Moisture Reduction on Essential Oil with Batch Distillation Process from A Polianthes Tuberos

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Abstract. Polyanthes tuberos mostly used as cut flowers, with limited time, many flowers become rotten and wasted as garbage, sought another alternative to the utilization it is an essential oil, with distillation process to produce essential oils. The results obtained in the distillation process are each adsorbent giving the results of a mixture of vegetable fats and animals with a moisture content of 0.2169%, the amount of water content indicating the level of hardness of adsorbent. The essential oil obtained crystal clear yellow, a very strong distinctive smell of them, did not leave the place on the filter paper and the results obtained 4.279%. The distillation process is a good technique for the acquisition of essential oils from it with a water content of 0.11%.

1. Introduction
Polianthes tuberos originated from Mexico, this plant in Indonesia is scattered in the district Bangil and Rembang, Pasuruan, East Java, until now the productivity is very high, the price ranges from IDR 400 to IDR 1000 per stalk, is cheaper than Rose and Jasmine [1]. Based on the arrangement of flowers, differentiated by single layered petals, double layered petals and semi-double flowers. There are equal together, so many rotten and wasted flowers are not used, other uses as essential oils [2, 3, 4, 5, 6, 7].

A good purification technique for essential oils on flowers is to use a distillation process, producing essential oils by capturing volatile essential oils from parts of the plant using a fat mixture as adsorbent at low temperatures. Increased tensile forces between esters from fats with essential oils can absorb volatile essential oils [8, 9], essential oils of leaf tobacco with batch distillation and a Flash distillation process [10, 11]. The distillation process ends when the saturated fat with the flower oil is approximately 10% of the volume used. The success of the distillation process depends on the fats used, the fats used to have a level of hardness for essential oils that are absorbed by the flowers [12] essential oils on tobacco leaves with water distillation [13, 14], essential oils with hydrolysis process [15, 16, 17].

This research aims to obtain the right type and composition of absorbent and has a high degree of hardness, and high essential oil yield with distillation process of them, essential oils were produced using distillation process. Fats are used as adsorbent to essential oils from the luscious flower of the night. There is essential oil yield in previous studies with goat fat adsorbent amounted to 0.81%, while using a cow's fat adsorbent of 0.71%. The mixing of fatty adsorbents (cows, goats and white butter) and a mixture of fats (cows, goats and palm oil) is expected to yield a 4% yield.
2. Distillation Process

Adsorbent (fat mixture) weighed 100 grams and applied on 2 glass surfaces of 5 mm each to 50 grams. A fine-weighted night interest is placed to one of the fat surfaces until evenly, combine the two chassis, close the meeting using the wrapper, store it in a place that is not exposed to the light source, the savory flowers are changed every 24 hours for 1 week. Organoleptic test or sensory test is a test using the human senses as the primary tool for the measurement of a product. Organoleptic tests conducted on this study included color testing, smell testing and spot testing. Result analysis is a comparison of the amount of oil produced and uses percent (%) units. Gas chromatography is a mixed separation technique of several compounds based on the pace of motion of the components that are to be separated by the process of flowing a gas flow through the silent phase, the difference in motion rate is caused by the difference in weight molecules, boiling points and astringent substance of each compound [18].

The filtrate has been obtained was inserted into the three-neck flask, place the three neck flask over the electro coat. Install the condenser and the adaptor and then distillation at ±78 °C to separate the alcohol and essential oils, up to the evening flower essential oil is obtained. After 7 days of taking adsorbent and insert into beaker glass, alcohol 96% added with a comparison of fat and alcohol 1:2. Then stir and melt using magnetic the stirrer with temperature no more than ±50 °C, then save for 24 hours, using what man filter paper until separate fat and produce filtrate.

