Analysis of Physical Properties and Chemical Contents in *Eucalyptus citriodora* PT. Toba Pulp Lestari, Tbk

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Abstract: *Eucalyptus citriodora* is one type of eucalyptus that has the potential to be developed. This plant originates from Australia, which is one of the most popular eucalyptus trees that grows in almost all of Australia, and is now found to grow in almost all tropical regions of the world including Indonesia. Content Analysis and determination of sineol Content in *Eucalyptus citriodora* leaf essential oil from PT. Toba Pulp Lestari Tbk has been carried out by the GCMS method. Eucalyptus leaves have been used for medicine, industry and perfume as well as Eucalyptus oil. The purpose of this study was to determine the compound content and levels of cineol in *Eucalyptus citriodora* leaves. The abundance of sineol levels 55-65% can be used as perfume, medicines and Eucalyptus oil. Stahl distillation was developed to produce The Eucalyptus oil. There are eleven compounds in *Eucalyptus citriodora* leaves, including α-Pinene, 1-Limone, 1.5 Cyclooctadiene, 1.8 Sineol, 1-P-Menthen-8-YL-Acetate, 3- Cycloheksane, 1-Methanol, α-Terpinyl Acetate, Phenol, Butylhidroxytoluena, Trans-Methyl-dihydrojasmonate, ISO - Cytronellate, ISO - Propyl Myristat, Propyltetradekanoate, Octadecanoic Acid. The mximum abundance is 1,8-cineol (60.29% v/v). The physical properties was obtained used titrimetric method yields of acidity and ester numbers respectively 2.8025 and 12.0 (T= 26.010C).

1 INTRODUCTION

Eucalyptus citriodora plant is one of the essential oils producing plants that are important for the essential oil industry in Indonesia. The main products produced from this plant are Eucalyptus oil obtained from the distillation of leaves. The total production of Eucalyptus oil produced in Java in 2018 is 300 tons (Mukriz Damanik, 2009)

PT.Toba Pulp Lestari Tbk is one of the Industrial Plantation Forest companies in Indonesia, especially the North Sumatra province which is the Industrial Plantation Forest with the largest area in North Sumatra where the total area reaches 188,055 Ha and the main commodity developed is the Eucalyptus Plant (*Eucalyptus* spp) Eucalyptus belongs to fast-growing plants or better known as Fast Growing Species (Beyranvand, 2015). Besides that, Eucalyptus is also a plant that has better benefits in terms of stems, branches, and leaves (Mahmoud and Ali, 2015).

PT Toba Pulp Lestari Tbk has developed Eucalyptus plants to be used as paper raw materials (Anders et al., 2018). However, now what is still used from Eucalyptus plants is still in the wood and branches while the leaves are not utilized or just left alone (Han Roliadi, Dulsalam, 2010). Some previous studies have also analyzed the content of essential oils from Eucalyptus leaves (Technology, 2013).

The quality of raw materials of eucalyptus leaves, especially in Java, is still low, only having a yield of 0.6% - 1.0%. While from the results of the study (Muyassaroh, 2016) with steam and water distillation methods the range of eucalyptus oil yields ranged from 0.84% to 1.21% (Abed, 2018).

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Distillation of Eucalyptus leaves to obtain Eucalyptus oil uses a principle based on the properties of essential oils that can evaporate if heated with hot steam (Muyassaroh, 2016). The flow of steam will carry essential oils contained in the leaves and when the steam comes into contact with cold media, changes will occur into moisture so that water and oil will be obtained in a separate state. In distillation by using direct steam there is a process of transporting essential oils from the ingredients together with hot steam which is blown directly (Beyranvand, 2015). This method is similar to the steamed method but water is not filled in the distillation kettle. The hot steam produced from the boiler is flowed through a porous steam pipe located above the filter in the tank or distillation kettle (Hadji et al., 2017). Several factors affect the amount of oil that evaporates together with water vapor, namely: the amount of vapor pressure used, the molecular weight of each component in the oil and the speed of oil coming out of the material. In distillation of steam at atmospheric pressure, the distillation temperature is slightly above or below 100^0C, which depends on whether the saturated vapor pressure or steam through each distillation process can be adjusted so that it runs below 100^0C, provided that the pressure is 1 atmosphere (Ghasemian, 2018).

Mukriz Damanik, (2009) has compared the yield of oil and the quality of oil from the same type of Eucalyptus, Europhylia based on differences in plant age. And it was obtained that the older the plant age, the more oil yield will be obtained, but the oil quality will decrease. Oil is obtained from steam distillation and obtained low levels of cineol, 30-35% and 40-45%. At harvest time in industrial plantations requires fast growing both for wood and also for the quality of oil in the leaves. Sunanto., 2003, the optimum length of eucalyptus oil refining is 3-4 hours. The factors that influence the operational steam are as low as possible, even though the production speed is determined by the temperature of the amount of water that is in direct contact with the distilled material. Use the least amount of water that can be connected or in direct contact with plant material. The size of the material, by chopping the plant material before distillation, is attempted so that the filling into the kettle is as homogeneous as possible (Kumar Tyagi et al., 2014).

