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FORMULASI SEDIAAN NANO-GEL SPEARMINT OIL *(Mentha spicata* L.*)* DENGAN VARIASI KONSENRASI HPMC

FORMULATION OF NANO-GEL SPEARMINT OIL *(Mentha spicata* L.*)* PREPARATION WITH VARIATIONS OF HMPC CONCETRATION

Sinta Yuliana[1*], Ratih Guswinda Lestari[2], Hanindya Fidela Ulayya[3], Nabilla Putri Auliyah[4]
Pharmacy Study Program, Faculty of Health Sciences, Kusuma Husada University, Surakarta, Surakarta, Indonesia
Nomor Handphone Untuk keperluan koordinasi : 0878-2550-7363

Corresponding Author:
Sinta Yuliana
Pharmacy Study Program, Faculty of Health Sciences, Kusuma Husada University, Surakarta, Surakarta, Indonesia
Email:sinta@ukh.ac.id

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ABSTRAK

Mentha spicata L., yang dikenal sebagai spearmint, tanaman kaya akan minyak esensial (1,2% - 1,5%) dengan kandungan utama senyawa *myrcene, L-limonene, cis-dihydrocarvone,* dan *L-carvone* yang memiliki aktivitas antiinflamasi, antioksidan, dan antifungi. Penelitian ini bertujuan untuk memformulasi sediaan nanogel minyak spearmint guna meningkatkan penetrasi zat aktif ke kulit dan mengetahui pengaruh variasi konsentrasi HPMC (3%, 5%, dan 7%) terhadap karakteristik fisik sediaan serta nilai persen transmitannya. Metode penelitian dilakukan secara eksperimental. Karakterisasi sediaan meliputi uji organoleptik, homogenitas, pH, viskositas, daya sebar, daya lekat, serta uji persen transmitan menggunakan spektrofotometri UV-Vis untuk mengukur kejernihan sediaan nanoemulsi. Hasil penelitian menunjukkan bahwa nanoemulsi spearmint memiliki ukuran partikel rata-rata 9,6 nm dengan nilai persen transmitan mencapai 98,23%, yang mengindikasikan terbentuknya sistem berukuran nano yang jernih. Variasi konsentrasi HPMC berpengaruh signifikan terhadap sifat fisik gel; semakin tinggi konsentrasi HPMC, maka viskositas dan daya lekat akan meningkat, sementara daya sebar akan menurun. Formula III dengan konsentrasi HPMC 7% disimpulkan sebagai formula terbaik karena memenuhi seluruh parameter persyaratan mutu fisik sediaan gel yang ideal, meliputi pH yang aman bagi kulit (6,42), viskositas yang stabil (340 dPas), serta daya sebar dan daya lekat yang sesuai literatur.

ABSTRACT

The *Mentha spicata* L., commonly known as spearmint, is a plant rich in essential oils (1.2% – 1.5%) with major compounds including myrcene, L-limonene, *cis*-dihydrocarvone, and L-carvone, which possess anti-inflammatory, antioxidant, and antifungal activities. This study aims to formulate a spearmint oil nanogel preparation to enhance the skin penetration of active substances and to evaluate the effect of varying HPMC concentrations (3%, 5%, and 7%) on the physical characteristics of the preparation and its percent transmittance value. The research was conducted experimentally. The characterization of the preparation included organoleptic tests, homogeneity, pH, viscosity, spreadability, adhesion capacity, and percent transmittance testing using UV-Vis spectrophotometry to measure the clarity of the nanoemulsion preparation. The results showed that the spearmint nanoemulsion had an average particle size of 9.6 nm with a percent transmittance value reaching 98.23%, indicating the formation of a clear nano-sized system. Variations in HPMC concentration had a significant effect on the physical properties of the gel; higher HPMC concentrations resulted in increased viscosity and adhesion capacity, while spreadability decreased. Formula III, containing 7% HPMC, was concluded to be the best formula as it met all the parameter requirements for the physical quality of an ideal gel preparation, including a skin-safe pH (6.42), stable viscosity (340 dPas), as well as spreadability and adhesion capacity consistent with the literature.

