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In-vitro Sun Protecting Factor of Rice Bran Oil and Its Formulation as Compact Powder

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ABSTRACT: This study aims to determine the effective concentration of rice bran oil (RBO) which can protect against ultraviolet (UV) rays as well as formulate it in compact powder preparation followed by determination of its Sun Protecting Factor (SPF) value. Rice bran samples were extracted using the Soxhlet extraction method with n-hexane: ethanol (1:1) as the solvent. Further identification of the γ-oryzanol in RBO was carried out using TLC silica gel GF254 with eluent of n-hexane:ethyl acetate (3:1). The obtained γ-oryzanol in RBO was used as the ingredient to develop the compact powder which is made into five formulas with 0.05 %-0.25 % concentration. All formulas were characterized, including homogeneity, adhesion and crack test. UV-Visible spectrophotometry was used to determine the SPF value of RBO and its compact powder. The identification results demonstrated a positive presence of the chemical γ-oryzanol in the RBO. At concentrations of 500, 1000, 1500, 2000, and 2500 ppm, RBO may protect against UV rays with SPF values ranging from 1.741 to 11.884. The result showed that all formulas dispersed homogeneously, performed well in terms of compactness, and had no breaks or cracks discovered. Meanwhile, the SPF from five variation of compact powder formula showed value in the range of 1.390- 2.178. The effective concentration of RBO from five concentration variations to protect against ultraviolet (UV) rays is 2500 ppm. However, the developed formulation compact powder using RBO 2000 ppm shows an SPF value that is sufficient to protect the skin from sun exposure which is included in shallow protection and are included in the minimal SPF category (2-4).

Keywords: **rice bran; γ-oryzanol; compact powder; SPF.**

Introduction

Cosmetics that contain sunscreen have the aim to protect the human body from harmful ultraviolet (UV) rays, prevent or minimize damage caused by the radiation to human health such as actinic ageing or cutaneous cancer [1]. Sun Protecting Factor (SPF) evaluates the effectiveness of product containing sunscreen compounds. SPF is the ratio between the time needed to cause erythema on the skin protected by sunscreen with the not protected one. This SPF value ranges from 0 to 100, and the ability of sunscreens considered good is above 15 (SPF15), which it can be filters about 94% of UVB rays [2].

Rice bran is a component of raw rice that is discharged from the endosperm portion obtained during the rice milling process. Rice bran oil (RBO) is a unique component among vegetable oils because it is rich in commercial sources. It contains essential nutrients such as oryzanol, lecithin, tocopherol, and tocotrienols [3]. γ-oryzanol has biological activity, especially antioxidants, and is often used in cosmetic formulations as a sunscreen [4,5]. It acts as a protective agent against UV rays caused by lipid peroxidation and can, therefore, be used as a sunscreen agent [6].

Face powders are a cosmetic preparation used to cover up minor flaws in the skin and improve facial appearance by covering the shiny skin due to moisture or grease from the secretion of sebaceous and sweat glands or preparations used on the skin. The essential properties of face powder are to mattify oily skin and have a characteristic of smooth feeling for a long time. Therefore, the preparation must be able to adhere for a long time, so no need for frequent application [7].

There are two primary forms of face powder, which can be classified into loose powder and compact powder. Loose powder has a sort of fine powder, soft texture, homogeneous so that it is easily sprinkled or evenly applied to the facial skin by a puff or large brush. Meanwhile, compact powder is a powder that is pressed into a cake

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compacted shape, soft, homogeneous, and easily applied evenly on the skin with a puff. This compact powder composition is similar to loose powder, but a binder is added to the preparations. The binders enhance their adhesion to the skin and to make the pressed powder compact. In addition, compact powder forms are best used because of their ease of application and practical packaging [8].

The face powders are made using a blend of ingredients such as talc, kaolin, magnesium stearate, zinc oxide, titanium oxide, and pigments. Additional ingredients that can be added such as preservatives, fragrance, and UV filters, namely materials that can protect against sunlight [9].

