Characterization of Essential Oils from Tuberose Flowers Waste (*Polianthes tuberosa L.*)

Arita D. Nugrahini, Aisyah L. Ristanti, and Jumeri
Department of Agroindustrial Technology, Faculty of Agricultural Technology, Universitas Gadjah Mada, Yogyakarta, Indonesia
Email: {arita.dewi.n, ais.linda14}@gmail.com, {arita.dewi.n, jumerimw}@ugm.ac.id

***Abstract*—Tuberose flower (*Polianthes tuberosa L.*) is known widely as fresh cut flower and perfume raw material. The purposes of this research were to find out whether waste of the tuberose flowers still contain valuable essential oil when extracted by maceration method, and also to determine the characteristics and antioxidant activities of produced essential oil. Maceration used hexane and petroleum ether as solvent to extract volatile compound from tuberose flower. The solvent and flower petals were soaked with ratio of 1:2. Maceration process carried out at ambient temperature for 24 hours. The solvent and flower petals were separated by filtration. The solvent was evaporated to obtain the extract and then measured the yield and checked the characteristics of the oil. It consist of color, refractive index, specific gravity, acid value, and the ester value. Examination of antioxidant activity used DPPH as free radicals. Yield of maceration with hexane (0,12%) is higher than petroleum ether (0,08%). Based on statistical test with 95% of confidence level indicates that characteristics of tuberose essential oil with hexane and petroleum ether was not different. Percentage of antioxidant showed that the yield by extraction with hexane solvent (13,13%) is higher than petroleum ether (9,27%).**

***Index Terms*—tuberose flower wastes, essential oils, maseration, antioxidant**

I. Introduction

Tuberose is a flower species developed by many of cut flower business owners. This is caused by the high demand which is influenced by variety advantages. The scent of tuberose is able to relieve the stress, hence it urges the healing process using aromatherapy. In addition to be useful as cut flower, tuberose is also useful as a raw material for making essential oil [1].

For the daily need, the demand of tuberose are predominantly in big cities such as Jakarta, Bandung, Medan, Bogor, Solo, Semarang, Yogyakarta, Malang, Denpasar, and Ujung Pandang, which is mostly used as flower bucket or room decoration. In Java, the necessity of tuberose on a large scale is not continuously required. Discontinuity of demand affecting the business owner's revenues and accumulation of material.

Cut flower center in Yogyakarta is located on Ahmad Jazuli St., Kotabaru, Yogyakarta, Indonesia. This cut flower center has 25 member of florists. Within a day, they can produce about a truck full of tuberose waste for a single region. This waste is still raw, means the flowers were just thrown away. This waste consists of wilted and rotten flowers and plants, and also cutted stems. This waste can pollute the environment, so it needs an appropriate management. A method to gain the flower's benefit is through essential oil extraction. Essential oil is one of the agro-industrial products which is prospective to be developed. According to the Ministry of Trade, the export value of essential oils and perfumes has been increased since 2010 to 2014 per year.

The purposes of this research were to determine the sucrose content of tuberose extract, to determine the characteristics of tuberose waste extract by its color, density, refractive index, acid value, ester value, and antioxidant activity using DPPH.

II. Research Methods

The raw materials for this research were tuberose flower waste which were collected from Kotabaru flower market, Yogyakarta. This research used hexane and petroleum ether as solvents. The other materials were methanol, KOH 0.1 N, HCl 0.1 N, aquadest, and DPPH (2,2-diphenylpicrylhydrazyl).

This research used maceration method. The solvent and flower petals were soaked with ratio of 1:2. Maceration process carried out at ambient temperature for 24 hours. The solvent and flower petals were separated by filtration. The solvent was evaporated to obtain the extract and then measured the yield and checked the characteristics of the oil. It consist of color, refractive index, specific gravity, acid value, and the ester value. Examination of antioxidant activity used DPPH as free radicals.

For antioxidant examination, this research used DPPH method. Then analyzed the data with SPSS to test the differences between the two solvents using Independent Sample Test.

III. Result And Discussion

A. The Process of Tuberose Waste Extraction

First step on this research was tuberose flower waste selection. This research used the tuberose between red and blue line (See Fig. 1). It was because the tuberose

Manuscript received May 13, 2016; revised November 3, 2016.

doi: 10.18178/joaat.4.1.53-56

above the red line were too young (bud), recognized through the color which were still green, and when the bud was opened, it produced the smell of sap instead of its authentic fragrance. The tuberose under the blue line was not used because it usually already wilted or had been rotten. Tuberose waste which was used in this research were those with white buds or almost wilted tuberoses.

