

## FORMULATION AND EVALUATION OF MANALAGI APPLE PEEL (*Malus sylvestris* Mill) SUNSCREEN SPRAY AS HALAL COSMETICS

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### ABSTRACT

Manalagi apple peel is known to contain flavonoid compounds that are active as photoprotectors, as well as antioxidants that can absorb UV-A and UV-B exposure and release free radicals. Apple peel has the potential to be formulated as an active ingredient in natural sunscreen, which is considered safer and less likely to irritate when applied to the peel. In this study, halal sunscreen preparations from 70% ethanol extracts of manalagi apple peel with varying concentrations of manalagi apple peel extract were 15%, 20%, and 25%. The purpose of this study was to determine the critical halal point of sunscreen spray preparation, physical characteristics (organoleptic, homogeneity, pH, viscosity, and spray pattern), and SPF value. The data analysis methods in this study were descriptively and quantitatively performed. Based on the results of this study, no halal critical point ingredients were used in the formulation. The physical characteristic test results of all spray sunscreen formulas met the requirements of good physical characteristics criteria, and the resulting SPF values of F1, F2, and F3 were 22.66, 26.51, and 31.22, respectively. The SPF value of the Manalagi apple peel waste sunscreen spray formula was very high (>15).

**Keywords:** *Halal, cosmetics, manalagi apple peel, SPF, spray sunscreen*

### INTRODUCTION

Apples are an agricultural commodity in East Java that has become an icon of Batu Tourism City in East Java Province (Dore *et al.*, 2021). The most widely grown type of apple in Batu City is the Manalagi apple, commonly known as the Malang apple. Manalagi apples are consumed for their flesh because they have a distinctive sweet flavor, the fruit is easily found in the Batu City area, and the price is affordable. The apple production in Batu City could be a business opportunity. Apple chips are an example of an apple-based processed product. However, the amount of apple peel waste generated from processing apple chips is quite large, approximately 42.308% of the total apples, which causes a buildup of waste that can be harmful to the environment (Surjowardojo *et al.*, 2016).

Apple peel itself is known to contain many flavonoid compounds that act as photoprotectors and antioxidants that are beneficial for human skin. Photoprotectors work by absorbing UV light at UV-A and UV-B wavelengths, while antioxidants counteract free radicals in the skin due to exposure to UV-A and UV-B waves (Prasiddha *et al.*, 2016). Until now, phenolic compounds, especially flavonoid compounds, have been known to have the potential to be used as ingredients for sunscreen preparations because they contain compounds with chromophore groups (conjugated double bonds), allowing them to absorb UV-A or UV-B rays (Salwa *et al.*, 2020). In addition, sunscreen protects the skin by absorbing the solar radiation energy that hits the skin, so that solar radiation does not hit the

skin directly. Sunscreen also serves to protect the body's skin from the effects of free radicals due to UV exposure; therefore, sunscreen is also closely related to its antioxidant activity. According to the United States National Cancer Institute, apples contain more flavonoids than other types of fruit. Apple peel contains phytochemical compounds, such as flavonoids and phenolic acids, that can fight free radicals (Baiti *et al.*, 2021). Apples are rich in antioxidants in both flesh and peel. However, apple peel has a higher antioxidant content than the flesh of the fruit (Octaviany *et al.*, 2017). Apple peels contain several phytochemical compounds such as catechins, phloridzin, chlorogenic acid, and quercetin. Nurcahyati's research stated that only apples on the skin contain quercetin (Nurcahyati, 2014). Quercetin is the most abundant flavonol in nature compared to other types. Quercetin in apple peel can be used as an antioxidant and an anti-aging agent (Pertiwi *et al.*, 2016).