![Distillation apparatus](image)

Description: 1. Electro Coat 2. Thermometer 3. Flask 4. Condenser 5. Water out 6. Clamp 7. Water inlet 8. Adapter 9. Erlenmeyer

3. Results and Discussion

Essential oils from the luscious flowers use adsorbent mix of animal and vegetable fats at room temperature with distillation process. The separation of essential oils from alcohol uses the distillation process. Goat fat and fat mixture with a ratio of 0.0833 have a high moisture content compared to other fats. If the fats used as adsorbents have a sufficiently high moisture content, low oil yield is obtained. This is due to the fat that the high water content tends to be softened and will be easily attached to the flowers and will be wasted at the time of interest turnover.

Organoleptic tests carried out include color testing, appearance, smell and spotting. There are three different colors: yellow, brownish yellow, and brown. Yellow color, much resulting from a fresh night flower condition. While the color of brownish yellow and brown color is produced from the condition
of the savory flower dry night. In the smell test, there are three criteria that are the distinctive fragrance of the savory flower of the night is very smelled (+ +), fragrant typical night flowers that are vaguely smell (+), and smell fat (-). In the sighting test there are two criteria i.e. oil appears clear or oil appears cloudy. If the essential oil obtained is cloudy, then the essential oil obtained still contains oil or grease from adsorbent [19]. It is also supported with spotting test.

Table 1. Moisture content on cow fat, goat fat, white butter and palm oil

| No. | Adsorbent type     | Moisture content (%) | Ratio  |
|-----|--------------------|----------------------|--------|
| 1   | 1 LS : 1 LK : 2 MP | 0.17                 | 0.5000 |
| 2   | 1 LS : 2 LK : 3 MP | 0.58                 | 0.1667 |
| 3   | 1 LS : 3 LK : 4 MP | 2.89                 | 0.0833 |
| 4   | 2 LS : 1 LK : 3 MP | 0.21                 | 0.6667 |
| 5   | 3 LS : 1 LK : 4 MP | 0.05                 | 0.7500 |
| 6   | 1 LS : 1 LK : 2 MY | 0.28                 | 0.5000 |
| 7   | 1 LS : 2 LK : 3 MY | 0.30                 | 0.1667 |
| 8   | 1 LS : 3 LK : 4 MY | 0.10                 | 0.0833 |
| 9   | 2 LS : 1 LK : 3 MY | 0.40                 | 0.6667 |
| 10  | 3 LS : 1 LK : 4 MY | 0.71                 | 0.7500 |
| 11  | MP                 | 0.05                 | -      |
| 12  | LS                 | 2.89                 | -      |
| 13  | LK                 | 0.21                 | -      |
| 14  | MY                 | -                    | -      |

Description: MP = White butter, LS = Beef fat, LK = Goat fat, MY = Palm oil

Spotting test is a common form identification in essential oils that are done with the essential oil in a hard strain. If the essential oil obtained does not leave stains on the filter paper because it evaporates, it indicates that the sample obtained is true essential oil, whereas if the essential oil leaves the stain spots on the filter paper, it signifies that which is acquired is not seed but an oil or fat derived from adsorbent. In this research, the savory interest of the night used as much as 350 grams using distillation process for 7 days. The savory flower is adsorbed with a mixture of fat (fat cow + fat goat + white butter) and a mixture of fat (fat cow + fat goat + oil palm).

Table 2. Color test, apparition, smell of spotting tires savory flower night in fresh flower conditions

| No. | Ratio | Color | Sightings | Smell | Test spots |
|-----|-------|-------|-----------|-------|------------|
| 1   | 0.17  | Yellow| Clear     | +     | No         |
| 2   | 0.58  | Yellow| Clear     | +     | Yes        |
| 3   | 2.89  | Yellow| Clear     | ++    | No         |
| 4   | 0.21  | Yellow| Clear     | ++    | No         |
| 5   | 0.05  | Yellow| Clear     | +     | Yes        |
| 6   | 0.28  | Yellow| Clear     | +     | Yes        |
| 7   | 0.30  | Yellow| Clear     | +     | No         |
| 8   | 0.10  | Yellow| Clear     | +     | Yes        |
| 9   | 0.40  | Yellow| Clear     | +     | Yes        |
| 10  | 0.71  | Yellow| Clear     | -     | Yes        |
The fat mixture that has been saturated with the savory flower essential oil is extracted with an alcoholic solvent. It is then distilled to separate alcohol and essential oils.