In this work, we want to analyze the chemical content, physico-chemical properties of acid numbers, esters and determination of cineol levels to be able to utilize leaf waste from *Eucalyptus Citriodora* plants in the PT. Toba Pulp Lestari Tbk area. analysis of chemical content and determination of cineol levels using the GC-MS method. Then a study was conducted to determine the physico-chemical properties of acid numbers, Esters. and the content analysis is carried out at the beginning to see the main compound. Then the determination of the level is done as a comparison that *Eucalyptus Cittriodora* has the potential as eucalyptus oil and also the highest% content obtained based on retention time.

2 MATERIALS AND METHODS

2.1 Collecting Sample

The process of collecting samples (Eucalyptus Citriodora fresh leaves) is taken directly from the PT. Toba Pulp Lestari located on Jl. Indorayon Desa Dolok Nauli, Kecamatan Parmaksian, Toba Samosir Regency, North Sumatra Province. Identification of Eucalyptus Cittredora leaves at the FMIPA - USU Herbarium Medanense (MEDA) Laboratory. Oil distillation from Eucalyptus Citriodora leaves was carried out with the Stahl Distillation at the FMIPA - USU Organic Chemistry Laboratory. Content Analysis and Determination of Sineol Levels were carried out at the Organic Chemistry Laboratory of FMIPA - UGM using GCMS method.

2.2 Extraction Process

100 grams of *Eucalyptus citriodora* leaves that have been cut into small pieces and put into a 1000mL bottom flask, then added enough aquadest until the entire sample is submerged, connected to a Stahl distiller, and boiled for ± 5-6 hours at ± 100 ° C to produce oil and distillation is terminated when the distillate that comes out is clear yellow. The essential oil obtained is accommodated in erlenmeyer. The distillate obtained is a mixture of oil and water. Then $CaCl_2$ anhydrous layer of oil is added to bind water which may still be mixed with essential oil, the oil layer is decanted and put into vial bottles, then the essential oil is stored in the refrigerator in a bottle and tightly closed.

The essential oils obtained were analyzed for their contents and determined by the level of cineol using GCMS tools.

2.3 Physical Properties Analysis

The physical properties analysis used titrimetri method (acidity and ester numbers).

2.4 Gcms Analysis

The Specifications Instrument GC-MS QP 2010S Shimadzu, using Column 5MS with type of ion source Electron Impact, Injector Temperature: 300^0C, Carrier Temperature: 50^0C, Carrier: Helium, Gas flow rate of carrier: 1.0 mL / min, Temperature oven: 500C for 5 minutes then 2400C for 7 minutes., Ionization electron: 70 ev.

The solution of each 1 μL standard sineol series was inserted into the syringer to be injected into the GCMS. Only the conditions adjusted to the conditions of each piece of equipment and then observed Mass Chromatogram data generated interpreted data. Obtained data then in Perform calculations to get the calibration curve and do the determination of levels through the equation.

3 RESULTS AND DISCUSSIONS

The result of identification shows that eucalyptus leaf is eucalyptus of Robusta species with plant taxonomy as follows: *Kingdom: lantae, Clade: Angiosperms, Clade: Eudicots, Order: Myrtales Family: Myrtaceae, Genus: Eucalyptus, Species: Eucalyptus grandis* W. Hill ex Maiden

Based on the research result, Eucalyptus grandis leaf essential oil of distillation with stahl tool is 1.8 mL with leaves of 450 grams and the percentage is 0.4%. Eucalyptus Oil Quality Requirements According to SNI 06-3954-2006 as Table 1.

Table 1: Eucalyptus Oil Quality Requirements (According to SNI 06-3954-2006).

No	Type of test	Unit	Terms
1	**Condition**		
1.1	colour	-	Clear until greenish yellow
1.2	odor	-	typical *Eucalyptus*
2	density	-	0,900 – 0,930
3	Refractive index	-	1,450 – 1,470
4	Solubility in Ethanol	-	1:1 up to 1:10 clear
5	Rotary optic	-	-4˚ up to 0˚
6	Cineole Content	%	50 – 65

Table 1 showed that then in terms of physical properties, the color of essential oil leaves Eucalyptus grandis was obtained a clear yellow solution of weak yellow. Compared to Table 1 above, the essential oils are in accordance with Eucalyptus oil requirements. The odor produced by the essential oil is almost the same as the smell of Eucalyptus oil that has a spicy, fresh and breathable scent. The smell of essential oils in accordance with the requirements of Eucalyptus oil. The weight of the essential oil is 0.9143. The refractive index was obtained 1.4653. Cineole content of essential oil is 0.08827 g or 8.827% v / v, with a percentage area of 36.55%. Based on SNI No. 06-3954-2006, cineole content is 50% - 65%. Cineole in volatile oil is one of the main constituent compounds alfa - Pinene as the main compound. In this study the method used is the distillation of water with stahl equipment which is a very simple method and has a weakness less vacuum so it can cause evaporation on essential oils during the process that allows evaporate cineole.