Therefore, the main purpose of the present study is to determine the effective concentration of bran oil (*Oryza sativa* L.) which can provide protection against UV rays and formulate it to compact bran oil powder as well as assess its effectiveness based on SPF values.

Materials and Methods

Materials

Rice bran (*Oryza sativa* L.) was collected from Masamba Village, Poso Coastal District, Poso Regency. γ-oryzanol (TCI, Japan), ethyl acetate (Merck®, Germany), n-hexane (Merck®, Germany), ethanol 96% (Merck®, Germany), kaolin (Bratachem, Indonesia), magnesium carbonate (Bratachem, Indonesia), magnesium stearate (Bratachem, Indonesia), nipagin, corn starch (Bratachem, Indonesia), titanium dioxide (Bratachem, Indonesia), BHA, Crodamol™ ISIS (Croda Inc, UK), silicon oil and talcum (Bratachem, Indonesia). All the reagent used were analytical grade and commercially available.

Plant Determination

Determination is carried out at the Indonesian Institute of Sciences, Bogor Botanical Gardens Conservation Center.

Rice Bran Preparation

Rice bran samples have been collected, wiped from dirt, then washed and rinsed using running deionized water until clean and then dried by aerating in the open air for 2 days to prevent enzymatic decomposition [10].

Soxhlet Extraction

The soxhlet extraction was carried out from 1.2 kg of dried rice bran using a mixture n-hexane:ethanol (1:1). The rice bran samples were wrapped in filter paper, then inserted into a thimble, and kept immediately for extraction in the soxhlet extractor. The soxhletation was carried out for 6 hours. The collected liquid bran extract was concentrated in a rotary vacuum evaporator (Eyela® OSB-2100, Malaysia). Then, the obtained rice bran oil (RBO) was weighed [11].

Identification of γ-Oryzanol Compounds in TLC Plates

The RBO and γ-oryzanol were dissolved in ethanol and n-hexane (1:1). RBO and γ-oryzanol solution were spotted on GF254 TLC silica gel plates then dried at 37 °C. After drying, it was eluted using n-hexane: ethyl acetate (3:1). The results were observed under UV (254 and 366 nm) [12].

Determination of The In Vitro SPF of Rice Bran Oil

Determination of in vitro SPF of RBO using the UV-Visible spectrophotometric method [13]. RBO (1.0 g) was transferred to volumetric flask (size 100 ml) subsequently diluted with ethyl acetate. The solution was stirred until mixed and then filtered with membrane filter (stock solutions). A quantitative solution of 0.25 ml, 0.5 ml, 0.75 ml, 1.0 ml, and 1.25 ml were pipetted, transferred to volumetric flask (size 5 ml) and then diluted with ethyl acetate to obtain a solution with a concentration of 500 ppm, 1000 ppm, 1500 ppm, 2000 ppm and 2500 ppm. The absorbance was measured by a UV-Visible spectrophotometer (Spectroquant®, Germany) at UV B wavelengths (280-310 nm) and UV A (320-400 nm). The SPF value was determined by calculating the area under curve (AUC) at a wavelength of 290-400 nm with an interval of 10 nm. AUC was calculated according to equation [14]:

$$[AUC]_{\lambda_{p-a}}^{\lambda_p} = \frac{A_{p-a} + A_p}{2} \times (\lambda_p - \lambda_{p-a})$$

where:

A_{p-a} = absorbance at the lower wavelengths between consecutive wavelengths

A_p = absorption at a higher wavelength between two consecutive wavelengths

λ_p = higher wavelengths between two consecutive wavelengths

λ_{p-a} = lower wavelengths between two consecutive wavelengths

The SPF value of each concentration was determined according to equation [14]:

$$\text{Log SPF} = \frac{\sum \text{AUC}}{\lambda_n - \lambda_1}$$

where:

$\sum$ AUC = the total of areas under curve of the wavelength range $\lambda_n - \lambda_1$ graph

λ_n = the largest wavelength between wavelengths of 280 nm to above it which has a minimum absorption value of 0.050

