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The Effect Of Hpmc Gelling Agent Concentration On The Physical Properties Of Anti-Acne Serum Formulation With Lemon Peel Extract

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ABSTRACT
Lemon peel contains compounds that can provide antibacterial effects, namely flavonoids and alkaloids, which can cause the emergence of acne This study aims to investigate how varying concentrations of HPMC gelling agent F1 (4%), F2(6%) and F3 (8%) affect the physical properties of anti-acne serum formulations containing lemon peel extract. The research method employed is descriptive, based on the evaluation of the physical properties of the serum formulations. Organoleptic observations of F1 and F2 show a yellowish-brown color, solid form, and characteristic smell of lemon extract, while F3 is brown, slightly thick, and has the characteristic smell of lemon peel extract. The pH values of F1, F2, and F3 are 5, indicating neutrality. The homogeneity test shows that all formulations are homogeneous. The adhesive strength of F1, F2, and F3 are 2.16, 2.65, and 3.35 seconds, respectively. The spreading power of F1, F2, and F3 are 7, 6, and 5. The specific gravity of F1, F2, and F3 are 1.508, 1.640, and 1.648 g/mL and the viscosity values are 385.95, 439.79, and 500.63 cps. The variation in HPMC concentration affects the results of the serum formulation tests, including organoleptic observations, spreading power, adhesive strength, specific gravity, and viscosity.

Key words: Lemon Peel, HPMC, Formulation Evaluation

INTRODUCTION
The skin is a layer that covers the entire surface of the body. On the surface of the skin, sweat glands excrete waste products released through skin pores in the form of sweat. Acne is a condition in which pores become blocked, leading to inflamed pus-filled pockets (Maharani, 2015). Acne vulgaris arises due to inflammation of the pilosebaceous follicles, characterized by the appearance of comedones, pustules, and nodules on the face, shoulders, chest, upper back, and upper arms (Adhi et al., 2018). The exact cause of acne is not fully understood, but various factors have been identified, including stress, hereditary factors, hormones, medications, and bacteria. Bacteria such as Propionibacterium acnes, Staphylococcus aureus, and Malassezia furfur play a role in the etiology of acne. One of the microorganisms that cause acne is Staphylococcus aureus, which is a normal flora on the skin, especially on the face, belonging to the group of

Corynebacterium bacteria and classified as Gram-positive bacteria (Putri et al., 2020).

Lemon (*Citrus limon* L.) is one type of plant that can have antibacterial properties. Lemon contains a variety of bioactive compounds, such as flavonoids, limonoids, phenolic acids, coumarins, furocoumarins, carotenoids, tannins, terpenoids, and essential oils. The essential oil content in lemon consists of limonene, which exhibits antibacterial activity (Russo *et al,* 2015; Yeni *et al,* 2015). Flavonoids inhibit energy metabolism by blocking the use of oxygen by bacteria, while alkaloids disrupt the components of bacterial peptidoglycan, causing the cell wall layers not to form completely, leading to cell death (Tuberta, 2019).

One of the evolving cosmetic formulations today is serum. Serum is a formulation with low viscosity, and due to its low viscosity, serum is categorized as an emulsion preparation. Serum has the advantage of containing a high concentration of active ingredients, allowing for faster absorption into the skin, providing a more comfortable effect, and easier spreadability on the skin surface due to its relatively low viscosity (Kurniawati, 2018). One of the advantages of using serum formulations is that they contain a higher concentration of active ingredients compared to other cosmetic formulations, making them faster and more effective in addressing skin concerns (Hasrawati et al., 2020).

One important factor in serum formulation is the gelling agent with its characteristics. Hydrophilic gel bases can enhance the activity of extracts in a formulation, such as methylcellulose, carboxymethylcellulose (CMC), and some derived from synthetic polymers like carbopol. The advantage of serum in anti-acne formulations is that it is a semi-solid formulation containing a high amount of water, allowing it to penetrate the cell walls of Gram-positive bacteria, which are more polar. The characteristics of the gelling agent used should be tailored to the type of formulation because the higher the concentration of the gelling agent used, the higher the viscosity of the gel due to the stronger gel structure (Ardana et al., 2015). HPMC (Hydroxypropyl Methylcellulose) is a semi-synthetic gelling agent derivative of cellulose that is resistant to phenol, stable at pH 3-11, can form clear gels, is neutral, and has stable viscosity during long-term storage (Rowe et al., 2009). Additionally, HPMC has good swelling properties in water, making it a good hydrogel-forming material (Yati et al., 2018)

The use of HPMC as a gelling agent in the formulation of anti-acne serum with lemon peel extract is expected to result in an anti-acne serum with good physical properties. Furthermore, the aim of this research is to investigate the effect of the gelling agent on the physical characteristics and anti-acne activity of the serum formulation.

MATERIAL AND METHODS

Material

Lemon peel extract, HPMC (Hydroxypropyl Methylcellulose), methylparaben, Na-EDTA (Sodium ethylenediaminetetraacetate), aquadest, 96% ethanol, glycerin, and NaCl (Sodium chloride).

