Trad. Med. J., September-December 2022
Vol. 27(3), p 247-256
ISSN-p : 1410-5918 ISSN-e : 2406-9086

Submitted : 17-12-2022
Revised : 20-12-2022
Accepted : 22-12-2022

Optimization of Gelling Agent of Sunflower (*Helianthus annuus*) Seed Oil Gel and Its Stability and Activity Test *In Vitro* as Sunscreen

A. Karim Zulkarnain*, **Feela Faridhotu, Ifthary Naqsya PR**
Faculty of Pharmacy, Universitas Gadjah Mada

ABSTRACT

Excessive ultraviolet (UV) radiation from the sun can harm human skin, such as skin erythema or sunburn, premature aging of the skin, darker skin discoloration, and damage to skin cells. Skin protection, like sunscreen, is needed to prevent the negative effects of UV rays. This study aims to evaluate the optimum formula, the physical properties, and the activity of a sunscreen gel preparation of sunflower seed oil in vitro. The method in this study was optimization with Design Expert version 10 of the carbopol gelling agent and sodium carboxymethyl cellulose. The optimum formula obtained was tested for its physical properties and activity as a sunscreen in vitro with a spectrophotometer. The results showed that the physical properties of the optimum formula were good, and stable during storage, pH, spreadability, adhesion, and viscosity were close to predictions, and the in vitro SPF test obtained a value of 15.60.

Keywords: Optimization; gel; SPF; sunflower

INTRODUCTION

Sunlight is a source of energy that is beneficial to human life. However, ultraviolet radiation from the sun needs to be cautious because it may have harmful effects on the skin. Ultraviolet light (UV) is an electromagnetic wave that has a wavelength between 100 – 400 nm. Based on its biological activity, UV light can be divided into UVC (190-290 nm), UVB (290-320 m), and UVA (320-400 nm). Most UVB radiation and almost all UVA radiation can reach the earth (Elsner *et al*., 2016). UVA (ultraviolet A) and UVB (ultraviolet B) cause skin melanoma, burning, photo aging, skin pigmentation, and various other painful effects. (Kulkarni, 2014).

The intensity of excessive exposure to UV rays can be reduced by using a topical sunscreen. Sunscreens can be in the form of gels, sprays, or other topical products. It helps protect the skin from UV radiation from the sun, reduces sunburns, and other skin damage. Based on its protective mechanism, sunscreen can be divided into chemical, by absorbing UV radiation, and physical, by reflecting/reflecting UV light (Gupta, 2010).

Sunflower plants (*Helianthus annuus* L.) of the Asteraceae family are plants that have therapeutic activity in various treatments, including wound healing, antioxidant, anticancer, antidiarrheal, antihistamine, anti-inflammatory, and as analgesic (Juniarti, et al., 2017). The sunflower seed oil has antioxidant activity due to its relatively high vitamin E content of 58.7 and β-carotene of 1.87. This indicates that sunflower seed oil has the potential to be formulated into sunscreen preparations.

A gel, sometimes called a Jelly, is a semisolid system consisting of a suspension made up of small inorganic particles or large organic molecules, penetrated by a liquid (Anonymous, 2014). The choice of gel preparation formulations in this study was because gel preparations have several advantages, including their ability to spread well on the skin, cause a cooling effect, not inhibit hair function physiologically, are easy to wash, and have good drug release (Voight, 1994).

Gelling agents can affect the physical properties and stability of gel preparations. Carboxymethylcellulose sodium is used because it is a stable material and can be used as a gel base (Rowe et al., 2009). The base is a cellulose derivative. This approach was used because cellulose derivatives are considered stable and have pseudoplastic properties where the gel will thicken when left standing but is easily applied to the skin surface.

The method used in this study was the simplex lattice design method with the ultimate goal to obtain an optimal formula that fulfills the good physical properties of the gel. The optimal sunscreen gel formula was then determined by using a UV-Vis spectrophotometer to determine the percentage of erythema transmission, and the percent transmission of pigmentation.

This research was conducted to determine the right formula to create sunflower (*Heliantus annuus*) seed oil with CMC-Na and carbopol as gelling agents. This research was also aimed to

***Corresponding author : A. Karim Zulkarnain**
Email : akarimzk@ugm.ac.id

determine the physical properties and SPF value of the gel formula that had been made.

Sunlight is important for humans, for example in the formation of vitamin D from pro-vitamin D which prevents polio or rickets. (Shovyana *et al*., 2013). Exposure to UV-A rays will stimulate the formation of melanin, which functions as a protective layer on the skin. UV-B radiation can penetrate the stratum corneum well, causing severe epidermal burning (erythema), especially in fair-skinned individuals.

