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Research Article

Physical Properties Investigation on Sunscreens with Red Dragon Fruit Peel Extract

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ABSTRACT

Red dragon fruit peel extract is reported as an active ingredient in sunscreen because of its ability to protect the skin. UV exposure to the skin can cause reddish skin to develop skin cancer. This study aimed to determine the characteristics of the cream made from ZnO and red dragon fruit peel extracts (*Hylocereus costaricensis*), such as sun protection factor (SPF), pH, stability, viscosity, spreadability, and adhesion. Red dragon fruit peel extract was obtained by maceration using methanol, and DPPH-scavenging activity resulted in an IC_{50} of 0.96 μg/mL. Measurement of diffuse UV-vis reflectance shows that ZnO has absorption at λex 385 nm, which correlates with a bandgap energy of 3.22 eV. F1 formulation cream (red dragon fruit peel extract and ZnO ratio of 0:1) has the highest SPF value of 17. The Kruskal-Wallis test shows that there is a significant difference in adhesion between the F1 formulation cream with F2 (p-value = 0.002) and F4 (p-value = 0.03).

INTRODUCTION

Indonesia is a tropical country with a high exposure to sunlight. Indonesian people who live along the equator need more skin protection compared to those who live in other regions. Sunlight spectrums such as ultraviolet light (UVA and UVB) have bad impacts on the skin (Svobodová et al., 2012). UVB radiation, for example, can cause skin cancer, while UVA radiation can reduce the elasticity of the epidermic layer of the skin, so it can cause photoaging (Jin et al., 2016; Shin et al., 2019; Yang et al., 2019). Both of these UV rays work synergistically, so protection is needed to prevent the adverse effects on the skin due to UVA and UVB radiation (Balakrishnan and Narayanaswamy, 2011). Sunscreen is used to prevent skin damage. Inorganic sunscreen such as zinc oxide (ZnO) has been used generally as sunscreen because of its ability to act as a barrier to UVA radiation, and its protection can be increased by combining it with titanium dioxide (Gutiérrez-Hernández et al., 2016; Kratošová et al., 2019). However, the addition of metal oxide concentrations to increase the effectiveness of sunscreens may hinder the ability of the product to spread evenly on the skin and cause white stains when the sunscreen is applied (Lusignan, 1998).

Inorganic sunscreens physically work by blocking a few or all of the UV radiation contemplated or scattered far from the surface of the skin (Sabzevari et al., 2021). Organic sunscreens work by absorbing a few or all of the UV radiation with the same purpose so that UV radiation does not reach the pores and the skin surface (Serpone et al., 2007). In this study, the sunscreen was made from inorganic filters such as zinc oxide and organic antioxidants (red dragon fruit peel). Red dragon fruit peel that has been analyzed in vivo can have different effects on oxidative free radical SOD, CAT, and glutathione-S-transferase enzymes. Red dragon fruit peel extract can be a natural antioxidant agent because of its health benefits (Jaiswal et al., 2013). Organic and inorganic ingredients are formulated and optimized along with other additives needed to make a stable o/w (oil in water) cream sunscreen. Sunscreen products are expected to protect the skin from exposure to UVA and UVB rays, repel water when applied while exercising or swimming, and be stable against light when used as filters or filter combinations. This study aims to make the optimum formulation of sunscreen cream with the addition of antioxidants from red dragon fruit peel extract and to characterize the physical properties of the resulting sunscreen cream.

METHODS

Manuscripts are written. All reagents, i.e., benzoic acid (pharmaceutical grade), stearic acid (pharmaceutical grade), hexadecane-1-ol (pharmaceutical grade), dimethicone (pharmaceutical grade), 2,2-diphenyl-1-picrylhydrazyl (Merck), ethanol (Merck), glycerin (pharmaceutical grade), isopropyl myristate (pharmaceutical grade), methanol (Merck), liquid paraffin (pharmaceutical grade), perfume, propylene glycol (pharmaceutical grade), spermaceti (pharmaceutical grade), triethanolamine (pharmaceutical grade), tween 80 (pharmaceutical grade), and zinc oxide (Merck) were of analytical grade and used without further purification. A UV-vis-spectrophotometer (JASCO V-730 UV-vis spectrophotometer) is used to measure the transmittance of the sunscreen.

