Formulation and Characterization of Sunscreen Lotion from Corn Cob

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Chapter 19: Formulation and Characterization of Sunscreen Lotion from Corn Cob (*Zea mays* L.) Extract

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Corn cob is the important type of agriculture residue. It contains phenolic compounds, protein, carbohydrates. We prepared and evaluated sunscreen lotion containing corn cob extract. Corn cob was extracted with reflux method using ethanol 80% for 8 hours. Several lotion containing 5% (F1), 7.5% (F2), and 10% (F3) extract was designed. These lotions were subjected to physical studies such as pH, viscosity, spreadability, adhesivity, organoleptic, and stability. The sun protection factors (SPF) were analyzed by ultraviolet (UV) spectrophotometry using samples irradiated with UV in B region. The results showed that the extract and formulations have minimum sunscreen protection. The extract showed SPF value was 4.95±0.86. The adding of corn cob extract in the lotion resulted in increasing SPF value of formulation. SPF values of F1, F2, and F3 were 1.20±0.06; 2.17±0.31; and 3.01±0.13, respectively. The formulation of sunscreen lotion containing corn cob extract meets the requirements of physical properties. The formulation containing 10% extract (F3) has the most effective sunscreen lotion. It showed the dark brown lotion with pH 7±0, viscosity 5244±20 cp, spreadability 5.06±0.02 cm, and adhesivity 1.0±0.04 s.

Keywords: *Zea mays* L., sunscreen lotion, sun protection factor

1.0 INTRODUCTION

A moderate sunlight exposure is well known to have some beneficial effects on the skin health. However, excessive exposure is also responsible for a destructive effect on the skin such as sunburn, skin cancer, aging, erythema, etc (Polefka et al., 2012). Sunlight radiation covers the ultraviolet (UV) to the infrared spectrum. The UV radiation from the UVA bands (320-400 nm) to the UVB (290-320 nm) is harmful to human skin. The carcinogenicity of UVB radiation is

well understood as a process of direct DNA photodamage. Instead, UVA radiation appears to act in indirect DNA photodamage.

Fortunately, the human skin has natural protection against sunlight radiation via the formation of melanin. The photoprotective role of melanin, especially eumelanin, is achieved by its ability to scatters UVR and reduces penetration of UV into dermal layers (Brenner and Hearing, 2008). However, the degree of melanin protection depends on the skin photo-type and age (Gonzales-Arjona et al., 2015).

The popular way to avoid the sunlight radiation's adverse effect was using sunscreen. Sunscreen is used to aid the body's natural defense to protect against UV-induced sunburns and its chemopreventive activity. Based on its ability to absorb, reflect or scatter the sun's rays, sunscreen protects the treated area from sunburn (Mishra et al., 2011). Sun Protection Factor (SPF) reflects sunscreen ability to prevent sunburn. Sunscreen is available in many forms like lotion, cream, gel, stick, spray, and lip balm. Lotions and creams were the most widely available and often used.

Now, the concept of alternative or complementary medicine appears to be increasing. The usage of herbal has been increased in product cosmetics. The researchers have claimed that herbal sunscreen cosmetics are more suitable for hyperallergic skin because they are less irritant and more easily adjustable to the skin. The role of herbal products and certain phytochemicals in the control of aging has been shown (Kapoor et al., 2009).

Several natural sunscreens, including aloe vera, tomato, pomegranate, green tea, cucumber, grapes, almond, saffron, have been reported to have UV filters. A few herbal oils including olive, coconut, castor, almond, mustard, chaulmoogra, sesame, peppermint, tulsi, lemongrass, lavender, orange, eucalyptus provides vital protection against harmful UV rays (Kaur and Saraf, 2010).

Corn cob is agriculture waste during corn grain harvest. It contains approximately 42.1% hemicellulose, 39.1% cellulose, 9.1% lignin, 1.7% protein, and 1.2% ash (Barl et al, 1991). Several lignans were identified as 7,7'-dihydroxy-3'-O-demethyl-4-methoxymatairesinol, β-sitosterol, β-sitosteryl-β-D-glucoside, 6β-hydroxycampest-4-en-3-one, 5α,8α-epidioxyergosta-6,22-dien-3β-ol, tricin, kaempferol and p-coumaric acid (Ashour et al., 2013). Previous studies suggested that corn cob extract exhibit antioxidant property (Dong et al., 2014; Wungkana et al, 2013; Fidrianny et al., 2016; Melo-Silveira et al., 2014; Sultana et al., 2007). The presence of phenolics including gallic acid, chlorogenic acid, cafeic acid, femlic acid, rutin, resveratrol, and kaempferol played important roles for strong antioxidant activities of corn cob (Dong et al., 2014). Furthermore, Lumempouw et al (2012) and Wungkana et al (2013) showed phenolic compounds in corn cob can act as a sunscreen. According to Dzialo's research, plant phenolics are promising metabolites in eliminating the causes and effects of skin disorders (Dzialo et al., 2016). There is a good correlation between SPF and antioxidant activity (Ebrahimzadeh et al., 2014).

