

Formulation of Peel-off Gel Mask with 70% Ethanol Extract of Dragon Fruit Peel (*Hylocereus polyrhizus* Haw.) as Antioxidant with Concentration Variation of PVA Gel Forming & Propylene Glycol Humectant

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Abstract. *The 70% ethanol extract of dragon fruit peel (*Hylocereus polyrhizus* Haw.) contains antioxidant activity in the form of flavonoids. This study aims to formulate a 70% ethanol extract of dragon fruit peel (*Hylocereus polyrhizus* Haw.) into a peel-off gel mask preparation with varying concentrations of PVA and propylene glycol, namely base without extract F1 (10%:10%), F2 (10%: 10%), F3 (10%:15%), and F4 (15%:10%). Extracts were made by maceration method with a solvent concentration ratio of 1:12. Determination of antioxidant activity was carried out by UV-Vis spectrophotometer using the DPPH method (2,2-diphenyl-1-picrylhydrazil) using quercetin as a comparison. This study showed that the 70% ethanol extract of dragon fruit peel (*Hylocereus polyrhizus* Haw.) has moderate potential as an antioxidant and can be formulated into a peel-off gel mask preparation. The manufacture of peel-off gel mask preparations with variations in the concentration of PVA and propylene glycol affects the parameters of the physical properties and antioxidant value of the peel-off gel mask. The higher the concentration of PVA, the smaller the dispersion value and the faster the drying ability. While the administration of propylene glycol with a high concentration will produce a mask preparation that has a high dispersion value and the ability to dry will be slower. While the antioxidant value of the mask preparation containing 70% ethanol extract of dragon fruit peel will be better if the concentration of the PVA gelling agent is higher than the humectant propylene glycol.*

Keywords: [70% ethanol extract of dragon fruit peel (*Hylocereus polyrhizus* Haw.), antioxidant activity, peel-off gel mask.]

INTRODUCTION

Facial skin is one of the most important external organs which is often exposed to ultraviolet rays which can cause skin problems such as wrinkles, aging, acne, and large pores, so it is important to take care of the skin itself (Sulastris and Chaerunisaa, 2018).

Free radicals are molecules that have one or more unpaired electrons. Therefore, the unpaired electrons will bind to the electrons of other cell molecules causing free radicals to become very reactive (Agustina, 2017). Examples are often found in the surrounding environment, such as metals (eg iron and copper), cigarette smoke, drugs, packaged food, and additives (Alhabsyi and Suryanto, 2014).

One of the antidotes to the bad effects of free radicals is antioxidants. Antioxidants are compounds that can donate electrons which can prevent the oxidation process from occurring (Pratiwi and Wahdaningsih, 2018). One way to prevent skin damage caused by free radicals is to consume fruits or vegetables that contain antioxidant compounds such as flavonoids, polyphenols, vitamin C, and vitamin E (Jani, Hakim, and Juliantoni, 2020).

A fruit that has natural antioxidant activity is dragon fruit (*Hylocereus polyrhizus*). Dragon fruit peel has greater activity of antioxidant compounds, this is because dragon fruit peel contains secondary metabolites of flavonoids which act as antioxidants. The value of total flavonoid content produced from 70% ethanol extract of dragon fruit peel was 88.695 ± 0.0922 mgQE/g extract ($8.87 \pm 0.01\%$) (Pujiastuti and El'Zeba, 2021).

The most used topical dosage forms for skin care are masks. In terms of use, the gel peel-off facial mask has the advantage of being easy to apply and clean because the mask preparation is in the form of an elastic membrane (Rahmawanty, Yulianti, and Fitriana, 2015). The benefits of using a peel-off gel mask are that it can repair and treat skin problems from aging, and acne, and can shrink pores. In addition, peel-off gel masks can clean and moisturize the skin (Sulastris and Chaerunisaa, 2018).

PVA functions as a gelling agent which can produce a gel that dries quickly and forms a thin, strong, and plastic film, providing good contact between the skin and the active substance as well as increasing temperature and blood circulation in the skin. (Santoso *et al.*, 2020). The addition of propylene glycol

as a humectant will maintain the stability of the preparation through the absorption of moisture from the environment and reduce water evaporation in the preparation, to maintain the stability of the preparation and maintain skin moisture (Sulastris and Chaerunisaa, 2018).