Extraction

The dried powdered red dragon fruit peel (1.37 kg) was macerated with 1.6 L of 70% methanol in a container. The maceration was carried out for 96 hours at room temperature with occasional shaking. The extract was separately filtered. The filtrate was dried using a rotary vacuum evaporator. The crude extract was weighed.

DPPH Radical Scavenging Activity

The free radical scavenging activity of red dragon fruit peel extract was determined using the DPPH radical scavenging method. A series of volumes of extract (0.1, 0.2, 0.3, 0.5, and 0.7 mL) were added to 3.8 mL of DPPH in methanol (19.6 mg/L). After incubation at 37°C for 20 minutes, the absorbance of each solution was determined (λ = 517 nm) using a UV-vis spectrophotometer. The method is described in the works of Kesuma et al. (2020).

$$\%RSA = [\frac{A_T - A_E}{A_T}] \times 100$$

where AT is the absorbance of the control reaction and AE is the absorbance of the sample. The IC_{50} was calculated from the curve of percent scavenging plotted against the concentration.

Preparation of Emulsion of The Sunscreens

The lipid phase is composed of hexadecane-1-ol, spermaceti, and stearic acid. The mixture was melted at 85°C. When the temperature of the lipid phase and the liquid phase is equal, the two are mixed and stirred quickly so that they are evenly mixed. The composition of the emulsion is mentioned in Table 1.

Physical Evaluation of The Sunscreens

Sun Protection Factor (SPF) determination.
The sun protection factor of the sunscreens was determined by measuring the transmittance after passing through a film using a diffuse reflectance spectrophotometer. A rapid and reliable in vitro approach to calculating the SPF is to screen the transmittance of the product between 290 and 320 nm with an interval of 1 nm. Each formula was tested three times.

Table 1. The Compositions of The Sunscreens

No	Ingredients	Amount of each ingredient (%)				
		F1	F2	F3	F4	F5
1	Red dragon fruit peel extract	0	20	10	5	15
2	ZnO	20	0	10	15	5
3	Distilled water	31.8	31.8	31.8	31.8	31.8
4	Benzoic acid	0.1	0.1	0.1	0.1	0.1
5	Stearic acid	5	5	5	5	5
6	Dimethicone	1	1	1	1	1
7	Glycerin	5	5	5	5	5
8	Hexadecan-1-ol	5	5	5	5	5
9	Isopropyl myristate	5	5	5	5	5
10	Liquid paraffin	5	5	5	5	5
11	Perfume	0.1	0.1	0.1	0.1	0.1
12	Propylene glycol	10	10	10	10	10
13	Spermaceti	2	2	2	2	2
14	Triethanolamine	5	5	5	5	5
15	Tween 80	5	5	5	5	5

