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Extraction of Essential Oil from Moringa Oleifera Leaves Using Steam Distillation and Soxhlet Extraction Method

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ABSTRACT

Essential oil is one of Indonesia's agro-industrial export commodities, one of which is essential oil from Moringa oleifera leaves. This study aims to study the results of the yield and analyze the essential oil content of Moringa oleifera leaves using the steam distillation method and Soxhlet extraction using ethanol and isopropyl alcohol as a solvent. The fresh feed was either directly extracted or pre-treated using an oven and then followed by several stages of extraction. The pre-treatment of fresh feed was carried out in the oven at 70°C for 1 hour. Atmospheric steam distillation was carried out at 100°C for 6 hours using 500 ml of distilled water. In the case of soxhlet extraction, the temperature was set at the boiling point of the solvent for 6 hours. Once soxhlet extraction was complete, the essential oil was separated from the solvent by distillation. After various extraction processes, the essential oils were analyzed for their compound content using GC-MS. Based on the results of the study, the highest yield of 29.04% was obtained from the treated feed using two stages of soxhlet extraction variable with ethanol as solvent.

***Keywords*:** Essential oil, moringa leaves, soxhlet extraction, steam distillation, yield.

1. INTRODUCTION

Essential oils are known as etheric oils or volatile oils, as well as aromatic oils produced by plants. This oil is a secondary metabolite in the vegetable oil group which has a liquid or solid state with varying compositions and boiling points but easily evaporates [1]. The volatility of this essential oil occurs initially at room temperature without decomposition and has a pungent taste, giving off a fragrant aroma according to the original essential plant [2]. Essential oil is one of Indonesia's potential agro-industrial export commodities. Almost all parts of a plant can produce essential oils with the help of heat, water and solvents [3]. Essential oils are produced as raw materials for the perfume, cosmetic, health, and food industries [4]. There are at least 150 types of essential oils traded on the international market, of which 40 types are produced in Indonesia, but only 15 types of essential oils are produced commercially and enter the international market [5].

In Indonesia, it has one of the plants as a basic ingredient for essential oils from the genus Moringa, namely *Moringa oleifera* or better known as the Moringa plant specifically for the leaves of this plant (Moringa oleifera leaves). This moringa plant can be found in various regions of the world, one of which is Southeast Asia where this plant can also function as a natural herb that has been traditionally used by the community [6]. This plant has a soft tree trunk and has been used as traditional medicine in various countries for more than 1 century. Unlike other plants, Moringa leaves are often used as a source of food reserves in the dry season because the leaves can be eaten and cooked without losing their nutrients [7]. Moringa leaves are anti-bacterial and anti-inflammatory. Tea brewed from Moringa leaves can treat ulcers and diarrhea. Moringa leaves are a good food

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source for people with malnutrition because of their high protein and fiber content. Moringa leaves are also able to treat fever, bronchitis, eye and ear infections, and inflammation of the mucous membranes. The high iron content is used for the treatment of anemia and is used in the treatment of skin disease scurvy. The leaves are the most nutritious part of the plant, being an important source of B vitamins, vitamin C, provitamin A as beta-carotene, vitamin K, manganese, and protein, among other important nutrients [8].