Radiation with a wavelength longer than 350 nm begins to penetrate the dermis thereby stimulating the formation of melanin and producing a tan of the skin as a result of direct sunburn. Even though UVA rays have lower energy than UVB rays, in reality, UVA can penetrate further into the hypodermis, which can cause elastosis and other skin damage that has the potential to cause skin cancer (Shaath, Nadim, 2005).

Sunscreen is a cosmetic preparation that is used to actively reflect or absorb sunlight, especially in the emission areas of ultraviolet and infrared waves, to prevent skin disorders due to UV rays (Draelos and Thaman, 2006). Based on the mechanism, active sunscreen ingredients are divided into two, namely physical blocking mechanisms (reflecting solar radiation) and chemical absorbing mechanisms (absorbing solar radiation). Physical sunscreens work by reflecting ultraviolet radiation. Their ability is based on particle size and layer thickness. They can penetrate the dermis to the subcutaneous or hypodermis layers and are effective in the spectrum of UV-A, UV-B, and visible light. Meanwhile, the mechanism of chemical sunscreens is to absorb ultraviolet radiation and convert it into a form of heat energy. It can absorb nearly 95% of UV-B radiation which can cause sunburn (erythema & wrinkles) (Lavi, 2012).

According to the FDA (Food Drug Administration), the division of sunscreen ability levels (Damogalad, 2013) is as follows: At a minimum, if the SPF is between 2-4; Medium, if the SPF is between 4-6; Extra, if the SPF is between 6-8; Maximum, when the SPF is between 8-15 and Ultra when the SPF is more than 15

Gels were made by selecting a base that could affect the character of the gel formed. Gels require a mixture of 2 or more gelling agents (base) to obtain a gel with certain characteristics according to its intended use (Sulaiman and Tambunan, 2018). Sunflower (*Helianthus annuus*) is a popular annual plant of the Asteraceae tribe, both as an ornamental plant and as an oil-producing plant. The flowers of this plant are very distinctive: large, usually bright yellow, with large flower heads (up to 30 cm in diameter). These flowers are compound flowers, composed of hundreds to thousands of small flowers on one head. Sunflowers also have distinctive behavior, like always facing the sun or heliotropism (Neti S, 2013).

This sunflower plant has a therapeutic activity in various treatments, including: as a wound healer, antioxidant, anticancer, antidiarrheal, antihistamine, anti-inflammatory, and as analgesic (Juniarti, *et al*., 2017). Based on previous research, it is known that sunflower seed oil contains active substances such as omega 9, omega 6, Vitamin E, tocopherols, and carotenoids. The content of vitamin E and β-carotene activity as an antioxidant (Susanti *et al*., 2019).

METHODOLOGY

Instruments and Materials

The tools used in this study included: a water bath, and an electric stove (Robusta). Mixer (Cucina Philips® and Power Supply IC Regulated model ad 01), analytical balance (Precise 2000C – 2000D1), Viscometer series VT 04 (Rion Co, LTD, Japan). oven (Shimadzu), excitator, glassware, porcelain cup, stamper mortar, spreadability test equipment (Pharmaceutical Technology Lab, Faculty of Pharmacy, UGM), adhesion test kit (Pharmaceutical Technology Lab, Faculty of Pharmacy, UGM), stopwatch (Alba Digital Stopwatch), micropipette, UV 254, UV 366, dispersing tool (ULTRA-TURRAX T25® Janke & Kunkel Ika®-Labortechnic), pH-meter.

The main ingredients used in this study were sunflower seed oil, CMC-Na, Carbopol, propylene glycol, Polysorbate 80, triethanolamine, sunflower seed oil hydantoin, stearic acid, glycerin (all pharmaceutical grades) span 80, cetyl alcohol, glyceryl monostearate, stearyl alcohol, mineral oil, methylparaben, propylparaben, citric acid, distilled water, all pharmaceutical grades, ethanol, ethyl acetate, chloroform, methanol (all analytical grades).

Means of Research

Gel preparation formulation

Water phase preparation

The aquadest is slowly put into the porcelain cup. The other water phase materials were then added, stirred, and heated at 70^{o}C until completely mixed.

Oil phase preparation

All the oil phase ingredients were mixed, stirred, and heated at 70^{o}C until homogeneous.

