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Development of Coffee Fruit Skin (*Coffea canephora*) Formula as Antioxidant peel-off Masks

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ABSTRACT: The outer skin of the robusta coffee bean (*Coffea canephora*) was a processing waste containing phenolic compounds with an antioxidant role. This activity is relevant when used in cosmetic preparations such as peel-off masks, which form a thin elastic layer on the facial surface and are easy to remove. One of the crucial ingredients of peel-off masks is a plasticizer such as sorbitol, characterized by flexibility, not easily fragile, and elasticity. Therefore, this research aimed to develop a peel-off formula from robusta coffee skin by influencing sorbitol variations on the characteristics of masks in the form of a gel. A total of six gel mask formulas were made with sorbitol variations of 10%, 12.5%, and 15%. The two types of gel base used were Carbopol 940 and HPMC (Hydroxy Propyl Methyl Cellulose), at concentrations of 0.25% and 4%, respectively. The results showed that sorbitol variations affected the physical characteristics ($p > 0.05$) but did not influence the antioxidant activity of robusta coffee skin extract peel-off masks.

Keywords: **coffe bean skin; peel-off mask; sorbitol; physical characteristics.**

Introduction

An antioxidant is a chemical compound capable of preventing free radicals to slow down facial skin aging and produce a youthful effect [1]. Many natural ingredients have been proven to possess antioxidant activity, including robusta coffee bean skin (*Coffea canephora*). Generally, coffee skin is a waste from coffee bean processing that has not been used optimally. This waste has high efficacy as a natural antioxidant due to the content of secondary metabolite compounds such as caffeine and polyphenols [2-4]. The polyphenolic compound in robusta coffee skin is chlorogenic acid, which possesses antioxidant activity [1, 5]. Chlorogenic acid has potent antioxidant activity, anti-aging, and photoprotective abilities. The content of these beneficial compounds means that coffee bean skin can be used as an active ingredient in cosmetic formulations. [6]. According to previous research, robusta coffee skin extract can capture free radicals of approximately (IC_{50}) 72.96 ppm. This leads to its inclusion in the potent antioxidant category due to the IC_{50} value being between 50 and 100 ppm [6]. Its compound content and properties mean that coffee bean skin can be an active ingredient in cosmetic formulations.

The use of robusta coffee skin extract can be increased in cosmetic preparations of facial masks, namely peel-off masks in gel form. These masks are easily applied to skin facial layers, peeled, or lifted as an elastic membrane [7]. The benefits associated with the masks include the prevention of aging, skin hydration, improved health status, and treating wrinkles, acne, and shrinking pores [1,8]. Components that influence the physical properties of peel-off masks are gelling agents, film formers, and plasticizers. Examples of gelling agents include Carbopol 940 and Hydroxy Propyl Methyl Cellulose (HPMC). Carbopol 940 is a synthetic polymer capable of forming a stable gel with high viscosity and can be used as a thickener [9,10]. During manufacturing, Carbopol 940 is dispersed in water, which cannot be diffused until it is

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neutralized with a suitable base to form a gel with a neutral pH. Due to the low pH of 4.5 – 5.5, there is a need to add a base such as triethanolamine to achieve a neutral value of 5-6 and prevent skin irritation [11]. Previous research has established that the concentration of Carbopol 940 suitable for use as a gelling agent is 0.1 - 2.0% [12].

HPMC is a semisynthetic polymer that can be used as a gelling agent in gel preparation [13-15]. HPMC produces a stable gel preparation that meets the standard requirements. Additionally, it is a hydrophilic base capable of making good skin dispersion and a cooling effect, not clogging skin pores, and is easy to wash with water. It has a promising drug release profile [16,17]. HPMC is categorized based on molecular weight and the number of methoxy and hydroxypropyl groups, producing different viscosities and physical properties [18]. This research used high-viscosity type HPMC because a concentration of 2-4% could produce peel-off masks that met standard requirements [19].