Therefore, researchers have innovated the processing of apple peel waste into a product that can be useful as a cosmetic product in the form of a sunscreen spray that is beneficial for skin health. Spray preparation was chosen because it has not been widely circulated in the market and has many advantages, such as being lightweight, easy, and fast to apply, suitable for use in large areas of the body, can be used over makeup, has a long contact time with the skin due to the gelling agent, and is also able to minimize the presence of bacterial contamination that can come from hands when applied to the skin (Kresnawati *et al.*, 2022). To produce and develop good and correct cosmetic products, it is necessary to evaluate product preparation. In the production process, it is necessary to test the physical properties of spray sunscreen preparations, such as organoleptic tests, pH tests, homogeneity tests, spray pattern tests, and viscosity tests, which show that the preparations produced have met predetermined standards. In addition to sunscreen products, the SPF value test is an evaluation that is no less important to do is the Sun Protection Factor (SPF) value test. The effectiveness of a sunscreen is based on its Sun Protection Factor (SPF) value. The SPF value describes the ability of sunscreen to protect the skin from erythema. Sunscreen activity can be determined in vitro using UV-Vis spectrophotometry (Zulkarnain *et al.*, 2013).

Indonesia is a country with a majority Muslim population, where Islam requires that the use of cosmetics be permissible on the condition that the ingredients must be pure and halal, which is allowed according to Sharia law, and does not cause harm. Thus, Islam requires its followers to use halal cosmetics. It is not only prayer that requires a person to clean his body from the unclean (Hasibuan *et al.*, 2019). Cosmetics that contain halal ingredients are very important, especially for Muslims. Therefore, researchers are very interested in innovating new products in the form of sunscreen spray with the main ingredient using waste from natural sources, which is of course halal with the title "Formulation and Testing the SPF Value of Sunscreen Spray Preparations from Waste".

## RESEARCH METHODS

### Equipment and Materials

The tools used in this research are UV-Vis Spectrophotometry (Shimadzu), Analytical Scales (Shimadzu), Measuring Flasks (Iwaki), Pipettes (Pyrex), Stir Rods (Pyrex), Beakers (Pyrex), Magnetic Stirrer, Hot Plate (Heidolph), Measuring Cups (Pyrex), Thermometers, pH Meters (Mettler Toledo), Watch Glasses, Viscometers Brookfields, Mica Plastic, Spray Bottle.

The materials needed for this research are Apple Peel Extract, Propylene Glycol (PT. Brataco), methylparaben (Golden Era), propylparaben (Golden Era), aquades (PT. Brataco), TEA (Merck), carbopol (Sanare Lab), and Apple Fragrance (PT. IFF), and 96% ethanol (PT. Brataco).

### Research Procedure

#### 1. Manalagi Apple Peel Extraction

Extraction was performed using the maceration method with 70% ethanol for 3 × 24 hours. The dry simplicia used was 1170 grams with a solvent amount of 8775 mL. The extracts were then organoleptically observed and tested for pH (Pertiwi *et al.*, 2016).

## 2. Identification of Halal Critical Points

The identification of halal critical points was performed by identifying and tracing each ingredient used in the formulation of spray sunscreen preparations. The identification results were then adjusted to the LPPOM-MUI Halal Assurance System Guidelines and the Decree of the Minister of Religion, which are based on Islamic rules related to food halalness.

## 3. Spray Sunscreen with Manalagi Apple Peel Extract Formulation

The finished extract was then formulated at different concentrations. Formula 1 (15%), formula 2 (20%), and formula 3 (25%). The spray sunscreen formulations are presented in [Table I \(Kreasnawati \*et al.\*, 2022\)](#).

**Table I. Formulation of Manalagi Apple Peel Extract Sunscreen Spray**

Component	Formula (b/v)		
	F I	F II	F III
Manalagi Apple Peel Extract	15 %	20 %	25 %
Propylene glycol	15 %	15 %	15 %
Methyl paraben	0.18 %	0.18 %	0.18 %
Propyl paraben	0.2 %	0.2 %	0.2 %
TEA	1%	1 %	1 %
Carbopol 940	0.5 %	0.5 %	0.5 %
Fragrance	Qs	qs	qs
Aquades	ad 50 mL	ad 50 mL	ad 50 mL

## 4. Organoleptic Tests

Organoleptic testing is carried out by visually observing the physical appearance of the spray preparation, which includes shape, odor, and color. The requirements for a good spray preparation are that it has a slightly viscous liquid form and a distinctive color and odor according to the active ingredient used ([Angelia \*et al.\*, 2022](#); [Fitriansyah \*et al.\*, 2016](#)).