Table I. Formula for optimization of carbopol-CMCNa gels

Materials (%b/v)	A	B	C	D	E	F	G	H
Sunflower seed oil	10	10	10	10	10	10	10	10
carbopol	1.25	2	1.5	1	1.5	1.75	1	2
Na CMC	2,75	2	2.5	3	2.5	2.25	3	2
Stearic Acid	5	5	5	5	5	5	5	5
Trietanolamin	2	2	2	2	2	2	2	2
Propylene glycol	15	15	15	15	15	15	15	15
Gliserin	2	2	2	2	2	2	2	2
DMDM hydantoin	1	1	1	1	1	1	1	1
Aqua ad (ml)	100	100	100	100	100	100	100	100

Notes: Formulas E, G, and H were used to validate the SLD equation

Mixing

Mixed into the water phase mixture, the oil phase mixture was added while stirring at 70°C. When the mixture was homogeneous, it was cooled to 45°C, added with sufficient water, and homogenized for 5 minutes. The resulting formulation was then put into a tightly closed container for 24 hours.

The gel formulation results were observed for 4 weeks of storage including

Viscosity test

The preparation was put into a tubular container, then a number 1 or 2 rotor was installed and it was ensured that the rotor was immersed in the test preparation. The viscometer was turned on and it was confirmed that the rotor could rotate. The pointer of the viscometer was observed when it pointed to the viscosity scale of the available rotor number 1 or number 2. When the needle pointed to a stable number, then that number was the viscosity and was recorded in units of dPa.S., that is (1 dPa.s = 1 Poise). The viscosity test for the gel was carried out every week for one month (Marchaban *et al*., 2016).

Spreadability test

Preparations weighing 0.5 grams were placed in the middle of a round glass scale. Another round glass was placed on top of the material, then it was let stand for 1 minute and the spread was recorded. Each stage was added with a weight of 50 grams and allowed to stand for 1 minute and then the distribution was recorded. Ballast was added up to 300 grams. The spread was recorded through 4 sides. The spreadability test was carried out every week for one month (Marchaban *et al*., 2016)

Stickiness test

A total of 0.1 grams of the preparation was smeared over a glass object 2 x 2 cm wide. On top of the preparation, another glass object was placed and pressed with a load of 1 kg for 5 minutes. The glass object was mounted on the test equipment, the weight weighing 80 grams was released and the time was recorded until the two glass objects were released. The adhesion test was carried out every week for one month (Marchaban *et al*., 2016).

Test the pH value of the preparation

As much as 20 grams of the preparation was put in a beaker glass, then the tip of the pH meter that had been calibrated in pH 4 and pH 7 was put in the preparation. The number on the pH meter was recorded in a stable position and that number was the pH value of the preparation.

Test of the separation volume ratio

The preparation was put into a test tube with a scale up to a certain scale. Scaled test tubes containing bases were stored at room temperature. Separation volume

The basis at room temperature was recorded every other day until day 28. If no separation occurred at room temperature, an accelerated emulsion stability test was performed. The separation volume can be calculated using the formula in the equation below.

$$F = \frac{Ht}{Ho} = \frac{Vt}{Vo} = \frac{L \times Ht}{L \times Ho} \quad \text{.....................................(2)}$$

Notes : F : Separation volume ratio; Ht : Emulsion height at time t; Ho : Initial emulsion height; Vt : Emulsion volume at time t; Vo : Initial emultion volume; L : The cross-sectional area of the container. (Mollet & Grubenmann, 2001).

Stability test with cycling test method

The preparations were put in the refrigerator at 8°C on the first day, on the second day the preparations were put in the oven at 45°C, and alternately placing the preparations while observing whether there was a phase separation on the surface of the preparations. The giving of stress conditions was at 45°C and 8°C alternately (1 cycle = 24 hours at 45°C and 24 hours at 8°C). After every 3 times, the viscosity was tested. The viscosity testing was carried out for up to 4 weeks.

Data Analysis and Processing

The data obtained from the results of the research on viscosity, spreadability, and adhesion, the separation volume ratio were analyzed using SPSS 25 statistical data analysis. Data were analyzed with a 95% confidence level preceded by the Kolmogrov-Smirnov suitability test to determine whether the data were normally distributed or not.

Normally distributed data were tested with one-way ANOVA and continued with the Tukey test. Data that are not normally distributed were tested by non-parametric analysis. The research results were also evaluated organoleptically and the activity test was analyzed descriptively.

The analysis of the data obtained from testing the various parameters was carried out by comparing the data obtained with the requirements in the literature and a linear regression test was carried out with a 95% confidence level.

RESULTS AND DISCUSSION

Sunflower seed oil sunscreen activity

The sunflower seed oil has an SPF value of 37.5; % a TE value of -0.11%; and the %TP value is -0.21%.