The technique for extracting essential oils from plants is currently known to have several extraction methods which are further divided into two categories. Namely conventional methods such as: hydro distillation, steam distillation, soxhlet extraction, and cold pressing. Also, for newer or sophisticated methods such as: supercritical fluid extraction, ultrasound assisted extraction, microwave assisted extraction, and solvent free microwave extraction [9]. Steam distillation is a separation process for temperature sensitive materials such as oils, resins, hydrocarbons, etc., which are insoluble in water and can decompose at their boiling point. This process, as the most widely used method for the extraction of essential oils from plants, has been carried out actively since the early 1980s. The basic principle of this process is to allow a compound or a mixture of compounds to be distilled at temperatures significantly below the corresponding boiling point. Essential oils contain substances with boiling points up to 200°C or higher, but in the presence of steam or boiling water, these substances evaporate at the boiling point of water (100°C) at atmospheric pressure [10]. Meanwhile, soxhlet extraction is a piece of laboratory equipment designed in 1879 by Franz von Soxhlet. The soxhlet extraction equipment consists of a round bottom flask, siphon tube, distillation line, expansion adapter, condenser, cooling water inlet, cooling water outlet, heat source and thimble. In this method, the sample is placed in a porous bag or "thimble" made of tough filter paper or cellulose, which is placed in the thimble chamber of the soxhlet apparatus. The extraction solvent is taken in a round bottom flask and heated using a heating source such as a heating mantle. The heating temperature is built upon the solvent used for extraction. Due to the heat, the solvent in the lower flask evaporates into the condenser and then drips back into the sample thimble. When the liquid content reaches the siphon arm, the liquid content is emptied back into the lower flask and the end of the process is shown as a clear solution in the siphon tube [11].

2. RESEARCH METHODS

The methods that will be used in the extraction of essential oil from moringa leaves is steam distillation and soxhlet extraction. The pre-treatment before the extraction is drying using the oven at 70°C for 1 hour. The variable for this experiment is 1) extraction method (steam distillation and soxhlet extraction), 2) pre-treatment (fresh leaves and oven dried), 3) solvent for soxhlet extraction (ethanol and isopropyl alcohol), and 4) extraction stage (single-stage and multi-stage 2 times).

2.1. MATERIALS

Moringa leaves used for this experiment come from local market at Surabaya. The solvent used for soxhlet extraction is a food grade quality solvent with a concentration of 96%. Both solvent, ethanol and isopropyl alcohol were bought from local chemical store at Surabaya.

2.2. STEAM DISTILLATION

The extraction process using steam distillation method (as shown in Figure 1) is carried out with an operating temperature of 100°C and atmospheric pressure of 1 atm. After the water reaches its boiling point, the water becomes steam and will be contacted with moringa leaves. The steam will lift the compounds inside moringa leaves. Thereafter, steam will be condensed in

condenser creating two layers. The top layer will be the essential oil while the bottom layer is the hydrosol. The extraction process is carried out for 6 hours.

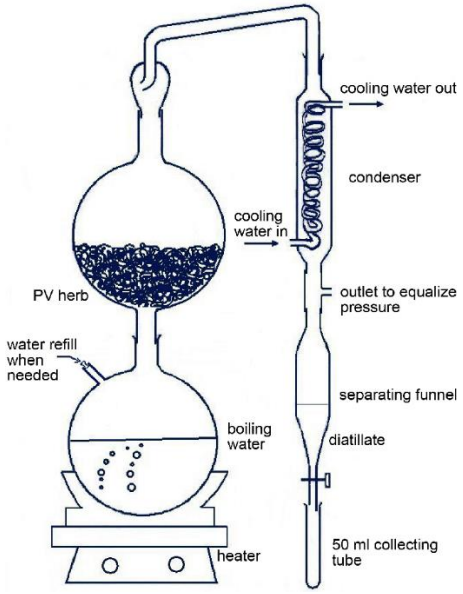

Figure 1. Steam Distillation Schematic.

2.3. SOXHLET EXTRACTION

For soxhlet extraction (as shown in Figure 2), the operating temperature adjusted according to the boiling point of each solvent. After the solvent reaches the boiling point, the solvent will evaporate and rise to the condenser. The condensed steam will drip down and encounter the Moringa leaves. Inside the thimble, the solvent in contact with the moringa leaves will extract the compounds contained in the moringa leaves, and this extraction process will run repeatedly, with repeated cycles of heating, circulating the solvent, and returning the saturated solvent to the flask. The extraction process is carried out for 6 hours. After that, the solvent and essential oil will be separated by distillation. This distillation process is carried out until the soxhlet extraction results are completely separated from the solvent in order to obtain pure essential oils.

