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Photoprotective Cream Containing Brown Seaweed (*Sargassum* Sp.) Extract: Optimization By Simplex Lattice Design

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Abstract: Sunlight containing ultraviolet (UV) rays is harmful to human skin health. Sunscreen is one of the trendy and attractive cosmetics. Brown seaweed extract (*Sargassum* sp.) has the potential to be developed into a sunscreen product. This study aimed to optimize the formula of a cream containing brown seaweed extract and its in vitro photoprotection. A simplex lattice design approach was used to factor (triethanolamine and stearic acid) and responses (pH, viscosity, adhesiveness, and spreadability). The concentration of variation in stearic acid and triethanolamine affects the parameters of pH response, viscosity, adhesion, and spreadability. The simplex lattice design predicts the optimal formula with stearic acid (17%) and triethanolamine (4%). The verification proved that no significant difference between the predicted and actual results. The in vitro photoprotective evaluation results indicate that the brown seaweed extract cream is categorized as providing high sun protection and serving as a sunblock. Optimization of the brown seaweed extract cream formula using stearic acid and triethanolamine factors has been successfully achieved. The conclusion is that brown seaweed extract cream has the potential to be developed into a sunscreen cosmetic.

Introduction

Sunlight containing ultraviolet (UV) rays is harmful to human skin health. It can cause cancer, hyperpigmentation, and erythema (1). UV rays reach the Earth's surface, with approximately 95% being UVA, 10% being UVB, and UVC being almost entirely absorbed by the ozone layer (2). Therefore, the use of sunscreen is one of the trendy and attractive solutions (3). Chemical sunscreens have been studied previously and have been found to have relatively high toxicity effects, such as those associated with oxybenzone, ethylhexyl methoxycinnamate, homosalate, and octisalate (4). Natural sources are known to have low side effects. The use of natural sunscreens has become an alternative to replace chemical sunscreens (5). Natural sunscreen can be obtained from marine sources such as artemia, plankton, algae, *L. ochroleuca*, *T. thermophillus* ferment, *N. gaditana, Spirulina platensis* (6)

Brown seaweed (*Sargassum sp.*) contains flavonoids, alkaloids, terpenoids, tannins, and saponins (7, 8). Previous research has shown that brown seaweed extract has a total phenolic content of 149.04 ± 5.14 mg GAE/g. A concentration of 1.6% brown seaweed extract has a sun protection factor value of 33.2 ± 3.11 (9). Flavonoid compounds have activity as sunscreens. Sun protection factor (SPF) is an indicator used to assess a material's ability to protect the skin from sunlight radiation (10). The SPF value of brown seaweed extract is categorized in the high sun protection product category (SPF > 30) (11). From these various backgrounds, brown seaweed extract needs to be formulated into a sunscreen cream.

The physical properties of the cream are influenced and controlled by the proportion of the emulsifier (12). The use of a combination of stearic acid and triethanolamine as emulsifiers produces a homogeneous and stable cream (13). However, stearic acid and triethanolamine exhibit opposite properties in terms of cream characteristics, including pH value, viscosity, spreadability, and adhesiveness (14). Therefore, the proportion of stearic acid and triethanolamine requires further study in brown seaweed extract cream. Simplex lattice design is an optimization method used to determine the optimum formula for a mixture of ingredients (15). Optimization of the proportion of stearic acid and triethanolamine in sunscreen cream can be achieved using the simplex lattice design method (16).

Sunscreen cream containing brown seaweed extract is an alternative to overcome the dangers of UV rays. This study aimed to optimize the stearic acid and triethanolamine content in the brown seaweed extract cream formula using the simplex lattice design method. The optimization responses used were pH, viscosity, spreadability, and

adhesiveness. Additionally, evaluating the photoprotective ability requires assessing parameters such as SPF value, erythema transmission percentage (%Te), and pigmentation transmission percentage (%Tp).

