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Original Research Article

Formulation of Facial Clay Mask from Red Glutinous Rice (*Oryza sativa* L. var. glutinosa) Bran Extract

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ABSTRACT

Red glutinous rice (*Oryza sativa* L. var. glutinosa) bran, a byproduct of rice processing, is known for its antioxidant properties. This study aimed to enhance the utilization of red glutinous rice bran extract as an antioxidant by formulating it into a clay mask for facial application. Five formulations (F1 – F5) of clay mask were prepared from varying concentrations of red glutinous rice bran extract; F1 (0%), F2 (2.5%), F3 (5%), F4 (7.5%), and F5 (10%). The formulations were assessed for their organoleptic properties, and physicochemical characteristics, including homogeneity, pH, spreadability, adhesion, drying time, viscosity, and flow properties. The antioxidant activity of the formulations was evaluated using the 2,2-diphenyl-1-picryl hydrazyl (DPPH) radical scavenging assay. The clay mask of red glutinous rice bran extract was successfully formulated with a smooth clay-like consistency and a characteristic clay odour. The physicochemical properties of the clay mask formulations were greatly influenced by the concentration of red glutinous rice bran extract, but varied within acceptable limits. The formulations were homogenous with pH ranging from 4.74 – 6.00, spreadability 2.7 – 4.2 cm, adhesion 6.14 – 7.44 seconds, drying time 12.14 – 15.21 minutes, and viscosity 123,333 – 283,666 cps. The formulations exhibited strong antioxidant activity with IC_{50} ranging from 204.114 ppm to 19.443 ppm. Among the formulations, F5 exhibited the most favorable results, meeting the desired clay mask specifications and having the highest antioxidant activity. This study highlights the potential application of red glutinous rice bran extract in cosmetic formulations, particularly as a natural antioxidant in clay mask preparations.

Keywords: Red glutinous rice bran, Clay mask, Physicochemical properties, Antioxidant activity.

Introduction

The importance of topical formulations with antioxidant effect on the skin cannot be overstated. Antioxidants are substances that neutralize free radicals, thereby preventing, slowing, and delaying the oxidation process at low concentrations.[1] Antioxidant compounds donate an electron to unstable free radicals, neutralizing them, and preventing their interference with the biological system.[2] Substances exhibiting antioxidant activity are classified based on their IC_{50} values, these include; exceptionally strong antioxidants with IC_{50} less than 50 ppm, strong antioxidants with IC_{50} between 50 ppm and 100 ppm, weak antioxidants with IC_{50} between 100 ppm and 150 ppm, and very weak antioxidants with IC_{50} values ranging from 151 ppm to 200 ppm.[3] Red glutinous rice bran extract (2.5%) cream formulation has been shown to exhibit an exceptionally strong antioxidant activity with IC_{50} of 31.7525 ppm, demonstrating superior antioxidant activity compared to black glutinous rice bran, which had an IC_{50} of 434.7525 ppm.[4]
Red glutinous rice bran is a byproduct of rice milling, and has been shown to contain several bioactive components, including tocopherol, tocotrienol, gamma oryzanol, phenolic antioxidants, and anthocyanins.[1,5]