1. INTRODUCTION

Indonesia is rich in natural resources that are increasingly being developed for medicinal purposes and disease prevention, particularly to minimize side effects during long-term use. One plant with great potential is *Mentha spicata L.*, or spearmint, which is known to contain approximately 1,2% to 1,5% essential oil [1]. Spearmint oil contains active compounds such as myrcene, L-limonene, cis-dihydrocarvone, and L-carvone, which offer various pharmacological benefits, including anti-inflammatory, anti-irritant, antioxidant, and antifungal properties [2]. Previous studies have shown that spearmint essential oil is effective in inhibiting pro-inflammatory biomarkers in human skin cells, making it a promising candidate for skincare products [3]. Previous studies demonstrated that spearmint oil at concentrations of 0,5%–1,0% effectively reduced skin inflammation in test rabbits [4].

However, the use of active ingredients in conventional dosage forms is often less effective in reaching therapeutic targets. Therefore, the development of nanotechnology-based drug delivery systems, such as nanogels, has become a widely discussed solution due to their ability to increase the penetration of active substances into the skin and provide controlled release [5]. Nanogels are delivery systems utilizing nanoparticles within a hydrated polymer network, typically ranging from 1 to 100 nanometers, which can enhance the stability and bioavailability of lipophilic active ingredients [6].

In nanogel formulation, the choice of a gelling agent is crucial in determining the physical characteristics of the preparation. Hydroxypropyl Methylcellulose (HPMC) is a semi-synthetic cellulose derivative polymer frequently used for its ability to form stable gel matrices through hydrogen bonding [7]. Variations in HPMC concentration are known to affect viscosity, pH, spreadability, and homogeneity. Earlier research indicates that specific concentrations of HPMC can produce viscosity and physical quality that meet pharmaceutical standards [8].

This study aims to formulate a spearmint oil nanogel by varying HPMC concentrations (3%, 5%, and 7%) to determine its effect on physical characteristics and delivery system efficiency [9]. The testing parameters include organoleptic properties, viscosity, pH, spreadability, adhesion, and percent transmittance as an indicator of clarity and particle size within the nano-system. Through this research, it is expected that the best nanogel formula will be identified to optimize the potential of spearmint oil as an effective topical agent [10].

2. METHOD

Materials

UV-Vis spectrophotometer (Shimadzu 1800, Japan), magnetic stirrer (C MAG HS 7), sonicator (Elmasonic P30H), viscometer (Rion VT-06), pH meter (Ohaus), and various laboratory glassware (Pyrex). Spearmint oil, Tween 80, PEG 400, HPMC, Propylene Glycol,

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Methylparaben, Propylparaben, and distilled water (Aquadest) were all from Haiap Chemicals.

Methods

1. **Preparation of Nanoemulsion**

Spearmint oil nano-emulsion was made by mixing surfactants and cosurfactants, which used Tween 80 and PEG 400. Using a magnetic stirrer at a speed of 200 rpm for 5 minutes to homogenize. Spearmint oil was then added little by little to the surfactant and cosurfactant mixture and stirred using a magnetic stirrer for 30 minutes. Add distilled water slowly and while stirring to 100 mL. Then the sonication process was carried out for 30 minutes at a temperature of 37°C [11].

Table 1. Spearmint oil Nano-Emulsion Formulation

	Formula (%)			
Material	**F1**	**F2**	**F3**	**Function**
Spearmint oil	0,5	0,5	0,5	Active substance
Tween 80 : PEG 400 (8 : 1)	60	60	60	Surfactans & Cosurfactans
Aquadest ad	100	100	100	Solvent

2. **Mixing nano-emulsion into gel base**

The gel is made by gradually dispersing HPMC into hot water (80°C). It is then left to swell, then stirred to form a gel. Dissolve methylparaben and propylparaben in propylene glycol and then mix it into the gel. The spearmint oil nano-emulsion is slowly added while stirring until homogeneous [11].