## 5. Homogeneity Test

A homogeneity test of the spray preparations was carried out by spraying the preparation in a spray bottle onto a glass object. The spray results were then observed to determine whether there were unmixed or clumped particles during the preparation. The criterion for homogeneity of a good spray preparation is that there are no unmixed or clumped particles ([Kresnawati \*et al.\*, 2022](#)).

## 6. pH Test

The pH test was performed using a tool in the form of a pH meter. The pH required for topical preparations for the skin is a pH that is adjusted to the pH conditions of normal human skin, namely 4.5–6.5 ([Kresnawati \*et al.\*, 2022](#); [Fitriansyah \*et al.\*, 2016](#)).

## 7. Viscosity Test

The viscosity test was performed using a Brookfield viscometer with a size 40 spindle at a speed of 2 rpm. The viscosity requirement for good spray preparation is less than 150 cP ([Fitriansyah \*et al.\*, 2016](#)).

## 8. Test Spray Pattern

The spray pattern test was carried out using transparent mica plastic, and millimeter paper was placed beneath it as a measuring tool. The sunscreen spray preparation was then sprayed on a plastic mica sheet at spraying distances of 3, 5, 10, 15, and 20 cm. The spray patterns formed at each spray distance were then measured in diameter and compared. The criteria for a good spraying pattern are that the preparation is easy to spray, and the sprayed particles can be evenly distributed (Kresnawati *et al.*, 2022).

#### 9. SPF

Determination of the SPF value was performed *in vitro* using UV-Vis spectrophotometry. Performed at a wavelength of 290–320 nm with intervals of 5 nm. The way to measure is by diluting each sunscreen spray preparation formula 0.5 grams in 10 mL of 96% ethanol solvent and measuring the absorbance at a wavelength of 290–320 nm every 5 nm interval. The blank solution contained 96% ethanol. The absorbance value was calculated using the SPF equation of Mansur (1986).

#### Data Analysis

Data analysis in this study was performed descriptively and quantitatively. Descriptive data will be analyzed by describing and comparing them with existing references. In this study, descriptive data included the values of the physical characteristics (organoleptic, homogeneity, pH, viscosity, and spray pattern). Meanwhile, quantitative data from this study were obtained by determining the SPF value of the preparation tested and then using the division of the protection level of the sunscreen preparation based on the reference SPF value to describe the effectiveness of the preparation.

## RESULTS AND DISCUSSION

### Manalagi Apple Peel Extraction

Extraction was performed using the maceration method with 70% ethanol for 3 × 24 hours. The yield of the extraction process was 38.63%. From the extraction results, the extract was obtained in the form of a thick extract with a brown color and a distinctive apple odor and a pH of 4.12.

### Identify Critical Halal Points

The halal critical point of a product is the stage in the production process that allows for changes from what was originally a halal product to a haram product. The critical point of halalness of a product can be identified from several factors, such as the ingredients used in the formulation of the product, which include raw materials and additional ingredients. The identification of critical points refers to the LPPOM MUI decision letter regarding the list of non-critical ingredients (List of Positive Halal Ingredients) SK12/Dir/LPPOM MUI/VI/20 and the Decree of the Minister of Religion of the Republic of Indonesia Number 1360 of 2021 concerning materials that are exempted from the obligation to be halal certified (Table II). In the formulation process of apple peel spray sunscreen preparations, the ingredients used are all clear about their halal status and are contained in SK12/Dir/LPPOM MUI/VI/20, as well as in the Decree of the Minister of Religion Number 1360 of 2021, except for apple fragrance products. Fragrance or fragrance additives are included in Decree of the Minister of Religion Number 748 of 2021 concerning Types of Products that Must be Halal Certified). The apple fragrance used in the formulation was produced by PT Essence Indonesia (IFF). PT Essence Indonesia (IFF) is a halal certified company. Thus, it can be said that the apple fragrance used is not included in the ingredients that have critical halal points.