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Oryzanol, exclusively present in bran, is a chemical that acts as an antioxidant. The mechanism of action of gamma oryzanol involves donating hydrogen atom to neutralize free radicals like reactive oxygen species (ROS).[6] The efficacy of brown rice bran extracts as antioxidants can be enhanced by making them into a mask preparation.
Masks are facial applications used to have a firming impact and remove accumulated debris.[7] Clay mask is designed to rejuvenate the skin, absorb excess sebum, constrict pores, eliminate blockages inside the pores, and remove dead skin cells and pollutants.[8] Clay face masks are extensively used for their exceptional capacity to rejuvenate the skin. Skin alterations are perceived as the mask initiates its action, causing tension in the skin layer as it dries. This sensation invigorates the skin, allowing the clay mask to eliminate debris from the face.[9] The debris are removed when the mask is rinsed from the facial skin. The effects of clay mask usage result in bright and clean skin.[10]
Antioxidants inhibit or prevent cellular degeneration, damage, or destruction resulting from oxidative processes. This study evaluated the antioxidant activity of clay mask preparation using the DPPH technique. The DPPH technique is a straightforward and rapid assay for evaluating antioxidant activity. This method uses DPPH free radicals to evaluate an antioxidant molecule that scavenges free radicals.[11,12] The metrics employed to illustrate antioxidant activity include the 50% inhibitory concentration (IC_{50}), which refers to the concentration of an antioxidant that induces a 50% reduction in the radical character of DPPH. This study used red glutinous rice bran extract as the active agent at varying concentrations of 0%, 2.5%, 5%, 7.5%, and 10% to formulate a clay mask product, and assessed its physical characteristics and antioxidant activity.

Materials and Methods

Materials
The materials used in this research include red glutinous rice bran extract (Research Institute on Medicinal and Aromatic Plants of Indonesia), Bentonite (Merck), Kaolin (Merck), Methyl Paraben (Merck), Xanthan Gum (Merck), Triethanolamine (PT. Sumber Berlian

Chemical), Sodium Lauryl Sulphate (Merck), Propylene Glycol (Merck), Oleum Rosae, Vitamin C (Merck), Methanol (Merck), and 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical (HiMedia Laboratories Pvt. Ltd).

Collection of rice bran
Red glutinous rice (*Oryza sativa* L. var. glutinosa) bran was obtained from the Research Institute on Medicinal and Aromatic Plants, Indonesia in 2024 (Latitude -6.57701254281639°, Longitude 106.78625218147363°).

Preparation of rice bran extract
Powdered glutinous rice bran was macerated with 70% ethanol in a dark glass container at room temperature for 5 days. The extract was filtered, concentrated in a rotary evaporator below 50ºC. The dried extract was stored in an air-tight opaque bottle until needed for further analysis.[13]

Assessment of organoleptic properties of the rice bran extract
The organoleptic properties, including morphology, hue, aroma, and flavour of the red glutinous rice bran extract was assessed.[14,15]

Determination of ash content
Rice bran extract (2 g) was weighed into a porcelain crucible of known weight. The extract was evaporated to dryness on a hot water bath. The dried sample was transferred to an electric furnace and incinerated at an initial temperature of 400°C for 4 hours. Subsequently, the temperature was increased to 800°C, and heating was continued for an additional 4 hours. The sample was allowed to cool in a desiccator and was subsequently weighed repeatedly until a constant weight was obtained.[16]

Phytochemical screening
Qualitative phytochemical tests were performed to detect the presence of some secondary metabolites in red glutinous rice bran extract. The phytochemicals tested include alkaloids, flavonoids, and tannins according to standard procedures.[17]

Preparation of clay mask
Bentonite and kaolin were initially sieved through a 100-mesh sieve and weighed (Table 1). The bentonite was dissolved in hot water and left to stand for 24 hours. Methylparaben was dissolved in heated distilled water. After 24 hours, the bentonite solution was combined with methylparaben and stirred until homogeneous.
Kaolin was gradually dispersed in water while mixing at 1,200 rpm until a uniform mixture was achieved. Xanthan gum was dispersed in propylene glycol, then added to the bentonite, methylparaben, and kaolin mixture and stirred rapidly.