Determination of Optimum Gel Base Formula

The parameters used to determine the optimum formula in this study were pH, viscosity, and spreadability. The three parameters observed had a significant model and the results of the lack of fit test were not significant so they could be used as parameters for determining the optimum formula. The goal for pH and viscosity parameters was in range. The goal for the spreading power parameter was to maximize. Software Design Expert® version 10.0 provides a formula solution that matches the desired optimization target, namely a solution with a desirability value of 0.996. The optimum formula chosen was a formula with a carbopol composition of 1.0%; sodium carboxy methylcellulose of 3.0%. The optimum formula was predicted to have a pH of 5.40; viscosity of 90.350 dPa.s; and spreadability of 32.670 cm2.

The component that had the smallest positive effect on the pH response was carbopol. This could be because carbopol has the lowest pH when compared to CMC-Na dispersions (Rowe et al., 2009).

The component that had the greatest positive effect on the viscosity response was carbopol. It is because carbopol gel has a structure that is more difficult to flow than CMC-Na even at lower levels than CMC-Na (Fujiastuti and Sugihartini, 2015).

The component that had the greatest positive effect on the scattering response was the interaction between carbopol and CMC-Na. Carbopol would form a gel structure that was difficult to flow at a relatively neutral pH and CMC-Na would form a gel structure that was difficult to flow at a relatively neutral to alkaline pH. Carbopol and CMC-Na have a relatively acidic pKa, so the addition of these two materials will result in the formation of a gel structure of carbopol and sodium carboxypropylene glycol that flows easily. The addition of propylene glycol will certainly increase the flowability of the resulting gel preparation (Rowe *et al.*, 2009). The component that had the greatest negative effect on the scattering response was the interaction between carbopol. Carbopol will form a structure that is rigid and difficult to flow at a relatively neutral pH (Riddick *et al.*, 1985; Rowe *et al.*, 2009).

Optimum Formula Gel Base Verification

The optimum formula was verified by comparing the physical properties of the prepared gel preparation with the predicted physical properties of the gel using the one-sample t-test (if the data was normally distributed and homogeneous) or the Wilcoxon test (if the data was not normally distributed and homogeneous) with a 95% confidence level. Physical properties data were processed using IBM SPSS Statistics 25 software. The results of the statistical analysis stated that the values of pH, viscosity, and gel spreadability of the experimental results were not significantly different from the predicted values.

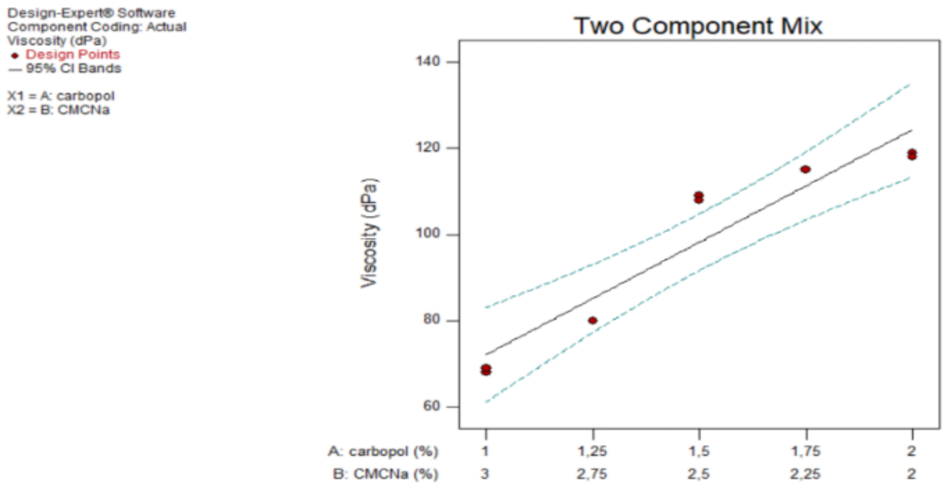

Figure 1. Profile Simplex Lattice Design viscosity of a mixture of Carbopol *and CMC Na gel preparations*