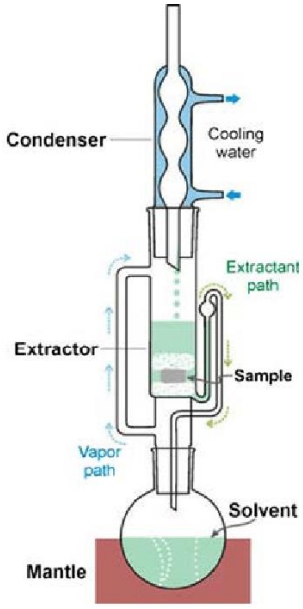

Figure 2. Soxhlet Extraction Schematic.

3. RESULTS AND DISCUSSION

The first variable in this experiment is the method used for essential oil extraction, which is steam distillation and soxhlet extraction.

Table 1. Mass and Yield of Essential Oil from Moringa Leaves using Soxhlet Extraction Method in Single Stage.

Solvent	Single Stage			
	Fresh Leaves		1 Hour Oven	
Ethanol	11.6674 g	23.33%	12.0190 g	24.04%
Isopropyl Alcohol	10.3403 g	20.68%	11.2233 g	22.45%

Table 2. Mass and Yield of Essential Oil from Moringa Leaves using Soxhlet Extraction Method in Double Stage.

Solvent	Double Stage			
	Fresh Leaves		1 Hour Oven	
Ethanol	14.2843 g	28.75%	14.5197 g	29.04%
Isopropyl Alcohol	12.5482 g	25.10%	13.2542 g	26.51%

In this experiment the steam distillation method failed to extract the essential oil, because there are no two layers of essential oil and hydrosol formed in the separator. This happens because the compounds contained in the essential oil of Moringa leaves cannot be vaporized with the boiling point of water, which is 100°C, for example, 1-2 benzene dicarboxylic acid which has a boiling point of 133°C and Glycerol tricaprylate which has a boiling point of 233°C. In addition, the compounds that make up the essential oil of Moringa leaves are polar. When Moringa leaves are extracted using the steam distillation method, these compounds will be lost because they are dissolved in water and do not evaporate due to their high boiling point [12]. Based on the data from Table 1 and 2, the yield using ethanol solvent is higher than isopropyl alcohol. The higher yield of the ethanol solvent shows that the polarity of ethanol has relatively the same polarity as the compounds found in Moringa leaf essential oil. Isopropyl alcohol solvent has a low dielectric constant value of 17.9 compared to ethanol which is 24.25, where the dielectric constant is a measure of the polarity of a solvent [13]. This causes the Soxhlet extraction yield to use less isopropyl alcohol than ethanol solvent. In addition, the yield also depends on the amount of active compound that reacts, so that the more yield produced, the more active compound content [14].

Table 3. Comparison of Most Components after GC-MS Test in Moringa Leaves Essential Oil from Various Extraction Methods.

Compounds	Extraction Methods					
	Soxhlet Extraction				Steam Distillation*	
	Ethanol		Isopropyl Alcohol			
	Single Stage	Double Stage	Single Stage	Double Stage		
	Oven	Oven	Fresh	Fresh	Oven	Fresh
	Percentage Area					
Neophytadiene		9.08%		48.22%		
Tricaprylin	9.31%	9.85%	4.73%	3.71%		
Ammeline	0.73%		32.04%	6.74%		
Thiophene-2-carbaldehyde oxime	1.58%	3.10%	9.06%	3.56%		
5-Hydroxycytosine	5.48%	7.80%	2.39%	8.20%		
3-Hydroxypyridine-2-thiol	1.71%		2.88%	4.26%		
Thioanisole	2.21%	4.26%	2.02%			
Piperazine, 1-methyl-4-[2-(p-tolylsulfonyl) ethyl]-	1.95%		6.98%			
Palmitic Acid				1.05%		
Undecane			0.06%			0.12%
1-(2,3,6-Trimethylphenyl)-3 buten-2-one		0.21%				3.44%
2-Ethyl-3,6-dimethylpyrazine		0.07%				0.12%
Phthalic Acid	57.16%					
1H-1,2,4-Triazole-1-carboxamide, 5-acetylamino-3-amino-			9.62%			

*Source: Ma et al. [15].