Table 2. Spearmint oil Nano-Gel Formulation

	Formula (%)			
Material	**F1**	**F2**	**F3**	**Function**
Nanoemulsion *Spearmint oil*	5	5	5	Active substance
HPMC	3	5	7	G*elling agent*
Propylenglikol	10	10	00	Humektan
Metylparaben	0,02	0,02	0,02	Preservative
Propylparaben	0,05	0,05	0,05	Preservative
Aquadest ad	100	100	100	Solvent

3. **Test characteristics**

 a. **Organoleptic Test**

Examination in the organoleptic test includes visualization of the nanogel preparation, including texture, odor, and color which are observed directly [11].

 b. **Homogeneity Test**

As much as 0,1 grams of the preparation is placed on a glass object and then observed to see if there are any lumps, if there are none then it is declared homogeneous [11].

 c. **Spreadability Test**

A 0,5 grams sample is placed in the center of the test apparatus, left for 1 minute, and its diameter is recorded. Add a load of 50 to 100 grams, observe the diameter, and record the test result. For gels, a diameter of 5 to 7 cm is typically sufficient for acceptable results [6].

d. Adhesion Test

A 0,5 grams preparation is placed in the center of the test apparatus. A 1-kg weight is applied to the slide and left for 15 minutes. Then, replace the weight with an 80-gram one, pull the lever, and observe how long it takes for the slide to detach. For gel preparations, this usually takes less than 4 seconds for accurate results. Record the time as the test result [6].

e. pH test

Measurements are made using a pH meter and the readings are observed. For gel preparations, the readings are between 4,5 and 6,5 [6].

f. Viscosity Test

Measurements are made using a Rion viscometer and an appropriate spindle. For gel preparations, a range of 200 to 700 dPs is typically required for satisfactory results [6].

4. Transmittance Percentage Test

Percent transmittance is a unit used in chemical analysis using a spectrophotometer to express the amount of light or electromagnetic radiation that successfully passes through or is transmitted by a sample [6]. The percent transmittance test is used for clarity testing in nanotechnology. A %T value approaching 100% is used as an indicator that the sample is very clear and the particles are very small [8].

Data Analysis

Data obtained from the characteristic and transmittance tests of spearmint oil nano-emulsions were analyzed descriptively. The results of the pH, viscosity, adhesiveness, and spreadability tests of spearmint oil nanogels were analyzed statistically using ANOVA [12].

3. RESULT

Table 3. Organoleptic Results of Spearmint Oil Nano-Emulsion

Test	Organoleptic
Color	Clear yellow
Texture	A little thick
Smell	Soft mint

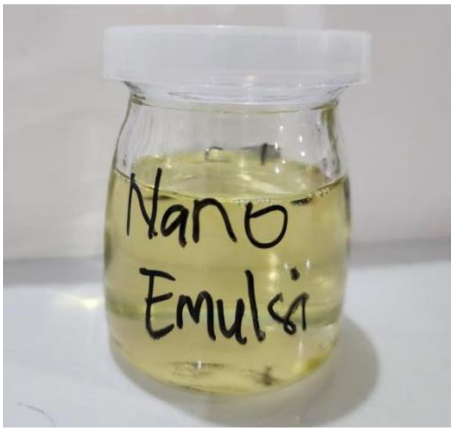

Figura 1. Spearmint oil nano-emulsion

Table 4. Physical Quality Results of Spearmint Oil Nano-Emulsion Gel

Formula	Organoleptic test		
	Color	**Texture**	**Smell**
I	Almost clear slightly pale yellow	The gel is quite runny	Soft Mint
II	Almost clear, slightly yellow	The gel is a little runny	Soft Mint
III	Almost clear more yellow	Gel is denser	Soft Mint

