FORMULATION AND PHYSICAL STABILITY EVALUATION OF ESSENTIAL OIL PARFUME

Oleh
Yelfi Anwar[1*], Efa Bonita[2], Andrianopsyah Mas Jaya Putra[3]
[1,2]Faculty of Pharmacy, University of 17 August 1945 Jakarta, Nort Jakarta, Indonesia
[3]Institut Sains dan Teknologi Al-Kamal (ISTA), Jakarta Barat, Indonesia
e-mail: [1]*yelfi.anwar@uta45jakarta.ac.id

Abstrak

The need for essential oils will continue to increase by up to 8–10% in line with the increasing production of perfume. This shows that there is a public need for the perfume, which is increasing day by day. This research design is experimental and aims to determine a symptom or effect that arises as a result of certain treatments, to produce eau de toilette-type formulas and perfumes that have good quality and are liked by respondents. The evaluation was carried out using several tests, namely the organoleptic test, specific gravity test, fragrance resistance test, spreadability test, sensitivity test, physical stability test, pH test, and hedonic test. The results showed that organoleptically, all formulas produced a yellow color, were homogeneous and free of particles, and had a slightly pungent smell of fresh oranges. The specific gravity of all formulas met the requirements, with the largest specific gravity F2 of 0.86 at cold temperatures. The fragrance resistance test still smelled after 4 hours. The biggest spot test results were on F1, with a diameter of 49.17 mm. The sensitivity test did not show any allergic reactions or irritation of the skin during the use of the perfume, and the physical stability test of the entire formula did not show any separation or change in shape over a period of 7 weeks, both in room temperature storage and at cold temperatures. The entire formula has a liquid texture, is not sticky, and is easy to apply. The results of the evaluation of pH testing are in the range of 5–6 and have fulfilled the requirements of SNI 16-4399-1996, cosmetic preparations must have a pH according to the pH of the skin, which is in the range of 4.5–6.5. And the most preferred formula is Formula 1.

Kata Kunci: Eau De Toilette, Cosmetics, Citrus Scent

PENDAHULUAN

According to the FDA (United States' Food and Drug Administration), Fragrances are chemical compounds resulting from a combination of certain formulas that give a different aroma to each perfume, including those used in other products. Fragrances play a very important role in increasing attractiveness. Refreshing aromas affect comfort and have a significant impact on the overall cosmetic evaluation (Primadina 2013).

Currently, one of the agro-industrial products that has bright prospects for development is essential oil. Essential oils, commonly known as "etheric oils" or "flying oils," are produced by plants and can be obtained from the roots, stems, leaves, and flowers of plants. Essential oils in industry can be used as cosmetics, perfumes, antiseptics, medicines, and aromatherapy (Sriandila 2020).

Essential oil is one of the export commodities with quite high yields. Essential oils are now getting more attention because they have many benefits, are relatively safe, and can be widely accepted by society. The benefits and natural activities of this essential oil are related to the chemical content in it. This component determines the commercial

value of a raw material in industry (Pujiarti et al. 2015).

Patchouli essential oil is a type of essential oil that is commonly used in perfumes. However, the source of these raw materials has not been fully utilized by industry players to process them into products in the form of perfume in Indonesia. Indonesia has not been able to manage these raw materials until now due to limitations, so that until now it has only imported finished products in the form of perfume worth as much as US 19.9 million (Ginting, Ishak, and Ilyas 2021).

The prospect of exporting patchouli oil from Indonesia in the future is still quite large, which is in line with the increasing demand for perfumes and cosmetics. *trend mode* and the lack of development of materials for patchouli oil substitution in the perfume and cosmetics industries. Patchouli oil has a development strategy in the perfume industry, considering that the world market requires 1,200–1,400 tons of patchouli oil each year, while the available production has only reached 1,000 tons per year (Harli 2017).

Based on existing data, the need for essential oils will continue to increase, reaching up to 8–10% in line with the increasing production of perfume. On the global market, there are 70 different types of essential oils.Indonesia has 40 types of essential oil-producing plants, but only 14 species have a real role as export commodities (Idris, Ramajura, and Said 2014).

There has been a rapid increase in the amount of perfume production in the last 20 years; even the perfume industry is estimated to be able to obtain sales of 25–30 million dollars per year (Gunawan and Rahayu 2021).