Another component of peel-off masks is a plasticizer capable of changing the physical properties of preparation, has a high boiling point, and does not quickly evaporate. In this context, sorbitol is a plasticizer that plays a significant role in peeling masks from the face after drying, increasing elasticity and preventing cracking [20]. Therefore, this research aimed to develop a peel-off formula from robusta coffee skin by influencing sorbitol variations on the physical characteristics of masks in the form of a gel.

Methods

Materials

The materials used in this research were robusta coffee skin (*Coffea canephora L.*) obtained from Sukawangi Village, Bogor Regency, Carbopol 940 (Corel Pharma Chem; Ahmedabad, India), HPMC (Making Cosmetics; Redmod, USA), TEA (Merck; Darmsdat, Germany), and PVA (Chang Chun Petrochemical; Taiwan, China). Other material included Sorbitol (Gujarat Ambuja Exports; Ahmedabad, India), Phenoxyethanol (PCI Innovative Chemicals; Selangor, Malaysia), Aquadest, DPPH powder (Tokyo Chemical Industry; Tokyo, Japan), Methanol p.a (Merck; Darmsdat, Germany), Ethanol 96% technical, Ascorbic Acid, and Gallic Acid (Sigma Aldrich; St. Louis, MO).

Preparation Coffe-skin Extract

The extraction preparation starts with determining the robusta coffee fruit plant (*Cofeea canaphora*) at the Herbarium Bogoriense, Biosystematics and Evolution Research Sector, BRIN, Cibinong. Subsequently, the coffee skin was separated from the dirt, and the outer part was collected and dried. The extraction process used the maceration method at 96% ethanol solvent. One part of dry simplicia powder was mixed with 10 parts of 96% ethanol (1:10) and evaporated with a rotary evaporator at 50^{0}C to obtain a thick extract. The robusta coffee skin mixture is left for 24 hours, filtered with a sterile filter and funnel to separate the filtrate from the sediment/dregs. The remaining robusta coffee skin dregs are re-macerated with a new solvent. Then, evaporation is carried out using a rotary evaporator at a temperature of 50^{0}C to obtain a thick extract. The results obtained were weighed, followed by calculating the yield value. The next stage of extract characterization included organoleptic, pH testing using a pH meter (Hanna Instruments), as well as determining water and ash content [21], phytochemical screening, and qualitative testing of phenol content using TLC [22].

Robusta Coffee Skin Extract Peel-off Masks Formula

The preparation of coffee skin extract peel-off masks in the form of gel was carried out using Carbopol 940

Table 1. Robusta coffee skin extract peel-off masks formula.

Composition	Concentration (% b/b)					
	F1	F2	F3	F4	F5	F6
Robusta Coffee Skin Extract	5	5	5	5	5	5
HPMC	-	-	-	4	4	4
Carbopol 940	0.25	0.25	0.25	-	-	-
TEA	0.75	0.75	0.75	-	-	-
PVA	10	10	10	10	10	10
Sorbitol	10	12.5	15	10	12.5	15
Phenoxyethanol	0.5	0.5	0.5	0.5	0.5	0.5
Aquadest ad	100	100	100	100	100	100

and HPMC, with varying concentrations of sorbitol as the plasticizer. The peel-off mask formula conducted in this research is shown in Table 1.

The making of peel-off masks started with developing each gelling agent. In the first stage, Carbopol was dispersed in distilled water for 24 hours until it swelled completely and added with TEA (M1.a) [23]. The HPMC process was carried out by dispersing in sorbitol and developing in heated distilled water at 80°C, which was left for one night (24 hours) as mass 1 (M1.b). In the second stage, PVA was dispersed in hot distilled water at 120°C until it became a thick and clear gel with constant stirring for 1 hour using a magnetic stirrer (Wisestir) at a speed of 350 rpm (M2) [19]. Furthermore, M1.a and M1.b are mixed with M2 in a beaker glass with sorbitol and phenoxyethanol, followed by stirring until homogeneous. The final stage included the addition of robusta coffee skin extract, which had been dispersed with the remaining distilled water into the homogeneous gel mixture and stirred at a speed of 1200 rpm for 3 minutes until evenly mixed.