Based on the above background, research was conducted on optimizing the addition of PVA and propylene glycol on the physical properties of peel-off gel mask preparations of 70% ethanol extract of dragon fruit peel (*Hylocereus polyrhizus* Haw.) and testing the antioxidant activity of the preparations.

METHODS

Types of research

This type of research is experimental research with a research model that uses a quantitative approach. The drying method of red dragon fruit peel (*Hylocereus polyrhizus* Haw.) is carried out by utilizing sunlight and then macerating *Simplicia* using 70% ethanol solvent. Phytochemical screening and antioxidant value determination were carried out using a UV-Vis spectrophotometer on extracts, and formulated into peel-off gel mask preparations with varying concentrations of PVA gelling agent and propylene glycol humectants. Data analysis used SPSS version 20 using the non-parametric Mann-Whitney and Kruskal-Wallis tests, and using the TUKEY post hoc parametric test.

Place and time of research

The research was conducted at the Pharmaceutical Technology Laboratory of the Institut Teknologi Kesehatan Cendekia Utama Kudus and the Pharmaceutical Chemistry Laboratory from February to April 2022.

Population and sample

The population used in this study was dragon fruit peel (*Hylocereus polyrhizus* Haw.) which was obtained from the Pandangan market, Kragan District, Rembang Regency, Central Java Province. The sample used in this study was the 70% ethanol extract of dragon fruit peel (*Hylocereus polyrhizus* Haw.) which was randomly selected with the criteria that dragon fruit has red flesh, ripe, red peel, not rotten, and there are not many wounds on the peel.

Tools

Flacon, grinder, 40 mesh sieve, stove, thermometer, stainless bowl, moisture balance, filter paper, measuring flask, measuring cup, stir bar, glass funnel, micropipette, dropping pipette, beaker glass, analytical balance, blue tip, n-spectrophotometer UV-Vis, stick pH indicator, spreadability glass, porcelain, and sensory exchanger.

Materials

70% ethanol extract of dragon fruit peel, PVA, propylene glycol, carbopol 940, PEG-400, triethanolamine, methylparaben, 96% ethanol, pro-analysis ethanol, Mg, concentrated HCl, 1% FeCl₃, quercetin, DPPH (1,1-Diphenyl-2-Picrylhydrazil), aluminum foil, and filter paper.

Research procedure

Sample processing

Dragon fruit is washed clean and separated from the peel. Then cut the dragon fruit peel into small pieces and dry it. Drying was carried out under the sun with a black cloth cover to protect the sample from dust and other foreign matter. Sorting is done on the dry *simplicia* to avoid impurities. Furthermore, the dried *Simplicia* was crushed using a grinder. The *simplicia* powder was then sieved using a 40-mesh sieve (Handayani, Ahmad, and Sudir, 2014). The *simplicia* powder obtained is then calculated for drying shrinkage using the formula:

$$\text{Drying shrinkage} = \frac{A - B}{A} \times 100\%$$

Information :

A = *simplicia* weight before heating (gr)

B = *simplicia* final weight (gr)

Simplicia powder moisture content

Measurement of water content was carried out using a moisture balance device at 105°C for 10 minutes. Replication was carried out 3 times. The percent water content will be shown automatically on the moisture balance. Drying results are considered good if the moisture content is <10%. Water content that is too high allows the emergence of microbial growth thereby reducing the quality of preparations for pharmaceutical purposes (Wiendarlina, Rahminiwati, and Gumelar, 2019).

Extraction

Dragon fruit peel simplicia (*Hyloversus polyrhizus* Haw.) was extracted by maceration for 3×24 hours using 70% ethanol solvent with a ratio of 1:10 and the remaceration process was carried out using a ratio of 1:2, then evaporated with a stove using the water bath method. Keeping the temperature constant at 50°C so that the compound components present in the extract are not damaged when heated. Then the % yield of the extract is calculated using the formula:

$$(\%) \text{ Rendemen} = \frac{A}{B} \times 100\%$$

Information:

A = extract weight (gr)

B = initial weight of fresh simplicia (gr)

Phytochemical screening

Flavonoid testing

A total of 2 ml of the test solution was evaporated. Then added Mg powder and concentrated HCl. If the formation of orange, red, or yellow color means it contains flavonoid compounds (Mulu, 2018).

Phenol testing

A total of 2 ml of the test solution was evaporated. Then added 3-4 drops of FeCl₃. The formation of bluish-black color indicates phenolic compounds (Harliananda, Halimatussakdiah, and Amna, 2019).

