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Formulation And Evaluation Of Black Glutinous Rice Clay Mask (*Oryza sativa* L. Var Glutinosa) Using Bentonite And Kaolin Variations

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ABSTRACT

Black glutinous rice is a staple cuisine consumed frequently by Indonesians. Black glutinous rice's high anthocyanin content can stimulate collagen production. This study seeks to determine the effects of the kaolin and bentonite combination on different concentrations as a clay mask and obtain the optimal composition formula. Optimizing the clay mask formula using the factorial design procedure. The evaluation of a formula includes organoleptic, homogeneity, pH, irritation, and drying time tests. The research findings indicated that all preparations satisfied the evaluation criteria before the pH test. The results showed that kaolin can elevate the pH level, while bentonite can lower the pH. Preparations F1 and F2 satisfy the requirements, whereas F3 fails to fulfil the standards following the pH test. F1 (5 % bentonite and 15 % kaolin) and F2 (3 % bentonite and 25 % kaolin) were optimal clay mask formulas for black glutinous rice.

INTRODUCTION

Premature aging is a physiological process that cannot be avoided. Based on world population data related to premature aging, there was an increase of 11% of the world's population experiencing premature aging in 2009 and is estimated to reach 20% in 2050 (Wahid et al., 2022), considering that the skin is part of the body most often exposed to external factors and is also the first thing that appears on an individual when interacting with other people (Ahmad *et al.*, 2018).

One of the leading causes of premature aging is free radicals. On the skin, free radicals can destroy the collagen and elastin structure, making the skin less elastic. Wrinkle lines arise, disturbing the distribution of the pigment melanin and melanocytes. Hence, the pigmentation is uneven, and destroying the macromolecules that makeup cells, i.e., proteins, carbohydrates, fats, and DNA, can cause skin cancer (Chen et al., 2021; Safilla et al., 2022). The best way to keep the number of free radicals in the body from being excessive is to limit exposure is by using cosmetics.

The need for cosmetics in life is increasing day by day. The cosmetics industry is competing to produce cosmetic preparations that are safe, effective, and of course, easy to use based on the ever-increasing demand for cosmetics, especially among women who care about the health of their facial skin. as well as raw materials for active substances that are easily obtained from the environment are the main attractions (Husna et al., 2023).

Natural masks are made using natural ingredients used for facial care. Clay mask is a type of mask and product skincare that is popular because it reduces excess oil production,

regenerates skin cells, and restores skin health (Febriani et al., 2022; Gomes et al., 2020; Kumalasari, 2023).

Black glutinous rice (*Oryza sativa* L. Var Glutinosa) is a plant used as a staple food consumed by more than one-half of the world's population (Dewi et al., 2019). The main secondary metabolite in black rice is anthocyanins. Anthocyanins is a compound in a class of flavonoids with many hydroxyl groups that allows proton donation to free radicals resulting in the activity of great antioxidants. The use of anthocyanins in plants is more widely used in the food sector, health (pharmaceutical preparations) and industry (cosmetics) because it has no dangerous effect (Dewi et al., 2019; Nurtiana, 2019).

Numerous research indicate that anthocyanins may be able to shield the skin from UV ray damage. Topical application of anthocyanins at a modest dose could reduce the quantity of UVB radiation that reaches the epidermis and prevent UV skin damage (Correia et al., 2021; Khoo et al., 2017; Nurtiana, 2019).

Based on the background above, it is necessary to formulate black glutinous rice into a clay mask dosage form so that it is more practical to use and stable in storage. The purpose of this research aims to determine the basic formula for the best clay mask and the stability of the preparation after adding black glutinous rice(Febriani et al., 2022; Syamsidi et al., 2021).

METHODS

Tools

Digital scales (Vernier), drying cabinets, blenders (Philips), rotary evaporators, desikator (Duran), glass objects (Onelab), pH meters (Benchtop), and laboratory glassware (Pyrex), Filter paper (Whatman).

Material

Black glutinous rice (purchased from the native market of Ternate), bentonite, xanthan gum, glycerin, sodium lauryl sulfate, titanium dioxide, nipagin, kaolin, sodium metabisulfite, aquadest (all excipients are technical grade obtained from PT. Sumber Rejeki, Makassar).

Preparation of Sample

The glutinous rice is washed until clean and dried, then ground until it can pass through a 60-sieve. The next stage is an extraction process using 70% ethanol, which has been acidified (adding 1% acetic acid). The filtrate obtained was then evaporated at a temperature of 60^{0}C at 60 rpm until all the solvent had evaporated. (Suhartatik et al., 2019).