Evaluation of Robusta Coffee Skin Extract Peel-off Masks

Evaluations carried out on peel-off masks were organoleptic observations, pH testing using a calibrated digital pH meter, viscosity with the Anton-Paar Viscometer (Type Visco QC 300 R), spreadability test [23], adhesive power [24], and drying time [7]. Testing elongation and tensile strength is an important evaluation stage for peel-off masks, which is determined using a Tensile Tester (Shimadzu AGS-X series 10kN) [25].

Antioxidant Activity Testing

Determination of antioxidant activity was carried out in stages on coffee skin extract and masks using the DPPH method. The test was performed using a solution of coffee skin extract at several concentrations. DPPH solution was added to each sample, stirred with a vortex, and incubated at 37°C for 30 minutes in a dark room. Furthermore, the absorbance of the sample was read at the maximum DPPH wavelength using UV-Vis spectrophotometry (Shimadzu-UV-19001). The results obtained were entered into the equation to obtain the % inhibition value:

% inhibition =(Blank absorbance - Absorbance of the Sample)/(Blank absorbance) x 100

The IC_{50} value as a parameter of the strength to inhibit free radicals was determined using a linear regression equation obtained by plotting the sample concentration value (x-axis) and the % inhibition value (y-axis) [26,6]. The same procedure was carried out on the coffee skin extract peel-off masks that had been physically evaluated.

Result and Discussion

Characterization of Coffee Skin Extract

Characterization results showed a thick extract with a dark brown color and a distinctive odor. The extract yield obtained was 10.59%, fulfilling the value requirement above 10% [21]. The results of phytochemical screening showed that the extract contained phenolic compounds with the formation of a dark green color when reacted with $FeCl_3$ solution [27]. Qualitative testing was carried out

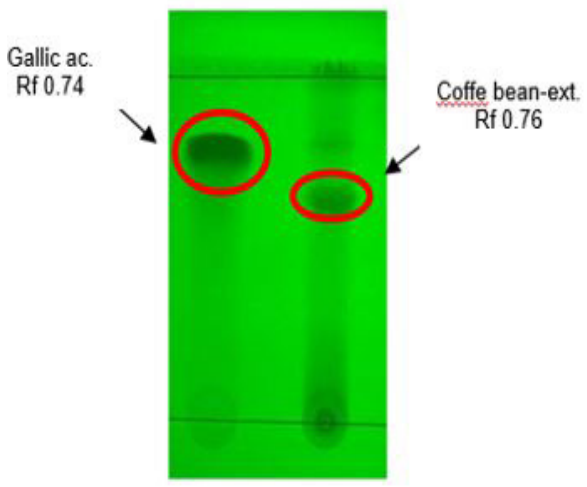

Figure 1. TLC test results for coffee skin extract.

Table 2. Characteristics of robusta coffee skin extract.

Characteristics	Result	Requirements
Form	Thick	-
Color	Dark chocolate	-
Smell	Typical	-
Extract Yield	10.59 %	> 10% [21]
Phytochemical Screening (Phenol)	(+) dark green	Dark green
Extract pH	3.71	-
TLC Phenol Extract	0.76	0.2-0.8
Gallic acid phenol TLC	0.74	0.2-0.8
Water content	15.45 %	5-30 %
Ash content	7.46 %	< 10% ([21]

using gallic acid as a comparison with the TLC method. Gallic acid has a high reactivity towards $FeCl_3$ and is a derivative of hydroxybenzoic acid, a natural and stable phenol [28,29].

Based on the spots observed under 254 nm U.V. light, the Rf value of extract and gallic acid was 0.76 and 0.74, respectively, as shown in Figure 1. This value showed that the two compounds had close Rf values, indicating the same characteristics or compound content [30].