λ_1 = smallest wavelength (280 nm)

Compact Powder Formulations

The compact powders were formulated using RBO of: (F1) 0.05%; (F2) 0.1%; (F3) 0.15%; (F4) 0.2%; and (F5) 0.25%; (F6) as a negative control. First, RBO and BHA (0.02%) were mixed, then magnesium carbonate (5%), kaolin (20%) titanium dioxide (1%) and nipagin (0.2%) were added. The mixture was mashed and crushed homogeneously. Talc moderately ground, then passed through mesh no. 200. After sifted the talc, added to the mixture with magnesium stearate (10%), Crodamol™ ISIS (5%), and silicone oil (5%). Finally, the powder mixture was blended for 5 minutes and sieved using mesh no. 60. Powders with average weight of 15 g were compressed and stored in container for evaluation (Table 1).

Physical Evaluation of Compact Powder [15]

Homogeneity Test

The color dispersion was determined by spreading the powder on the surface of white paper and observed using a magnifying glass. The dispersion was homogen if the color was spread evenly.

Adhesion Test

Powder shade was determined by applying powder to the back part of the hand. Shade test was good if the powder properly distributed and well adhered. It is characterized by two times application resulting intense and homogeneous color.

Crack Test

The crack test was carried out by dropping the powder on the wood surface several times at the height of 8 inches. The good result was claimed if after dropped several times, it is not cracked or broken.

Determination of the in vitro SPF of compact powder

The UV-Visible spectrophotometric method was used to determine the in vitro SPF of compact powder [13]. Each sample (0.05%, 0.1%, 0.15%, 0.2%, 0.25% and negative control) weighed 100 mg then put in volumetric flask (size 50 ml) and diluted with ethyl acetate. The solution was mixed and then filtered (stock solutions). Each of 5 ml was pipetted, transferred to volumetric flask (size 10 ml), and then diluted with ethyl acetate. The absorbance was measured by a UV-Visible spectrophotometer at UV B wavelengths (280-310 nm) and UV A (320-400 nm). SPF value was calculated using the same equation as in determination the SPF value of bran oil.

Data Analysis

Data from the test results were subjected to statistical analysis using the Statistical Program Service Solution

Table 1. Composition of compact powder formulations

Ingredients	Formulations (%)					
	F1	F2	F3	F4	F5	F6
Rice bran oil (RBO)	0.05	0.1	0.15	0.2	0.25	-
BHA	0.02	0.02	0.02	0.02	0.02	0.02
Nipagin	0.2	0.2	0.2	0.2	0.2	0.2
Titanium dioxide	1	1	1	1	1	1
Magnesium carbonate	5	5	5	5	5	5
Crodamol™ ISIS	5	5	5	5	5	5
Silicone oil	5	5	5	5	5	5
Magnesium stearate	10	10	10	10	10	10
Kaolin	20	20	20	20	20	20
Talc	53.73	53.68	53.63	53.58	53.53	53.78

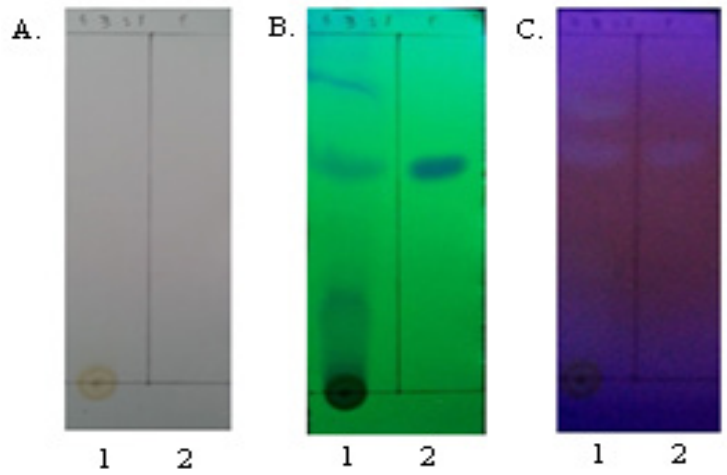

Figure 1. TLC of (A) Visible light (B) UV light 254 nm (C) UV light 366 nm. (1) RBO, (2) γ-oryzanol (standard)

(SPSS) 26.0. Data analysis was carried out through the One-Way ANOVA test with a significance level of 5%.