Methods

Preparation of Lemon Peel Extract

The lemon is washed under running water until clean. Once cleaned, it is peeled to separate the peel from the flesh. The lemon peel is thinly sliced and dried for 4 days. After drying, the dried lemon peel is finely ground and sifted using a No. 100 mesh sieve. The powdered lemon peel is then extracted using the maceration method. 181 g of powdered lemon peel is soaked in 96% ethanol at a ratio of 1:7.5 for 3 days with occasional stirring. After 3 days, the soaked sample is filtered using filter paper to obtain filtrate 1 and residue 1. The residue obtained is then added with more 96% ethanol, covered with aluminum foil, and left to soak for another day with occasional stirring. After 1 day, the soaked residue is filtered again to obtain filtrate 2 and residue 2. The filtrate 1 and filtrate 2 are combined and evaporated using a rotary evaporator at 60°C for 50 minutes until a thick extract forms. Once done, the extract is left until all the ethanol solvent evaporates at room temperature, resulting in a thick extract. Based on the obtained results, the extract yield can be calculated, which is the ratio of the weight of the extract produced to the weight of the powdered lemon peel used.

Formulation

The formulation of ethanol extract of lemon peel into a serum preparation with antibacterial function involves three variations of HPMC concentration: 4, 6 and 8%, along with predetermined additional ingredients. The materials are weighed and a mucilage is prepared. A Na-EDTA solution is added along with glycerin and stirred until homogeneous. After achieving homogeneity, NaCl and methyl paraben are added to the mixture. Then, the ethanol extract of lemon peel is added to the serum mass and stirred until homogeneous. Finally, the serum is transferred into serum containers. The formulation of serum preparation can be seen in Table 1.

Table 1 Serum Formula

Material	Concentration (%)			Function
	F1	F2	F2	
Lemon peel extract	3	3	3	Active Ingredient
HPMC	4	6	8	Gelling Agent
Gliserin	1	1	1	Humectant
Na-EDTA	10	10	10	Complexing Agent

Metyl Paraben	1	1	1	Preservative
NaCl	2	2	2	Viscosity Regulator
Aquadest	Add 100	Add 100	Add 100	Solvent

Physical evaluation

Organoleptic Test

The organoleptic test is conducted visually, assessing the appearance, color, and odor of the prepared product.

Homogeneity Test

The homogeneity test involves applying the substance to be tested onto a suitable surface, such as a glass slide, and observing for a uniform distribution without coarse particles.

pH Test

0.5 g of serum is diluted with 5 mL of distilled water, and then a pH strip is immersed for 1 minute. The color change on the pH strip indicates the pH value of the product. The measurement should target the pH range suitable for skin, which is 4.5-6.5.

Spreadability Test

The spreadability test evaluates the ability of the serum to spread rapidly when applied to the skin. Greater spreadability ensures broader contact between the active ingredients and the skin. A serum exhibits good spreadability if it meets the criteria of skin spreadability, typically within a range of 5-7 cm.

Adhesion Test

The adhesion test assesses whether the product adheres or sticks to the skin surface, which is crucial for the effectiveness of topical applications. The longer a product adheres to the skin, the greater its pharmacological effect. Ideally, the adhesion time for topical formulations should be less than 4 seconds.

Viscosity Test

The viscosity test determines the thickness of the serum, which influences its consistency and spreadability. The principle involves measuring the time required for a fluid to pass through two designated points in a vertical capillary tube. The Ostwald viscometer operates based on the concept of fluid flow velocity in a tube. Lower flow velocities indicate higher viscosity. This test aims to assess the thickness of the serum and its impact on consistency and spreadability. Higher viscosity values indicate thicker consistency and reduced spreadability.

RESULT AND DISCUSSION

The preparation of lemon peel simplisia begins with wet sorting to separate dirt or other particles. Then, the lemon fruit is peeled to obtain its peel. The lemon peel is cut into small pieces and dried by airing while covered with black cloth for approximately 4 days. In this study, the dried simplisia weighs 258 g, allowing the calculation of the moisture content of the simplisia, which is approximately 0.48%. The dried lemon peel, weighing 181 g, is then macerated using 96% ethanol. Maceration is conducted for 3 days with stirring once a day. The filtrates are combined, then concentrated in a Rotary Evaporator and obtain a thick extract weighs 22.82 g.

HPMC, or Hydroxypropyl methylcellulose, is a gelling agent commonly used in cosmetic products and is a hydrogel-forming substance that can float in water (Maryani & Eka, 2023). Gelling agents are among the important basic components in serum production, where they aggregate with molecules and coils, resulting in viscous properties that affect the physical characteristics of the serum. The serum formulation of lemon peel (*Citrus limon* L.) is made with three different concentrations of HPMC: 4, 6 and 8% (Table 1). Subsequently, the serum formulation undergoes evaluation, including organoleptic assessment, pH testing, homogeneity assessment, spreadability test, adhesion test, and viscosity test.