The essential oil of *Eucalyptus citriodoras* leaf obtained from the study was analyzed by using GC-MS to determine the chemical content contained therein, the results were adjusted with Library Wiley 229 and NIST Library 12, the chemical content of *Eucalyptus grandis* leaf contained twelve compounds in guess, that is : α-Pinene (45.21%), Camphene (1.38%), β-Pinene (1.11%), Camphogen (0.74%), 1,8-Cineole (36.55%), α-Campholene Aldehyde (0.73%), Pinocarvone (0.83%), α-Terpineol (8.87%), β-Caryophyllene (1.72%), Spathulenol (0.84%), Elemol (0.85%), 1–Nonadecene (1.17%).

The titrimeric method was done to analysed of the physical properties. This method yields of acidity and ester numbers respectively 2.8025 and 12.0 (T= 26.010C).

Based on the analysis with GC-MS that has been adapted to Library Wiley 229, the 1.8-Cineole spectrum is shown in Figure 1 below:

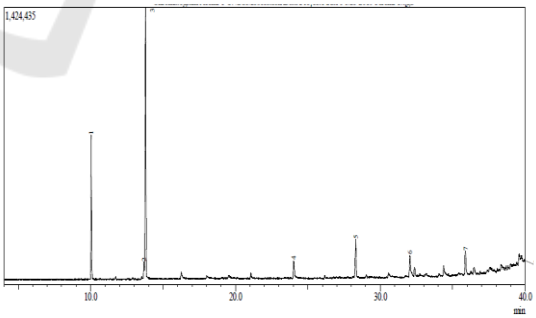

Figure 1: Chromatogram 1,8 sineol.

Peak Chromatogram with a retention time of 15.266 minutes is a compound with the formula $C_{10}H_{18}O$ molecule. The spectrum shows the molecular ion peak at m / e 154 followed by fragments at m / e 154, 139, 125, 108, 84, 81, 69, 43, 41, 27. By comparing the spectrum obtained with the standard spectrum data library, which allows is

1.8 - Cineole as much as 36.55% with the wake formula as in Figure 2 follows:

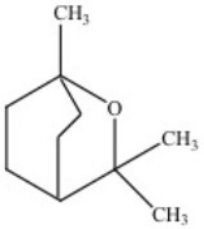

Figure 2: Structure of 1,8 – Cineole.

The fragmentation pattern of the 1.8 - Cineole compounds is most likely as shown in Figure. 3 below:

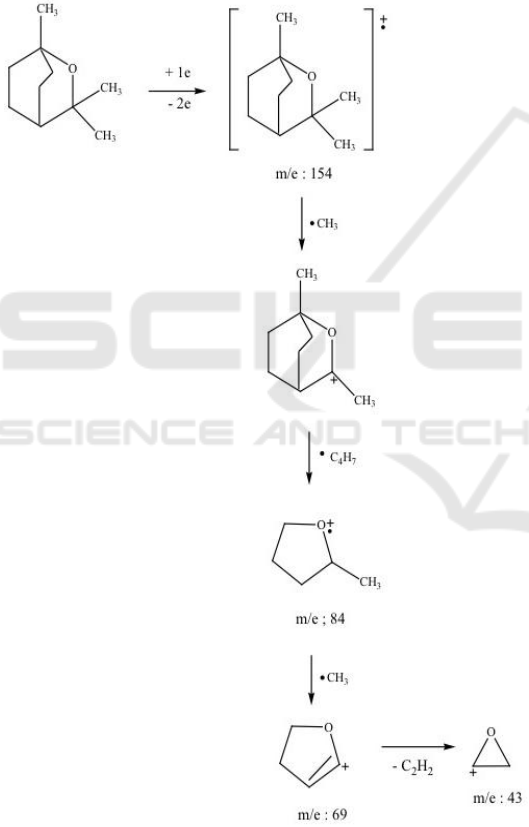

Figure 3: Fragmentation Pattern of 1,8 – Cineole.

4 CONCLUSIONS

Eucalyptus citriodora is plant originates from Australia, which is one of the most popular in Australia. Determination of sineol content in *Eucalyptus citriodora* leaf essential oil from PT. Toba Pulp Lestari Tbk has been carried out used GCMS method. There are eleven compounds in *Eucalyptus citriodora* leaves, including α-Pinene, 1-Limone, 1.5 Cyclooctadiene, 1.8 Sineol, 1-P-Menthen-8-YL-Acetate, 3- Cycloheksane, 1-Methanol, α-Terpinyl Acetate, Phenol, Butylhidroxytoluena, Trans-Methyl-dihydrojasmonate, ISO - Cytronellate, ISO - Propyl Myristat, Propyltetradekanoate, Octadecanoic Acid. The mximum abundance is 1,8-cineol (60.29% v/v). The physical properties was obtained used titrimetric method yields of acidity and ester numbers respectively 2.8025 and 12.0 (T= 26.010C).

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