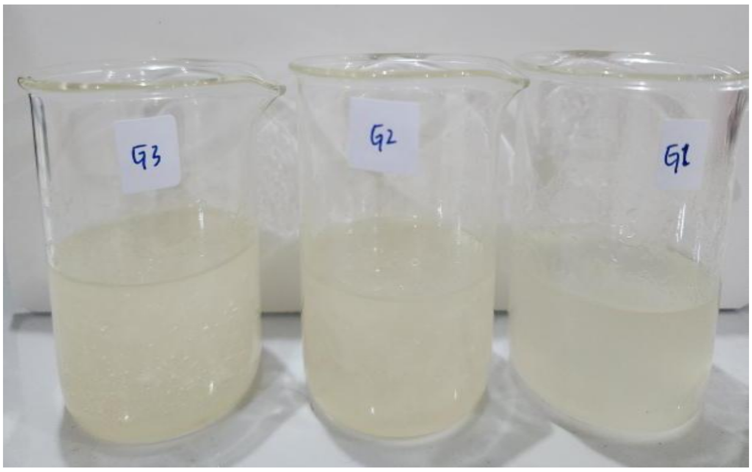

Figura 2. Spearmint oil nano emulsion gel

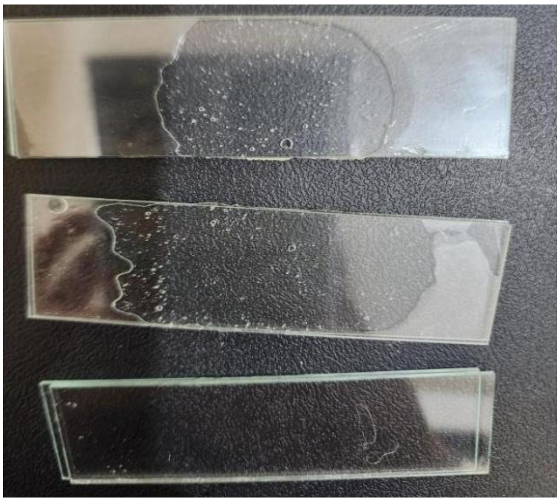

Figura 3. Homogeneity test

Table 5. Transmittance percentage test results

Replikasi	Percentage Transmittance Results (%)
I	98,3
II	98
III	98,4

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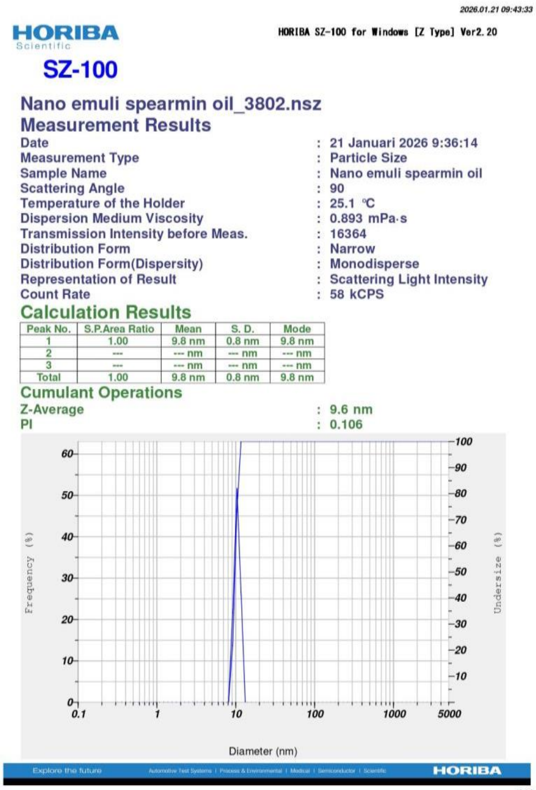

Figura 4. Results of spearmint oil nano-emulsion particle size

Table 5. Spreadability test (Coverage in cm)

Formula	Spreadability Test (Coverage in cm)		
	Replikasi I	Replikasi II	Replikasi III
Formula I	2	2,3	2,2
Formula II	1,4	1,5	1,5
Formula III	0,5	0,8	0,7

Table 6. Spreadability test (50g in cm)

Formula	Spreadability Test (50g in cm)		
	Replikasi I	Replikasi II	Replikasi III
Formula I	4	4,1	4
Formula II	3,5	3,3	3
Formula III	2,8	2,7	2,9

Table 7. Spreadability test (100g in cm)

Formula	Spreadability Test (100g in cm)		
	Replikasi I	Replikasi II	Replikasi III
Formula I	6,9	6,5	6,7
Formula II	6,1	6	6
Formula III	5,2	5,4	5,2

Table 8. Adhesion Test

Formula	Adhesion Test (Seconds)		
	Replikasi I	Replikasi II	Replikasi III
Formula I	0,63	0,69	0,62
Formula II	0,99	0,97	1,01
Formula III	1,15	1,09	1,12

Table 9. pH test

Formula	pH test		
	Replikasi I	Replikasi II	Replikasi III
Formula I	5,84	5,80	5,88
Formula II	6,13	6,17	6,10
Formula III	6,42	6,39	6,45

Table 10. Viscosity test

Formula	Viscosity test (dPas)		
	Replikasi I	Replikasi II	Replikasi III
Formula I	200	200	200
Formula II	280	280	280
Formula III	340	340	340

4. DISCUSSION

Organoleptic testing was conducted to evaluate the formula using the five human senses, including smell, color, and texture [6]. The results of the organoleptic test on the nano-emulsion showed a soft minty odor, a slightly thick texture, and a clear yellow color. The organoleptic test on the nano-emulsion gel formula, with increasing HPMC concentration, resulted in a whiter color and a denser texture, but the odor remained the same, namely soft mint [3]. The homogeneity test for the three homogeneous formulas is indicated by the absence of grains under the deckglass used for the homogeneity test.

