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Formulation of Anti-Blackhead Rubber Mask from Purified Gambier (*Uncaria gambir* [Hunter] Roxb.)

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ABSTRACT: The purified was isolated from *Uncaria gambir* [Hunter] Roxb. that contains more than 90% of catechins. Its antioksidant and antibacterial properties have potential to formulate as cosmetics. Dry form formulation of cosmetic was one approach to minimize cathecins degradation in a product. In this study, three formulation of rubber mask containing 5% of purified gambier was done. Each formulation was formulated with calcium sulfate, propylene glycol, magnesium carbonate, citrus essential oil and variation of HPMC and chitosan as film former. The three formulas had a brown color, smooth textures, the gambier odor, visually homogeneous, particle sizes at 112.24 – 116.39 µm, moisture values at 3.9 – 7.04%, pH of 6, drying times of 8 to 15 minutes, adhesive power of > 10 seconds and remained stable for 21 days at 4±2°C and 27±2°C. The actual catechin content in formula I, II, and III were 0.89%, 1.09%, and 1.39%, respectively. The best formula was formula III, which contained 40% HPMC and 10% chitosan due to the fastest drying time of 8.36 minutes ± 19 seconds and the highest catechins content at 1.39%.

Keywords: **cosmetics; HPMC; purified gambier; powdered masks; peeling-off.**

Introduction

Rubber mask is a powdered facial mask that requires mixing with water to create a thick paste before application to the face. Once it dries, this mask forms a thick and elastic layer with peeling-off properties, allowing it to be easily removed from the skin. The peeling-off characteristic of the rubber mask when it dries has made it popular in South Korea for blackhead treatment because it provides a tangible sensation to users regarding the blackhead removal process [1]. Blackhead (*comedonal acne*) can caused by acne vulgaris or triggered by androgen hormones [2–4]. Routine facial skincare is necessary for someone experiencing blackhead complaints, especially rubber mask.A natural ingredient that can provide a solution for blackhead issues is gambier (Uncaria gambir [Hunter] Roxb.). The catechin content in gambier had potential as antioxidant that can inhibit sebum production in the sebaceous follicles by suppressing sebocyte differentiation. Antioxidant compounds are known to inhibit sebum production in the sebaceous follicles by suppressing sebocyte differentiation [5]. Gambier also exhibits moderate to strong antibacterial potential against *P. acnes* [6]. The use of gambier gel peel-off masks has been proven to have a positive impact on the treatment of comedonal facial skin [7].

The formulation of purified gambier into a rubber mask is based on the higher possibility of enzymatic browning reactions in semisolid preparations compared to powder formulations [8].

The presence of oxygen groups in water causes semisolid preparations to contain higher levels of oxygen compared to powder formulations. This leads to lower stability in gel masks compared to powder. Regarding to Anggraini (2013), stability of anti-acne gel mask from ethyl acetate extract of gambier was at formulation with percentage of 0.5 and 1% of extract during room temperature and cold temperature of storage conditions [8]. Therefore, purified gambier with a catechin content of ≥ 90% is expected to be suitable for formulation as a rubber mask in the blackhead removal process.

In this study, purified gambier rubber masks were formulated

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into three formulas, each containing 5% purified gambier. These three formulas were varied based on the percentage of their film formers, namely Hydroxypropyl methylcellulose and chitosan. The combination of two film formers in this preparation aimed to enhance the strength and elasticity of the film so that the mask could be completely peeled off from the skin without breaking, providing maximum effect and comfort for users. Thus, this research is expected to bring innovation to the formulation of rubber masks using purified gambier.

Methods

Materials

Purified gambier was obtained from Andalas Sitawa Fitolab, West Sumatera Indonesia. Some other excipients were hydroxypropyl methylcellulose (MakingCosmetics, Washington), chitosan (Biotec Surindo, Indonesia), calcium sulfate (Karya Muda Indochem, Indonesia), propylene glycol (Dow Chemical Pacific, Singapore), magnesium carbonate (Techno Pharmchem, India), diatomaceous earth (Dicalite Minerals Corp, United States), citrus essential oil (Darjeeling, Indonesia), catechin (Andalas Sitawa Fitolab, Indonesia) and pro-analysis methanol (Merck, Indonesia).