Methodology

Materials

Brown seaweed collected from the coast of Suraga Village, Cinangka District, Serang Regency, Banten Province, Indonesia. Etanol 96% (PT. Jayamas Medica Industri Tbk, Indonesia), distilled water (PT. Brataco, Indonesia), triethanolamine (Petronas Chemicals Marketing (Labuan), Ltd., Malaysia), stearic acid (PT. Wilmar Nabati Indonesia), cetyl alcohol (PT. Ecogreen Oleochemicals, Indonesia), glycerin (PT. Wilmar Nabati Indonesia, Indonesia), propylene glycol (SK Picglobal, South Korea), methylparaben (Ueno Fine Chemicals Industry, Ltd., Japan), and propylparaben (Ueno Fine Chemicals Industry, Ltd., Japan).

Preparation of Brown Seaweed Extract

Brown Seaweed dry powder was macerated for 3 days using 96% ethanol (ratio 1:10) with occasional stirring. The extract was concentrated using a rotary evaporator (50 °C, 50 rpm) and a water bath. Remaceration was carried out to obtain maximum extract yield.

Design of Experiment

Optimization of the stearic acid and triethanolamine proportion was conducted using the simplex lattice design approach, employing Design-Expert software version 13 (trial edition). Lower and upper concentration limits were set at 17-19% for stearic acid and 2-4% for triethanolamine. The simplex lattice design experiment process suggested 8 formulas are shown in **Table 1**. The responses for the optimum formula were pH, viscosity, spreadability and adhesiveness.

Formulation of Brown Seaweed Extract Cream

The oil phase, consisting of stearic acid, liquid paraffin, cetyl alcohol, and propyl paraben, was melted in a beaker and heated on a hot plate at a temperature of 60–70 °C with continuous stirring until a homogeneous mixture was obtained. Simultaneously, the water phase, comprising triethanolamine (TEA), methyl paraben, propylene glycol, and distilled water, was prepared in a separate beaker and stirred until it was uniform. The water phase was then gradually added to the oil phase with constant stirring until a thick cream mass was formed. Once the mixture cooled to approximately 45 °C, brown seaweed extract was incorporated into the cream base with gentle stirring until a homogeneous formulation was achieved.

Physical Evaluation of Brown Seaweed Extract Cream

pH Test

The cream's pH measurement was performed using a calibrated pH meter. The electrode was immersed in the cream, and the pH results were displayed on the pH meter screen (17).

Spreadability Test

A portion of the cream was placed at the center of a transparent glass plate and covered with another transparent glass plate for 1 minute. A load of 250 grams was then applied to the top plate and left in place for an additional 1 minute. Afterwards, the diameter of the resulting spread was measured to assess the spreadability of the cream (18).

Adhesiveness Test

A portion of the cream was placed on a glass object and covered with another glass object. A load of 500 grams was applied to the top glass object and maintained for 5 min to allow adhesion. Subsequently, a load of 80 grams was suspended, creating a downward force that pulled the bottom glass object. The time required for the two glass objects to separate was recorded to evaluate the adhesiveness of the cream (17, 19).

Viscosity Test

Viscosity measurements were performed using a digital Brookfield viscometer (model BDV-9s). The appropriate spindle was immersed in the cream sample and allowed to equilibrate. Viscosity readings were then recorded directly from the instrument’s display (20).

Simplex Lattice Design Analysis

The simplex lattice design method was employed to optimize the cream formulation by varying the proportions of stearic acid and triethanolamine (14). The measured responses included pH, viscosity, adhesiveness, and spreadability. The lack-of-fit value was used to assess the suitability of the selected model in representing the response data (21). The optimum formulation was predicted based on the desirability value, followed by verification through comparison of predicted and observed results (22). The optimized cream

Table 1. Brown seaweed extract cream formula.

Ingredients	Concentration (%)							
	F1	F2	F3	F4	F5	F6	F7	F8
Brown seaweed extract	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6
Stearic acid	**18**	**17.5**	**19**	**19**	**17**	**17**	**18.5**	**18**
Triethanolamine	**3**	**3.5**	**2**	**2**	**4**	**4**	**2.5**	**3**
Propylene glycol	10	10	10	10	10	10	10	10
Propyl paraben	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Cetyl alcohol	4	4	4	4	4	4	4	4
Liquid paraffin	5	5	5	5	5	5	5	5
Methyl paraben	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Distilled water ad	100	100	100	100	100	100	100	100

$$\mathrm{SPF} = \mathrm{CF} \times \sum_{290}^{320} \mathrm{EE} \times \mathrm{I} \times \mathrm{Abs}$$