Table 1: Clay Mask Preparation Formula

Ingredient	Percentage composition (%)					Function
	F1	F2	F3	F4	F5	
Brans Extract	0	2.5	5	7.5	10	Active Substance
Bentonite	6	6	6	6	6	Mineral Clay
Kaolin	35	35	35	35	35	Oil Absorbent
Xanthan Gum	0.1	0.1	0.1	0.1	0.1	Thickener
Propilenglikol	7	7	7	7	7	Humectant
SLS	1	1	1	1	1	Cleaning Agent
TEA	1	1	1	1	1	pH buffer
Methyl Paraben	0.1	0.1	0.1	0.1	0.1	Preservative
Rose oil	Qs	Qs	Qs	Qs	Qs	Perfume
Distilled Water	100	100	100	100	100	Solvent

Sodium lauryl sulfate was incorporated and mixed slowly. Red glutinous rice bran extract was added as follows: F1 with no extract, F2 with 2.5% extract, F3 with 5% extract, F4 with 7.5% extract, and F5 with 10% extract. Oleum rosae was then added for fragrance and mixed gently. The remaining water was added and mixed until uniform. The pH of the mask was measured, and if necessary, triethanolamine (TEA) was added to adjust the pH to the desired range of 4.5–6.5.[18] The mask preparation was stored in an air-tight container until needed for further analysis.

Evaluation of physical properties of clay mask preparation
Organoleptic properties
The clay mask preparation was assessed for odour, colour, and consistency.[8]

Homogeneity test
The clay mask was uniformly applied to a transparent glass and exposed to light. Subsequently, the sample was examined from three different points on the glass surface. The absence of visible coarse grains indicate homogenous sample.[18]

pH test
The pH of the clay mask preparation was measured using a pH meter (Starter 3100 pH Bench, Ohaus, USA).[19]

Spreadability test
The clay mask (1 g) was placed in a Petri dish, sealed, and 1-gram, 2-gram, 3-gram, and 5-gram load were applied on the clay mask for 1-2 minutes, after which the diameter of the spread was measured with each subsequent load.[19]

Adhesion test
The adhesive properties of the clay mask formulations were evaluated using a method previously described. Each of the slay mask formulations (0.25 g) was applied to two pre-designated glass slides. A 1 kg weight was placed on top of the slides for 5 minutes to ensure uniform contact. The slides were then secured onto the testing apparatus, and an additional 80 g load was applied. The time taken for the clay mask to detach from the glass slides was recorded for each formulation.[19]

Determination of drying time
Drying time of the clay mask was determined by applying the clay mask from each concentration to the dorsal side of the hand and allowed to dry. The time interval from the application to the point at which the mask was completely dried was taken as the drying time.[8]

Determination of viscosity and flow properties
The viscosity of the clay mask was determined using a Brookfield RV-DVE viscometer, USA, equipped with a spindle and suitable speed. Viscosity data was documented, and the flow characteristics (Rheology) were assessed.[20]

Evaluation of antioxidant activity
The antioxidant activity of the clay mask preparation was evaluated using the 2,2-diphenyl-1-picryl hydrazyl (DPPH) radical scavenging assay according to the method previously described by Lestari *et al*. (2020).[21] Briefly, A solution of 0.1 mM DPPH in methanol was prepared, and 3.0 mL of this solution was mixed with 3.0 mL of clay mask preparation in different concentrations (5 ppm, 10 ppm, 15 ppm, 20 ppm, and 25 ppm). The mixture was incubated for 30 minutes in the dark. After incubation, the absorbance of each sample was measured using a UV-VIS spectrophotometer (Shimadzu 1780, Japan) at 515 nm. Ascorbic acid (8 ppm, 10 ppm, 12 ppm, 14 ppm, and 16 ppm) was used as reference standard. The blank solution consists of 3 mL DPPH solution and 3 mL of methanol. The DPPH scavenging activity was calculated using the following equation:

$$\text{DPPH radical scavenging activity (\%)} = [(A_0 - A_1)/(A_0)] \times 100$$

Where; A_0 is the absorbance of Blank
A_1 is the absorbance of DPPH radical + sample
The 50% inhibitory concentration value (IC_{50}) was derived from the linear regression equation: y = ax + b.

Statistical analysis
Data were analyzed using one-way analysis of variance (ANOVA), followed by post-hoc analysis using the Tukey-HSD (Honestly Significant Differences) test at 95% confidence level ($\alpha = 0.05$). The statistical analysis was conducted using IBM SPSS Statistics, Version 38 (Chicago, IL, USA).