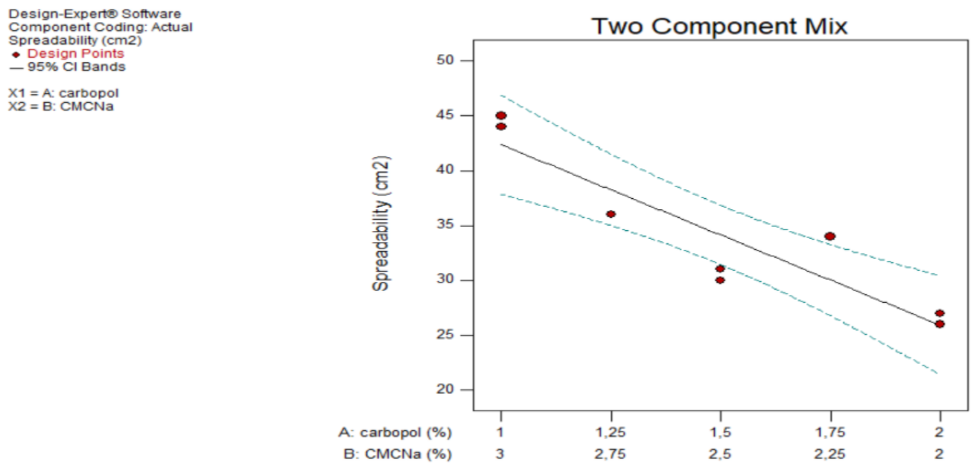

Figure 2. Simplex Lattice Design profile Spreadability of Carbopol *and CMCNa mixture of gel preparations*

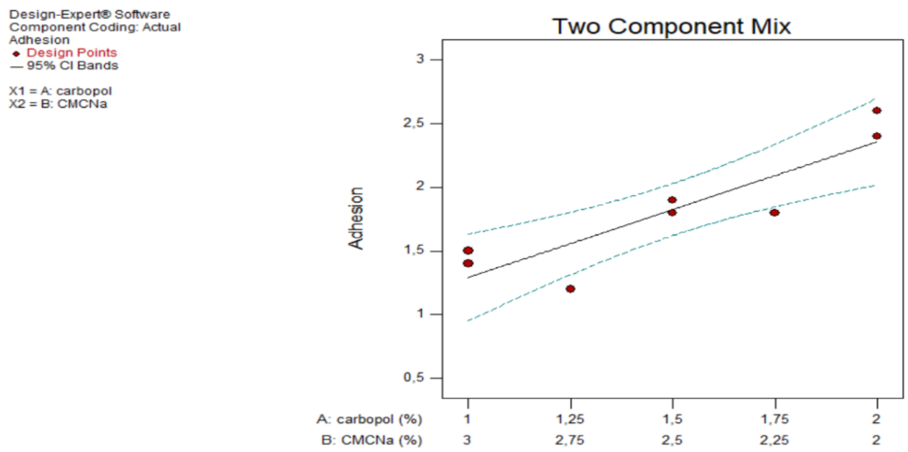

Figure 3. Profile Simplex Lattice Design adhesiveness of Carbopol mixture *and CMCNa mixture of gel preparations*

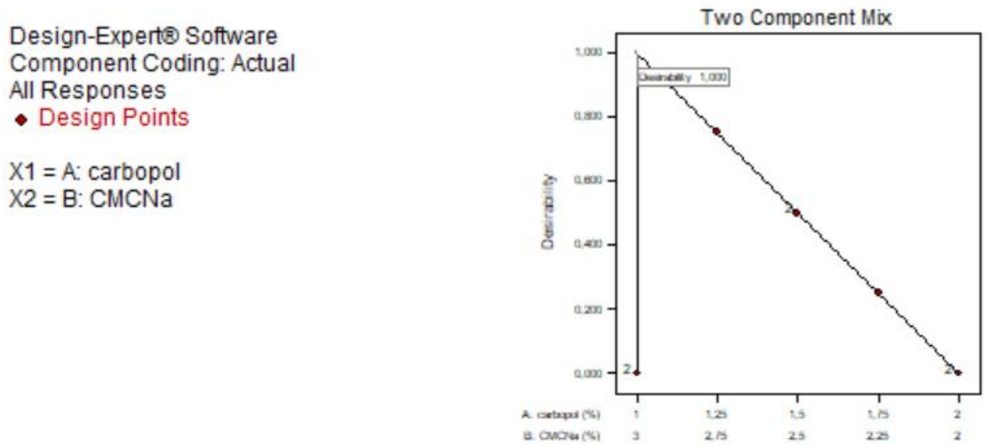

Figure 4. Simplex Lattice Design desirability profile of a mixture of carbopol and CMC Na gel preparations

Response plot of the physical properties of the optimum formula suggested from the combination of ingredients carbopol content was of 1.0% w/w; CMC-Na with a concentration of 3.0% w/w

Table II. Results of verification of the optimum physical properties of the formula

Response	Prediction	Test	*Sig. (2-tailed)*	Notes
pH	5.40	5.50	0.073	Not significantly different
Viscosity (dPa.s)	90.35	91.26	0.067	Not significantly different
Spreadability (cm^2)	32.67	33.14	0.056	Not significantly different