Equation 1 | *CF* is the correction factor (set at 10), *EE(λ)* is the erythemal effect spectrum, *I(λ)* is the solar intensity spectrum, and *Abs(λ)* is the absorbance of the sample at each wavelength. The product *EE* × *I* is considered constant for each wavelength interval.

$$\%\mathrm{Te} = \frac{\mathrm{Ee}}{\Sigma\,\mathrm{Fe}} = \frac{\Sigma\,(\mathrm{T} \times \mathrm{Fe})}{\Sigma\,\mathrm{Fe}}$$

Equation 2 | Product of transmission (*T*) and erythemal effectiveness (*Fe*) at each wavelength, then dividing by the total erythemal effectiveness (*ΣFe*), where *Ee* is the effective erythema energy transmitted.

$$\%\mathrm{Tp} = \frac{\mathrm{Ee}}{\Sigma\,\mathrm{Fp}} = \frac{\Sigma\,(\mathrm{T} \times \mathrm{Fe})}{\Sigma\,\mathrm{Fp}}$$

Equation 3 | Product of transmission (*T*) and pigmentation effectiveness (*Fp*) at each wavelength, then dividing by the total pigmentation effectiveness (*ΣFp*), where *Ee* is the effective pigmentation energy transmitted.

formulation was further evaluated for *in vitro* photoprotective activity.

In Vitro Photoprotective Evaluation

The *in vitro* photoprotective evaluation was conducted by determining the Sun Protection Factor (SPF), the percentage of erythema transmission, and the percentage of pigmentation transmission. The cream was dissolved in 96% ethanol to obtain a 1% solution (23). SPF determination was carried out using UV spectrophotometry, following the method developed by Mansur (24). The absorbance of the sample was measured across wavelengths ranging from 290 to 320 nm at 5 nm intervals (10). SPF values were calculated using **Equation 1**.

The cream was dissolved in 96% ethanol to obtain a 1% solution. The erythema transmission percentage (%Te) and pigmentation transmission percentage (%Tp) were determined using the equations developed by Cumpelik (25, 26). The %Te was measured over the wavelength range of 292.5–337.5 nm, while the %Tp was measured over 322.5–372.5 nm. These calculations are presented in **Equations 2** and **3**. In the equations, %Te represents erythema transmission, Fe denotes the flux of erythema (considered constant), and %Tp represents pigmentation transmission.

Results and Discussion

Preparation of Brown Seaweed Extract

Brown seaweeds are distributed globally, with greater abundance in shallow, rocky coastal regions and are typically harvested during low tide (27). Due to their availability and bioactive potential, brown seaweeds have strong prospects for development and industrial applications. In this study, the seaweed was processed into a dry powder to reduce moisture content, thereby inhibiting microbial growth. The powdered form also increases the surface area of the simplicia particles, enhancing solvent contact and penetration, which facilitates the extraction of more compounds. The extraction process yielded 2.34%, which is lower than that reported in previous studies (6.67%) (9). Factors such as extraction temperature, duration, and milling method are known to influence yield (28). The resulting extract exhibited a greenish color, a thick consistency, and a characteristic odor reminiscent of seaweed.

Formulation of Brown Seaweed Extract Cream

All formulations produced homogeneous cream with a green color, a characteristic seaweed odor, and a soft texture. The presence of brown seaweed extract contributed to the cream's distinctive color and scent. The cream formulation was adapted from previous research with several modifications (29). Stearic acid and triethanolamine were used in combination as emulsifying agents to form a stable oil-in-water (O/W) emulsion. The evaluation of the cream preparation was conducted to ensure compliance with pharmaceutical standards and to assess the quality and suitability of each formulation. These results were further used to identify the optimal formulation of the brown seaweed extract cream. The creams demonstrated good homogeneity, indicated by a uniform color and the absence of visible particles (30). Homogeneity refers to the even distribution of all components within a preparation. It is influenced by factors such as the solubilization of ingredients and the effectiveness of the stirring process, which ensures the active ingredients are uniformly dispersed throughout the cream.