**Table II. Results Identification of Critical Points of Apple Peel Extract Sunscreen Spray Ingredients**

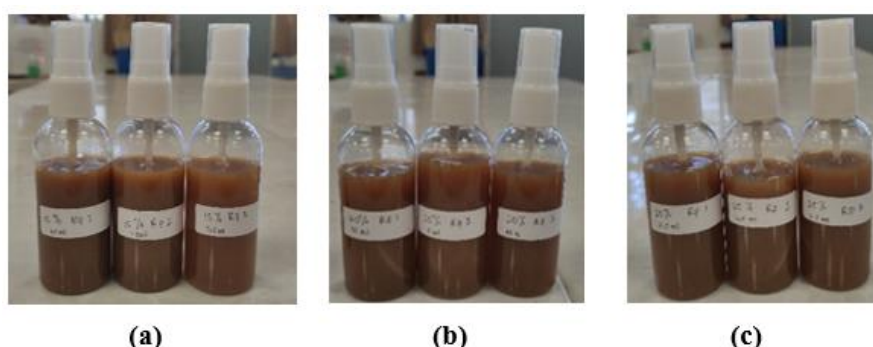
Material Name	CAS	SK12/Dir/LPPOM MUI/VI/20	Decree of the Minister of Religion 1360 of 2021	Halal Critical Point
Propylene glycol	57-55-6	√	No. 424	-
Methyl paraben	99-76-3	√	No. 422	-
Propyl Paraben	94-13-1	√	No. 426	-
Triethanolamine	102-71-6	√	No. 2948	-
Carbopol 940	9003-01-4	√	No. 3863	-
Aquades	7732-18-5	√	No. 2	-
Apple fragrance oil (alcohol free)	-	-	-	-

The extraction process used a filter solution in the form of ethanol. The ethanol solvent based on the latest MUI Fatwa No. 10/2018 on Food and Beverage Products Containing Alcohol/Ethanol states that only ethanol derived from khamr is haram and unclean, whereas other types of ethanol may be used with the restrictions set out in the fatwa. Examples include synthetic ethanol and non-khamr fermentation products. The ethanol used in the extraction process was industrial ethanol, as permitted by LPPOM-MUI. In addition to the main ingredients and additives, the place of production and tools used in the formulation also need to be considered because they can affect the halal status of a product. The place and tools used in the formulation process of this study were free from unclean and haram materials; therefore, it can be concluded that the apple peel extract sunscreen spray products have a clear halal status.

### Evaluation Results of Spray Sunscreen Preparations

#### Organoleptic Test

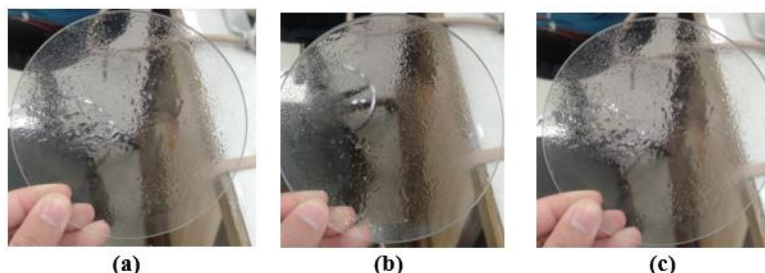
The organoleptic test results obtained from the three formulas (F1, F2, and F3) had a liquid dosage form that was slightly thick, brown, and had an apple aroma. These results are based on the requirements for a good spray preparation, namely having a slightly thick liquid form and a distinctive color according to the active ingredient used. The difference between each formula is that the higher the concentration of the extract, the more intense is the color produced from the preparation.