The transmittance percentage test on the nano-emulsion was obtained three times, all of which approached 100%, which can be interpreted as indicating that the active ingredient is encapsulated in the surfactant and cosurfactant [11]. The particle size test (Particle Size Analysis) obtained an average particle size of 9,6 nm, which means that below 100 nm is considered to have formed a nano-sized [5].

The spreadability test on a 100 gram load stated that a good gel has a spreadability test result between 5 to 7 cm which means the formula already has a good spreadability test result with a result between 5,2 to 6,9 cm [13]. The good gel adhesion test for topical preparations is more than 1 second in formula III. The adhesion test is too short, namely less than 1 second, then it can be stated that the absorption of the active substance is too short, it will reduce the treatment effect [14]. Both of these tests are influenced by the concentration of HPMC, where the higher the concentration of HPMC, the smaller the spreadability and the higher the adhesion [10]. The concentration of HPMC (Hydroxypropyl Methylcellulose) in the gel preparation has a relatively small to almost no significant effect on the pH of the gel preparation itself, as long as the HPMC used is of pharmaceutical quality and dissolved in a neutral solvent (such as distilled water).

HPMC influences physical quality tests, especially viscosity, adhesion, and spreadability. As the HPMC concentration increases, more polymer chains are hydrated and cross-linked, forming a denser three-dimensional gel matrix that resists the movement of water molecules. Spreadability is inversely proportional to viscosity. Gels with higher HPMC concentrations have a rigid matrix structure and greater internal resistance. Therefore, the higher the HPMC concentration, the lower the spreadability. Consequently, the gel becomes more difficult to flow and requires greater pressure to spread on the skin surface. HPMC's adhesive properties are good mucoadhesive/bioadhesive due to the presence of hydroxyl groups (-OH) in its cellulose structure. These groups are able to form hydrogen bonds with glycoproteins in skin tissue or mucous membranes, thus increasing the adhesiveness [9].

Data from the spreading power and adhesion tests obtained were analyzed statistically using SPSS. The data results were concluded to be normal and homogeneous because sig>0,05 with a sig value of 0,996 in the spreading power test and a sig value of 0,458 in the adhesion test. Then, the One Way ANOVA test showed a sig value <0,05 with a sig value of 0,005 in the spreading power test and a sig value of 0,001 in the adhesion test (Rahayu, Rahmah and Yanti, 2025), so it can be concluded that there are significant differences in each formula in the spreading power test and the higher the adhesion due to variations in the gelling agent [11].

The pH test of the three formulas is all good, namely between 4,5 to 6,5, where the pH of the Spearmint oil nano-emulsion gel formula is not too acidic and not too alkaline when applied to the skin so it will not irritate the skin [14]. The viscosity test here HPMC greatly affects this viscosity test because the more HPMC concentration used, the greater the viscosity value will be, the good gel viscosity test results are 200 to 400 dPas [13], here the results obtained are quite good, namely 200 to 340 dPas.

Data from the pH and viscosity tests obtained were analyzed statistically using SPSS. The data results were concluded to be normal and homogeneous because sig>0,05 with a sig value of 0,916 for the pH test and a sig value of 0,177 for the viscosity test. Then, the One Way ANOVA test showed a sig<0,05 with a sig value for both tests of 0,000 [12], so it can be concluded that there are significant differences in each formula in the pH and viscosity tests due to variations in the gelling agent [11].

5. CONCLUSION

The transmittance percentage test yielded a result of 98,23%, indicating that the preparation is truly clear and it can be confirmed that the particle size is indeed very small. Supported by the particle size test, the average particle size obtained was 9,6 nm. Formula III produced good results from all physical quality tests tested, starting from the organoleptic test of almost clear color; typical texture of gel preparation; soft mint odor, homogeneity of formula III was declared homogeneous because there were no granules in the gel preparation, spreadability of 5,2 cm at a load of 100 grams, pH 6,42, and viscosity of 340 dPas, and adhesive power of 1,12 seconds; all tests provided results consistent with the literature for gel preparations.

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