Table III. Comparison of Physical Properties of Optimum Formula Gel Base and Sunflower Seed Oil Gel

Formula	pH	Viscosity (dPa.s)	Spreadability (cm^2)	Adhesion (second)
Gel basis	5,36 ± 0,001	92,15 ± 0,33	30,44 ± 23	1,97 ± 1.14
Sunflower seed oil gel	5,50 ± 0.008	91,26 ± 0.69	33,14 ± 0.25	4,88 ± 0.21

Comparison of Physical Properties of Optimum Formula Gel Base and Sunflower Seed Oil Gel

Responses to the physical properties of the optimum formula gel base and sunflower seed oil independent samples (if the data were normally distributed and homogeneous) or the Mann-Whitney test (if the data were not normally distributed). The physical properties compared were pH, viscosity, spreadability, and adhesion.

The results of statistical analysis showed that there was no significant difference between the pH and viscosity of the two gels. Sunflower seed oil added to the optimum formula gel base did not significantly affect the pH of the gel base. The results of the statistical analysis did not show that there was no significant difference between the spreadability and adhesion of the two gels. The optimum formula gel base and sunflower seed oil gel have the following pH, viscosity, and spreadability values (Table III).

Physical stability test and syneresis test

The results of organoleptic observations showed that there was no change in color, odor, and homogeneity of the optimum formula base gel and sunflower seed oil gel. The two gels were stable for four weeks of storage.

The results of statistical analysis showed that the pH of the two gels did not change significantly in the first week of storage. The pH of the two gels indicated that they were stable for four weeks of storage at room temperature. The resulting gel structure was stable under storage conditions.

Table IV. Sunscreen activity test results

Ingredients/Formula	SPF Value	%TE Value	%TP Value
Sunflower seed oil	37,50 ± 0.14	-0,11 ± 0,02	-0,21 ± 0,03
Optimum formula gel base	0,06 ± 0.02	97,17 ± 0,62	95,65 ± 0,24
Sunflower seed oil gel	15,60 ± 0.04	21,23 ± 0,07	0,38 ± 0,05

Table V. The results of the t-test of one sample response with results gel preparation test

Response	Prediction	Test	Significance	Description
Viscosity	90.35	91,26 ± 6.18	0.279	Not significantly different
Adhesion	4.87	4,88 ± 1.26	0.572	Not significantly different
Spreadability	32.67	33,88 ± 1.11	0.363	Not significantly different

The results of statistical analysis of the viscosity showed that the viscosity of the optimum gel base formula experienced no significant change from the first week to the fourth week of storage, sunflower seed oil gel did not change significantly during the three storages. The viscosity of the two gels indicated that they were stable for four weeks of storage at room temperature.

The results of statistical analysis showed that the spreadability of the optimum formula gel base did not change significantly during four weeks of storage at room temperature and the spreadability of sunflower seed oil gel did not change significantly during storage. The optimum spreadability of the gel base formula indicated that it was stable for four weeks of storage at room temperature and the spreadability of sunflower seed oil gel was stable for four weeks of storage at room temperature. Sunflower seed oil was able to maintain the stability of the resulting gel structure,

The results of statistical analysis showed that the adhesion of the two gels did not change significantly after being stored for four weeks at room temperature. The adhesion of the two gels indicated that they were stable for four weeks of storage at room temperature.

The syneresis test results showed that the optimum base gel formula and sunflower seed oil gel gave a syneresis percentage of 0%. Both gels were stable during storage at 10°C because no syneresis events were observed.

Accelerated stability test

The results of the accelerated stability test using the cycling test method and the accelerated stability test using the centrifugation treatment showed that the optimum formula gel base and sunflower seed oil gel gave an F value of 1.

The two gels indicated that they were stable under extreme conditions in the form of changes in storage temperature and styling.

Evaluation of the optimum formula gel base sunscreen activity and sunflower seed oil gel

The optimum SPF values of the gel base formula with a concentration of 0.5% w/v in a solvent and sunflower seed oil gel with a concentration of 10% w/v in a solvent were 0.06 and 15.60, respectively. The optimum formula gel base could not provide protection, while sunflower seed oil gel could provide protection.

The optimum %TE and %TP gel base formula values with a concentration of 0.5% w/v in a solvent were 97.17% and 95.65%, respectively. The %TE and %TP values of sunflower seed oil gel with a concentration of 10% w/v in a solvent were 21.23% and 0.38%, respectively. The optimum formula gel base with a concentration of 0.5% w/v in solvent did not provide a protective effect against UV rays. Sunflower seed oil gel with a concentration of 10% w/v in solvent was categorized as a sunscreen, based on the %TE value produced, and categorized as a sunscreen based on the %TP value produced.