This shows that there is a public need for perfume, which is increasing day by day from a secondary need to a primary need. So, based on the background above, the researcher intends to utilize essential oils in Indonesia to formulate selected essential oils, namely Citrus hystric essential oil as the top note, Cananga ordorata essential oil as the middle note, and Patchaouli essential oil as the base note in perfume and cologne.

METODE PENELITIAN

The materials used in this study were 3 types of essential oils obtained from farmers and refineries in Lembang, West Java, namely, orange oil, ylang ylang oil, and patchouli oil. as well as other supporting materials, namely 96% alcohol, 0.3% benzophenone-2, 3% aqua dest, 3% propylene glycol, 1% phenoxyethanol, and 3% PEG-40 HCO. 1 set of GC-MS brands Aligent 7890B (GC) and 5977A (MSD), an analytical balance, parchment paper, a measuring cup, a beaker glass, a 100mL bottle, a dropper pipette, a watch glass, a measuring flask, a pycnometer, a caliper, a funnel, a paper test, filter paper, and HVS paper were used in this study.

Research design

The research design is quantitative and experimental in nature, with the goal of determining a symptom or effect that occurs as a result of a specific treatment (Gunawan and Rahayu 2021).The treatment is in the form of an Eau de Toilette perfume formulation (F1, F2, F3), which has citrus characters as the top note, ylang ylang as the middle note, and patchouli as the base note. This formulation is carried out through trial and error throughout the aging process in order to optimize the perfume's quality.

Evaluation of the quality of the preparation includes organoleptic tests (color test, homogeneity, particle-free, aroma test), specific gravity tests (SNI 16-4949-1998), fragrance resistance tests (Mustakim, Sari, and Kholis 2019), spot tests (Gunawan and Rahayu

2021), sensitivity test, pH stability test, and preference test.

This research was conducted at the University Laboratory on August 17, 1945, in Jakarta in November 2022–January 2023, with the target outcome of this research being to obtain good eau de toilette perfume formulas and products that meet the physical quality requirements based on SNI 16-4949-1998 and are liked by respondents.

A. Organoleptic Test

1. Color

Take 1 mL of the formula sample using a measuring pipette, then drop it on the watch glass and observe it against a black background.

2. Homogeneity

Take 1 mL of the formula sample using a measuring pipette, then drop it on the watch glass and observe it against a black background.

3. Particle free

Take 1 mL of the formula sample using a measuring pipette, then drop it on the watch glass and observe it against a black background.

4. Aroma test

Do it visually by spraying it on *paper with* as much as 2 sprays, then letting it stand for 1 minute, then doing the aroma assessment by inhaling it directly through the nose. The results are said to meet the requirements if the perfume smell has the desired characteristics (Gunawan and Rahayu 2021).

B. Specific gravity test

Evaluation of specific gravity according to SNI 16-4949-1998 for perfume is carried out by:

- Take 25 mL of perfume using a 25 mL volumetric flask, put it in a 25 mL pycnometer, and weigh it on an analytical balance.
- Then do the calculation using the formula.

$$\rho = \frac{W2 - W0}{W1 - W0}$$

Information :

P = denotes the perfume's specific gravity.

W2 = pycnometer weight + perfume

W1 = pycnometer weight + aqua dest

W0 = empty pycnometer weight

C. Fragrance durability

A perfume resistance test is done by spraying perfume on paper test, and then an assessment is carried out. Starting from the first (1st) hour to the fourth (4th) hour, the results are said to meet the requirements. If, after the fourth (4th) hour, the fragrance of perfume can still be detected by the sense of smell, then these results are considered usable (Mustakim et al. 2019).

D. Spot Test

Spraying a sample of perfume on colored paper and taking measurements is how Test Spot Perfume is done. Using an electric vernier caliper to measure the

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diameter of the produced spot (Mustakim et al. 2019), the wider the spread of the perfume, the better the quality of the perfume.

E. Temperature stability testing

Examine by observing changes every time, which in this experiment was carried out for 7 weeks (week 0, week 1, week 2, week 3, week 4, week 5, and week 6) at various storage temperatures, namely room temperature (20–25^{o}C) and cold temperatures (2–8^{o}C), (Farhamzah and Aeni Indrayati 2019).

F. Sensitivity test

This test was carried out directly by the researcher with an open test on the arm by spraying the perfume formula three times a day and leaving it open for 10 hours, then being observed.

G. pH Test

The pH test was carried out using a pH meter. The electrode is immersed in the perfume preparation. The pH value that appears on the screen is then recorded. Measurements were made at room temperature (Iswandana and Sihombing 2017).