Results and Discussion

The yield oil percentage of rice bran extraction using hexane and ethanol (1:1) was 3.26%. This extraction process was influenced by solvent, time and temperature of extract.

SPF is a quantitative measurement of the effectiveness of a sunscreen formulation. To be effective in preventing sunburn and other skin damage, a sunscreen product should have a wide range of absorbance between 290 and 400 nm [14]. The sunscreen product should have SPF value higher than 2. However, US Food and Drug Administration (FDA) recommend sunscreen products have SPF values of 15 or above to help reduce the risk of skin cancer and early skin aging caused by the sun [16].

To ensure that the extraction results were contained γ-oryzanol, a chromatographic analysis was performed.

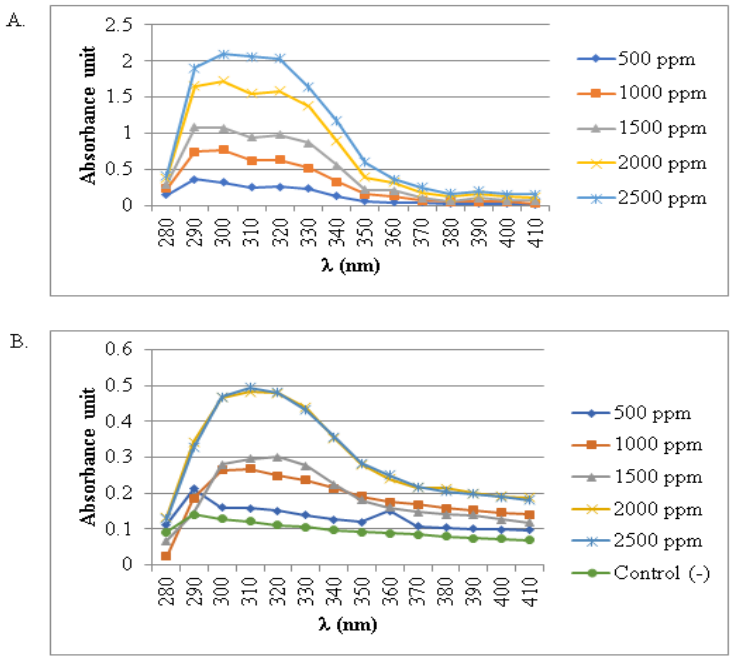

Figure 2. UV-absorbing from 280-410 nm. (A) Rice bran oil (B) Cosmetic product of rice bran (compact powder) com-pare with negative control

Table 2. Absorbances of RBO

Wavelength (nm)	Absorbances				
	500 ppm	1000 ppm	1500 ppm	2000 ppm	2500 ppm
280	0.145	0.227	0.292	0.376	0.417
290	0.359	0.746	1.077	1.657	1.902
300	0.320	0.764	1.073	1.726	2.098
310	0.252	0.622	0.945	1.553	2.061
320	0.253	0.631	0.977	1.588	2.038
330	0.233	0.519	0.862	1.374	1.633
340	0.127	0.325	0.550	0.888	1.167
350	0.053	0.147	0.213	0.387	0.598
360	0.039	0.130	0.206	0.320	0.364
370	0.036	0.064	0.100	0.177	0.250
380	0.025	0.051	0.058	0.114	0.163
390	0.023	0.050	0.103	0.150	0.192
400	0.021	0.047	0.068	0.115	0.156
410	0.017	0.026	0.069	0.113	0.149

The TLC showed that RBO has the same band with γ-oryzanol (standard). These results are shown in Figure 1. As a result, under visible light did not show any spots on the plates that had been bottled and eluted by eluents. Nevertheless, at UV 254 and 366 nm showed the color of band is dark purple and light blue (Figure 1). Retention factor of RBO and γ-oryzanol is 0.64, it indicates that RBO contains γ-oryzanol [17].