Moreover, most people in rural areas cannot afford the expensive cosmetic products, they tend to use the natural resource for sun protection and beautification. Herbal products are cheaper than chemical based sunscreen, therefore an attempt has been made in the present study to prepare lotion sunscreen using corn cob. Present work reports the extraction, characterization, and formulation of sunscreen lotion of corn cob.

2.0 MATERIALS AND METHODS

2.1 Materials

The corn cob was collected in Purbalingga, Central Java, Indonesia and authenticated by taxonomist at the Faculty of Biology, Universitas Jenderal Soedirman, Indonesia. Ethanol, stearic acid, triethanolamine (TEA), and disodium edetate were purchased from Merck (Germany). Propylene glycol, methylparaben, propylparaben, cera alba, lanolin, and distilled water were from Brataco Chemica (Indonesia).

2.2 Preparation of Extracts

Corn cob was dried at room temperature for 1 week and made into a fine powder of 40 mesh size using the laboratory mill. Extraction of 750 g of powdered sample was performed by reflux using ethanol (80%) for 8 h at 78-90 ^{0}C. The extract was then separated from the sample residues by filtration through Whatman No. 1 filter paper. The resulting extract was concentrated over a rotary evaporator and concentrated extracts were dried to obtain actual yields. The extract was transferred into a dark glass bottle, and then stored at 4 ^{o}C.

2.3 Lotion Formulation

The ingredients used in the formulation of lotion are given in Table 1. The lotion was prepared by adding ingredients A to the ingredients B with fast stirring to avoid separation of water and oil phase. Ingredients A were first melted together and slowly added to the preheated mixture (70-82 ºC) of ingredients B. Finally, the volume was made up to 100 mL by adding distilled water. In the experiment, the commercial sunscreen Marina was employed as a positive control. An extract free lotion was also prepared as a negative control.

Table 1. Content of the Formulations (%, w/v)

Ingredients	Formula			
	I	II	III	K(-)
Ingredients A				
Corn cob extract (g)	3	4.5	6	-
Lanolin (g)	3.0	3.0	3.0	3.0
Cera alba (g)	2.50	2.50	2.50	2.50
Stearic acid (g)	4	4	4	4
Propylparaben (g)	0.050	0.050	0.050	0.050
Ingredients B				
Methylparaben (g)	0.10	0.10	0.10	0.10
Disodium edetat (g)	0.050	0.050	0.050	0.050
Propylene glycol (g)	5.0	5.0	5.0	5.0
Triethanolamine (g)	1.0	1.0	1.0	1.0
Distilled water (ml)	60	60	60	60

2.4 Organoleptic Characteristics

All formulations were tested for physical appearance including color, texture, and odor. These characteristics were evaluated by visual observation for 30 days, and characteristics assessed on the 1st, 2nd, 3rd, and 4th weeks.

2.5 pH Values

Lotion pH was measured with pH indicator stick. One gram of formulation was dispersed in 25 mL deionized water. Measurements were made in triplicate.

2.6 Viscosity Measurement

Viscosity was evaluated in Brookfield viscometer using an LV-2 spindle. The rotation rate was adjusted to 60 RPM. Viscosity was observed for 30 days and assessed on the 1st, 2nd, 3rd, and 4th weeks. All measurements were made in triplicate.

2.7 Spreadability

Spreadability of the lotion was determined by measuring the spreading diameter of 0.5 g sample between two circle glass plates (diameter 15 cm) after one minute. The standard weight applied to the upper plate was 50 g. The increase in the diameter due to lotion spreading was noted. Each formulation was tested three times.

2.8 Adhesivity

One gram of formulation was placed between two glass plates. The load (1 kg) was applied to the upper plate for 5 minutes. Further, the load was removed. Afterward, the plate was given a load release 80 g. The timing of the release of both plates was noted. Each formulation was tested three times.