H. Hedonic test

In distributing questionnaires to collect data, researchers distributed media questionnaires. *Google Forms* as well as directly by giving *paper* tests to respondents. This hedonic test used 35 respondents using descriptive analysis.

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HASIL DAN PEMBAHASAN

Parfume Formulation

Table 1. Parfume Formulation

No	Ingredient	Formulation (%)		
		F1	F2	F3
1	*Essential Oil Citrus hystric*	5	4	3
2	*Essential Oil Cananga odorata*	2	1,6	1,2
3	*Essential Oil Pogostemon cablin Benth*	3	2,4	1,8
4	Benzophenone-2	0,3	0,3	0,3
5	Aquadest	3	3	3
6	Propilenglikol	3	3	3
7	Phenoxyethanol	1	1	1
8	PEG-40 HCO	3	3	3
9	Ethanol 96% Add	100	100	100

Evaluation of perfume quality

A. Organoleptic

According to SNI 16-4949-1998, the organoleptic requirements for the quality of non-aerosol perfume include color, homogeneity, particle-free status, and aroma. The results of the visual tests that have been carried out by the researchers on all perfume samples placed on the watch glass using a black background produce a perfume color that is not cloudy or clear (light yellow), the fragrance ingredients and additional ingredients are also mixed as a whole to form one homogeneous solution phase, there are no foreign particles that enter the perfume preparation (particle free), and the perfume has a final aroma that is a fresh citrus scent.

These results are the same as research (Hardiyati, Fajar, and Novitasari 2020), which states that the visual test results of all samples show fairly good color consistency, as well as aroma testing with a distinctive pungent odor. Likewise, with research from (Gunawan and Rahayu 2021), the organoleptic test for all parameters (clarity, homogeneity, particle-free, and aroma) showed a result of 100% fulfilling the requirements.

According to (Maimunah et al. 2018), organoleptic observations of all preparations showed that the preparations did not have a significant change, namely a light brown color, a

characteristic odor of perfume, and a clear and transparent appearance. Observations in this parameter were said to be stable before; the components in the preparation during storage did not experience a reaction between one material and another, so that there are no signs of reaction from changes in color, appearance, or smell. The results of the organoleptic evaluation carried out by the researchers for all parameters over 7 weeks also showed that the results met the requirements.

B. Specific gravity

Based on the test results, the average specific gravity of all formulas meets the requirements according to SNI 16-4949-1998, namely that the quality requirements for non-aerosol perfumes are 0.7–1.2. can be seen in figure 1 & 2.