Saponin testing

As much as 10 ml of the test solution is heated and then shaken for 10 seconds to form foam. The foam will last for 10 minutes and then be dripped with 1 drop of 2N HCl, the foam will not disappear (La, Sawiji and Yulawati, 2020).

Tannin testing

A total of 2 ml of the test solution was evaporated. Then it is added to a 1% iron (III) chloride solution. If the color shows greenish black then it contains tannin compounds (Sutomo *et al.*, 2016).

Dragon Fruit Peel 70% Ethanol Extract Gel Mask Formulation

Table 1 Peel-off Gel Mask Formulations

Materials (gram)	F1	F2	F3	F4
Dragon fruit peel extract	-	10	10	10
PVA	10	10	10	15
Propylene glycol	10	10	15	10
Carbopol 940	1	1	1	1
PEG 400	6	6	6	6
Triethanolamine	0,5	0,5	0,5	0,5
Methylparaben	0,2	0,2	0,2	0,2

Information:

F1: Base does not contain extract,

F2: Contains 10% extract, with variations of 10% PVA and 10% propylene glycol,

F3: Contains 10% extract, with variations of 10% PVA and 15% propylene glycol,

F4: Contains 10% extract, with variations of 15% PVA and 10% propylene glycol.

The procedure for preparing PVA was developed in hot aquadest (80°C). Carbopol 940 was developed with hot aquadest. Dissolve methylparaben with 96% ethanol. Propylene glycol, developed

carbopol 940, PEG 400, methylparaben solution, TEA were added sequentially into the PVA. Then add the extract which has been dissolved with the remaining aquadest. Stir until homogeneous.

Organoleptic observations

The mask preparation that has been made is observed for its shape, color, and smell (Mulu, 2018).

Homogeneity testing

The homogeneity test was carried out by smearing the sample on the glass and then observing whether the preparation was evenly mixed (Daswi, Stevani, and Santi, 2018).

pH testing

Dip the pH indicator stick into the diluted peel-off gel mask preparation, then wait until the pH paper changes color, and then adjust it to the indicator.

Spread ability testing

As much as 1 gram of gel is placed on a glass measuring 20x20 cm. Then it is covered with another glass and a ballast is used on it until the weight reaches 125 grams, then the diameter is measured after 1 minute. (Voight, 1994).

Dry time testing

The dry time test was carried out by applying 0.1 gram of gel evenly with an area of 2.5 x 2.5 cm on the arm and observing the time it took for the preparation to dry, namely the time from when the gel mask was applied until a dry and elastic layer was formed. can be peeled off from the skin surface without leaving a gel mass (Andini, Yusriadi, and Yuliet, 2017).

Antioxidant Activity Testing

Preparation of Quercetin Comparison Solution

Weigh 50 mg of quercetin and then put it into a measuring flask and add 50 ml of ethanol p.a to obtain a concentration of 1000 ppm. Then 1 ml pipette was dissolved with ethanol p.a up to 10 ml to obtain a quercetin stock solution with a concentration of 100 ppm (Handayani, Ahmad, and Sudir, 2014).

Preparation of DPPH Control Solution

Weigh 9.8 mg of DPPH and then put it into a measuring flask and add 250 ml of ethanol p.a to obtain a DPPH concentration of 0.1 mM (Molyneux, 2004).

Wavelength Determination

Take 500 μ l of 2 ppm quercetin stock solution. Then 4 ml of DPPH solution was added to the flacon which had been covered with aluminum foil. Then the absorbance was calculated using UV-Vis spectrophotometry at a wavelength of 400-600 nm.

Determination of Operating Time

Operating time was determined by taking 500 μ l of 2 ppm quercetin stock solution. Then 4 ml of 0.1 mM DPPH solution was added to the flacon which had been covered with aluminum foil. Then the mixed solution is shaken until homogeneous. Then read the absorbance for 1 hour at a wavelength of 513 nm.

DPPH Absorbance Determination

A 4 ml of DPPH solution was put into a closed flacon. After that, the absorbance of the solution was read at a maximum wavelength of 513 nm. To measure antioxidant activity, this solution is used as a control for the reference solution and the test solution.