In this experiment, we evaluated the effect of pre-treatment in the form of drying in an oven with a temperature of 70°C for 1 hour, on the amount of yield of moringa leaves essential oil produced. Based on the data from Table 1 and 2, the moringa leaves that have been dried in the oven produce more yield than fresh moringa leaves. This is because decreased water content due to drying. This reduced water content can be determined by the gravimetric method. The following [16] equation can be used to determine the reduced water content,

$$\%Moisture = \frac{initial\ sample\ (gr) - final\ sample\ (gr)}{initial\ sample\ (gr)} \times 100\%$$

The water content reduced after moringa leaves has been oven dried at 70°C for 1 hour is 68,84%. The water content contained in the leaves protects the essential oil content in plant tissues. So, if the water content is too much, it will be difficult for the essential oil to be extracted which in other words will reduce the amount of yield produced [17].

During this research we extract the essential oil by single-stage and multi-stage (2) for the soxhlet extraction. Multi-stage extraction was carried out by repeating the soxhlet extraction process twice, using a fresh solvent each time with the aim of increasing the amount of essential oil yield. Based on the data from Tables 1 and 2, the amount of yield on multi-stage variables is more than single-stage extraction. This can happen because, the levels of compounds in Moringa leaves that are left in the dregs of the previous extraction, are extracted at the next extraction stage. Although it produces more yield than single-stage extraction, the difference in yield amount between single-stage and multi-stage extraction is relatively small, so multi-stage extraction with the Soxhlet extraction method to extract essential oil from Moringa leaves is considered less efficient and less economical.

In determining the quality of moringa leaf essential oil using the Soxhlet extraction method and steam distillation, the chemical properties of the resulting essential oil were tested. Test analysis was carried out using GC-MS (Gas Chromatography-Mass Spectrometry) to identify the composition of the compounds contained in essential oil from moringa leaves. Based on the results of the GC-MS analysis (Table 3), the compounds that appear will be compared based on the method and solvent used.

There are several compounds present in the 4 samples, such as Tricaprylin, which are useful for treating metabolic disorders associated with Alzheimer's disease, both mild and moderate [18]. In addition, there are Thiophene-2-carbaldehyde oxime and 5-Hydroxycytosine compounds that appear in each analysis sample. The benefits that can be used from the Thiophene-2-carbaldehyde oxime compound in the essential oil of Moringa leaves are as nitriles for pharmaceutical needs such as the use of organophosphate nerve poisoning drug synthesis [15]. Whereas the 5-Hydroxycytosine compound is useful as a secondary antioxidant defense [19]. n the compounds found from the steam distillation extraction experiments, there are several compounds that also have similarities with Soxhlet extraction, including: Undecane, 1-(2,3,6-Trimethylphenyl)-3-buten-2-one, and 2-Ethyl -3,6-dimethylpyrazine [20]. One of the uses of the compounds mentioned, namely: 1-(2,3,6-Trimethylphenyl)-3-buten-2-one, is as an aroma compound to give flavor or aroma to food [21].

4. CONCLUSION

Extraction of the essential oil from moringa leaves has been carried out using steam distillation and soxhlet extraction. The steam distillation method was not successful in extracting essential oil from moringa leaves. Ethanol as a solvent in soxhlet extraction produced more yield than isopropyl alcohol. The main compound in moringa leaves essential oil are Tricaprylin, Neophytadine, and 1H-1,2,4-Triazole-1-carboxamide, 5-acetylamino-3-amino- which all of them beneficial for health and is commonly used as a medical raw material. Moringa leaves that has been dried produce more essential oil yield than fresh moringa leaves. The multi-

stage extraction produced more essential oil yield than single-stage extraction.

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