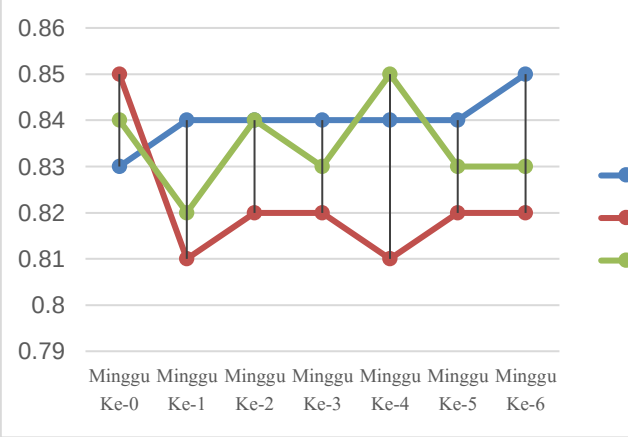

Figure 1. Specific gravity at room temperature

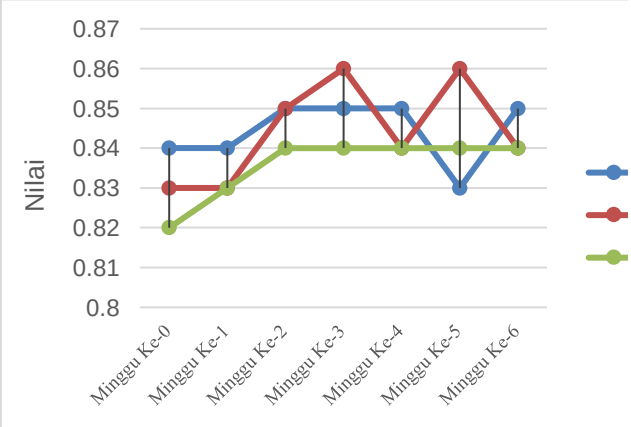

Figure 2. Specific gravity at cold temperatures

The results of the specific gravity evaluation show that the entire formula meets the requirements according to SNI 16-4949-1998. This is due to the factor of using the right solvent, 96% ethanol, with an average weight of 0.80 g/mL, while the specific gravity of fragrances and excipients is not much different with an average value of 0.7–1.2. The highest specific gravity occurs in formula-2, ieat cold temperature storage in the third week of 0.86 and storage at room temperature in the 0th week of 0.85. Specific gravity is closely related to the viscosity of a fluid; the greater the density, the greater the viscosity. However, the greater the viscosity of preparation, the more difficult it will be for the perfume to be removed through the sprayer and the narrower the spread of the perfume will be**.** These results are not much different from the study (Gunawan and Rahayu 2021), with the largest specific gravity of 0.86 in F5 with a 10% concentration of eau de toilette.

C. Fragrance durability

The results of the evaluation of fragrance resistance showed that the entire perfume formula still smelled after 4 (four) hours from week 0 to week 6. where the aroma produced is categorized as strong to rather strong. The same thing was explained (Ginting et al. 2021), who said that the overall formula has almost the same aroma staying power for approximately 2 days with formulas $B_1S_1T_1M_1$, $B_1S_1T_4$, and $B_1S_1T_5M_5$.

D. Spot Test

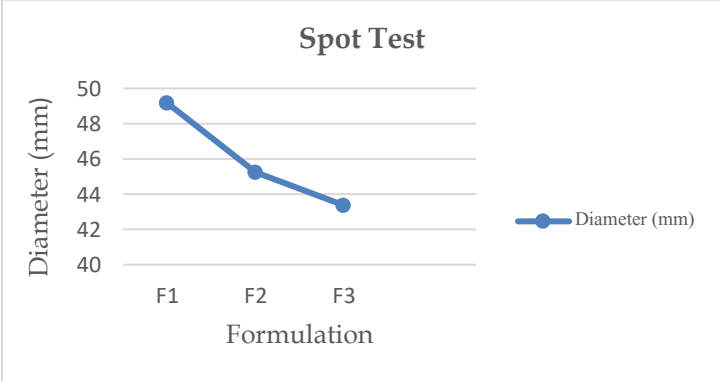

Figure 3. Spot test

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The average spot test result was 45.92mm. Based on SNI 16-4949-1998, perfume preparations (cologne) do not require a diameter with a certain value. The same is true for research (Gunawan and Rahayu 2021), but the spot test yields larger results, with an average of 75.22 mm. Both look different; this refers to the diameter of the spray tool used. The diameter of the perfume spread is influenced by the shape and diameter of the sprayer used. The greater the spread of perfume, the better the perfume used (Mustakim et al. 2019).

E. Ph Test

This pH test aims to determine the safety of perfume preparations so that, when used, they do not irritate the skin. The compatibility of the pH value of a topical preparation with the pH of the skin will affect the skin's readiness for a preparation. The results of the pH test can be seen in Figure 4.

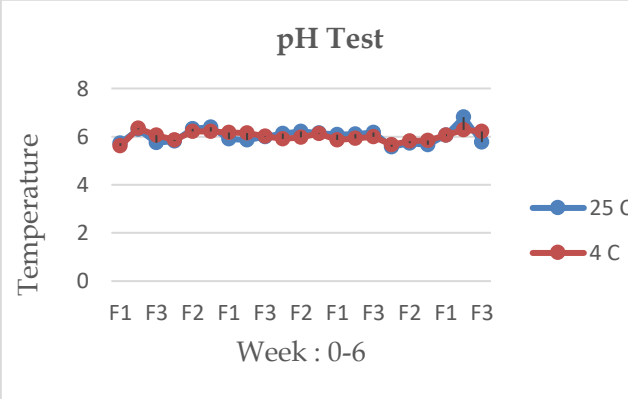

Figure 4. pH testing from week 0 to week 6

According to SNI 16-4399-1996, cosmetic preparations must have a pH according to the pH of the skin, which ranges from 4.5 to 6.5 (Kusantati, Prihatin, and Wiana 2018). Because if the pH is too acidic, it can cause skin irritation, and if the pH is too large, the skin will become itchy or scaly. The pH value of the preparations produced during these 7 weeks of storage showed a difference but was still within range according to skin pH standards. This shows that the results of the evaluation of the pH of the perfume preparations have met the requirements for pH testing.