Results and Discussion

Rice bran extraction yield
Extraction of red glutinous rice bran produced an extract with a percentage yield of 13.27%.

Physical characteristics of red glutinous rice bran extract
Organoleptic assessment of the rice bran extract revealed a viscous extract, with a characteristic aroma, acrid flavour, pH of 7.01, and a dark red hue. The moisture content was 12.86%, and the ash content was 0.89%.

Phytochemical constituents of rice bran extract
Phytochemical screening of red glutinous rice bran extract revealed the presence of alkaloids, flavonoids, and tannins (Table 2).

Table 2: Phytochemical constituent of red glutinous rice bran extract

Phytochemical	Inference
Alkaloids	+
Flavonoids	+
Tannins	+

Physicochemical characteristics of red glutinous rice bran extract clay mask
Organoleptic properties
The assessment of organoleptic properties of clay mask derived from red glutinous rice bran extract included visual inspection for colour, as well as other sensory attributes such as odor, form, and texture. The organoleptic properties assessment was done for the five clay mask formulations (F1 – F5). The formulations exhibited a smooth clay-like consistency without presence of coarse grains, which facilitated easy application to the face. In terms of aroma, formulation F1 exhibited the characteristic odour of clay mask bases, whereas formulations F2, F3, F4, and F5 exhibit the usual rice bran aroma. F1 exhibited a milky white hue, attributable to the absence of extract in the formulation, whereas F2 and F3 exhibit a brownish-white tint, while F4 and F5 displayed a brown colouration due to a higher concentration of extract. These observations indicated that the variation in the concentration of the red glutinous rice bran extract did not significantly affect the colour, aroma, and texture of the formulations (Figure 1).

Figure 1: Red glutinous rice bran extract clay mask

Homogeneity
Homogeneity test was conducted to check for the presence or absence of non-homogeneous particles in the clay mask formulations. Homogeneity test is necessary due to the impact of non-homogenous formulation on consumer comfort. In the present study, all the clay mask formulations appeared to be physically homogeneous, and no discernible roughness was observed.

pH
pH is an essential parameter in formulations intended for topical application, as this influences user comfort during application. A low or acidic pH might irritate the skin, while a high or alkaline pH can lead to skin dryness upon application. Therefore, the pH of topical formulations should closely approximate the pH of the skin, which ranges from 4.5 to 6.5. The red glutinous rice bran extract clay mask formulations have pH adjusted to match that of facial skin. Results showed that formulas F1 to F5 had pH ranging from 4.74 to 6.00 (Figure 2), which aligned with normal pH of facial skin. Although the pH of F1 was significantly lower compared to the other formulations, it was still within the normal pH range of the skin (4.5 – 6.5).[22] The pH of F2 to F5 did not differ significantly from one another, suggesting that the red glutinous rice bran extract did not influence the pH of the formulations. The pH of the red glutinous rice bran extract was found to be neutral at 7.01 (Figure 2).