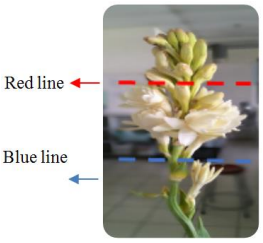

Figure 1. Tuberose flower waste selection

After the selection, the tuberoses were cut to widen their surface, and then they were measured per 500 grams. After that, 1.000 liters of hexane was served. In this research, the employed ratio between material and solvent was 1:2. Then, cut tuberose petals were put into a glass jar and poured with measured solvent. After that, tuberose and solvent were stirred together. Next, the jar was covered with aluminium foil and left it in room temperature for 24 hours (See Fig. 2).

Figure 2. Maceration process

After 24 hours, solvent and tuberose petals were sieved using strainer. The sieving process resulted two layers as shown in Fig. 3. The under layer was the water that also extracted by solvent, meanwhile the above layer was the solvent and essential oil. Both layers had to be separated because the water could make the essential oil to be rancid and would impede the evaporation. Both liquid were separated using a separation funnel.

Figure 3. Separated tuberose waste extract

The tuberose extract then was evaporated using rotary vacuum with temperature between 40-45°C and 170-175 mmHg of pressure to separate the solvent. The result of evaporation then was analyzed based on its characteristic and antioxidant activity. The same method was employed for petroleum ether.

The maceration result was extracted in transparent container. This extraction was in semi-solid, light yellow colored, and had a tuberose fragrance. From the extraction of tuberose essential oil, it resulted pasta (semi-solid) extract. This solid extract is named as concrete sucrose content. Concrete sucrose content is sucrose content consists of mixed essential oil and wax, albumin, and colored essence in a small quantity [2].

B. Sucrose Content from Extraction

Sucrose content is the ratio (quantity) of produced oil and the aromatic plants extract. Higher value of sucrose content shows higher essential oil production [3]. Sucrose content from the extraction using both solvents can be seen in Table I.

TABLE I. SUCROSE CONTENT FROM EXTRACTION

Solvent	Average weight of the extract (g)	Average sucrose (%)
Petroleum eter	0,36 ±0,16	0,08 ±0,04
Hexane	0,62 ±0,25	0,12 ±0,05

In the research of Suyanti and Muhajir [4], they used the solvent extraction method and resulted sucrose content concretes of 0,22-0,31% and absolute sucrose content of 0.03-0,11% from fresh tuberose extraction. In their research, the resulted sucrose concrete is 0,12% for hexane and 0,08% for petroleum ether. There is a big gap in both researches, which is caused by decreasing quality of the tuberose. In Table I, the deviation standard on average of extract weight is considered as big, since the extract results were in different weight. The differences of the results were influenced by the species of flowers. The types of flowers that was used in this research came from the same source (Kotabaru), however the specification of the types was unknown. The different age in harvesting could affect the amount of the essence. In this research, the used sucrose content was concretes, in which there were still waxes. Higher amount of wax, the resulted sucrose concrete is also higher. Based on Nopalas [5], the difference of climate, soil, harvesting age, solvent, and oil storage method, can influence the result of extraction.

C. The Characteristics for Extraction Result of Tuberose Waste

This study implemented 5 parameters of tuberose essential oil characteristics in terms of color, refractive index, specific gravity, acid value, and ester value. The result of examination can be seen in Table II.

TABLE II. CHARACTERIZATION OF EXTRACTION RESULT

Characterization	Test Result	
	Petroleum eter	Hexane
Color	Yellow	Yellow
Refractive Index at 15°C	1,334	1,334
Specific gravity at 15°C	1,027 ±0,001	1,015 ±0,002
Acid value (mg KOH/g sample)	23,84 ±1,99	16,83
Ester value (mg KOH/ g sample)	357,64 ±9,91	392,70

1) Color

Color is the crucial parameter in determining the quality of essential oil. The color intensity is determined by the amount of certain pigment in the essential oil. The color of new extracted essential oil is usually reddish, greenish, and brownish, depends on the species of extracted plants [6]. In this research, the produced essential oil was yellow colored.

2) Refractive index

Refractive index is the ratio between sine of angle of incidence and sine of the angle of refraction when a certain wavelength of light entering oil from the air with certain unchanged angle in maintained temperature. The measurement of refractive index is purposed to decide the oil purity [7].

The refractive index of tuberose oil is 1,4916 in the temperature of 25°C [8]. This value is higher than the research's results. The low refractive index in this research was caused by the decreasing quality of the flower. This research did not use fresh tuberose flower and they were not sold. The different climate, soil, harvesting age, solvent, and oil storage method will influence the result of essential oil [5]. The value of refractive index is also influenced by the water that found in the essential oil. If water intensity is higher, the refractive index will be lower. It is because water easily refracts the coming light. Hence, the essential oil with bigger refractive index is better than essential oil with smaller refractive index.