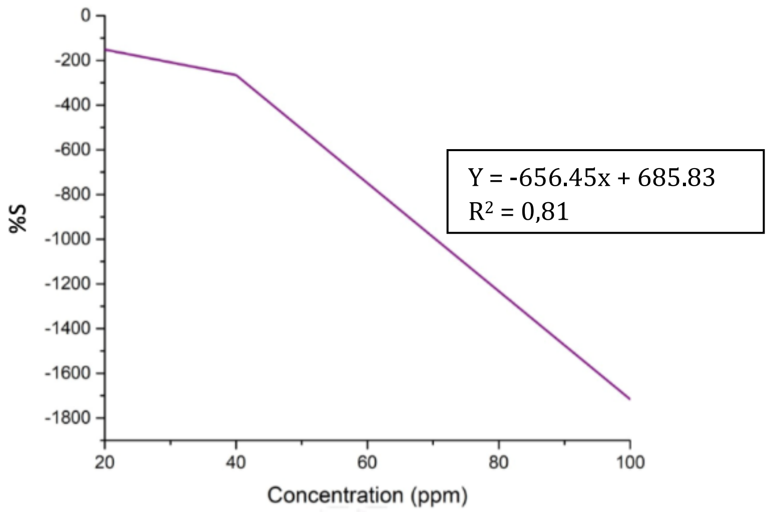

Figure 1. Antioxidant activity of red dragon fruit peel extract

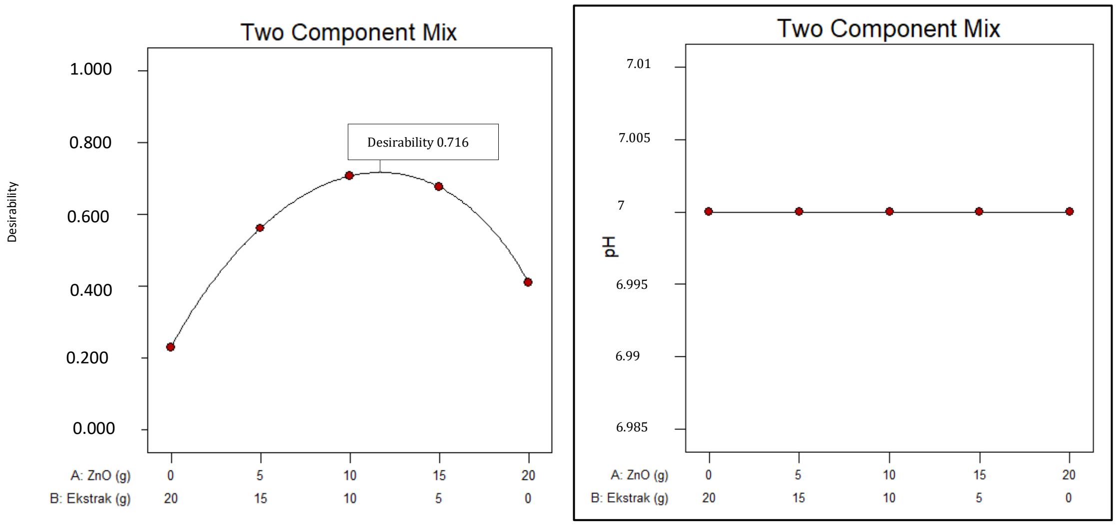

Figure 2. The SLD result of desirability and pH measurement of products

The SPF can be calculated using the Mansur equation (Malsawmtluangi et al., 2013):

$$\text{SPF} = \text{CF x} \sum_{290}^{320} \text{EE}(\lambda) \text{x I}(\lambda) \text{x Abs}(\lambda)$$

Spreadability
The spreading diameter of 0.5 g of a sample between two horizontal glass plates (10 cm x 20 cm) was measured after one minute. The standard weight applied to the upper plate was 0, 50, 100, 150, 200, 250, and 500 g. Each formula was tested three times (Chen et al., 2016).

pH values
Zero-point twenty-five gram of each formulation was dispersed in 10 mL of distilled water and pH was determined using a universal pH indicator. Each formula was tested three times (Chen et al., 2016).

Adhesion
Zero-point of one gram of a sample between two horizontal glass plates (2 cm x 2 cm). A weight of 1 kg was given to the glass plates for 5 minutes. Then, the glass plates were tied to the rope with the standard weight of 40 g on both sides. The time when the glass plates were separated was measured using a stopwatch. Each formula was tested three times (Kesuma et al., 2020).

Stability
One gram of each formulation was taken into 10 vials that were tightly closed. Five vials were used as controls kept at 25^{o}C, and five vials were

used for the freeze and thaw cycles with a storage temperature of 4°C in the first 12 hours and at a temperature of 40°C for the next 12 hours. These cycles were repeated three times. After three cycles, the organoleptic test, including physical appearance, color, texture, phase separation, homogeneity, and immediate skin feel of each formulation, has been conducted (Chen et al., 2016).

Viscosity

Viscosity measurements were carried out to see the viscosity of sunscreens produced due to the influence of the addition of other ingredients such as ZnO and red dragon fruit peel extract. Viscosity measurement using a cone and plate viscosimeter Stationary plates from the bottom of a removable sample cup are filled with 0.5–2.0 mL of sunscreen. The system is accurate within ± 1.0% of the full-scale range. Reproducibility: ± 0.2% The tool works in the temperature range of 0-100°C. Samples are placed in the sample cup, and the sample is ensured to be free of bubbles and spread evenly on the surface of the cup. Furthermore, the sample cup is reassembled on the viscometer, the viscometer is turned on, and then left for a while until the reading is stable (Chen et al., 2016).

Statistical Analysis

The results were reported as mean ± standard deviation. The average contents of adhesion, dispersion and SPF of sunscreens were statistically investigated using Statistic R 3.5.2. A statistical probability (p-value < 0.05) indicated a statistically significant result.

RESULTS AND DISCUSSION

DPPH Radical Scavenging Activity

The antioxidant activity of dragon fruit peel was measured using UV-vis spectrophotometer. It can be seen in Figure 1. The IC_{50} value is 0.96 μg/mL. This extract showed strong DPPH scavenging activity. Meanwhile, reported the IC_{50} value of red dragon fruit peel extract at 118 μg/mL (Wu et al., 2006).