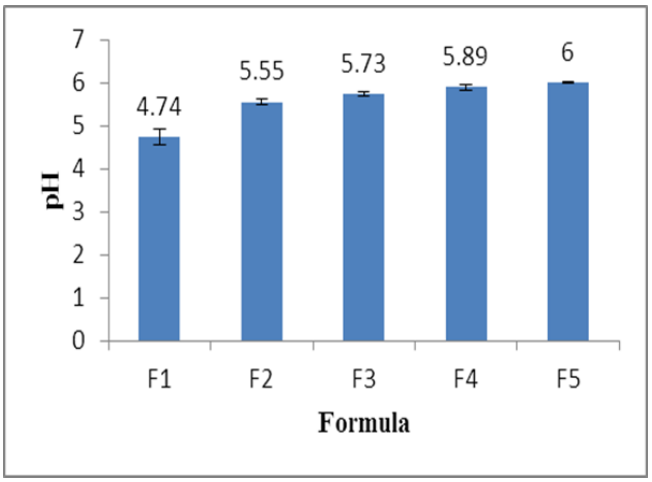

Figure 2: pH of red glutinous rice bran extract clay mask formulations

Spreadability
The spreadability of the red glutinous rice bran extract clay mask formulations is illustrated in Figure 3. The results indicate a significant disparity in the spreadability of formulation F1 and F5, as the spreadability decreased gradually from F1 to F5. The spreadability assessment of a semisolid formulation is directly correlated with its viscosity; a higher viscosity indicates reduced spreadability. The results indicated that an increased extract concentration led to a more viscous formulation. A highly viscous preparation impedes flow, hence reduces its spreadability. Conversely, a thinner preparation will enhance flow and increase its spreadability. The spreadability of all the red glutinous rice bran extract clay mask formulations remain within the acceptable limit for clay mask formulations, which is 5 – 7 cm (Figure 3).[23,24]

Adhesion
Adhesion test is conducted to assess the efficacy of the mask preparation, specifically the ability of the red glutinous rice bran extract clay mask to adhere to the skin. The adhesion test seeks to determine the duration of contact between the preparation and the skin's surface; a preparation is deemed adequate if it exhibits substantial adhesion. An inadequate adhesion property of the preparation will lead to its easy detachment from the skin, resulting in suboptimal efficacy. The adhesion values of the red glutinous rice bran extract clay mask formulations are presented in Figure 4. The results indicate a substantial increase in adhesion time in formulations F3, F4, and F5 compared to F1, and F2. This can be attributed to the viscosity of the extract. The extract is viscous, and its inclusion decreases the volume of water required in the formulation, thus prolonging the adhesion time. The

adhesion test results indicate that all formulations (F1 – F5) met the minimum adhesion requirement of 4 seconds for topical formulations.[25]

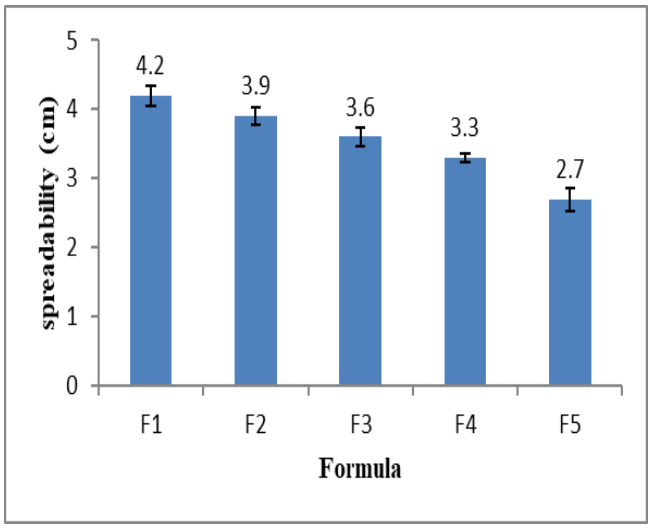

Figure 3: Spreadability of red glutinous rice bran extract clay mask formulations