**Table 3.** Color test, apparition, smell of spotting tires savory flower night in dry flower conditions

| No. | Ratio | Color                  | Sightings | Smell | Test spots |
|-----|-------|------------------------|-----------|-------|------------|
| 1   | 0.17  | Brownish yellow        | Murky     | -     | No         |
| 2   | 0.58  | Brownish yellow        | Murky     | -     | Yes        |
| 3   | 2.89  | Brownish yellow        | Murky     | -     | Yes        |
| 4   | 0.21  | Brownish yellow        | Murky     | -     | No         |
| 5   | 0.05  | Brownish yellow        | Murky     | -     | Yes        |
| 6   | 0.28  | Brownish yellow        | Murky     | -     | Yes        |
| 7   | 0.30  | Brownish yellow        | Murky     | -     | Yes        |
| 8   | 0.10  | Brownish yellow        | Murky     | -     | No         |
| 9   | 0.40  | Brownish yellow        | Murky     | -     | No         |
| 10  | 0.71  | Brownish yellow        | Murky     | -     | Yes        |

**Description:**

(++) = Fragrant flowers are very smell

(+) = Fragrant flowers are sketchy, smelly smell

(-) = smelling fat

**Table 4.** Level analysis of savory flower oil night on fresh flower conditions

| No | Ratio   | Weight of essential oils (grams) | Essential oils levels (%) |
|----|---------|----------------------------------|---------------------------|
| 1  | 0.5000  | 7,532                            | 2,152                     |
| 2  | 0.1667  | 7,144                            | 2,041                     |
| 3  | 0.0833  | 2,108                            | 0,602                     |
| 4  | 0.6667  | 15,040                           | 4,297                     |
| 5  | 0.7500  | 6,553                            | 1,872                     |
| 6  | 0.5000  | 4,244                            | 1,213                     |
| 7  | 0.1667  | 6,925                            | 1,979                     |
| 8  | 0.0833  | 4,182                            | 1,195                     |
| 9  | 0.6667  | 4,996                            | 1,428                     |
| 10 | 0.7500  | 4,148                            | 1,185                     |

On the Figure 2 the blue colored chart indicates the mixture adsorbent LS: LK: MP. From the starting point at ratio 0.0833 to a ratio of 0.6667 charts increased. Ratio 0.6667 is the highest point on this blue chart. And further, the ratio of 0.75 decreases. The increase and decline caused by the texture in the mixture of adsorbenta, in the ratio of 0.0833 adsorbent mixture used has a high enough water
content that is 2.89% that can be said that the tektur of the Adsorbennya is very mushy so low of 0.602% [20]. The chart at a ratio of 0.0833 to a ratio of 0.6667 is increased which indicates that the texture belonging to adsorbent is getting better for a good night flower essential oil. Can be seen from the size of the amount obtained at ratio 0.6667 which has a water content of 0.21% is 4.297%. While in ratio 0.75 decreased, caused by moisture content contained in the low adsorbennya mixture of 0.05% which indicates that the texture of the Adsorbennya very hard and the harsh texture will complicate the absorption good night flower essential oil because the flowers do not stick well on the surface adsorbent so that the lowland obtained is low enough 1.213%[21].

![Graph](image)