Optimization of Gel Preparations

Formula determination used Design Expert software version 10. The response of adhesion, spreadability and viscosity were used as the basis for determining the optimum formula.

The data were analyzed resulting in the response of viscosity, adhesion, and spreadability of the gel. Based on these predictions, one formula was produced with a desirability value of 0.99 with a concentration of 1.0% carbopol, and 3.0% NaCMC. The response prediction obtained from the

Table VI. Stability test results for the optimum physical properties of the formula

Week	Spreadability (cm2)	Adhesion (second)	Viscosity (dPas)
0	33,19 ± 1.01	4,81 ± 0.75	91,33 ± 2.37
1	33,15 ± 1.74	4,84 ± 0.67	91,29 ± 1.43
3	33,16 ± 1.80	4,86 ± 2.18	91,28 ± 3.54
4	33,14 ± 1.58	4,89 ± 1.33	91,26 ± 6.45

optimum formula was a viscosity of 90.35 dPas, an adhesive power of 4.87 seconds, and a spreading power of 32.67 cm2.

Verification of optimization results and statistical analysis

The experimental results of testing the physical properties of the optimum gel formula were compared with the predicted response obtained from the Design Expert® analysis. The one-sample test was used to test the difference in the average data with the hypothesis value. The results of the t-test for one sample of the response to the optimum formula for prediction compared to the optimum formula for the experiment are presented in Table V.

The experimental results of the optimum formula physical property test compared to the predicted response obtained insignificantly different values for the response of viscosity, adhesion, and spreadability with a significant value of > 0.05. The response of viscosity, adhesion, and spreadability of the experimental results was in accordance with the predicted response obtained with Design Expert® version 10.

The optimum formula was then tested for its physical stability. Physical stability tests included organoleptic, spreadability, adhesion, and viscosity. The stability observation was carried out for 4 weeks.

Optimum formula physical stability test

Viscosity test

The optimum formula viscosity test during storage from week 0 to week 4 showed a stable value. The results of the one-way ANOVA test were to see the stability of the optimum formula from its viscosity response. The results of the analysis obtained a significant value > 0.05 which indicates that the optimum formula viscosity data is not significantly different. The LSD results from the ANOVA test indicated that the optimum formula viscosity was stable. Carbopol was well developed and perfect so that when stored there was no longer any absorption of water. Completely expanded carbopol did not release water so the optimum viscosity of the formula was stable during storage.

Spreadability test of gel preparations

Based on the spreadability test, the optimum formula during storage from week 0 to week 4 tends to be stable. The spreadability value was inversely proportional to the viscosity value. The higher the viscosity, the lower the spreading power value. The spreadability test data were analyzed statistically using one-way ANOVA to see their stability. The results of the analysis obtained a significant value > 0.05 which indicated that the data was not significantly different, which means that the spreadability of the optimum formula was considered to have no significant difference each week or to be stable during storage.

Gel adhesion test

Based on the adhesion test, the optimum gel formula during storage from week 0 to week 4 tends to be stable. The adhesive power value was proportional to the viscosity value. The higher the viscosity, the higher the stickiness value. Stickiness test data were analyzed statistically using one-way ANOVA to see their stability. The results of the analysis obtained a significant value > 0.05 which indicated that the data was not significantly different, which means that the adhesion of the optimum gel formula was considered to have no significant difference every week or was stable during storage.

Accelerated stability test

An accelerated gel stability test can be carried out using the cycling test method. This method is to provide gel treatment under stress conditions in the form of extreme temperature changes.

The result of the cycling test was that there was a separation of the emulsion after being given an extreme temperature difference treatment which was indicated by the volume ratio value of the separation. With extreme temperature changes, the ingredients in the gel could be damaged, hence it could damage the stability of the emulsion. The cycling test was carried out for 3 cycles at 40º C and 8º C (1 cycle = 24 hours at 40º C and 24 hours at 8º C). From the test results, the separation volume ratio was equal to 1. The gel preparation did not undergo liquid separation during the accelerated stability test. The gel is stable under extreme conditions.

pH preparation Test

The results of testing the pH of the sunflower seed oil optimum formula obtained a stable pH value of 5.50. For the physiological pH of the skin (pH range 4.2 – 6.5), the optimum gel formula tends to be acidic. The gel still provides a safe effect. Topical preparations must have a pH range between 4 – 7 to avoid irritation or reduce comfort when applied to the skin.