The results showed that the water content of robusta coffee skin extract of 15.45% met the specified requirements, namely the range of 5-30% [21]. Generally, low extract quality is usually affected by mold due to excessive water content. In this research, the ash content test was conducted to determine the mineral content. The results obtained from the coffee skin extract were 7.46%, which fulfilled the requirements below 10%. This value showed that the extract had high purity when ash content was low, indicating suitability for use [31]. Detailed results of robusta coffee skin extract characteristics are shown in Table 2.

Antioxidant Activity Test of Robusta Coffee Skin Extract

Antioxidant activity is a preliminary test performed to determine the ability of the extract to inhibit free radicals. It serves as an indicator of the presence of phenol and flavonoid content, according to a report by a previous study [29]. The test uses the DPPH method and vitamin C solution as a comparison. The initial stage was to determine the maximum wavelength of the DPPH solution, which was obtained at 516.3 nm, and a linear regression equation. Coffee skin extract was made

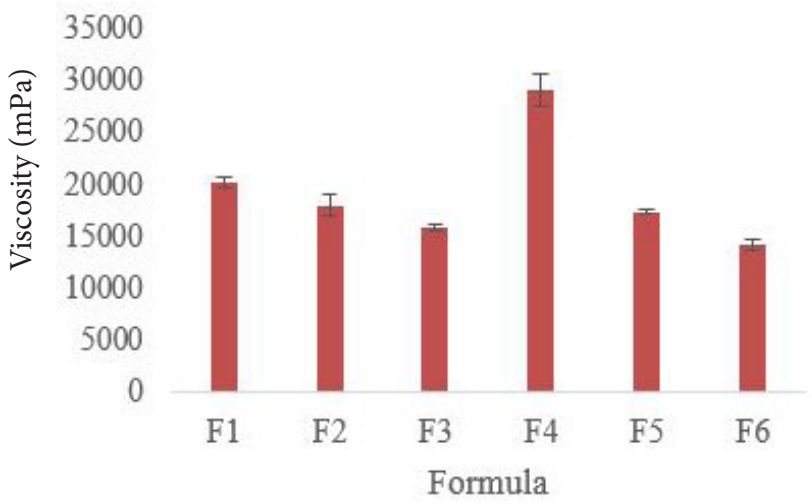

Figure 2. Viscosity value of robusta coffee bean skin extract peel-off masks.

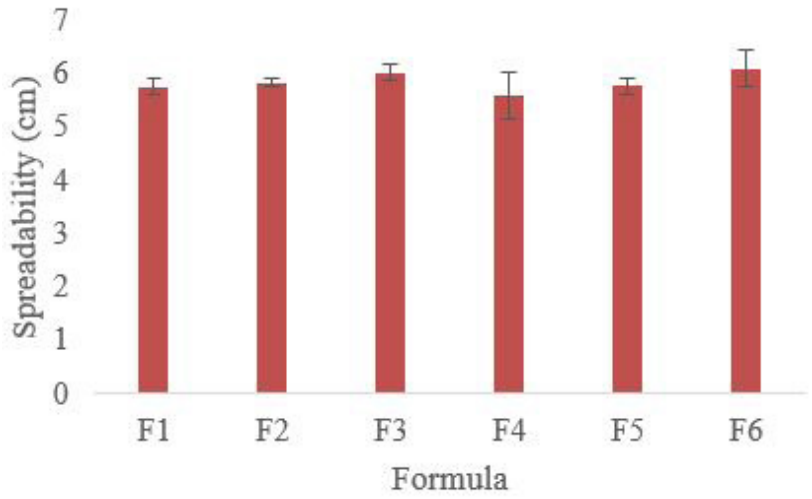

Figure 3. Spreadability value of robusta coffee bean skin extract peel-off mask.

in several concentrations (ppm), followed by calculating the inhibition ability of the extract solution, known as Inhibition Concentration (IC_{50}). The IC_{50} value for robusta coffee skin extract was obtained at 24.48 ppm, which showed that the antioxidant activity was potent due to an IC_{50} value < 50 ppm. Vitamin C is used as a comparison due to its use in topical preparations at a concentration of 5% with the potential as an antioxidant [32].