2.9 Preliminary Study of Stability

The formulated lotion was stored at different temperatures of 27 ºC (at room temperature) and 4 ºC for a period of one month and studied for color, odor, and creaming.

2.10 Determination of SPF value

Efficacy of sunscreen lotion was measured by an in-vitro method using UV Visible spectrophotometer. 1.0 % solution (w/v) of corn cob extract, formulated sunscreen lotions, and commercial sunscreen lotions were prepared. 1.0 g of all samples were weighed, transferred to a 100 mL volumetric flask, and diluted to volume with ethanol. Further, it was kept for ultrasonication for 5 min and then filtered through a cotton filter, discarded the ten first mL. Afterward, A 5.0 mL aliquot was transferred to 50 mL volumetric flask and the volume was adjusted with ethanol. Then a 5.0 mL aliquot was transferred to a 25 mL volumetric flask and the volume completed with ethanol (Dutra et al., 2004). The absorption of samples in solution was measured in the range of 290 to 320 nm, every 5 nm, and the same was performed in triplicate for each sample, followed by the Mansur equation to calculate the SPF (Mansur et al., 1986).

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

Where, CF (correction factor) = 10, EE = erythemal effect spectrum, I (λ) = intensity of solar light of wavelength λ, Abs (λ) = spectrophotometric absorbance values at wavelength λ. The value of EE x I are constant and predetermined by Sayre et al. (2003).

2.11 Statistical Analysis

Data obtained was expressed as mean ± SD (standard deviation). The analysis of variance (ANOVA) test was used to assess if there were any difference in the data obtained. P-values less than 0.05 were considered statistically significant.

3.0 RESULTS

Ethanol has been widely used to extract compounds from plant sources. The extraction yields of corn cob by ethanol 80% is 11.4%. The extracts showed brown color, typical corn odor, and sweetish taste.

Three different lotions were formulated using different corn cob concentrations. The organoleptic properties, including texture, color, and odor of lotion formulations, are displayed in Table 2. Results showed that the lotion had smooth texture, a color from light to dark brown and characteristic odor of corn. The lotion indicated a homogeneous composition and no visible coarse grains. This was confirmed by visual examination and by touch. The lotions were easy to remove after application by washing with water. The formulations were able to produce uniform distribution of extracts in the lotion (Figure 1).

Tabel 2. Physical Characteristics of the Formulations

Formula	Texture	Color	Odor
I	Semisolid	Light brown	Characteristic of corn
II	Semisolid	Brown	Characteristic of corn
III	Semisolid	Dark brown	Characteristic of corn

All the fabricated lotions were then evaluated for their pH, viscosity, spreadability, and adhesivity. The results were tabulated in Table 3.

Tabel 3. Evaluation of Prepared Lotions

Evaluation Parameters	F1	F2	F3
pH	7±0	7±0	7±0
Viscosity (cps)	2756±15	6948±23	5244±20
Spreadability (cm)	7.55±0.04	5.51±0.02	5.06±0.02
Adhesivity (s)	0.36±0.01	0.74±0.04	1.00±0.04

The preliminary study of stability was observed under room temperature and cold conditions after the first month of storage. Results of the physical stability of lotion formulations are presented in Table 4. We only observed changes in the color and odor at cold temperatures. The color and odor of the formulation were considered stable under cold conditions. There is no creaming occurs on storage. However, the result showed that F1 requires more attention and protection from temperature changes and environmental stresses (Table 4).

Tabel 4. Evaluation of Prepared Lotions Stored at Room Temperature for One Month

Evaluation Parameters	F1	F2	F3
pH	8±0	7±0	7±0
Viscosity (cp)	3656±13	8396±25	6624±15
Color	No change	No change	No change
Creaming	No	No	No
Odor	No change	No change	No change