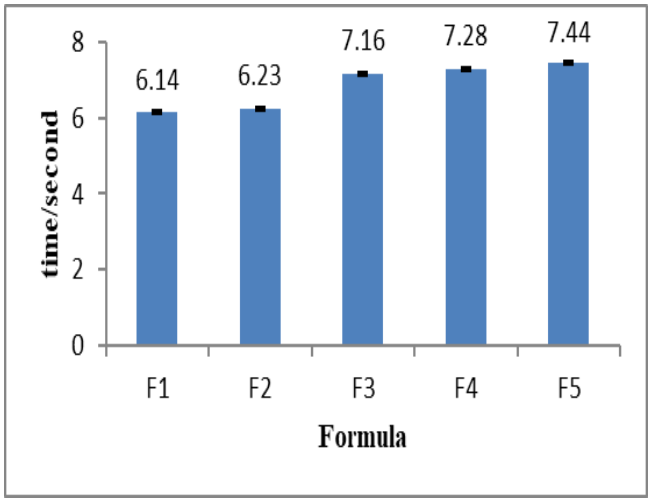

Figure 4: Adhesion of red glutinous rice bran extract clay mask formulations

Drying time

The drying time test seeks to measure the duration needed for clay mask preparation to dry from when the mask is applied on the skin until a dry layer is formed. The drying time for the red glutinous rice bran extract clay mask formulations is illustrated in Figure 5. From the results, it was observed that the drying time decreased gradually from 15.21 s to 12.14 s in the formulations F1 to F5, which indicated that decreasing drying time correlated with increasing red glutinous rice bran extract concentration in the formulations, which can be attributed to diminishing water content in the preparation. Formulation F1 had an extended drying time due to its lower concentration of rice bran extract and water content. In addition, drying time could also be influenced by excipients capable of water absorption, such as Bentonite. Bentonite functions as an absorbent, particularly for water, decreasing the moisture content in clay mask formulations. As a result, the drying time is expedited. The drying time for all formulations met the drying time criteria for clay mask, specifically within the range of 10 to 20 minutes (Figure 5).[8]

Viscosity

The consistency and flow properties of the clay mask were assessed using a Brookfield RV viscometer. A thickness test was conducted to determine the viscosity of the clay mask formulation. Viscosity is crucial in topical formulations as it determines ease of application and consumers' comfort. Optimal consistency facilitates effortless application.

The viscosity of a formulation is inversely related to the dispersion/spreadability of the formulation; a lower viscosity leads to a broader spreading diameter and vice versa. The viscosity of the rice bran extract clay mask formulations is presented in Figure 6. The results indicate that the viscosity of the clay mask formulations increased significantly as the concentration of the rice bran extract increases. There was a discernible increase in the viscosity from 123333 cps in formulation F1 to 283666 cps in formulation F5.

Furthermore, the inclusion of propylene glycol in each formulation also influences viscosity. Propylene glycol enhances the viscosity of formulation due to its hydrophilic properties.[26] This is particularly apparent in formulation F5, which possesses a greater concentration of the extracts, resulting in higher viscosity (Figure 6).