Physical Evaluation of Brown Seaweed Extract Cream

pH Test

The pH of the cream formulation was measured to assess its compatibility with the skin's pH. Creams with low pH values (1-4) can cause skin irritation, while those with high pH values (8-14) may lead to skin dryness. The acceptable pH range for topical creams is 4.5–6.5 (31). The results of the pH measurements are presented in **Table 2**. Analysis using the simplex lattice design was performed, and the corresponding ANOVA results are shown in **Table 3**. Based on the ANOVA analysis, a linear model was selected to evaluate the pH response, with a significant p-value of 0.0001 ($p < 0.05$). The lack-of-fit test yielded a p-value of 0.2628 ($p > 0.05$), indicating that the model was not significantly different from pure error. Thus, there was no significant discrepancy between the observed and predicted data. The resulting **equation from** the simplex lattice design is shown in **Table 4**.

According to the equation, triethanolamine was found to have a dominant effect on increasing the pH compared to stearic acid. The response surface graph is presented in **Figure 1**, illustrating that increasing the concentration of stearic acid results in a more acidic formulation, while higher levels of triethanolamine yield a more alkaline pH. These findings are consistent with previous research indicating that triethanolamine acts as an alkalizing agent, whereas stearic acid contributes to acidity (32, 33). Chemically, triethanolamine ($C_6H_{15}NO_3$) contains a tertiary amine functional group, which imparts basic properties (34). In contrast, stearic acid contains a carboxyl group (-COOH), classifying it as a carboxylic acid (35).

Table 2. Physical evaluation of brown seaweed extract cream.

Characterization	F1	F2	F3	F4	F5	F6	F7	F8
pH	5.39	5.42	4.86	4.83	6.02	5.96	4.93	5.11
Spreadability (cm)	5.48	5.49	5.17	4.47	5.99	5.69	5.30	5.36
Adhesiveness (second)	7.64	7.51	8.76	8.95	6.71	6.86	8.35	7.71
Viscosity (dPas)	592.26	396.44	990.86	988.82	247.52	203.26	790.53	592.43

Table 3. Results of the p-value model and lack of fit.

Response	P-value	
	Model	Lack of Fit
pH	0.0002	0.2628
Spreadability (cm)	0.0061	0.8933
Adhesiveness (second)	0.0001	0.2267
Viscosity (dPas)	0.0001	0.6646

Table 4. Response equations and models.

Response	Equation	Model
pH	Y = 0.172619(X1) + 0.735952(X2)	Linear
Spreadability (cm)	Y = 0.187877(X1) + 0.662321(X2)	Linear
Adhesiveness (second)	Y = 0.516726(X1) - 0.496607(X2)	Linear
Viscosity (dPas)	Y = 83.37595 (X1) - 300.16738(X2)	Linear