Characterization of Robusta Coffee Skin Extract Peel-off Masks

The characterization of peel-off masks was determined based on pharmaceutical standard preparation requirements. Physical observations include organoleptics, determination of pH, viscosity, spreadability, and stickiness. Meanwhile, as peel-off masks, observations continued with the drying time, elongation, and tensile strength. An antioxidant activity test was also carried out to ensure the effectiveness of mask preparation in inhibiting free radicals.

Organoleptic Test

The results of organoleptic observations, including the smell, color, and consistency of each formula, showed that differences in sorbitol variations caused a significant difference. As the sorbitol concentration increased, the preparation formed became more liquid [33] due to sorbitol's high hygroscopic and large molecular weight. This showed that greater molecular weight caused more significant gaps between water molecules, thereby

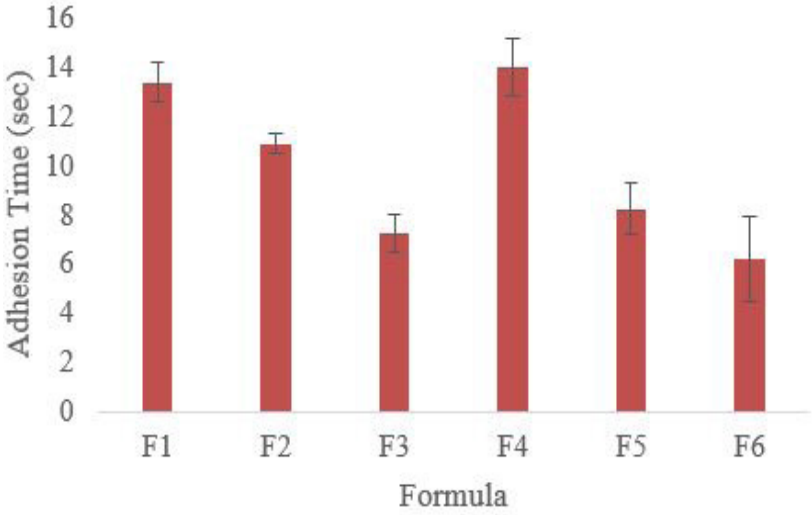

Figure 4. Test results for the adhesive power of the robusta coffee bean skin extract peel-off mask.

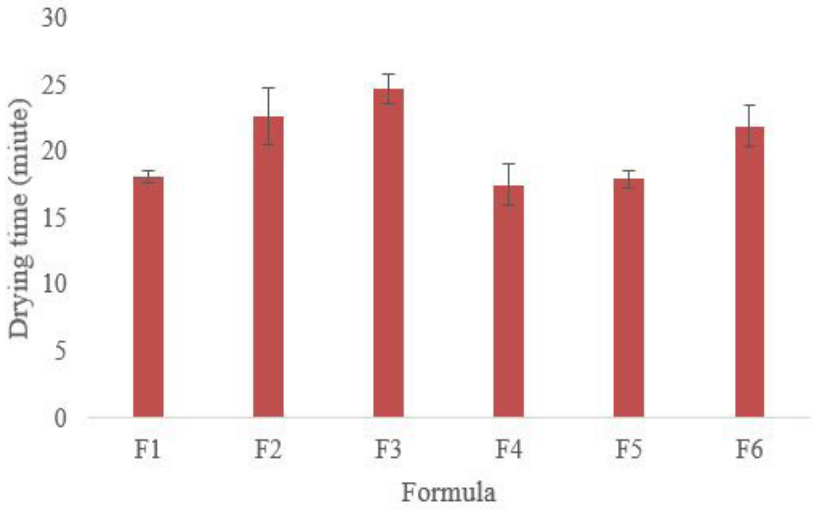

Figure 5. Drying time for robusta coffee bean skin extract peel-off masks.