**Figure 1. Results of organoleptic test for manalagi apple peel extract sunscreen spray (a) formula 1 (b) formula 2 (c) formula 3**

### Homogeneity Test

The results of the visual observations from the homogeneity test for each formula showed that all preparations had good homogeneity. All particles in the preparation were mixed evenly and no clumped particles were found, so it can be said that there was no difference in the level of homogeneity between each formula along with the increase in the concentration of the extract used.



**Figure 2.** results of homogeneity test for manalagi apple peel extract sunscreen spray  
(a) formula 1 (b) formula 2 (c) formula 3

### pH Test

A pH test was carried out to determine whether the acidity level of each formula (F1, F2, and F3) was suitable for human skin conditions. A pH that is either too high or too low can cause skin irritation. The results of the pH test were as follows.

**Table III.** Results of pH test for Manalagi Apple Peel Extract Sunscreen Spray

Formula	pH $\pm$ SD
F1 (15%)	5.95 $\pm$ 0.08
F2 (20%)	5.57 $\pm$ 0.04
F3 (25%)	5.27 $\pm$ 0.07

The pH requirements for topical preparations are adjusted to normal human skin conditions, namely 4.5–6.5. From the pH test results, all preparations met the pH range requirements for topical preparations. Between formulas 1, 2, and 3, there was a decrease in the pH as the extract concentration increased. This is because apple peel extract has a fairly low pH due to its content of apple peel extract, which is rich in organic acids such as chlorogenic acid, caffeic acid, and other organic acids (Sa'adah and Estiasih, 2015).

### Viscosity Test

Viscosity testing was performed by using a Brookfield viscometer. The results obtained from the viscometer test are as follows:

**Table IV.** Results of Viscosity Test for Manalagi Apple Peel Extract Sunscreen Spray

Formula	Viscosity $\pm$ SD
F1 (15%)	68.46 $\pm$ 1.15
F2 (20%)	42.00 $\pm$ 1.19
F3 (25%)	20.95 $\pm$ 0.38

The viscosity requirement for good spray preparation was less than 150 cp. An excessively high viscosity can make the preparation difficult to spray and spread evenly. Meanwhile, preparation with low viscosity makes the preparation drip easy. From the viscosity test results, it was found that all formulas were within the required range, namely, below 150 cp (Fitriansyah *et al.*, 2016).

From the three formulae, it is known that the higher the concentration of the extract produced, the lower the viscosity produced. This decrease in viscosity was due to the relatively acidic pH of the Manalagi apple peel extract. The addition of the extract to the gel base (carbopol) can cause the pH of the gel base to decrease further and reduce the viscosity of the carpol itself. Carbon is often used as a viscosity enhancer or thickening agent in the formulation process. Carbon has acidic properties and its development is greatly influenced by the ionization process, where carbon does not form a stable gel mass if it is in an acidic environment (Rahmatullah *et al.*, 2020). Additionally, the addition of extracts can reduce the viscosity of the preparation because the extract has liquid properties (Indriarini *et al.*, 2021). A decrease in viscosity can also occur owing to the synergy process, namely the release of liquid trapped in the gel, which causes the liquid to move to the surface, which causes the viscosity of the preparation to decrease (Astuti *et al.*, 2017).

**Spray Test Pattern**

The spray test pattern was obtained by spraying each preparation on a millimeter block of paper that had been previously coated with mica plastic. The spray pattern distances were 3, 5, 10, 15, and 20 cm. The diameter of the obtained spray pattern was then calculated and observed to determine whether the pattern formed was spread evenly. The results obtained from the test are as follows

**Table V. Results of Diameter of Spraying Pattern for Manalagi Apple Peel Extract Sunscreen Spray**

Formula	Spray Pattern Diameter ± SD				
	3 cm	5 cm	10 cm	15 cm	20 cm
F1 (15%)	3.60 ± 0.52	6.76 ± 0.25	12.33 ± 0.57	14.90 ± 0.79	17.26 ± 0.25
F2 (20%)	4.96 ± 0.25	7.36 ± 0.15	13.83 ± 0.28	15.56 ± 0.51	19.50 ± 0.50
F3 (25%)	5.73 ± 0.25	8.26 ± 0.68	14.26 ± 0.25	17.63 ± 0.35	20.76 ± 0.68