In this research, RBO and the cosmetic product of rice bran (compact powder) was evaluated with in vitro UV spectrophotometry method. The wavelength range for determination SPF is 280-400 nm [18]. RBO can absorb UV rays at 280-350 nm and the highest absorbance at 290-300 nm (Figure 2A). The SPF calculation for the five RBO concentrations showed a statistically significant difference ($p < 0.05$) to the SPF value of the RBO. SPF values obtained using the UV spectrophotometric method are shown in Table 2 and 3 which showed that 2500 ppm of RBO has the highest SPF value at 11.884±0.027.

This study indicated that the compact powder absorbs UV rays at 280-400 nm and the highest absorbance at 290-320 nm (Figure 2B). SPF determination of compact powder was obtained at different value of each formulation. The 2000 ppm of RBO in compact powder showed the highest absorbance which was different from the absorbance results from RBO. Absorbance and SPF value of compact powder are shown in Table 4 and 5, respectively, using the UV spectrophotometric method. According to FDA guidelines, the criteria for the level of effectiveness of a product as a UV protector places SPF

Table 3. In vitro SPF values of rice bran oil

RBO concentration (ppm)	SPF	AUC
500	1.741±0.011	16.893
1000	2.394±0.009	41.762
1500	3.416±0.005	64.115
2000	7.199±0.010	102.933
2500	11.884±0.027	129.033

Table 4. Absorbances of compact powder

Wavelength (nm)	Absorbances					
	500 ppm	1000 ppm	1500 ppm	2000 ppm	2500 ppm	Negative control
280	0.112	0.024	0.067	0.134	0.128	0.091
290	0.211	0.186	0.143	0.343	0.327	0.140
300	0.159	0.265	0.279	0.466	0.468	0.128
310	0.158	0.267	0.295	0.483	0.495	0.121
320	0.151	0.249	0.300	0.478	0.480	0.110
330	0.138	0.236	0.277	0.439	0.432	0.105
340	0.127	0.213	0.224	0.354	0.357	0.097
350	0.119	0.191	0.181	0.279	0.283	0.092
360	0.150	0.176	0.159	0.240	0.250	0.088
370	0.107	0.169	0.148	0.216	0.217	0.084
380	0.104	0.158	0.140	0.213	0.204	0.079
390	0.100	0.153	0.138	0.199	0.197	0.074
400	0.099	0.146	0.127	0.191	0.189	0.073
410	0.096	0.140	0.118	0.188	0.181	0.068

value of 2-12 in the minimal sun protection category, 12-30 in the moderate sun protection category, and >30 in the high sun protection category. SPF value of F4 and F5 compact powder were 2.178±0.003 and 2.173±0.003, respectively. This result indicated that the SPF of compact powder lower than the RBO but according to FDA guidelines for sunscreen active ingredient to contribute a minimum determined SPF of not less 2 so those that meet the requirements are the formulas of F4 and F5 [16]. The SPF values of 2000 and 2500 ppm of RBO in compact powder did not differ significantly ($p > 0.05$). This is due to a decrease the concentration of γ-oryzanol in compact powder. Reduction of concentration can be occurred by instability of RBO during formulation and time storage as a result of the formation of oxidizing compounds such as singlet oxygen, superoxide anions or reactive compounds by photosensitization [19,20]. It also needed to consider for physicochemical interaction of active ingredient and vehicle component [14].

Furthermore, the physical evaluation of five formula of RBO with different concentration showed that all the formula has a good physical sunscreen product. The compact powder formulation of RBO is shown in Figure 3 and physical test results are represented in Table 6. In addition to, herbal extract and natural molecule use as a solar protection is a new trend in cosmetic industry.