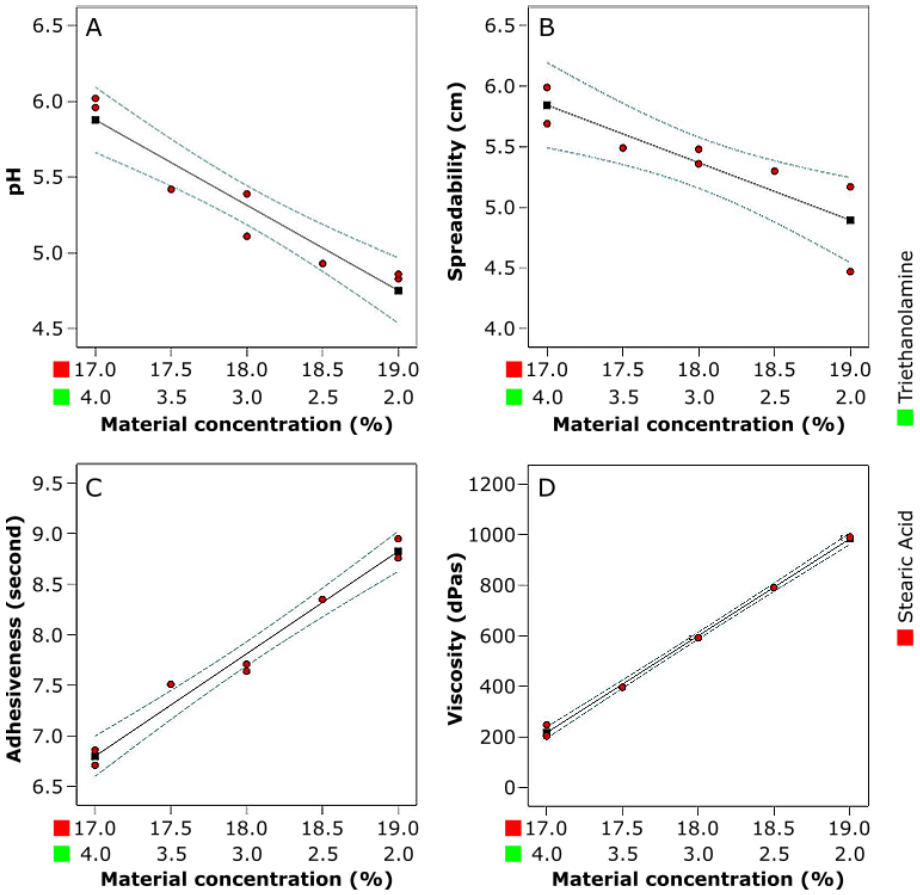

Figure 1. Response model of formula containing a mixture of stearic acid and triethanolamine with different ratio. Note: (A) pH, (B) spreadability, (C) adhesiveness, and (D) viscosity.

Table 5. Best formula prediction results.

Desirabilty	Stearic acid (%)	Triethanolamine (%)	pH	Spreadability (cm)	Adhesiveness (second)	Viscosity (dPas)
0.852	17.000	4.000	5.878	5.843	6.798	216.023

Table 6. Prediction and actual verification results.

Response	Prediction	Actual	P-Value
pH	5.878	5.756±0.050	0.840
Spreadability (cm)	5.843	5.680±0.495	1.000
Adhesiveness (second)	6.798	6.798±0.220	0.981
Viscosity (dPas)	216.023	247.926±0.055	1.000

Table 7. Photoprotection evaluation of brown seaweed extract cream.

Evaluation	Result	Categories
SPF	21.679±2.281	High sun protection
%Te	0.636±0.164	Sunblock
%Tp	0.468±0.250	Sunblock

Spreadability Test

The spreadability test was conducted to assess the ability of the cream to spread evenly when applied to the skin (36). Adequate spreadability ensures a uniform distribution of active ingredients on the skin, thereby enhancing their therapeutic efficacy. A linear model was selected to evaluate the spreadability response, as indicated by a significant p-value of 0.0061. The lack-of-fit test yielded a p-value of 0.8933, indicating no significant deviation from pure error and confirming the model's suitability.

According to the model, triethanolamine had a greater influence on increasing spreadability compared to stearic acid. The response surface graph, shown in **Figure 1**, illustrates that increasing the concentration of stearic acid reduces spreadability, while higher levels of triethanolamine enhance it. These findings align with previous studies, which report that stearic acid decreases spreadability due to its ability to increase viscosity by forming complexes with other formulation components (37). Spreadability is closely related to viscosity—higher viscosity typically results in lower spreading ability. Triethanolamine, being part of the water phase, has a lower viscosity compared to oil components, thereby contributing to improved spreadability (38).

Adhesiveness Test

The adhesiveness test was conducted to determine the duration the cream remains adhered to the skin surface (39). Adequate adhesiveness ensures that the cream remains in place, allowing for prolonged contact with the skin and optimal therapeutic effect. The minimum acceptable adhesion time for topical creams is greater than 4 s. A linear model was selected to evaluate the adhesiveness response, as indicated by a significant p-value of 0.0001. The lack-of-fit test produced a p-value of 0.2267. According to the model, stearic acid was found to be more influential in increasing adhesiveness compared to triethanolamine. Increasing the concentration of stearic acid leads to higher adhesiveness, whereas increasing the concentration of triethanolamine reduces it. Stearic acid, a saturated fatty acid, contributes to the formation of a hydrophobic layer that enhances the cream's ability to adhere to the skin (40). In contrast, triethanolamine is more hydrophilic (41), which may reduce the cream's adhesion by promoting faster dispersion or absorption, thereby lowering its retention time on the skin.