Formulation of Black Glutinous Rice Clay Mask

The mask is made in 100 grams, and the composition of each excipient is listed in **Table 1**. All ingredients are weighed according to the percentage in each formula. The preparation method is first to dissolve bentonite using 30 ml of aquadest into the mortar, then add black glutinous rice powder that has been mashed, then add xanthan gum, and grind quickly until homogeneous. Then added, glycerin and titanium dioxide into the mortar. Kaolin is added to the mortar while being crushed until the preparation is homogeneous. In a separate container, dissolve sodium metabisulfite and nipagin using 15 ml of hot water (container A) and sodium lauryl sulfate in 10 ml of distilled water (container B) and mix containers A and B into the core mixture. In the last part, add flavoring to the preparation. The procedure was repeated for other formulas with different amounts of kaolin and bentonite bases. Then an evaluation of the preparation of each formula is carried out.

Table 1. Formulation of Black Glutinous Rice Clay Mask (*Oryza Sativa* L. Var Glutinosa)

Ingredients	Concentration (%)		
	F1	F2	F3
Black Glutinous Rice	10	10	10
Bentonite	5	3	1
Xathan Gum	0.5	0.5	0.5
Glycerin	10	10	10
Sodium Lauryl Sulfate	1	1	1
Titanium Dioxide	0.5	0.5	0.5
Nipagin	0.1	0.1	0.1
Kaolin	15	25	35
Sodium Metabisulfite	0.2	0.2	0.2
Flavoring agent	qs	qs	qs
Distilled water	Add 100%		

Evaluation Of Availability

Organoleptic Test

Organoleptic observations included the clay mask preparation's shape, color, odor, and

consistency (Syamsidi et al., 2021; Wikantyasning et al., 2019).

Homogenity Test

The test is done by smearing the preparation on a piece of glass or other transparent material, where the material is taken from three parts: the top, middle, and bottom. The gel preparation is homogeneous if it shows no clumping or unmixed particles (Sayuti, 2015; Setiyadi et al.,, 2020).

pH Test

The pH of the preparations was measured using a pH meter with three repetitions and measured for 12 weeks of storage. The pH of the preparation was determined utilizing a pH meter. The instrument is initially calibrated by employing a buffer solution with a neutral pH of 7.01, as well as an acidic buffer solution with a pH of 4.01, until the instrument accurately displays the pH measurement. Subsequently, the electrodes were rinsed with distilled water and subsequently dried using a tissue. Formula is prepared at a concentration of 1% in distilled water. Next, the electrode is immersed into the solution. Allow the instrument to display the pH value until it reaches a state of equilibrium. The numerical value displayed on the pH meter corresponds to the pH level of the solution being analyzed. pH preparations that adhere to the specified skin pH range of 4.5-7.5 (F. Fauziah et al., 2020; Febriani et al., 2022).

Drying Time Test

The mask drying time was measured at room temperature ± 25°C by applying ± 1g clay mask preparation on the skin of the back of the volunteer's hand and measuring the time. The speed at which the practice dries and forms film-coating preparations is counted using a stopwatch. Good preparation drying time is 15-30 minutes (Setiyadi *et al.*, 2020; Febriani *et al.*, 2022; Syamsidi *et al.*, 2021).

Irritation test

The irritant test was conducted by topically applying the clay mask to the skin behind the ear. The applied area was then covered with Whatman paper and undisturbed for 24 hours. After this period, the paper layer was removed and left exposed for 30 minutes. Witness the chemical response taking place inside the designated region. Instances of irritation responses such as heat, itching, or discomfort are documented. Observations were conducted on a sample of ten women, all between the ages of 20 and 35. (Lestari et al., 2018, 2021)

RESULT AND DISCUSSION

Clay mask preparations from black glutinous rice are made using the standard pH-neutral clay face mask formula. This standard formula is modified to suit the shape of a clay mask with black glutinous rice powder as an active ingredient. Herbal cosmetics provide the benefit of being non-toxic, reducing the occurrence of allergic responses, and having a proven track record of effectiveness for many substances (Londhe et al., 2021). The amount of black Glutinous rice used is a concentration of 10%. The consistency of the clay mask preparation is in the form of a paste with a blackish-grey colour.