Table 5. In vitro SPF values of compact powder

Product	SPF	AUC
F1 (0.05%)	1.390±0.017	17.268
F2 (0.1%)	1.682±0.005	24.915
F3 (0.15%)	1.612±0.005	25.042
F4 (0.2%)	2.178±0.003	40.635
F5 (0.25%)	2.173±0.003	40.518
F6 (negative control)	1.274±0.002	12.707

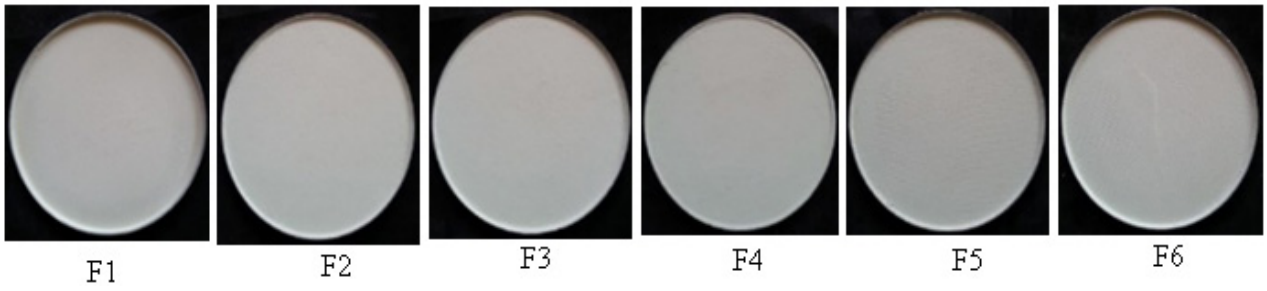

Figure 3. Formulation of compact powder, (F1 0.05%), (F2 0.1%), (F3 0.15%), (F4 0.2%), (F5 0.25%), (F6 negative control)

Table 6. Physical evaluation of compact powder

Formula	Homogenity Test	Adhesion Test	Crack Test
F1	Homogeneous	Good	Not crack
F2	Homogeneous	Good	Not crack
F3	Homogeneous	Good	Not crack
F4	Homogeneous	Good	Not crack
F5	Homogeneous	Good	Not crack
F6	Homogeneous	Good	Not crack

Vegetable oil in sunscreen formulation reported that it could be promising strategy for design product to protect from UV-rays [21].

Conclusion

In the present study show that the RBO could be contribute as a cosmetic ingredient for protection from UV rays based on SPF value. The effective concentration of RBO compact powder to have the sufficient sun protection effect was at 2000 ppm that has SPF value of 2.178±0.003. Although, the SPF of cosmetic products of RBO was found to be low in SPF value even though the developed compact powder showed a good homogeneity and adhesion to the skin. Furthermore, these studies suggest to improve the formula to protect the product from instability.

References

[1]. Kale S, Sonawane A, Ansari A, Ghoge P, Waje A. Formulation and in-vitro determination of sun protection factor of Ocimum basilicum, Linn. leaf oils sunscreen cream. Int J Pharm Pharm Sci. 2010;

[2]. Sambandan DR, Ratner D. Sunscreens: An overview and update. J Am Acad Dermatol. 2011;64(4):748–58. https://doi.org/10.1016/j.jaad.2010.01.005

[3]. Bernardi DS, Pereira TA, Maciel NR, Bortoloto J, Viera GS, Oliveira GC, et al. Formation and stability of oil-in-water nanoemulsions containing rice bran oil: In vitro and in vivo assessments. J Nanobiotechnology. 2011; https://doi.org/10.1186/1477-3155-9-44

[4]. Juliano C, Cossu M, Alamanni MC, Piu L. Antioxidant activity of gamma-oryzanol: Mechanism of action and its effect on oxidative stability of pharmaceutical oils. Int J Pharm. 2005;299(1–2):146–54. https://doi.org/10.1016/j.ijpharm.2005.05.018

[5]. Sapino S, Carlotti ME, Cavalli R, Ugazio E, Berlier G, Gastaldi L, et al. Photochemical and antioxidant properties of gamma-oryzanol in beta-cyclodextrin-based nanosponges. J Incl Phenom Macrocycl Chem. 2013;75(1–2):69–76. https://doi.org/10.1007/s10847-012-0147-3

[6]. Nagendra Prasad MN NP, KR S, Khatokar M S. Health Benefits of Rice Bran- A Review. J Nutr Food Sci. 2011; https://doi.org/10.4172/2155-9600.1000108

[7]. Mohiuddin AK. An extensive review of face powders: Functional uses and formulations. Int J Pharm Pharm Sci. 2019;1(1):1–12. https://doi.org/10.5281/zenodo.3547001

[8]. Mohiuddin AK, Trust MNM. Face Powders : Formulation Considerations. 2019;1–29.