Preparation Rubber Masks

Rubber mask formulations are in Table 1. In this study, three formulas (FI, FII, and FIII) were made for rubber mask of purified gambier with varying concentrations of two film formers, respectively, by 30, 35, 40 % for HPMC and 15, 12.5, 10 % for chitosan. The purified gambier was dispersed in propylene glycol in a mortar. Subsequently, citrus essential oil was added and homogenized. Afterward, diatomaceous earth was added gradually and homogenized. Magnesium carbonate and calcium sulfate were slowly added while continuously being homogenized with a pestle. After all the ingredients were thoroughly mixed, HPMC and chitosan were gradually added until the entire mass was uniformly blended. The mask mixture was then sieved through a 14-mesh sieve and dried in a drying cabinet for one hour, followed by drying at room temperature for 18 hours. This drying and sieving process was repeated three times. The rubber masks were placed into the container.

Evaluation

Organoleptic Test [9]

Organoleptic analysis was performed by observing the color, texture, and odor of the purified gambier rubber mask.

Particle Size Evaluation [9]

The purified gambier rubber mask was sieved using sieves with pore sizes of 212, 250, 355, 425, 600, 850, and 2000 µm, with 10 grams of the sample sieved for 5 minutes.

Moisture Test [10]

Five grams of rubber was put in to the plate of a precision moisture analyzer (Shimadu, Japan) and heat at 105°C. The process was considered concluded when the device's indicator light turned off, facilitating the determination of sample's percentage moisture content.

PH Test [11]

One gram of the rubber mask in paste form was pH-tested using universal pH strips. The color of the pH strips after the test was matched to the color range of the universal pH scale.

Table 1. Rubber masks formula

Ingredients	Formula (%)		
	FI	FII	FIII
Purified gambier	5	5	5
Hydroxypropyl methylcellulose (HPMC)	30	35	40
Chitosan	15	12.5	10
Calcium sulfate	10	10	10
Propylene glycol	10	10	10
Magnesium carbonate	4	4	4
Diatomaceous earth	25	22.5	20
Citrus essential oil	1	1	1

Homogeneity Test [9]

The mask in paste form was evenly spread on a glass microscope slide, and visual observations were made regarding the homogeneity of the paste.

Adhesive Strength Test [12]

One gram of the rubber mask in paste form was placed on a glass microscope slide. Another glass microscope slide was placed on top of it with a 50-gram metal calibration weight for 1 minute. One of the glass slides was then lifted, and the time it took for the sample to release from the glass slide was recorded.

Drying Time Test [17]

The rubber mask in paste form was applied to the back of the hand in a 4x4 cm area. The duration required for the mask to dry and form a peelable film layer on the skin was precisely measured.

Stability Test [18]

Samples were stored at a cold temperature (4±2°C) and room temperature (27±2°C) for 21 days. Systematic organoleptic evaluations were carried out on days 1, 7, 14, and 21. On the 21st day, comprehensive pH and drying time assessments were executed for all formulations considering both cold and room temperature conditions.

Determination of Catechin Content

Determination of Actual Catechin Content in Purified Gambier

Ten milligrams of purified gambier were dissolved in a 10 mL volumetric flask with methanol. Then this solution was diluted in methanol to made concentration of 35 μg/mL. t The solution was measured using a UV-Vis spectrophotometer at a wavelength of 280.5 nm. The catechin content was measured using the regression equation obtained from the catechin calibration curve.

Determination of Actual Catechin Content in the Formulations

Seventy milligrams of each rubber mask formulation were dissolved in a 25 mL volumetric flask with methanol. The solution was sonicated for 1 hour, and then filtered using filter paper. The filtrate was measured using a UV-Vis spectrophotometer at a wavelength of 280.5 nm. The catechin content was measured using the regression equation obtained from the catechin calibration curve.

Result and Discussion

The result of the organoleptic evaluation of the three rubber mask formulation were powder form, brown color, smooth texture, and only had a distinctive gambier purified aroma (Figure 1 and Table 2). The brown colour of the mask was formed due to the addition of 5% purified gambier into the formula. According to the Indonesian Herbal Pharmacopoeia edition II (2017), gambier extract is characterized by a surface color ranging from light brown to reddish dark brown [15]. The brown color in gambier is generated by flavonoid tannin compounds with the ability to impart shades of yellowish-brown and reddish-brown [16] All the rubber mask formulas retained the distinctive scent of purified gambier, even with the addition of citrus essential oil as a fragrance. The disappearance of the citrus essential oil aroma of the rubber mask was ascribed to the volatile nature of limonene as the characteristic aroma to citrus essential oil [17,18]. The volatility of limonene led to the transient nature of the citrus fragrance in all three formulas as it evaporated during the drying process. Consequently, the distinctive scent of purified gambier persisted in all three formulations of the rubber mask.