increasing the amount of bound water [34]. Large amounts of sorbitol used would produce peel-off masks that were more liquid but flexible [34,35].

pH Test

The acidity level is an essential parameter in skin care products. Low or too acidic value can cause skin irritation, while alkaline preparation can make the skin scaly [36]. The gel pH test results for all formulas were in the required range, thereby preventing skin irritation, with values ranging from 4.5 to 6.5 [38]. Based on the results, pH values in the six formulas ranged from 5.16 – 5.17, with a significant difference ($p > 0.05$).

Viscosity Test

Viscosity testing was carried out to determine the ability of the preparation to flow from the container and spread over the skin surface. Based on the results, the peel-off mask preparation showed good flowing consistency, with a viscosity ranging from 14096.67 – 29070 mPa. The viscosity value of all formulas meets the requirements because the value is 7100 - 83144 mPa [39]. Data analysis shows that different gelling ingredients influence ($p < 0.05$) all formulas. The test results can be seen in Figure 2. These results showed that Carbopol 940 at a lower concentration than HPMC produced gel that meets the requirements with a pH value of ± 5. The difference in sorbitol variations also affected the viscosity of preparation ($p < 0.05$). With increasing sorbitol variations, the use of both gelling agents caused a significant decrease in viscosity. This occurred due to the hygroscopic nature of sorbitol, as increasing concentration contributed to high viscosity

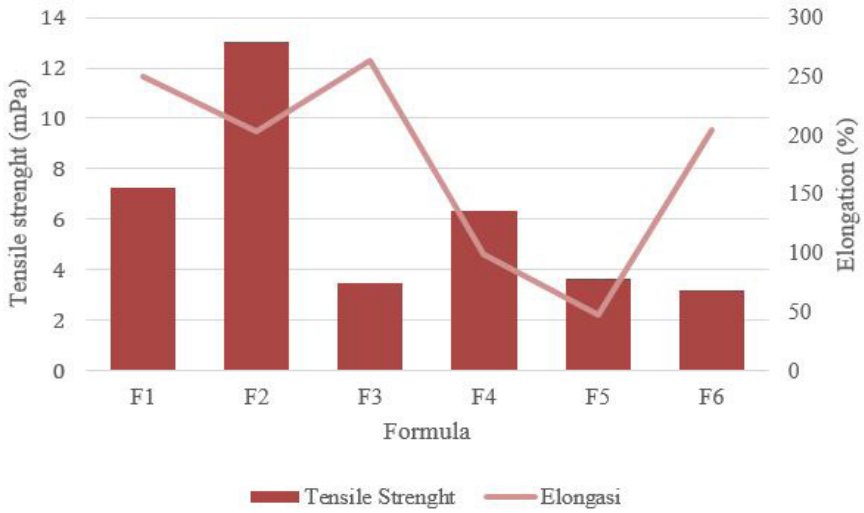

Figure 6. Mechanical properties test results of robusta coffee bean skin extract peel-off masks.

Table 3. Antioxidant activity test results of peel-off masks.

Formula	IC_{10} (ppm)
F1	92,80 ± 7,62
F2	94,90 ± 0,99
F3	96,92 ± 8,34
F4	90,38 ± 3,79
F5	98,86 ± 9,63
F6	91,08 ± 8,18

[34].

Spreadability Test

Spreadability testing was performed to determine the wide and even distribution of the formula when applied to the skin. The results showed that all formulas were not significantly different ($p > 0.05$), ranging from 5.60 to 6.10 cm, as presented in Figure 3. This showed that the formula was in a good spreadability range, according to the semisolid category, with values between 5-7 cm [37,38].