Preparation of Quercetin Standard Curves

Weigh 25 mg of quercetin and then put it into a measuring flask and add 25 ml of ethanol p.a to obtain a concentration of 1000 ppm. Then 1 ml pipette was dissolved with ethanol p.a up to 10 ml to

obtain a quercetin stock solution with a concentration of 100 ppm. Then, from the mother liquor, a concentration series of 2 ppm, 4 ppm, 6 ppm, 8 ppm, and 10 ppm was made (Al Kadri *et al.*, 2019).

Measurement of % Absorbance of Quercetin Inhibition, Extracts, and Masks

The quercetin analysis stage was carried out in the serial solution which was taken as much as 500 μ l and then reacted with 0.1 mM DPPH as much as 2ml. Then allowed to stand in a dark place and incubated for 31 minutes. After the incubation time is complete, the absorbance is calculated at a wavelength of 513nm. The absorbance measurement of dragon fruit peel extract samples was carried out by making a 1000 ppm mother liquor, which was then made into series solutions of 100 ppm, 200 ppm, 300 ppm, 400 ppm, and 5 ppm. From the serial solution, 500 μ l was taken and then reacted with 2ml of 0.1 mM DPPH. Then allowed to stand in a dark place and incubated for 31 minutes. After the incubation time is complete, the absorbance is calculated at a wavelength of 513nm. The treatment is the same as the peel-off gel mask.

Measurement of IC₅₀

IC₅₀ is the concentration of an antioxidant substance that can cause 50% of DPPH to lose its radical character. From the data obtained, a line equation was made indicating the relationship between concentration and inhibition (% inhibition) to determine the IC₅₀ antioxidant value. Based on the equation of the linear line, it can be determined that the IC₅₀ value of the dragon fruit peel extract is. Compounds that have an IC₅₀ value of <50 μ g/mL are said to be very strong, an IC₅₀ value of 50-100 μ g/mL is said to be strong, an IC₅₀ value of 100-150 μ g/mL is said to be moderate, an IC₅₀ value of 150-200 μ g/mL is said to be weak, an IC₅₀ value > 200 μ g/mL is said to be very weak and potentially has no antioxidant activity (Molyneux, 2004).

RESULTS AND DISCUSSION

Sample Processing

Drying shrinkage was carried out by weighing 10 g of simplicia powder in a porcelain cup, then heating it in an oven at 105°C for 1 hour. From the calculation results, the drying shrinkage of dragon fruit peel powder is 8.5%. The results of the simplicia drying shrinkage test using the sunlight method were following the standards required by BPOM (2014), namely <10%.

Water content

Calculation of the water content was carried out using a moisture balance tool which was carried out 3 times at the top, middle, and bottom of the simplicia powder, the goal was to determine the dry quality of the powder evenly. The average water content value of dragon fruit peel simplicia was 2.27%. This value meets the BPOM standard (2014) that the requirement for simplicia moisture content is <10%.

Extraction

The extract obtained from the extraction process of 250 grams of dragon fruit peel simplicia with 2.5 L 70% ethanol is as much as 56.3 grams. The % yield of the extract obtained through calculations is 22.52%. The characteristics of the extract obtained were brown, viscous, and had a distinctive odor.

Phytochemical Screening

Phytochemical screening is carried out to determine the presence of certain compounds in a sample (Noor, Yufita, and Zulfalina, 2016). The secondary metabolites contained in the 70% ethanol extract of dragon fruit peels include flavonoids and phenols. Flavonoids are a class of secondary metabolite compounds that act as antioxidants. Dragon fruit peel extract does not contain secondary metabolites of saponins and tannins.

Testing the Physical Properties of Gel Masks

Organoleptic Testing

The non-extracted peel-off gel mask preparation has a white color, is in the form of a gel, and has a

distinctive ethanol odor. And the formulation of peel-off gel masks with the addition of extracts has a brown color, is in the form of a gel, has a characteristic odor of ethanol, and has a characteristic odor of extracts.

Homogeneity Testing

The results of the homogeneity test showed that all the gel peel-off mask preparations were mixed homogeneously.

pH Testing

The results of the pH test showed that the 4-peel-off gel mask preparations of dragon fruit peel extract had a pH of 5 which was following the range of requirements set by SNI 16-6070-1999 that topical preparations should have a pH in the range of 4.5-6.5. This is because if the pH of the topical preparation is <4.5 or too acidic it can cause irritation to the skin, and if the pH is >6.5 or too alkaline it can cause the skin to become dry and scaly. So that from the results of the fourth pH test, the peel-off gel mask formula is classified as safe.