Viscosity Test

The viscosity test was conducted to determine the viscosity of each cream formulation, as appropriate viscosity is essential for ease of application and spreadability on the skin (42). The acceptable viscosity range for topical creams is between 50 and 1000 dPas. A linear model was selected to evaluate the viscosity response, supported by a significant p-value of 0.0001. The lack-of-fit test produced a p-value of 0.6646 ($p > 0.05$), indicating that the model was not significantly different from pure error, and thus accurately represented the observed data.

According to the model, stearic acid had a greater effect on increasing viscosity compared to triethanolamine. Increasing the stearic acid concentration led to higher viscosity, while increasing the triethanolamine concentration reduced viscosity. These findings are consistent with previous studies, which have shown that stearic acid can form complexes with other ingredients, contributing to increased viscosity (38). In contrast, triethanolamine is part of the aqueous phase, and since water has a lower viscosity than oil, its presence tends to reduce the overall viscosity of the emulsion.

Simplex Lattice Design Analysis

The simplex lattice design analysis considered the lack-of-fit results for each response variable. As previously described, the lack-of-fit values indicated that all response models were valid and could be used for further analysis in determining the optimal formulation. Using the desirability function approach, the optimization results identified the optimum formulation consisting of 17% stearic acid and 4% triethanolamine. These results were obtained from the desirability value generated by Design Expert software, where a desirability value approaching 1 indicates a highly optimized formula. This value reflects the predicted combination of the independent variables (stearic acid and triethanolamine) concerning the targeted responses: pH, spreadability, adhesiveness, and viscosity. The predicted response values for the optimal formulation are presented in **Table 5**.

Desirability serves as a function value that evaluates the software's ability to meet the desired criteria for the final product. To validate the optimization, verification was performed by comparing the predicted and experimental (actual) values using statistical analysis. The verification

results, shown in **Table 6**, indicate no significant difference between the predicted and observed values, as evidenced by p-values greater than 0.05. This confirms the reliability of the model in predicting the optimal cream formulation.

In Vitro Photoprotective Evaluation

In vitro photoprotective evaluation was carried out using the verification formula. The results of the photoprotective test are shown in **Table 7**. Photoprotective evaluation was conducted *in vitro* using spectrophotometry. Evaluation used parameters of SPF value, %Te and %Tp. The results of the photoprotective evaluation are shown in **Table 7**.

The SPF value obtained from the cream formulation indicates that it falls within the high sun protection category, defined as a sunscreen product with an SPF of 30 or above. The percentage of erythema transmission (%Te) and pigmentation transmission (%Tp) represent the proportion of ultraviolet radiation that penetrates the sunscreen and reaches the skin, potentially causing erythema and pigmentation, respectively (43). In this study, the %Te and %Tp values of the cream containing brown seaweed extract were found to meet the criteria for the sunblock category, characterized by %Te < 1% and %Tp ranging from 2% to 40% (44).

Brown seaweed is known to contain flavonoids (7), which contribute to photoprotection due to their ability to absorb ultraviolet (UV) radiation (45). Flavonoids possess aromatic chromophores (46), which are molecular structures capable of absorbing specific wavelengths of UVA and UVB radiation (47, 48). These chromophores play a critical role in mitigating UV-induced skin damage, thus supporting the potential of brown seaweed extract as a natural and effective sunscreen agent.

Conclusion

Optimization of the cream formula containing brown seaweed extract has been successfully achieved. Simplex lattice design analysis shows that there is an influence of stearic acid and triethanolamine on the responses (pH, viscosity, adhesiveness, and spreadability). The optimal formula was obtained with the proportion of stearic acid (17%) and triethanolamine (4%). The results of *in vitro* photoprotector evaluation showed that the cream has promising potential as a herbal sunscreen product.

Declarations

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability

The unpublished data is available upon request to the corresponding author.

Ethics Statement

Not applicable.

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Additional Information

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