The average diameters for formulas I, II, and III were 112.24, 114.86, and 116.39 μm, respectively (Table 2). Formulas I, II, and III met the particle size requirements for powdered mask formulations, which are less than 1 mm [19]. Data in Figure 2 was utilized to compare the

a b c

Figure 1. Physical appearance of rubber mask a. FI b. FII c. FIII

Table 2. The evaluation of rubber masks

Evaluation		FI	FII	FIII
Organoleptic	Form	Powder	Powder	Powder
	Colour	Brown	Brown	Brown
	Odor	Gambier aroma	Gambier aroma	Gambier aroma
	Texture	Smooth	Smooth	Smooth
Average Particle Diameter (µm)		112.24	114.86	116.39
Average Moisture (%) ± SD		3.90 ± 0.06	5.67 ± 0.10	7.04 ± 0.08
pH		6	6	6
Adhesion Stregth		> 10 seconds	> 10 seconds	> 10 seconds
Driying time ± SD		15.38 minutes ± 16 seconds	10.35 minutes ± 14 seconds	08.36 minutes ± 19 seconds

particle size distribution of rubber mask formulations I, II, and III. Samples with the same average particle diameter can exhibit different particle distribution patterns [24]. The slope of the lines formed by the graph of the three formulas was nearly parallel to the x-axis, indicating that all three formulas had a narrow particle size distribution. This type of particle size distribution suggested a high degree of uniformity in the particle size of formulas I, II, and III [20]. This was substantiated by the fact that over 95% of all three formulas could pass through the smallest sieve pore size, which was 212 µm. The size and distribution of particles impacted the physical characteristics of a preparation, influencing texture finesse, solubility during mask-water mixing, and the uniformity of active substances in the preparation [21]. As particle size decreased, the resulting mask texture became finer, and the increased particle surface area in contact with warm water during application enhanced solubility. This led to easier dissolution of the mask when mixed with warm water. Particle size also influenced the release process of active ingredients from the formulation. Smaller particle sizes contributed to a more efficient release of active ingredients from the rubber mask formulation, enabling better penetration into the skin and delivering the desired effects [22].

The moisture percentage of all three rubber mask formulas in Table 2 mets the quality standard for water content in facial mask formulations, as specified in SNI 16-6070-1999, which is less than 10% [23]. Low water content has the advantage of extending the shelf life of the formulation, whereas high water content can create a conducive environment for the growth of microorganisms such as fungi, which typically thrive in water content above 10% [24]. The average moisture content in all three formulas was directly proportional to the percentage of HPMC contained in each formula. The higher the HPMC percentage in a formula, the higher the moisture content of the mask. This was attributed to the hygroscopic nature of

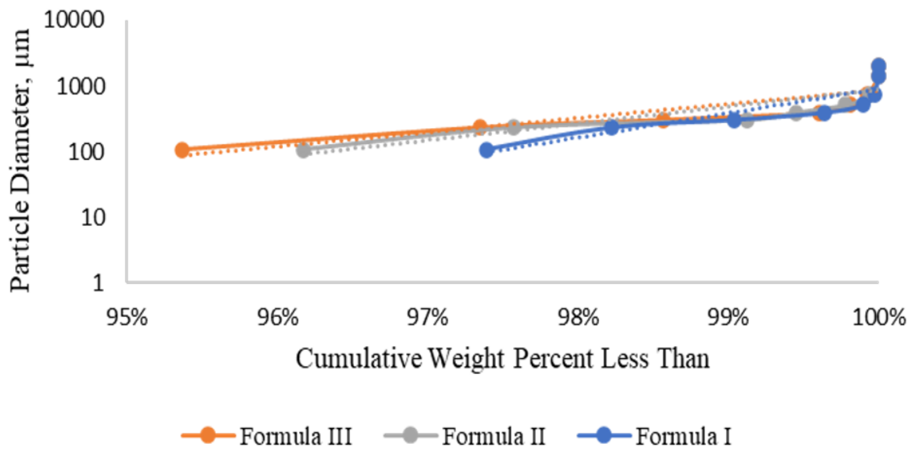

Figure 2. Logarithmic scale of particle size distribution

Table 3. Stability test of the rubber masks

Criteria		Temperature	FI	FII	FIII
Organoleptic	Form	4±2°c	Powder	Powder	Powder
	Colour		Brown	Brown	Brown
	Odor		Gambier aroma	Gambier aroma	Gambier aroma
	Texture		Smooth	Smooth	Smooth
	Form	27±2°c	Powder	Powder	Powder
	Colour		Brown	Brown	Brown
	Odor		Gambier aroma	Gambier aroma	Gambier aroma
	Texture		Smooth	Smooth	Smooth
pH		4±2°c	6	6	6
		27±2°c	6	6	6
Driying time ± SD		4±2°c	15.30 minute ± 9 seconds	10.43 minute ± 14 seconds	08.27 minute ± 13 seconds
		27±2°c	15.60 minutes ± 15 seconds	10.73 minutes ± 9 seconds	08.68 minute ± 4 seconds