F. Sensitivity test

The results of the sensitivity test were carried out for 7 weeks every 4 hours, and all formulas did not show any sensitive reactions such as redness or excessive itching. It's the same with research. The results of the irritation test (Macrina Enggar R. L., Hartati Soetjipto 2022), showed that the solid perfume used did not cause symptoms of irritation to the panelists so the perfume cream could be used safely**.** These results indicate that the resulting perfume meets the requirements.

G. Physical stability test

The results of the evaluation of the overall physical stability of the formula did not show any separation or change in shape during a period of 7 weeks, both at room temperature and at cold temperatures. The entire formula has a liquid texture, is not sticky, and is easy to apply. Research (Suryani 2020), shows that each formula does not show any separation or change, namely having a good shape, because the shape remains hard, does not melt or soften, and is not watery. Research (Farhamzah and Aeni Indrayati 2019), The stability test at room temperature for 12 weeks gave stable results for 3 formulas until the 8th week, but in the 12th week there was a slightly faded color change in formulas 2 and 3. This could be due to the influence of perfume reacting with astaxhantin, which gives an orange color to the product.

H. Hedonic Test

Evaluation of the perfume formula was carried out using a data collection sheet (*Google Form*) in the form of a questionnaire. In this test, the panelists expressed their personal responses and opinions about their likes and dislikes for the smell or aroma

produced and the packaging presented by the researcher.

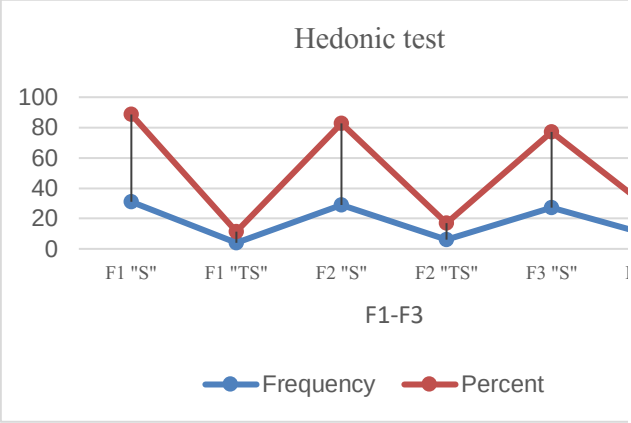

Figure 5. Aroma Hedonic test

The preference level test through weighting the overall value of the perfume on 35 respondents gave the result that the perfume in formula 1 had a better level of preference compared to formulas 2 and 3, namely, 31 people (88.6%) liked it and 4 people (11.4%) did not like it, while for formula 2, as many as 29 people (82.9%) liked it and 6 people (17.1%) did not like it. 27 people (77.1%) liked the aroma in Formula 3, and 8 people (22.9%) didn't. So that, from the results of the hedonic aroma test for the entire formula, formula 1 is the chosen formula for the scent that the respondent likes.

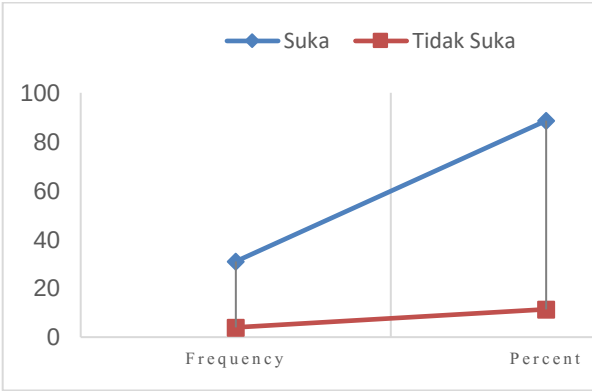

Figure 6. Packaging Hedonic test

Based on the diagram above, out of a total of 35 respondents who were selected, as many as 31 people liked the packaging presented by the researcher, or as much as 88.6% liked it, and 4 people, or 11.4%, did not like the packaging presented. This shows that more respondents quite like the packaging that has been provided by the researcher.

PENUTUP

Kesimpulan

All perfume formulas produced through product quality evaluation have met the requirements according to SNI 16-4949-1998, and formula 1 (5%,2%,3%) is the selected formula that is most preferred by respondents.

Future research can modify the formulation with differences in fragrance to be more varied on each note, and efforts need to be made to improve the quality of essential oils in order to have a higher added value.

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