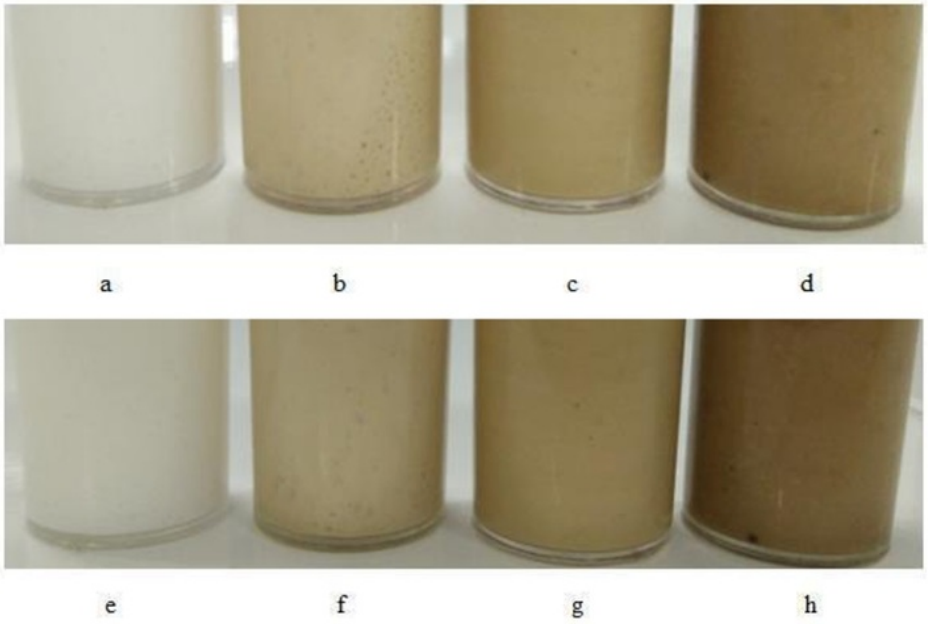

Figure 1. Formulated lotions: negative control (a), F1 (b), F3 (c), and F4 (d). After one month of storage: negative control (e), F1 (f), F3 (g), and F4 (h).

The extract of corn cob was tested in vitro at 1.0 % solution (w/v), and the SPF value obtained was 4.95±0.86. The absorbance values at various wavelengths (λmax) from 290 to 320 nm of extract are given in Figure 2. Furthermore, the lotion formulations with corn cob extracts were found to possess SPF in the range of 1.20 to 3.01. The results showed that the formulation F3 has better SPF value than other formulations (Table 5).

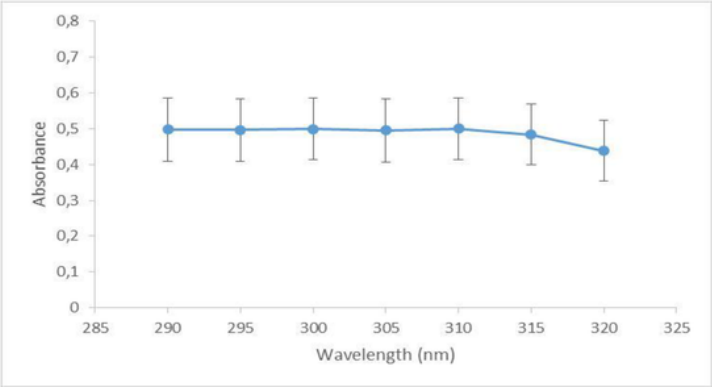

Figure 2. Absorbance Values of the Formulated Sunscreens

Tabel 5. SPF of the Extract and Formulated Lotion Sunscreen

Sunscreen	SPF
Extract	4.95±0.86
F1	1.20±0.07
F2	2.17±0.31
F3	3.01±0.13
Negative control	0.47±0.14
Positive control	7.32±0.18

4.0 DISCUSSION

People concerned with the environmental pollution are willing to spend more on environmentally friendly products, supporting the fast growth of the market for organic cosmetics. Plants are important sources of potentially useful constituents for the development of new therapeutic agents because most of them are safe with little side effects. Extraction of corn cob was conducted by reflux using ethanol as extraction solvent. Ethanol is permitted as solvents in the cosmetic and food industries. The reflux method is less time and solvent- consuming than maceration. Sharif and Bennett (2016) demonstrated that reflux extracts showed a better correlation between total phenolic content and antioxidant activity than the maceration extracts.

Corn cob extract gave a brown color to the formulations. The physical properties of formulations were assessed in accordance with the requirements of Standar Nasional Indonesia (SNI) for sunscreen No. 16-4399-1996.

A lotion is semisolid dosage forms proposed mainly for external use and usually consist of two immiscible phases, an oily internal phase, and an aqueous external phase. Because of emulsified nature of skin surface, extract formulated as lotion more effectively interact with skin

and more readily penetrate through biological membranes. Some of the plant extracts with sun protection activity have been previously formulated as topical lotions.