HPMC after drying. The hygroscopic property of HPMC after the rubber mask dried allowed the mask preparation to absorb water molecules from the surrounding environment. Therefore, the higher the percentage of HPMC in the rubber mask, the higher the water molecules absorbed from the surrounding environment, resulting in an increased moisture content of the rubber mask [25]. In addition to HPMC, other ingredients in the mask preparation that influenced the moisture content percentage were chitosan and diatomaceous earth, both of which also had hygroscopic properties [26,27]. However, the significantly greater percentage of HPMC compared to chitosan and diatomaceous earth caused the moisture content percentage of the rubber mask to increase with the increasing amount of HPMC in the formula. The temperature and drying duration had a significant impact on the moisture content of a preparation. High temperature and prolonged drying time resulted in reduced moisture content in the preparation [28].

The pH values for formulas I, II, and III were 6 (Table 2), falling within the range of 4.5 to 8, which met the quality requirements for mask formulations according to the SNI 16-4399-1996 standard [29]. The ideal pH range for skin is typically between 4.5 and 6.5. Maintaining the correct pH level in cosmetics is crucial to ensure the skin's protective layer, known as the acid mantle, functions optimally. The acid mantle is a thin layer on the skin's surface composed of lipids, amino acids, and natural skin oils. It plays a crucial role in preserving skin moisture, protecting the skin from pollution, dirt, pathogenic bacteria, and preventing inflammation, dehydration, and premature aging of the skin. Using a mask with the appropriate pH is essential to ensure maximum skin care benefits. Masks with high pH levels can lead to dry skin, while masks with excessively low pH levels can strip away natural oils and damage the skin's lipid barrier. Therefore, maintaining a cosmetic pH level within the range of 4.5 to a maximum of 8 is vital to ensure the effectiveness of a cosmetic product [30].

Formulas I, II, and III appeared to have been uniformly mixed without any coarse particles visible on the glass microscope slide (Figure 3). A cosmetic preparation is considered homogeneous when there are no coarse particles and no variation in color within the formulation [31]. The homogeneity of a preparation is closely related to the consistency of the active ingredients within the product during each use. This factor can subsequently

a b c

Figure 3. Homogeneity evaluation of rubber mask a. FI b. FII c. FIII

Table 4. The actual content of catechins in purified gambier and rubber masks

Sample	Absorbance (280.5 nm)	Content (%)	Average Content (%) ± SD
Purified Gambier	0.589	90.24	90.08 ± 0.014
	0.594	91.43	
	0.582	88.57	
FI	0.516	0.91	0.89 ± 0.020
	0.508	0.89	
	0.502	0.87	
FII	0.582	1.11	1.09 ± 0.015
	0.577	1.09	
	0.573	1.08	
FIII	0.680	1.40	1.39 ± 0.015
	0.669	1.37	
	0.676	1.39	

impact the effectiveness of the therapy provided by the preparation [32].

Formulas I, II, and III provided adhesion results that met the criteria, with adhesion times exceeding 10 seconds, and the masks remained attached to the glass microscope slide for more than 30 minutes (Table 2). The presence of HPMC in all three formulas played a role in enhancing the adhesion of the masks. It has been demonstrated that as the concentration of HPMC increases, the adhesion in each formula also improves [31]. The adhesive ability of the rubber mask has an impact on the therapeutic effect it delivers. The longer the rubber mask can adhere to the skin, the longer the therapeutic effect can last [32].

The drying time test results in Table 2 indicated that formulas I, II, and III met the criteria for the drying time of facial mask preparations, taking less than 30 minutes to dry and can be easily peeled off from the skin. The difference in concentrations of HPMC and chitosan had a significant effect on the drying time of the mask. Higher HPMC concentration in the mask preparation led to a shorter drying time, as did chitosan [33,34]. Therefore, all three rubber mask formulas in this study could achieve drying times of less than 30 minutes due to the significant impact of the combination of film formers, HPMC and chitosan on the drying time of the mask preparation.