**Figure 2.** Graph of relationship between adsorbent comparison with fresh flower conditions.

Orange-coloured graphics indicate that the adsorbent used is a mixture of palm oil with a mixture of animal fats (fat cow + fat goat). At ratio 0.0833 until the ratio of 0.1667 trend increased. The next point experienced a penuran at ratio 0.5 and increased slightly in ratio 0.667 then decreased again at ratio 0.75. The ups and downs of these graphs are caused by the moisture content contained in each adsorbent mixture, at the starting point of ratio 0.0833 has a fairly low water content of 0.10% which has a tektur quite hard so it is difficult to absorb essential oils To the low night interest of 1.195%. Ratio 0.1667 is the maximum point found on the chart with a large size of 1.979% of the water content contained at the rate of 0.30% in other words adsorbent used to have a tektur that fit not too soft and not too hard so can with maximum absorbing flowers essential oil night. And ratio 0.75 is the lowest result on this chart with a yield of 1.185%, this ratio has a fairly high water content of 0.71% when compared to the moisture content that belongs to a mixture of animal fat adsoben and coconut oil This palm so that it has a fairly mushy texture that results in low-acquired lowland. The result is high enough to be 4.279% compared to previous research conducted [22]. The adsorbent used is a mixture of cow fat and goat fat, getting a yield of 0.174%. The texture of a fat adsorbent greatly affects its adsorb power. Because if too dense fat will be difficult to absorb essential oils, because the petals of the flower not stick to the surface of fat. So the adsorb process cannot be maximized. And conversely, if the texture of fat is too soft then fat will stick to the flowers and will be wasted on the process of changing the flowers [23].

4. Conclusion

The highest yield essential oil of interest is obtained 4.297% in adsorbents with a ratio of beef fat: goat fat: white butter on fresh flower conditions, the distillation process is a good technique for the acquisition of essential oils from Polianthes tuberos with a water content of 0.11%, with adsorbent goat fat acquired essential oil 1.434%, cow adsorbent 2.169%, both have higher yield than the previous researcher 0.81%. With white butter adsorbent obtained 2.382% higher than the previous researcher was 0.7%. The best adsorbent type is the white adsorbent, odorless and has a suitable texture (not too
harsh and not too inert). In this study, the best adsorbent comparison gained was a mixture of white butter with animal fats (fatty goat fat cows) with comparisons (2 fat cows, 1 goat fat, and 3 white butter).

References

[1] Suyanti 2002 Post Harvest Technology Journal of Agricultural Research and Development volume 2 no1 p 30-35, Teknologi Pasca Panen Jurnal Penelitian dan Pengembangan Pertanian volume 2 no1 p 30-35

[2] M Ardiansyah, D Noerianti, and Muhandoyo 2013 The supply and demand of savory interest (Polianthes Tuberose L.) Village Rembang District Pasuruan Journal Primordial volume 9 no 2 p 54-55, Penawaran dan Permintaan Bunga Sedap Malam (Polianthes Tuberose L.) Desa Rembang Kabupaten Pasuruan Jurnal Primordial volume 9 no 2 p 54-55

[3] M Tutt, T Kikas, and J Olt 2012 Influence of different pretreatment methods on bioethanol production from wheat straw Agronomy Research Biosystem Engineering volume 1 p 269-276

[4] E C Bensah, and M Mensah 2013 Chemical Pretreatment Methods for the Production of Cellulose Ethanol: Technologies and Innovations Research Article Article ID 719607, 21 pages, http://dx.doi.org/10.1155/2013/719607

[5] F Battista, G Mancini, B Ruggeri, and D Fino 2016 Selection of the best pretreatment for hydrogen and bioethanol production from olive oil waste products Renewable Energy, volume 88 p 401-407

[6] Y Liu, H Zhou, S Wang, K Wang, and S Xiaojun 2015 Comparison of 󰀄-irradiation with other pretreatments followed with simultaneous saccharification and fermentation on bioconversion of microcrystalline cellulose for bioethanol production Bioresource Technology volume182 p 289-295

[7] R C Kuhad, R Gupta, Y P Khasa, and A Singh 2010 Bioethanol Production from Lantana Camara (Red Sage): Pretreatment, Saccharification, and Fermentation Bioresource Technology, volume 101 p 8348-8354

[8] R Faisal, R Purwanti, and N Chotijatun 2016 Pengaruh Jenis Adsorben dalam Proses Enfleurasi Minyak Atsiri Daun Kemangi. Jurnal Permata Indonesia volume 7 no 1 p 1-5

[9] N Brosse, M N M Ibrahim, and A A Rahim 2011 Biomass to Bioethanol: Initiatives of the Future for Lignin Review Article, Article ID 461482, page 10, doi:105402/2011/461482

