, Yogyakarta, Indonesia, pure CO_2 gas, and anhydrous sodium sulfate (Na_2SO_4) with pro analysis grade from Sigma Aldrich.

Preparation of Ginger Industrial Waste

Eight kg of ginger industrial waste were dried in an oven at 105 °C. The dried ginger industrial waste was divided into two parts, each containing 4 kg. Both raw materials were extracted using boiling distillation and CDSE methods. The physical form of ginger industrial waste is shown in Figure 1.

Figure 1. Physical appearance of the ginger industrial waste

Extraction Process of Ginger Essential Oil from Ginger Industrial Waste Using Water Distillation

The water distillation apparatus was completely assembled, with all connections tightly sealed to prevent steam leakage. Ginger industrial waste, as much as 4 kg, was placed in a kettle lined with cloth. The kettle was filled with 13 L of water until the sample was submerged. The distillation apparatus was heated using a gas stove. The initial heating was done using high heat, and after the first drop, it was heated to low heat. The resulting vapor flowed through the condenser and condensed into a liquid. The condensed liquid was collected in a collection flask. The distillation process lasted for 6 hours. After completion of the distillation process, the condensed liquid formed two layers: a water layer below and an essential oil layer above. The two layers were separated using a separating funnel. The essential oil was collected in a 100 mL beaker. Anhydrous sodium sulfate was gradually added to the essential oil until no water was left. Essential oils are stored in dark, airtight bottles, in a cool place protected from strong light.

Isolation of Ginger Essential Oil from Ginger Industrial Waste Using the CDSE Method

A total of 4 kg of ginger industrial waste, as much as 4 kg of powder, was placed into an extraction tube and then tightly sealed. A CO_2 gas pipe was installed and connected to the extractor. The valve of the CO_2 gas cylinder connected to the extractor was opened. CO_2 gas was circulated at a pressure of 120 bars at a temperature of 35 °C for an extraction time of 60 minutes. After 60 minutes, the valve to the separator was opened to release the CO_2 gas. The valve to the separator was slowly opened until the pressure in the extraction tube was reduced to 0 bar, then the ginger essential oil was extracted. The ginger essential oil was stored in a brown bottle. A schematic of the extraction process using CDSE is shown in Figure 2.

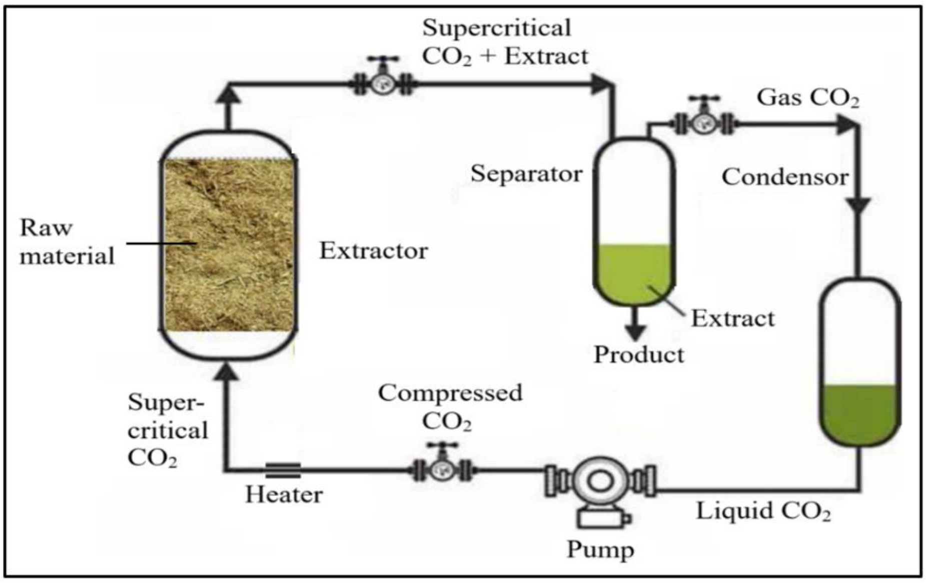

Figure 2. Schematic and steps of the extraction process using CDSE

Results and Discussion

Physical Appearance and Characterization of the Ginger Industrial Waste for Essential Oil