Formulas I, II, and III were found to maintain their

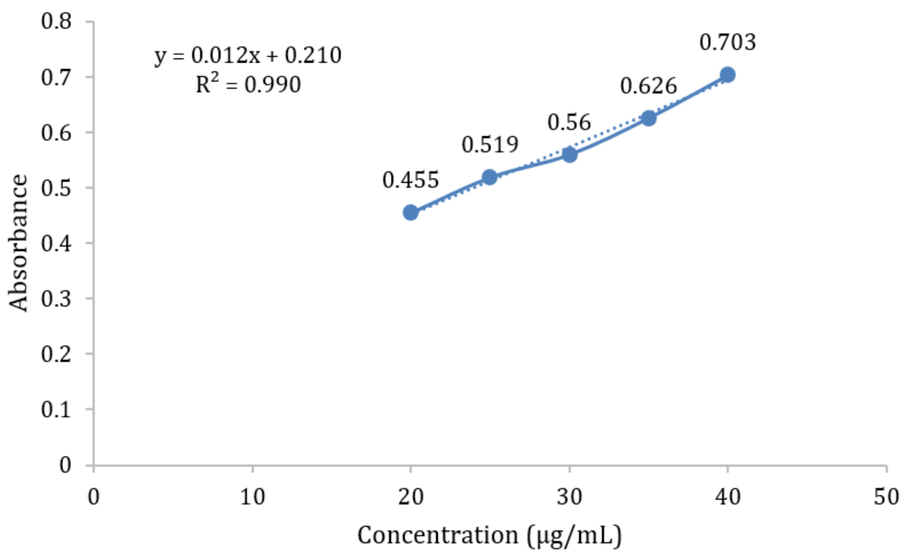

Figure 4. Catechins calibration curve

organoleptic properties throughout 21 days of storage at temperatures of 4±2°C and 27±2°C. All three formulas retained their powder form, brown color, smooth texture, and the characteristic aroma of gambier extract (Tabel 8). The powder preparations in this study demonstrated greater stability compared to the semisolid preparations in the previous research [8]. Catechin is a phenolic compound that is prone to oxidation. One crucial factor that influenced the stability of phenolic compounds was the presence of oxygen. The presence of oxygen groups in water resulted in gel mask preparations having a higher oxygen content compared to powder preparations. This caused the powder formulation to be less susceptible to oxidation compared to the gel formulation. Additionally, the narrow inter-particle spaces in the powder formulation, as opposed to the gel formulation, offer reduced opportunities for interaction with oxygen [35].

Formulas I, II, and III at both storage temperatures of 4±2°C and 27±2°C maintained a pH of 6 throughout the 21 days of storage (Table 3). The pH evaluation in the stability test of the powder mask proved more stable to the pH evaluation in the gel mask stability test. In the gel mask stability test, the ethyl acetate extract of gambier concluded that the pH value in the gel preparation decreased every week during storage [8]. Phenolic compounds found in catechins release H+ ions in gel preparations containing water, subsequently causing the pH value to become acidic. In this study, it is evident that the powder preparation is more stable in terms of pH compared to the gel preparation.

The drying time of rubber mask preparations for formulas I, II, and III remained unchanged for 21 days of storage at temperatures of 4±2°C and 27±2°C, consistently falling within the range of 8 to 15 minutes (Table 3). The combination of two film formers, HPMC at 30%, 35%, and 40%, and chitosan at 15%, 12.5%, and 10% in this formulation successfully maintained the drying time within the 8 to 15-minute range during 21 days of storage at temperatures of 4±2°C and 27±2°C.

The actual catechin levels in purified gambier could be observed in Table 4. The average catechin content in purified gambier was found to be 90.08% ± 0.014. This aligns with the certificate of analysis for purified gambier, which states that gambier contains ≥ 90% catechin.

The average catechin levels in formulas I, II, and III were 0.89%, 1.09%, and 1.39%, respectively (Table 4). The expected actual catechin levels were 4.51% in each formula. The lower actual catechin levels in the masks were due to some of the gambier in the mask forming a compact mass with other excipients. This impacted the solubility of purified gambier in the formulation in the methanol solution [36]. Consequently, the actual catechin levels obtained were lower when compared to the theoretical actual catechin levels in each formula. The catechin levels In all three rubber mask formulas still mets the percentage of gambier extract that can inhibit *Propionibacterium acne*. Antibacterial activity against *Propionibacterium acne* has been found with gambier extract at concentrations as low as 0.25% to 6%. Therefore, it is expected that the actual catechin levels in these three rubber mask formulas can inhibit the activity of *Propionibacterium acne*.

Conclusion

Purified gambier can be formulated into a rubber mask, and all three formulas created met the criteria for mask preparations remained stable for 21 days at 4±2°C and 27±2°C. Among the three rubber mask formulas formulated, the best formula was Formula III, which included a combination of 40% HPMC and 10% chitosan as film formers. This formula exhibited the fastest drying time, approximately 8.36 minutes ± 19 seconds, and the highest actual catechin content, which was 1.39%.

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