All three formulas had a spray form that spread evenly. Of the three formulas, F1, F2, and F3 have spray diameters that widen as the concentration of active ingredients increases. This is because the lower the viscosity of the preparation, the easier it is to spray and the wider the spray pattern. Thus, it can be concluded that the lower the viscosity, the wider is the spray pattern formed.

**SPF Value Test**

The aim of the SPF value test is to obtain the SPF value of the tested formula. The SPF value is important because it indicates the ability of a sunscreen product to protect against UV radiation. SPF value testing was performed using UV-Vis spectrophotometry. The output results of the tool are in the form of absorbance values at wavelengths of 290–320 nm. The absorbance value can be calculated using the Mansur equation (1986):

$$SPF_{spectrophotometric} = CF \times \sum_{290}^{320} EE(\lambda) \times I(\lambda) \times Abs(\lambda)$$

Description:

- EE : Erythematous effect spectrum
- I : Solar intensity spectrum
- Abs : Absorbance of sunscreen
- CF : correction factor

Subsequently, calculations were carried out using Microsoft Word Excel so that the SPF value of each formula and its replication were obtained as follows.

**Table VI. Results of SPF Value Test for Manalagi Apple Peel Extract Sunscreen Spray**

Formula	Replication			
	Replication 1	Replication 2	Replication 3	Average
F1 (15%)	23.35	23.20	21.43	22.667
F2 (20%)	27.66	26.04	25.82	26.511
F3 (25%)	30.91	32.20	30.54	31.220

The results of the SPF values listed in the table show that F1 (Formula 1) (15%) containing the 7.5 mL extract produced the lowest SPF value when compared to other formulas, with an average SPF of 22.66. F2 (Formula 2) (20%), containing 10 mL of extract, produced an average SPF value of 26.51. F3 (formula 3) (25%) containing a 12.5 mL extract produced the highest mean SPF value of 31.22. This can be interpreted as the greater the amount of apple peel extract added, the greater the SPF value produced during preparation. According to the FDA, the potential protection by sunscreen is categorized into 5 types, namely minimum protection (SPF value 2–4), medium protection (SPF value 4–6), extra protection (SPF value 6–8), maximum protection (SPF value 8–15), and ultra protection (SPF value >15) (Alrosyidi and Syaifiyatul, 2021). The SPF values produced from F1, F2, and F3 can be categorized as sunscreen with ultraprotection (SPF value >15).

The photoprotective ability of the apple peel extract sunscreen spray formula is produced by the presence of high antioxidants in the apple peel, which is higher than that of the fruit. The active compounds are flavonoids, which can counteract the negative effects of free radicals (Baiti *et al.*, 2021). Phenolic compounds, especially flavonoids and tannins, are known to have the potential to be used as ingredients in sunscreen preparations because they contain compounds with chromophore groups (conjugated double bonds), allowing them to absorb UV-A or UV-B (Salwa *et al.*, 2020).

## CONCLUSION

The spray sunscreen preparation of manalagi apple peel extract (*Malus sylvestris* Mill) in its formulation does not contain critical points of halal ingredients, so it can be said that the preparation has halal status. Spray sunscreen formulations with three variations of active ingredient concentrations of 15%, 20%, and 25% met the physical characteristics (organoleptic, homogeneity, pH, viscosity, and spray pattern) of good spray sunscreen preparations. Spray sunscreen preparations of manalagi apple peel extract (*Malus sylvestris* Mill) with extract concentrations of 15%, 20%, and 25% had SPF values of 22.667, 26.511, and 31.22, respectively. These results can be interpreted if the SPF value produced has an ultra-protection value (SPF value > 15).

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