3) Specific gravity

Specific gravity of essential oil is described as the ratio between weight oil and water in the same volume of water and oil [9]. The value of the specific gravity of tuberose flower waste extracts can be seen in Table II.

The specific gravity for essential oil is approximated between 0,698-1,188 on 15°C [7]. This value is still considered as essential oil. The specific gravity for essential oil is 1,007 and 1,003 in Guenther [8]. The research result showed higher value of specific gravity. But, based on statistic test with 95% of confidence level showed that specific gravity of tuberose flower waste using hexane and petroleum ether solvents was not different.

Density is affected by the types and components of essential oils. Each component has different specific gravity. The higher concentration of chemical, the higher the density [10]. Suspected both inert solvents, the components contained in the extract, so it does not affect the components of the oil produced. One of requirement in the selection of the solvent are inert solvents that do not react with the oil component of the flower [7].

4) Acid value

Determining the fatty acid can be used for knowing the quality of oil or fat, it is because the acid number is useful to measure and find the value of free fatty acid on material or sample. The higher value of fatty acid means there is a higher intensity of free fatty acid in the sample.

Based on the research conducted by Hesse and Elze in Guenther [8], the acid numbers were shown as 22 and 25. Those values are not too much different with the value in this research, especially for petroleum ether. However, this research gained lower value for hexane solvent, which is 16,83mg KOH/g sample. Based on statistic test with 95% of confidence level showed that acid value of tuberose flower waste essential oil which uses hexane and petroleum ether was not different. It is considered that the factor is not because the different solvent. Meanwhile, it is caused by the treatment after the extract was collected, specifically is because of the storage. The extract sample often be opened just for knowing the fragrance, so it will cause the oxidation.

5) Ester value

Ester value is the number of milligram of hydroxide (Mg KOH) that is needed to saponify the ester which contained in a gram of oil. The decision of ester value is very important to determine the value of essential oil [7].

The ester value in this research was 357,64mg KOH/g sample for petroleum eter and 392,70mg KOH/g sample for hexane. The ester value in this research was higher than the research of Hesse and Elze in Guenther [8], which are 224 and 230. The high ester value shows that the oil produces fragrance. The higher of ester value, the fragrance will be stronger. The tuberose is known for its fragrance. Based on statistic test with 95% of confidence level showed that ester value of tuberose flower waste essential oil which uses hexane and petroleum ether was not different.

D. Antioxidant Activity

Antioxidant is a chemical compound that is able to contribute one or more electron for free radicals, so the free radicals can be damped down [11]. This antioxidant examination was conducted using the DPPH method. The DPPH method is easy, quick, and responsive for antioxidant activity examination on certain compound or plants extract. The DPPH method measures the ability of specific compound in seizing the free radicals [12].

This research resulted higher percentage of antioxidant activity compared to research conducted by Baskara [13]. It is caused by different method. The maceration method applies solvent as the oil absorption media. The enfleurage method uses fat as oil absorption media. The tuberose waste extract using enfleurage method still consists of fat that is used in the extraction. The fat decreases the intensity of tuberose compounds, thus the antioxidant activity is lower. The enfleurage method also shows that percentage of antioxidant in hexane is higher than petroleum ether.

The result of antioxidant activity in this research is considered as low. The way of extracting natural antioxidant depends on the antioxidant type and the solvent also has to be matched with the polarity of extracted compounds. Besides, the type of organic solvent is influenced by the material authenticity and substrate stability. The solvent with medium rate of polarity is better than a non-polar solvent or high polarity solvent. The polarity index for petroleum ether and hexane are the same and have very small of value which is 0,1. It means that both solvent are non-polar. There is a possibility that petroleum and hexane cannot seize well

the compound of antioxidant source. It affects the antioxidant activity in tuberose extract of being low.

IV. Conclusion

Based on this research, the yield of tuberose extract by maceration with hexane is higher than petroleum ether, also the percentage of antioxidant showed that the extraction with the usage of hexane solvent is higher than petroleum ether.

References

[1] Suyanti, "Post-harvest technology flower tuberose," *Journal of Litbang Pertanian*, vol. 21, no. 1, 2002.

[2] N. S. Sani and R. R. Mahfud, "Jasmine essential oils extraction by enfleurage and volatile solvents extraction," *Journal of Pomits Technic*, vol. 1, no. 1, pp. 1-4, 2012.

[3] R. Armando, *Production of Good Quality Essential Oils*, Jakarta: Penebar Swadaya, 2009.

[4] Suyanti and I. Muhajir, "The influence of efflorescence and extraction time of essential oils contents of tuberose flower multiple cv.," Laporan Hasil Penelitian Balai Penelitian Tanaman Hias, 1998.

[5] R. Nopalas, "The influence of extraction time and level of flower efflorescence against quality of tuberose essential oils of single cultivars (Polianthes tuberose vase gracilis)," Thesis, Faculty of Agricultural Technology, IPB, Bogor, 1999.