Table 2. Physical Properties of The Sunscreens

Formula	Average value of SPF	Average value of adhesion	pH	Stability	Viscosity (Cp)
F1	17	3.84 s	7	stable	7.56
F2	15.6	73 s	7	less stable	12.92
F3	15.3	27 s	7	less stable	10.67
F4	16	44 s	7	less stable	37.19
F5	12.4	38 s	7	less stable	15.14

Table 3. Organoleptic Characteristics of Each Formula

Formula	Physical appearance	Color	Texture	Phase Separation	Homogeneity	Immediate skin feels
F1	Opaque	White	Smooth	No	Homogeneous	No grittiness, viscous, light, not greasy
F2	Transparent	Purple	Smooth	No	Homogeneous	Cool, not viscous, very greasy
F3	Transparent	Pale purple	Smooth	No	Homogeneous	Cool, less viscous greasy
F4	Transparent	Pale purple	Smooth	No	Homogeneous	Cool, viscous, greasy
F5	Transparent	Pale purple	Smooth	No	Homogeneous	Cool, less viscous, greasy

Table 4. Kruskal-Wallis Test of The Sunscreens

Response	*p*-value	Conclusion
SPF	0.01	Significant
Adhesion	0.02	Significant

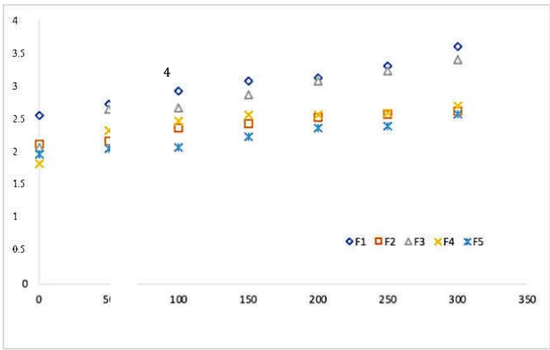

Figure 3. Spreadability values of the sunscreens

Optical Properties of ZnO

PXRD characterization was carried out to determine the morphology of zinc oxide crystals, as shown in Figure 2. The crystallinity of ZnO was 92%, with characteristic peaks at 2 Theta 31.64°, 34.3°, and 36.13°. ZnO crystallite size is 42.5 nm. Furthermore, characterization using the UV-vis spectrophotometer was used to determine the band gap energy (Eg) from ZnO. Zinc oxide has a band gap energy of 3.22 eV, which corresponds to the absorption at the wavelength of the UV region of 385 nm.

Physical Evaluation of The Sunscreens

Optimization of the ZnO sunscreen cream formula is done by varying the active ingredients, namely dragon fruit peel extract and ZnO, which are recommended by the Design Expert® program, as shown in Table 1. Prediction of the optimum formula is obtained from Design Expert® software version 7.0.0. Based on these predictions, the formula chosen with the highest desirability value is 1. The prediction of the resulting response is the SPF value, which is equal to 16 with a sticky power of 47 seconds. The optimum formula composition is the ratio of w/v ZnO and the extract of dragon fruit peels, respectively, 11.7 and 8.3.

Meanwhile, based on statistical results with Kruskal-Wallis, it was concluded that the data with a normal distribution and cream with F1 and F4 formulations were known to be better than F2, F3, and F5. The addition of dragon fruit peel extract to ZnO-based sunscreen cream is known to give similar results, where it can be seen that the SPF values produced are 17 and 16. In addition, there are differences in adhesion between creams F1 and F2 (p-value = 0.002) and F1 and F4 creams (p-value = 0.03).

Cream stability testing carried out by the freeze-thaw method is shown in Table 2. Based on observations, it is known that the cream with ZnO and fruit skin extracts that is 1:0 (F1) is stable, while other formulation creams are less stable. This is because in formulations F2, F3, F4, and F5, there are extracts from organic materials that are physically less stable.

The results of testing the spread of cream are shown in Figure 3. Based on the test results, it is known that creams with F1 and F3 formulations have the greatest spreadability. The increasing area of cream distribution may be caused by the decrease in viscosity of the cream.

CONCLUSIONS

The sunscreens were formulated with ZnO and red dragon fruit peel extract. The physical properties of the sunscreen have been investigated. The highest SPF value and the highest adhesion were obtained in F4. While the best spreadability was obtained in F1.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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