Adhesion Test

Adhesion testing was carried out to determine when the preparation adhered to the skin surface after application. Preparations with strong adhesion would remain in the skin layer for a long time and can clog skin pores. However, when the attachment time is short, the therapeutic effect of the preparation will not be achieved. Based on the results, all formulas adhered at no less than 4 seconds and were categorized as meeting the requirements [24]. Different types of gelling agents with the same sorbitol did not affect ($p > 0.05$) the adhesive power of peel-off masks. However, differences in sorbitol affected the adhesive power of peel-off masks, as shown in Figure 4.

Adhesion depends on the viscosity value and consistency of the preparation. This shows that when the viscosity meets reasonable standard requirements, it will retain the active substance of the preparation [39]. When the viscosity value is low, the preparation flows more efficiently, increasing the distribution and reducing the sticking force [40, 41].

Drying Time

After application, a drying time evaluation was carried out to determine the time required for masks to dry and form a thin, elastic layer on the skin layer. Based on the results presented in Figure 5, peel-off mask preparation took 17.48 – 24.65 minutes to dry and form a film layer. The results met the requirements, namely 15-30 minutes [38]. According to a previous study, peel-off masks in a gel form that dried quickly were preferred due to comfort on the skin surface [43].

Differences in sorbitol concentration influenced the drying time test results ($p < 0.05$). This occurs due to the evaporation of water from peel-off mask preparation, which is hampered because sorbitol can reduce internal hydrogen bonds in intermolecular bonds. Therefore, a greater sorbitol concentration correlates with high water content and longer drying time [44].

Elongation and Tensile Strength

Determination of the mechanical properties was carried out in the form of elongation and tensile strength. This served as a measure of the strength limit of peel-off masks to accept tension and survive before the film layer breaks. Subsequently, tests were carried out for all formulas, and the results showed tensile strength values in the 3.21 – 13.04 mPa range (Figure 6). Based on the JIS 1975 standard (*Japanese Industrial Standard*), the minimum value of 0.3922 mPa still meets the requirements. The results show that sorbitol is a suitable plasticizer because it can reduce the energy required for molecules to move, thereby causing a significant reduction in the stiffness and tensile strength values [44]. A Dosage form with a lower tensile strength value will not break easily, indicating good elastic properties. Otherwise, a dosage form with a high tensile strength value is susceptible to breaking easily.

Antioxidant Activity of Peel-Off Masks

The antioxidant activity of peel-off masks was evaluated by determining the IC_{50} value using the DPPH method, and vitamin C was used as a comparison. The sample solution containing antioxidant compounds was reacted with DPPH. Subsequently, an indication of the capture of free radicals was marked by a change in

the solution, which was initially purple to yellow. This identified the reaction of the DPPH molecule with the sample solution, namely reducing the DPPH solution and forming the compound 2,2-diphenyl-1-picrylhydrazine [45]. The antioxidant activity test results of peel-off masks with DPPH are shown in Table 3.

According to experience, antioxidant activity is potent when the IC_{50} value is less than 50 ppm [6]. All formulas had antioxidants in the strong category due to an IC_{50} value of less than 100 ppm, compared to vitamin C, which had a high IC_{50} of 14.82 ppm. The results indicate that the antioxidant activity of robusta coffee skin extracts peel-off masks was unaffected by variations in the gelling agent type and sorbitol ($p > 0.05$). These results prove that sorbitol is inert, which is one of the requirements for excipient ingredients in pharmaceutical preparations. It does not affect the pharmacological activity of pharmaceutical preparations [46]. This showed that robusta coffee skin extract had the potential as an antioxidant in the preparation of peel-off masks using Carbopol 940 and HPMC as gelling agents with sorbitol as a plasticizer.

Conclusion

In conclusion, this research showed that robusta coffee skin extracts peel-off masks in gel form with different types of gelling agents affected several physical properties of the formula. Based on the results, all formulas meet pharmaceutical characteristics and had intense antioxidant activity. Moreover, recommendations were made to determine the concentration value of the gelling agent, which provided better gel mechanical properties.

Conflict of Interest

All authors declare that there is no conflict of interest in this research.

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