[9]. Gabriella B, Kenneth S. A. Introduction To Cosmetic Formulation and Technology. Wiley. 2015. https://doi.org/10.1007/s13398-014-0173-7.2

[10]. Estefan G, Sommer R, Ryan J. Methods of Soil , Plant , and Water Analysis : A manual for the West Asia and North. Int Cent Agric Res Dry Areas ICARDA@cgiar.org. 2013; https://doi.org/10.1038/modpathol.3800701
[11]. Pandey R, Shrivastava SL. Comparative evaluation of rice bran oil obtained with two-step microwave assisted extraction and conventional solvent extraction. J Food Eng. 2018; https://doi.org/10.1016/j.jfoodeng.2017.09.009
[12]. Mariod A, Ismail M, Abd Rahman NF, Matthaus B. Stability of rice bran oil extracted by SFE and soxhlet methods during accelerated shelf-life storage. Grasas y Aceites. 2014; https://doi.org/10.3989/gya.109413
[13]. Khunkitti W, Satthanakul P, Waranuch N, Pitaksuteepong T, Kitikhun P. Method for screening sunscreen cream formulations by determination of in vitro SPF and PA values using UV transmission spectroscopy and texture profile analysis. J Cosmet Sci. 2014;
[14]. Rejeki S. The SPF Value Determination of Extract and Sunscreen Lotio of Lime Rind Extract by Spektrofometri UV-Vis Method. J Farm (Journal Pharmacy). 2018; https://doi.org/10.37013/jf.v1i1.58
[15]. Jarupinthusophon S, Anurukvorakun O. Development of jasmine rice flour properties as a safe and efficient ingredient for compact powder. Appl Sci. 2021;11(1):1–12. https://doi.org/10.3390/app11010248
[16]. FDA. Sunscreen drug products for over-the-counter human use [Internet]. Department of Health and Human Services. 2022. Available from: https://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?CFRPart=352&showFR=1#:~:text=The concentration of each active,the combination multiplied by 2
[17]. Sakunpak A, Suksaeree J, Monton C, Pathompak P, Kraisintu K. Quantitative analysis of γ-oryzanol content in cold pressed rice bran oil by TLC-image analysis method. Asian Pac J Trop Biomed. 2014;4(2):119–23. https://doi.org/10.1016/S2221-1691(14)60219-7
[18]. Dutra EA, Da Costa E Oliveira DAG, Kedor-Hackmann ERM, Miritello Santoro MIR. Determination of sun protection factor (SPF) of sunscreens by ultraviolet spectrophotometry. Rev Bras Ciencias Farm J Pharm Sci. 2004;40(3):381–5. https://doi.org/10.1590/S1516-93322004000300014
[19]. Min DB, Boff JM. Chemistry and reaction of singlet oxygen in foods. Compr Rev Food Sci Food Saf. 2002;1(2):58–72. https://doi.org/10.1111/j.1541-4337.2002.tb00007.x
[20]. Park JW, Jang EY, Kim JY, Yi BR, Kim MJ, Park KW, et al. Effects of visible light irradiation on the oxidative stability in rice bran. J Cereal Sci. 2013;58(1):178–81. https://doi.org/10.1016/j.jcs.2013.05.004
[21]. Montenegro L, Santagati LM. Use of vegetable oils to improve the sun protection factor of sunscreen formulations. Cosmetics. 2019;6(2). https://doi.org/10.3390/COSMETICS6020025.