Figure 3 shows that the essential oil extracted by water distillation has a clear yellow color, while the CDSE method is dark brown. The ginger essential oil extracted by water distillation is clear yellow because only volatile compounds are extracted. Ginger essential oil extracted by the CDSE method produces a dark brown color. This is because extraction with liquid CO_2 extracts all components in ginger industrial waste, such as fat, lignin, and other dyes. The CDSE extraction method requires an extraction process with ethanol to purify the ginger essential oil. Plants, including ginger, contain fat [37] and pigments [38]. Table 1 shows the results of the characterization of essential ginger oil by water distillation and CDSE. The parameters of specific gravity, refractive index, and optical rotation indicate that both essential oils comply with SNI 1312:2021. Chromatographic profile analysis showed that the zingiberene levels in ginger industrial waste samples with water distillation and CDSE were 9.53% and 8.31%, respectively. The different compositions of ginger industrial waste essential oils are caused by various factors in the extraction process.

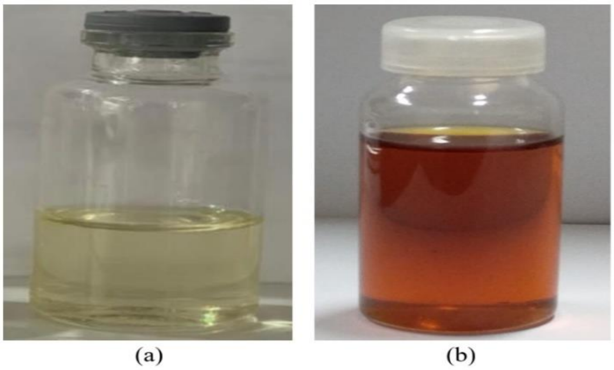

Figure 3. Ginger industrial waste essential oil obtained by (a) water distillation and (b) CDSE methods

Characterization of ginger industrial waste essential oil

Table 1. Characterization results of ginger industrial waste essential oil by water distillation and CDSE

No.	Parameters	Water distillation	CDSE	Quality standards according to SNI 1312:2021
1	Appearance	Clear liquid	Clear liquid	Clear liquid
2	Color	Pale yellow	Dark brown	Pale yellow to yellow
3	Odor	Ginger spice aroma	Ginger spice aroma	Characterized by ginger rhizome, with aromas of spices, pepper, and lemon.
4	Relative Specific Gravity	0,8790	0,8761	0,870-0,895
5	Refractive Index	1,4806	1,4808	1,480-1500
6	Optical Rotation	+4,325°	+3,175°	-30° - (+20°)
7	Chromatographic Profile	9,53%	8,31%	12%-30%

Analysis of Ginger Industrial Waste Essential Oil Using GC-MS

Figure 4 shows the chromatogram of ginger industrial waste essential oil extracted using the water distillation and CDSE methods. Identification of the compounds in the essential oil from ginger industrial waste using the boil distillation method using GC-MS is shown in Table 2. Thirty-five compounds were detected by GC-MS in the first sample of ginger industrial waste essential oil using the boil distillation and CDSE methods. The five main compounds dominant in the boil distillation sample were champene, 1,8-cineole, Z-citral, E-citral, and zingiberene. Compared to the compounds in ginger essential oil, the zingiberene content of the first sample did not fall within the minimum range of 12% according to SNI 1312;2021.