[10] N K Sari and E Dira 2016 Comparasion Production Bioethanol from Cellulose using Batch Distillation and Flash Distillation Process Journal of GEOMATE volume 58 no 01003 p 1-5

[11] S Patrisia 2017 Influence of type of fat and vegetable oil in the process of extraction system of essential oil characteristics of Cambodian flowers sandalwood (Plumeria Alba) Journal of Agro-Industry engineering and management volume 5 no 2 p 39-44, Pengaruh Jenis Lemak dan Minyak Nabati Pada Proses Ekstraksi Sistem Enfleurasi Terhadap Karakteristik Minyak Atsiri Bunga Kamboja Cendana (Plumeria Alba) Jurnal Rekayasa dan Manajemen Agro Industri volume 5 no 2 p 39-44

[12] S K Thangavelu, A S Ahmed, and F N Ani 2014 Bioethanol Production from Sago Pith Waste Using Microwave Hydrothermal Hydrolysis Accelerated by Carbon Dioxide Applied Energy, volume 128 p 277-283
[16] G S Geetha, and A N Gopalakrishnan 2011 Bioethanol production from Paper Fibre Residue Using Diluted Alkali Hydrolysis and the Fermentation Process E-Journal of Chemistry, volume 8 no 4 p 1951-1957

[17] F Teymouri, L L Peres, Alizadeh, and B E Dale 2005 Optimization of the Ammonia Fiber Explosion (AFEX) Treatment Parameters for Enzymatic Hydrolysis of Corn Stover Biorecs. Tech. volume 96 p 2014-2018

[18] R Maryan 2009 Fine oil Extraction Night Maceration Method distillation Vacuum journal Technology and Food volume 5 no 2 p 1-6, "Ekstraksi Minyak Sedap Malam Metode Maserasi Destilasi Vacuum Jurnal Teknologi dan Pangan" volume 5 no 2 p 1-6

[19] D Yulianingsih, Amiarsi1, and S Sabari 2004 Enefleuration techniques in the process of making Rose Oil Horticultural Journal volume 17 no 4 pp 39-45, "Teknik Enflouerasi dalam Proses Pembuatan Minyak Mawar Jurnal Hortikultura" volume 17 no 4 pp 39-45

[20] M Hetik, D Maghfoer, and T Wardiyati 2013 Influence of adsorbent type on quality essential oil in two cultivars savory flower night (Polianthes Tuberose) Journal of Crop Production volume 1 no 4 p 308-310, "Pengaruh Jenis Adsorben Terhadap Kualitas Minyak Atsiri Pada Dua Kultivar Bunga Sedap Malam (Polianthes Tuberose) Jurnal Produksi Tanaman" volume 1 no 4 p 308-310

[21] Sulong and M Faisal 2006 Extraction of Essential Oils from Jasmine Flower Using Solvent Extraction Method. Faculty of Chemical & Natural Resources Engineering, Thesis University College of Engineering & Technology Malaysia Johor Bahru Malaysia

[22] Suryani and Mutia 1999 Research on production process of the delicate flowers essential oil (Polianthes Tuberose Var Gracilis) with the method of Enflouration Faculty of Agricultural Technology Bogor Institute of Agriculture, West Java Indonesia, "Kajian Proses Produksi Minyak Atsiri Bunga Sedap Malam Tunggal (Polianthes Tuberose Var Gracilis) Dengan Metoda Enflouerasi Fakultas Teknologi Pertanian Institut Pertanian Bogor Jawa Barat Indonesia"

[23] R Prapassorn, U Dilokkunanant, U Sukkatta, and S Vajrodaya 2009 Extraction Methods for Tuberose Oil and Their Chemical Components Journal Kasetsart. volume 43 no 2 p 204-211

Acknowledgment

The authors would like to acknowledge the financial support of the Directorate Research and Public Service. The Directorate of Research and Development Strengthening. The Ministry of Research, Technology, and Higher Education of the Republic of Indonesia with the Research-Based Competence Grant. Contract Number: 083/SP2H/LT/DRPM/2018.