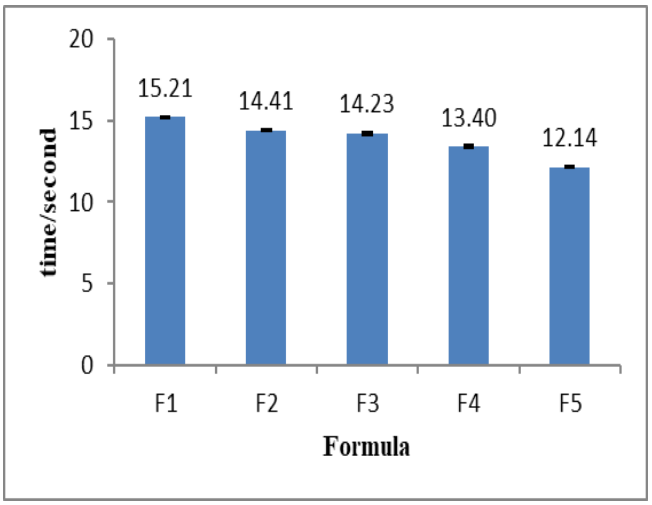

Figure 5: Drying time of red glutinous rice bran extract clay mask formulations

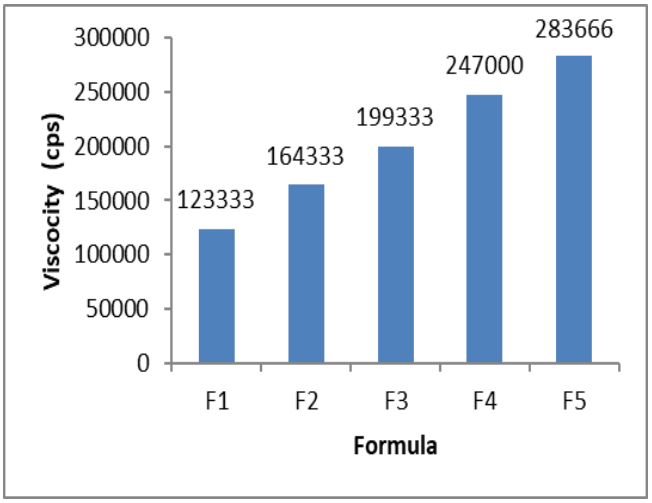

Figure 6: Viscosity of red glutinous rice bran extract clay mask formulations

Flow properties

Flow properties encompass the mixing and transferring of material into a container, pre-use transfer, and discharge from the tube. Thixotropic flow is characterized by increased viscosity with the application of force; nevertheless, once the force ceases, the particles do not settle rapidly, resulting in uniform particle dispersion. The thixotropic flow is

characterized by an ascending curve to the right of the descending curve. The flow characteristics of ecotropic are the preferred flow attributes in pharmaceutical formulations due to their consistency in containers and efficient dispersibility. The yield value is the numerical result derived from assessing the consistency of semisolid formulations. The yield value is the force or shear stress that must be surpassed for the material to flow.[27] The yield value signifies the flocculation capacity; a higher concentration of flocculated material in the formulation correlates with an increased yield value.[28] The yield values obtained for the red glutinous rice bran extract clay mask formulations are presented in Table 3.

Table 3: Yield values of red glutinous rice bran extract clay mask formulations

Formulation	Yield value (dyne/cm²)
F1 (0%)	11.3260
F2 (2.5%)	22.0508
F3 (5%)	29.4166
F4 (7.5%)	42.4531
F5 (10%)	46.9516

Antioxidant activity

Antioxidants in topical formulations are intended to neutralize free radicals that impact the skin. The results of the antioxidant activity test as measured by DPPH radical scavenging activity for the clay mask of red glutinous rice bran extract are presented in Table 4. The results revealed that red glutinous rice bran extract clay mask exhibited potent and dose-dependent antioxidant activity with the following IC_{50} values; 204.114 ppm, 124.019 ppm, 87.945 ppm, 23.676 ppm, 19.443 ppm for formulas F1. F2, F3, F4, and F5, respectively. As expected, the standard antioxidant compound vitamin C exhibited higher antioxidant activity compared to the clay mask formulations, with IC_{50} of 7.250 ppm.

The antioxidant activity of the clay mask derived from red glutinous rice bran extract may be attributed to antioxidant compounds such as gamma-oryzanol present in the rice bran extract. Gamma-oryzanol has been shown to possess strong antioxidant activity by donating hydrogen atom to reactive free radicals.[29]

Table 4: Antioxidant activity of red glutinous rice bran extract clay mask formulations

Formulation	IC_{50} (ppm)
F1 (0%)	204.114
F2 (2.5%)	124.019
F3 (5%)	87.945
F4 (7.5%)	23.643
F5 (10%)	19.443
Vitamin C	7.250

Conclusion

Red glutinous rice bran has been successfully formulated into a clay mask for topical application to facial skin. The physicochemical properties of the clay mask formulation were influenced by the concentration of the red glutinous rice bran extract. The physicochemical properties including homogeneity, pH, spreadability, adhesion, drying time, viscosity, and flow properties of all the formulations (F1 – F5) were within acceptable limits for topical formulations. The clay mask formulations of red glutinous rice bran extract exhibited potent antioxidant activity with formulation F5 having the highest activity. Therefore, red glutinous rice bran clay mask demonstrates promising potential as a natural cosmetic ingredient with antioxidant properties.

Conflict of Interest

Authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

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