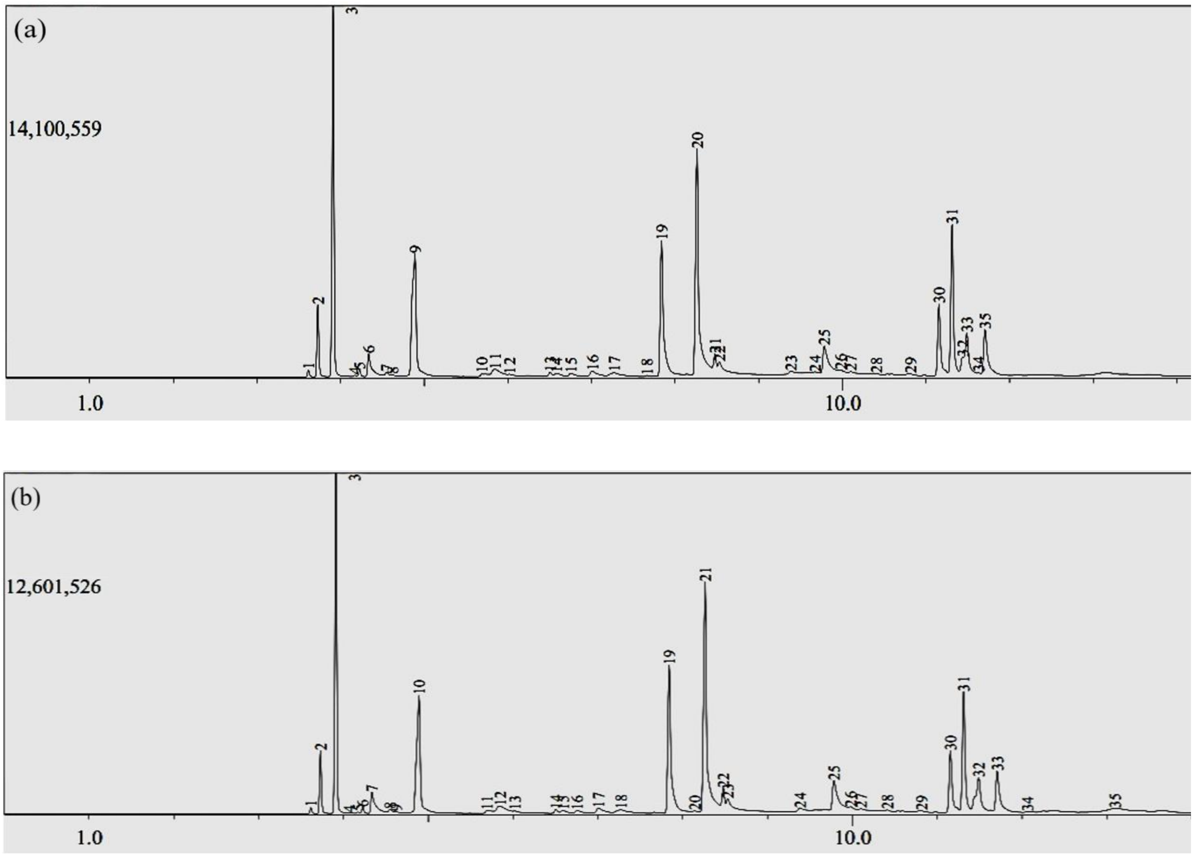

Figure 4. Chromatogram of ginger industrial waste essential oil extracted using the water distillation (a) and CDSE (b) methods.

Table 2. Identification of Components of Ginger Industrial Waste Essential Oil Extracted Using Water Distillation and CDSE

No	Compound name using water distillation	(%area)	Compound name using CDSE	(%area)
1	Tricyclene	0.32	Tricyclene	0.28
2	α pinene	3.22	α pinene	2.86
3	Champene	16.98	Champene	16.24
4	β Phelandrene	0.07	1,5,8-p-menthatriene	0.08
5	1-β pinene	0.33	β phellandrene	0.09
6	β mycrene	2.18	β pinene	0.33
7	1-Phellandrene	0.30	β mycrene	2.38
8	DELTA.3-Carene	0.10	1-phellandrene	0.38
9	1,8-Cineole	12.04	DELTA.3-Carene	0.16
10	α-Terpinolene	0.47	1,8-Cineole	10.56
11	Linalool	1.39	α-Terpinolene	0.23
12	Methyl Bronil Ether	0.14	Linalool	1.81
13	Champor	0.28	Methyl Bronil Ether	0.19
14	Citronella	0.38	Champor	0.28
15	Trans-Carane	0.26	Citronella	0.39
16	Isogeraniol	0.75	Verbenol	0.35