Table 2. The Result of Organoleptic, Homogeneity, Spreadability, Adhesion and Viscosity Test

	Organoleptic	Homogeneity	pH	Spreadability (cm)	Adhesion (seconds)	Viscosity (cps)
F1	Brownish-yellow, lemon scent, liquid	Homogeneous	5	7	2.16	385.95
F2	Brownish-yellow, lemon scent, liquid	Homogeneous	5	6	2.65	439.79
F3	Dark brown lemon scent, thick	Homogeneous	5	5	3.35	500.63

Organoleptic testing aims to observe and assess the physical characteristics such as color, shape, and odor of the prepared formulation. The results of the organoleptic evaluation of lemon peel extract serum (*Citrus limon* L) shown in Table 2 indicate that formulations F1 and F2 have a liquid form, while F3 is slightly viscous. All three formulations have the characteristic scent of lemon peel. F1 and F2 are yellowish-brown in color, while formula 3 is dark brown. The greater the amount of HPMC gelling agent in each formula, the thicker the consistency of the gel mass, with slight

differences in color, although the intensity of the color differences in each formula may not be significant (Afianti et al., 2015).

The pH testing aims to determine the acidity level of the serum preparation. A good serum should have a pH that matches the pH of facial skin, which is 4.5 – 6.5 (Raharjeng et al., 2021). The pH of the serum corresponding to the skin's pH aims to provide safety and comfort during use. If the pH of the preparation does not match the skin's pH, it may cause irritation or discomfort during use (Aprilianti et al., 2020). The pH testing results indicate that all three serum formulas have a pH within the range of facial skin pH: F1; F2; and F3 show a pH of 5. The pH testing data of the serum preparation from lemon peel extract indicates that there is no influence of HPMC concentration variation on the pH change of the serum.

Homogeneity testing is conducted to determine whether a formulation is homogeneous or not. In this case, a material is considered homogeneous if there is no difference in characteristic values between one part and another (Warnida *et al*., 2016). The results of homogeneity testing for all three formulations indicate homogeneous results. In Table 2, the homogeneity testing results indicate that there is no influence of HPMC concentration variation on the homogeneity of the gel.

The spreading power test aims to determine the ability of serum to spread quickly when applied to the skin. Table 2 shows the range of spreading considered good according to the spreading power test criteria, which is in the range of 5-7 cm (Pohan et al., 2019). For F1, the spreading power is 7 cm, for F2 it is 6 cm, and for F3 it is 5 cm. The results indicate that the higher the concentration of HPMC, the smaller the spread area. This decrease in spreading ability corresponds to an increase in gel viscosity. If the pressure applied is the same for each gel formula tested, the thicker the preparation, the smaller its spreading capability. Afianti et al., 2015, stated that lower concentrations of HPMC result in a more diluted form, thus gels with lower concentrations have a wider spread range.

The adhesive power test aims to determine whether the formulation can adhere or stick to the skin surface, which is crucial for the application site. Adhesive power can affect the effectiveness of active ingredients in a serum formulation, where the longer the adhesive power, the longer the intended activity will last. The adhesive power of gel formulations should ideally be more than 1 second (Yusuf et al., 2017). The results of the adhesive power test in Table 2 show that all concentration formulations meet the criteria for F1, F2, and F3. The results of the gel adhesion test in Table 2 indicate that the higher the concentration of HPMC used in each formula, the longer the gel adheres. This is because HPMC is capable of forming colloids with the addition of hot water (Rowe et al., 2009). Colloids are formed because the dispersed substance absorbs the dispersing medium, making it viscous and sticky. Therefore, the higher the concentration of HPMC, the more colloids are formed, increasing its adhesion.

The specific gravity test aims to provide an overview of the chemical content of the preparation based on the obtained specific gravity. In the three

formulations with varying concentrations of HPMC, they meet the standard specific gravity test of at least 1.020 g/mL. Among the three formulations, the best result is from F3 (8%), which is 1.648. The viscosity test aims to determine the thickness and spreading ability. The higher the viscosity value, the higher the thickness of the preparation. The viscosity test results can be seen in Table 2, where F1, F2, and F3 have viscosities of 385.43, 439.79, and 500.63 cps, respectively. The viscosity test results indicate an increase in consistency and viscosity with the addition of HPMC in the lemon peel extract serum. This is because HPMC is a derivative of cellulose. In cellulose derivative polymer dispersion, primary molecules enter the cavities formed by water molecules, resulting in hydrogen bonding between the hydroxyl groups (-OH) of the polymer and water molecules. These hydrogen bonds play a role in hydration during the polymer development process, so an increase in HPMC concentration leads to more hydroxyl groups and higher viscosity (Kibbe, 2004). The serum can be considered good if it meets the requirement, which is within the range of 230-1150 cps. The higher the viscosity of the preparation, the lower the effectiveness of the serum. This occurs because high viscosity of the serum can reduce the mobility of active ingredients towards the skin surface, causing a decrease in penetration speed into the skin (Dewi et al., 2019).

CONCLUSION

The different of concentrations of HPMC did not affect the results of the physical property tests for pH and homogeneity. However, in organoleptic evaluation, spreadability, adhesiveness, and viscosity tests, the concentration of HPMC as a gelling agent had an influence. Based on the evaluation of the physical properties of the serum preparations, the best formula is F3 with an HPMC concentration of 8%.

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