Giving carbopol 940 as a gelling agent is carried out to obtain mask preparations that have a pH according to quality requirements. Carbopol has an acidic pH, which is 2.5-3, so an alkali is needed to increase the pH of carbopol (Tsabitah *et al.*, 2020).

The stable pH value in the peel-off gel mask formula is influenced by the presence of an additional ingredient in the form of triethanolamine which acts as an alkalizer and gives a stable pH to carbopol 940. In line with research Andini *et al.* (2017) that giving carbopol 940 plus triethanolamine can produce mask preparations that have a pH of 5-6 according to the requirements.

Spread Ability Testing

Table 2 Data on the Spread Ability Test of Peel-off Gel Masks

Formula	Average±SD (cm)	Requirement (SNI, 1992)	Information
F1	4,4±0,05	5-7 cm	Not fulfilled
F2	6,6±0,05		Fulfilled
F3	7±0,05		Fulfilled
F4	4,8±0,1		Not fulfilled

Source: Processed primary data (2022)

The results of the spreadability test measurements showed that from the replication results, F1 had a value of 4.4 cm which did not meet the requirements because F1 is a base that has a viscous dosage form so it produces little spreadability. Whereas F2 has a value of 6.6 cm and F3 has a value of 7 cm which means that the formulation shows that the spreadability value has met the specified requirements. The results of F4 replication have a value of 4.8 cm where this value does not meet the specified requirements.

The spreadability value of the preparation was affected by variations in the concentration of PVA gelling agent and propylene glycol humectants. The addition of PVA affects the spreading power in a preparation where the higher the PVA value, the less spreading ability of the preparation, because the dosage form will be thicker if it has more PVA concentrations. And the administration of propylene glycol at a high concentration will cause the preparation to have a soft texture so that the spreadability value is even greater (Yogesthinaga, 2016).

Dry Time Testing

Table 3 Data on Dry Time Test Results for Peel-off Gel Masks

Formula	Average±SD (minute)	Requirement (SNI, 1992)	Information
F1	13.14±0,12	15-30 Minutes	Not fulfilled
F2	23.26±0,23		Fulfilled
F3	28.19±0,2		Fulfilled
F4	15.12±0,09		Fulfilled

Source: Processed primary data (2022)

The results of the dry time test on F1 were 13.14 minutes where this value was less than the predetermined requirements of 15-30 minutes, this was because F1 was a base without extracts that had a PVA concentration comparable to the concentration of propylene glycol so that the resulting dosage form was very viscous.

While the formulation of a peel-off gel mask that had the addition of extracts obtained results in 23.26 minutes (F2), 28.19 minutes (F3), and 15.12 minutes (F4). Where this value meets the requirements of SNI, (1992) that the dry time range for a good mask preparation is 15-30 minutes.

Antioxidant Activity Testing

Maximum wavelength Result

The results of UV-Vis spectrophotometry measurements at the maximum wavelength of 0.1 mM DPPH solution in the range of 400-600 nm, based on the optimization carried out, obtained the maximum wavelength of 513 nm. The maximum wavelength is indicated by the largest absorption or peak seen on the curve.

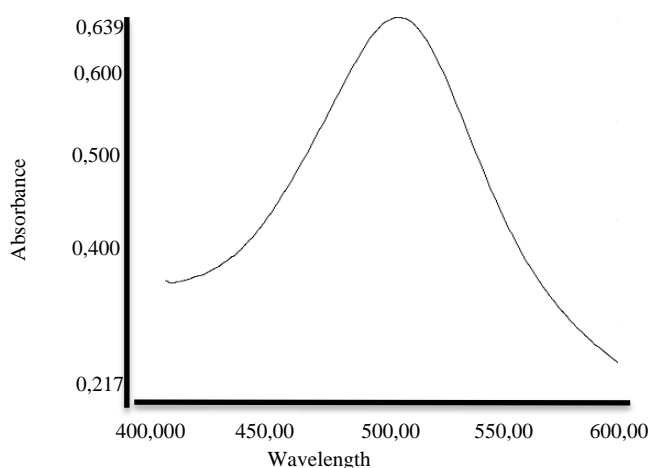


Figure 1 Wavelength Curve

Operating time results

Determination of the operating time of 0.1 mM DPPH solution in ethanol p.a aims to determine the most stable working time of DPPH compounds in ethanol p.a. The operating time reading was carried out for 60 minutes and was determined by looking at the most stable absorption. The operating time obtained was 31 minutes, during which time samples containing antioxidants could scavenge DPPH free radicals. The selection of operating time can be determined by looking at the absorbance value which is stable or has a slight difference sequentially.