SPF test of chalcone compound gel preparation *in vitro*

Based on the results of the SPF test, it is known that the higher the concentration, the higher the SPF value obtained. The SPF results show that sunflower seed oil gel has an activity as a sunscreen which can provide ultra protection with a concentration of 10% with an SPF value of 15.60.

CONCLUSION

The optimum formula obtained is a formula with the composition of the gelling agent carbopol with a concentration of 1.0% w/w; CMC-Na with a content of 3.0% w/w. Sunflower seed oil gel preparations have physical properties that meet the required physical stability criteria. Sunflower seed oil gel preparation with a concentration of 10% has an SPF value of 15.60 (ultra protection).

ACKNOWLEDGEMENT

We thank the Faculty of Pharmacy, Universitas Gadjah Mada, for the funding of this research.

REFERENCES

Anonim, 2014, *Farmakope Indonesia,* Edisi 5, Departemen Kesehatan Republik Indonesia, Jakarta.

Damogalad, V. Hosea Jaya Edy dan Hamidah Sri Supriadi, 2013, Formulasi Krim Tabir Surya Ekstrak Kulit Nanas (Ananas comosus L Merr) dan Uji In Vitro Nilai Sun Protecting Factor (SPF), *Pharmacon Jurnal Ilmiah Farmasi UNSRAT Vol. 2 No. 2*, Program Studi Farmasi FMIPA UNSRAT, Manado.

Draelos, Z. D., dan Thaman, L.A., 2006, *Cosmetic Formulation of Skin Care Product*, Taylor and Francis Group, New York.

Elsner, P., Sivamani, R.K., Jagdeo, J.R., Mailbach, H.I., 2016, *Cosmeceuticals and Active Cosmetics,* 3rd edition, CRC Press, New York.

Gupta, S.D., 2010, Cosmetic Attributes of Aloe Vera L. Gel, *Medicinal and Aromatic Plant Science and Biotechnology*.

Juniarti, R., Herdiana Y., 2017, 'Revier Article: Aktivitas Ekstrak *Helianthus annuus L.*', *Jurnal Farmaka Universitas padjajaran,* Volume 15 No. 2, Jatinagor.

Kulkarni, S. S., (2014) 'Herbal Plants in Photo Protection and Sun Screening Action: an Overview', *Indo American Journal of Pharmaceutical Research American Journal Of Pharm Research*.

Lavi, Novita, 2012. *Sunscreen For Travellers*, Departement Pharmacy Faculty of Medicine, University of Udayana, Denpasar.

Mollet, H. dan Grubenmann, A., 2001, *Formulation Technology, Emulsion, Suspension, Solid Forms*, Wiley-Vch, Toronto.

Marchaban, Fudholi, A., Sulaiman, T.N.S., Mufrod, Martin, R., dan Bestari, A.N., 2016, *Seri Buku Petunjuk Praktikum Teknologi Farmasi: Teknologi Formulasi Sediaan Cair Semi Padat*, Laboratorium Teknologi Farmasi Fakultas Farmasi Universitas Gadjah Mada, Yogyakarta.

Neti.S. (2013*). Ensiklopedia dan Tanaman Obat*, Rumah Ide, Malang

Rowe, R.C., Sheskey, P.J., Quinn, M.E., 2009, *Handbook of Pharmaceutical Excipients,* 6th edition, Pharmaceutical Press, British.

Shaath, Nadim. A. 2005. *Sunscreens*, Third Edition, Taylor & Francis Group, New York, Page 359.

Shovyana, H.H., Zulkarnain, A.K., 2013, Physical Stability and Activity of Cream W/O Etanolic Fruit Extract of Mahkota Dewa (*Phaleria macrocarpha* (scheff.) Boerl) as A Sunscreen, *Traditional Medicine Journal*, 18(2).

Sulaiman, T.N.S., Tambunan, S., 2018, 'Formulasi Gel Minyak tsiri Sereh dengan Basis HPMC dan Karbopol', *Majalah Farmaseutik Vol. 14 No. 2:87-95.*

Susanti Y., Purba A.N, Rahmat D., 2019, Nilai Antioksidan dan SPF dari Kombinasi Minyak Biji Wijen (Sesamum indicum L.) dan Minyak Biji Bunga Matahari (Helianthus annuus L.), *Majalah Farmaseutik Vol. 16 no.*

1: 107 – 110.
Voight, R., 1994, *Buku Pengantar Teknologi Farmasi*, diterjemahkan oleh Soedani, N., Edisi V, Gadjah Mada University Press, Yogyakarta.