The analysis of homogeneity for the formed clay mask preparations revealed that none of the preparations exhibited the presence of coarse grains upon application to transparent glass. This observation demonstrates that every formula exhibits a homogeneous structure

Table 2. The results of measuring the pH Test

Time (weeks)	The pH test of formula		
Mean ± SD (n=3)	F1	F2	F3
0	6.15±0.18	7.01±0.42	8.21±0.11
1	6.16±0.22	7.03±0.33	8.16±0.12
4	6.21±0.35	7.11±0.25	8.17±0.21
8	6.20±0.28	7.10±0.27	8.15±0.11
12	6.21±0.19	7.11±0.23	8.15±0.19

Note:
F1: Clay mask with 5% bentonite and 15% kaolin
F2: Clay mask with 3% bentonite and 25% kaolin
F3: Clay mask with 1% bentonite and 35% kaolin

Organoleptic evaluation includes odour and colour. Observations showed that the odour and colour of each formula did not change during 12 weeks of storage at room temperature. This is by the provisions stipulated in the Indonesian Pharmacopoeia Edition III.

Table 3. Results of mask drying time measurements

Formula	Drying Time (t) Mean ± SD (n = 3)
F1	10.28±0.22
F2	9.20±0.18
F3	8.41±0.36

Note:
F1: Clay mask with 5% bentonite and 15% kaolin
F2: Clay mask with 3% bentonite and 25% kaolin
F3: Clay mask with 1% bentonite and 35% kaolin

No change in odour and colour of the mask preparation indicates that the clay mask preparation is stable in storage.

The clay mask preparation's pH might be found to range from 6.0 to 8.5 (**Table 2**). The pH range that is acceptable is 4.5–7.5. Kaolin itself has a pH that tends to be alkaline (pH=6.0–8.0), the results of pH tests on each preparation produced variable results (D. W. Fauziah, 2017; Husna et al., 2023). Therefore, the more acidic the pH of the mask preparation, the lower the kaolin content in a formula. This assertion is consistent with the pH value increasing from F1 to F3, were F3 has the highest pH due to the highest concentration of kaolin. The pH produced by each formula is different, F1 and F2 are formulas that meet the pH requirements of skin preparations used on the face. However, F3 does not meet the pH requirements for mask preparations because it is too alkaline and can cause the skin to become dry (Hwang et al., 2022; Jurecek et al., 2021). Another factor that influences the pH of the preparation is how long the preparation is stored; the greater the possibility of the pH increasing.

The drying time test of the clay mask preparation aims to determine how long it will take for the gel preparation to dry on the skin's surface and form a film layer (Pratiwi, 2023). The back of the hand area was chosen because it is easy to observe. Measurements were repeated three times with different volunteers. **Table 3** shows the results of the drying time measurements. Based on the results of measuring the drying time, the results range from 5-9 minutes, where the higher the concentration of the active ingredient, the longer the drying time for the mask.

Based on irritation testing on volunteers (**Table 4**), the results showed no side effects on the skin when using a clay mask made from black glutinous rice on the back of the ear and leaving it for 24 hours. There were no visible side effects in the form of redness, itching and rough skin on the skin caused by all clay mask preparations.

Table 4. Results of irritation test on volunteers

Formula	Observation	Volunteers									
		1	2	3	4	5	6	7	8	9	10
F1	Red Skin	-	-	-	-	-	-	-	-	-	-
	Itching	-	-	-	-	-	-	-	-	-	-
	Swelling	-	-	-	-	-	-	-	-	-	-
F2	Red Skin	-	-	-	-	-	-	-	-	-	-
	Itching	-	-	-	-	-	-	-	-	-	-
	Swelling	-	-	-	-	-	-	-	-	-	-
F3	Red Skin	-	-	-	-	-	-	-	-	-	-
	Itching	-	-	-	-	-	-	-	-	-	-
	Swelling	-	-	-	-	-	-	-	-	-	-

Note:
(+) = Irritation occurs
(-) = No irritation occurs

CONCLUSIONS

The optimum formula for clay masks from black glutinous rice was obtained at F1 (5% bentonite and 15% kaolin) and F2 (3% bentonite and 25% kaolin).

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AUTHORS' CONTRIBUTIONS

Ermalyanti Fiskia conducted preformulation studies and prepared the manuscript. Amran Nur evaluated the claymask formula, while Cindhany D.F.U Mala conducted data analysis and interpretation. All authors thoroughly reviewed the results and approved the final version of the manuscript.

CONFLICT OF INTERESTS

The authors have no conflict of interests related to this publication.

ETHICAL CONSIDERATION

Evaluation in this research has obtained ethical clearance from the Research and Health Ethics Committee, Faculty of Pharmacy, Hasanuddin University, Makassar, South Sulawesi. (169/UN4.17.8/KP.06.07/2024).

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