DPPH Control Absorbance Value

Measurement of the absorbance value of 0.1 mM DPPH was carried out by observing the absorbance of 0.1 mM DPPH solution at a maximum wavelength of 513 nm. From the results of measuring the absorbance of the 0.1 mM DPPH solution, a value of 0.927 was obtained. This value can then be used to calculate the IC₅₀ of the sample to be tested for its antioxidant content.

Absorbance % Inhibition Measurement

Absorbance and % inhibition data from several concentrations showed that the greater the sample concentration, the smaller the DPPH absorbance value so the greater the concentration of DPPH free radical inhibitors.

Value measurement of IC₅₀

Table 4 Value Result Data of IC₅₀

Sample	Value Average of IC ₅₀ ($\mu\text{g/mL}$) \pm SD	Category (Molyneux, 2004)
Quercetin	17,18 $\mu\text{g/mL}$ \pm 0,85	Very strong
Dragon Fruit Peel 70% Ethanol Extract	175,76 $\mu\text{g/mL}$ \pm 5,75	Weak
F1 Mask	665,27 $\mu\text{g/mL}$ \pm 12,10	Very weak
F2 Mask	597,75 $\mu\text{g/mL}$ \pm 6,77	Very weak
F3 Mask	568,14 $\mu\text{g/mL}$ \pm 12,04	Very weak
F4 Mask	487,70 $\mu\text{g/mL}$ \pm 15,15	Very weak

Source: Processed primary data (2022)

These results show that as a comparison the quercetin sample has very strong activity with a value of $<50 \mu\text{g/mL}$. Quercetin is a flavonoid that has antioxidant activity. When the quercetin compound is reacted with the DPPH free radical, the quercetin will donate electrons so that the DPPH becomes non-radical. Therefore quercetin is very good at inhibiting free radicals. Meanwhile, the IC_{50} value obtained from the extract indicated that the 70% ethanol extract of dragon fruit peel was classified as weak, this was influenced by the low % yield of the extract, where the lower the % yield, the fewer compounds contained in the extract. The low % yield obtained could have been affected by damaged components in the drying process. Because flavonoid metabolites are easily damaged by high temperatures or $>50^\circ$.

The IC_{50} result value of all dragon fruit peel extract peel-off gel mask formulations has a value of $>250 \mu\text{g/mL}$ which indicates that these preparations do not have antioxidant activity (Molyneux, 2004). The lowest antioxidant content is in F1, this is due to the absence of additional extracts that help the preparation in counteracting free radicals. While the highest antioxidant value is in F4 which has the highest PVA content compared to the others. The difference in the value of this antioxidant is influenced by the amount of gel base in the preparation, namely PVA. The greater the concentration of PVA used, the ability to inhibit free radicals will increase. PVA has chain bonds that work to help extracts inhibit free radicals.

These results indicate that the provision of the base has an absorbance absorption value which can affect the antioxidant value of the preparation. In line with the research of Mutiara (2018) that the more emulsifier, the lower the antioxidant value obtained, this is because the active antioxidant substances will protect the emulsifier from being oxidized, causing a decrease in the antioxidant activity of the preparation.

CONCLUSION

Based on the results of the study it can be concluded that:

1. Dragon fruit peel extract has an antioxidant activity of $175.76 \mu\text{g/mL}$ which is classified as weak, while the mask preparation has a very weak antioxidant activity of $665.27 \mu\text{g/mL}$ (F1), $597.75 \mu\text{g/mL}$ (F2), $568.14 \mu\text{g/mL}$ (F3), and $487.70 \mu\text{g/mL}$ (F4) which have no potential antioxidant activity.
2. The 70% ethanol extract of dragon fruit peel can be formulated into a gel peel-off mask preparation that meets the parameters of good physical properties according to SNI standards.
3. Variations in the concentration of PVA and propylene glycol can affect the physical properties of spreadability and dry time. Where the higher the PVA concentration, the thicker the preparation will be, so the smaller the spreadability value obtained and the required dry time. While the addition of propylene glycol at a high concentration makes the preparation runny, so it takes a long time to dry and the spreadability value obtained will be even greater. And it can affect the antioxidant value, where the more PVA concentration, the smaller the IC_{50} value.

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