17	α terpaniol	0.69	Isogeraniol	0.88
18	2-Octanol	0.05	α terpaniol	0.77
19	Z-Citral	10.46	Z-Citral	11.70
20	E-Citral	19.44	2-Decenal	0.19
21	α- Fechyl acetate	0.74	E-Citral	18.66
22	Methyl nonyl ketone	0.54	Acetic acid 1,7,7-trimethyl-bicyclo hept-s-ylester	1.57
23	Citronellyl acetate	0.26	Methyl nonyl ketone	1.53
24	(+)- cycloisosativene	0.06	Citronellyl acetate	0.31
25	Geranil acetate	3.39	Geranil acetate	3.94
26	β-elemene	0.42	β-elemene	0.45
27	Zingiberene	0.13	Zingiberene	0.18
28	Trans-Caryophyllene	0.06	Trans β-Caryophyllene	0.08
29	β-sesquiphellandrene	0.19	β-sesqui phellandrene	0.09
30	α-Curcumen	5.10	α-Curcumen	4.74
31	Zingiberene	9.53	Zingiberene	8.31
32	Farnesene	1.20	β- Besabolene	4.75
33	β- Besabolene	4.02	β-sesqui phellandrene	4.15
34	EPI-Bicyclosesquiphellandrene	0.02	Beta,-copaen-4.alpha.-ol	0.07
35	β-sesquiphellandrene	4.50	Nerolidol	0.98

The first sample of ginger industrial waste essential oil using the CDSE method detected 35 compounds by GC-MS (Table 2). The five main compounds dominant in the first sample were champene, 1,8-cineole, Z-citral, E-citral, and zingiberene. Compared to the components of ginger essential oil, the zingiberene content in the second sample did not fall within the minimum range of 12% required by SNI 1312:2021. The zingiberene content in the ginger industrial waste the essential oil was lower than that in essential oil extracted from ginger rhizomes. This difference is due to the differences between the rhizome and the pulp, and possibly suboptimal extraction conditions.

The physical properties of ginger essential oil are indicated by its color. This color is related to the chemical composition of the ginger essential oil. Extraction of ginger essential oil by water distillation requires 6 hours for a single process. Ginger essential oil contains light fractions that can be extracted by boiling distillation without pressure. Ginger essential oil extracted using water distillation is colorless (clear), while ginger essential oil extracted using CDSE is brown. This color difference is due to the differences in the compounds extracted using the two methods. Water distillation extracts volatile compounds, while CDSE extracts compounds soluble in liquid CO_2. Non-volatile compounds in ginger essential oil are also extracted with CO_2 gas. Non-volatile compounds such as oil, fat, and lignin are extracted with CO_2 gas, producing a yellow color.

The advantages of the CDSE method over water distillation are yield and extraction time. The yield with the CDSE method is 0.36%, while the water distillation method produces a maximum yield of 0.18%. The main advantage is the faster extraction time, a maximum of 1 hour, compared to 6 hours with water distillation. The advantage of the CDSE method is that it does not require fuel that produces CO_2 gas. The CO_2 gas used for the extraction process in CDSE can be reused by releasing it as CO_2 in the gas phase. This process makes the CDSE method highly efficient and environmentally friendly [33]. The disadvantage of the CDSE method is that it produces ginger essential oil, which requires purification due to its non-volatile chemical compounds. Non-essential chemical compounds such as lignin, oils, and fats are also extracted from ginger essential oil [39]. The CDSE extraction method produces a higher yield than water distillation. This method also requires a shorter extraction time. The CDSE extraction method is preferred due to its high efficiency, shorter extraction time, and high selectivity [40,41].

The CDSE method in this study did not optimize the parameters of powder material, pressure, and temperature. Further research that can be conducted is optimizing various parameters that affect the quality and quantity of ginger essential oil. Important parameters affecting the CDSE extraction method are CO_2 gas flow rate, pressure, temperature, and extraction time [42]. The use of co-solvents and the particle size of the raw materials also affect extraction results [43].

Conclusions

Ginger industrial waste can be processed using water distillation and CDSE. Both methods produce essential oils with different physical and chemical properties. GC-MS analysis results show that the water distillation and CDSE methods produce ginger essential oil with nearly identical chemical compounds. Characterization of ginger essential oil using both methods also meets the quality standards for ginger essential oil according to SNI 1312:2021. The advantages of the CDSE method over the water distillation method include faster extraction time, no heating required, no pollutant gas production, and environmental friendliness. The CDSE method has the disadvantage of requiring further processing to separate volatile and non-volatile chemical compounds. This study recommends further research to optimize CDSE performance, separate non-essential compounds, and evaluate the economics of ginger essential oil.

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