The lotions have the same texture and odor but slightly different in color. The color of lotions formulated was reported to vary from light brown to dark brown as extract concentration was increased from the lowest to the highest level. It was observed that the formulation previously stored in the room and cold temperature, after being kept for about one month remain stable in color and odor (Figure 1). This was good sign for lotion and it was acceptable.

The pH of formulations was 7 that fulfills with that of skin's normal pH value. All the formulations were shown pH nearer to skin required. Lotion that is too alkaline can cause skin to be sensitive and dry, even leading to skin conditions like eczema. Furthermore, lotion that is too acidic tends to irritate the skin. In general, the pH values of the formulations did not change significantly over the period of 4 weeks, except for the F1. During one month of room temperature storage, the pH of F1 increased slightly. Various amounts of lotion ingredients, as well as concentration of extract, could affect the pH of formulations. The increase in concentration of stearic acid causes the decrease of the pH of the formulation (Gyawali et al., 2016).

The viscosity of the formulated lotions is given in Table 3. The viscosity was found to be in the range of 2756 to 6948. The formulation F2 showed the highest viscosity (6948 cp) followed by F3 (5244 cp), and F1 (2756 cp). Also, higher viscosity of the lotion reveals more protection since microbial progression. Various amounts of lotion ingredients, as well as concentration of extract, could affect the viscosity. In this study, we used triethanolamine (TEA) as an emulsifier. Gyawali et al. (2016) demonstrated that triethanolamine have negative effect on the viscosity whereas, the stearic acid has positive effect on the viscosity. The increase in the concentration of triethanolamine decreases viscosity and the increase in concentration of stearic acid increase the viscosity. Furthermore, the viscosity of the formulations had increased after storage in room temperature condition. There was a significant difference in the viscosity values between F1, F2, and F3 ($p < 0.05$).

The ability of a lotion to uniformly spread on the skin plays an important role in the administration of a medicated formulation to the skin (Garg et al., 2002). The bioavailability efficiency of a lotion also depends on its spreading significance. The spreadability of lotions was found to decrease with increasing of corn cob extract. Spreadability was found to be in the range of 5.06 to 7.55 cm (Table 4). It has been found that the lotions have good spreadability (5-7 cm). Spreadability was closely related to viscosity. The rate of spreading depends on the viscosity of the sunscreen formulations (Garg et al., 2002). According to the statistical analysis, significant differences were observed between the spreadability values of negative control, F1, F2, and F3 ($p<0.05$).

Adhesivity measurement revealed that the formulated lotions possessed fairly good stickiness, which is essential for its application and retention on the skin. The adhesivity of

lotions was found to increase with increasing of corn cob extract. Results revealed that there was a significant difference in the adhesivity values between F1, F2, and F3 ($p < 0.05$).

The formulations were able to remain stable after incorporating the active ingredients, which is corn cob extract, into them. The stability study indicated that the lotion formulations were able to maintain their integrity for 4 weeks without showing signs of instability.

The effectiveness of a sunscreen lotion formulation to protect skin from sunburn and other skin damage can be evaluated by the SPF value. The predictable SPF values provide awareness to consumers for how long they can stay in the sun before sun burning. The higher the SPF value, the more effective is the sunscreen in preventing sunburn and other skin damage. A sunscreen lotion product should have a wide range of absorbance between 290 and 320 nm.

Determination of SPF of formulated corn cob extract sunscreen was conducted by in vitro method using ultraviolet (UV) spectrophotometer. Extraction of corn cob was conducted by reflux using ethanol as extraction solvent. Dutra et al. (2004) demonstrated that the method is highly suitable and reliable. Among samples analyzed, the extract of corn cob exhibits a maximal absorbance higher than sunscreen lotion formulations, as it can be shown in Table 5. The extract has medium SPF of 4.95±0.86, which considered being medium protection sunscreen. The formulation containing 10% extract (F3) has the higher SPF values than the other formulations. This is probably due to the fact that F3 has a total amount of corn cob extract higher than the other formulations. The presence of photo-shielding phenolic makes it possible for the lotion formulations to get protection from UV solar light (Dong et al., 2014; Lumempouw et al., 2012; Wungkana et al., 2013). The lotion formulations studied have minimum protection sunscreen. According to the statistical analysis, significant differences were observed between the SPF values of negative control, F1, F2, and F3 ($p<0.05$). This study clearly indicated that formulations which have corn cob plant extracts were more potent than the base.

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