[6] D. F. Sihite, "Characterization of Jerangau essential oils (Acorus calamus)," Thesis, Dep. Forestry, Faculty of Agriculture, Univ. North Sumatera, Medan, 2009.

[7] E. Guenther, *Essential Oil: Part I*, Jakarta: Universitas Indonesia Press, 2011.

[8] E. Guenther, *Introduction to Essential Oils Technology*, Jakarta: Balai Pustaka, 1985.

[9] H. Sastrohamidjojo, *Chemistry of Essential Oils*, Yogyakarta: Gadjah Mada University Press, 2004.

[10] F. K. Hidayat, "Essential oil extraction of Kaffir lime leaves (Citrus hystrix DC) on a scale of pilot-plant," Thesis, Dept. Food Technology and Nutrition, Faculty of Agricultural Technology, Bogor Agricultural Institute, 1999.

[11] F. Febrianti, "The influence of belimbing wuluh fruit (Averrhoa bilimbi, L.) extract addition against black tea functional beverages making," Thesis, Dept. Agricultural Technology, Andalas University, Padang, 2013.

[12] E. R. Friatna, R. Achmad, and H. Tanti, "Antioxidant activity testing of citrus fruit's peel (Citrus sinensis) as an alternative material of face masks production," *Journal of Technic Faculty*, 2011.

[13] C. P. D. Baskara, "Utilization potency of tuberose flower waste (Polianthes tuberosa L.) for essential oils production," Thesis, Dept. Agroindustrial Technology, Faculty of Agricultural Technology, Gadjah Mada University, 2016.

Arita Dewi Nugrahini was born in Klaten, Indonesia on January 10th, 1986. She was studied Bachelor Degree on agro-industrial technology at Gadjah Mada University, Yogyakarta (2004) and graduated in 2009 with a degree of STP (Bachelor of Agricultural Technology). In 2013, she obtained a degree MT (Master of Engineering) from the Department of Chemical Engineering, Institute of Technology Bandung. Since 2010 until now she is active as a LECTURER in the Department of Agro-industrial Technology, University of Gadjah Mada. She was an expert in the field of the cocoa industry development in South Sulawesi and active in research in the field of agro-industries. Some publications of her research results were determination of storage conditions for cut roses, production of edible films from filtration waste of rice flour, and the production of medium chain triglycerides from coconut oil as functional food. Her research interests are in the fields of food engineering, by-product of waste agro-industry, and development of horticulture industry.
Ms. Nugrahini been awarded in terms of the local food industry development in her hometown. She is active in some seminars, including as committee on APTA national seminar (Agro-Industrial Technology Professionals Association) and as an author at the international seminar ICOA (International Conference on Agro-industry). Proceedings of the seminar have been published in Agriculture and Agricultural Science Procedia and KNE Life Sciences.

Aisyah Linda Ristanti was born in Magelang, Indonesia on December 14th, 1993. Now she is still studying on Bachelor Degree majoring in agro-industrial technology at University of Gadjah Mada, Yogyakarta.
Since 2011 until now she is active as STUDENT in Department of Agro-industrial Technology, Gadjah Mada University and will graduate in the year of 2016. She was active in student research activities and attended practical work in a company engaged in the field of herbal medicine industry. Her publication of the research results was about forecasting demand for herbal products and waste identification of cut flowers. Her research interests are in the field of agro-industries.
Ms. Ristanti been awarded as a finalist of the national mathematics competitions. She is active role in the activities of organizations and committees, including as coordinator of student activities.

Jumeri was born in Klaten, Indonesia on January 1st, 1971. He was studied Bachelor Degree on faculty of agricultural technology at Gadjah Mada University, Yogyakarta (1991) and graduated in 1996 with a degree of STP (Bachelor of Agricultural Technology). In 2002, he earned a degree M.Si (Master of Science) from faculty of agricultural technology, Gadjah Mada University.
Since 1997 until now he is active as a Lecturer in Department of Agro-industrial Technology, Gadjah Mada University. He has been a consultant at assessment agency of food, medicine, and cosmetics, as well as a staff of experts in research institutes and community service. His several publications were analysis of the potential of cassava as a raw material for making nata, and halal certification. His research interests are in the areas of food security and sovereignty, kosher products, as well as food technology.
Dr. Jumeri been awarded by obtaining several research grants. He is active role in the seminar, among them as an author on a national seminar APTA (Agro-Industrial Technology Professionals Association), author of the international seminar on development of coastal sandy area towards sustainable agriculture, and as an author on international seminar ICOA (International Conference on Agro-industry). Proceedings of the seminar have been published in Agriculture and Agricultural Science Procedia and APTA Journal.