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Formulation of Peel Off Gel Mask from Robusta Green Coffee Bean (*Coffea canephora*) Ethanolic Extract with Polyvinyl Alcohol

(Formulasi Masker Gel *Peel Off* dari Ekstrak Etanol Biji Kopi Robusta Hijau (*Coffea canephora*) dengan Polivinil Alkohol)

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Abstract: Robusta green coffee beans (*Coffea canephora*) have strong potential to be used as antioxidants which can inhibit free radicals that causes premature aging. The peel-off gel mask is a cosmetic product that increases the penetration of active ingredients, is easy to use, and maintains the natural moisture of the skin. This research aimed to examine the possibility of the ethanolic extract of Robusta green coffee beans into a peel-off gel preparation. A peel-off gel mask made from the ethanolic extract of Robusta green coffee beans was formulated using 3 polyvinyl alcohol (PVA) concentrations to obtain the best preparation, which were 10% (F1), 12% (F2), and 14% (F3). All preparations produced a clear yellowish gel. Only F3 met the qualifications for all evaluation tests. The results of a mechanical property test showed that F3 has the best value for tensile strength and elongation. There were statistically significant differences between the results of the evaluation tests before and after the stability test. The results of irritation tests showed that none of the formulas caused irritation. The best peel off gel mask preparation made with ethanolic extract of Robusta green coffee beans was formula F3 that used 14% PVA.

Keywords: Antioxidant, peel off gel mask, polyvinyl alcohol, robusta green coffee bean,

Abstrak: Biji kopi hijau robusta (*Coffea canephora*) memiliki potensi kuat untuk digunakan sebagai antioksidan yang dapat menghambat radikal bebas penyebab penuaan dini. Masker gel *peel off* merupakan produk kosmetik yang meningkatkan penetrasi bahan aktif, mudah digunakan, dan menjaga kelembapan alami kulit. Penelitian ini bertujuan untuk mengkaji kemungkinan ekstrak etanol biji kopi hijau Robusta menjadi sediaan gel *peel off*. Masker gel *peel off* berbahan ekstrak etanol biji kopi hijau Robusta diformulasikan dengan menggunakan 3 konsentrasi polivinil alkohol (PVA) untuk mendapatkan sediaan terbaik yaitu 10% (F1), 12% (F2), dan 14% (F3). Semua sediaan menghasilkan gel bening kekuningan. Hanya F3 yang memenuhi kualifikasi untuk semua parameter evaluasi. Hasil uji sifat mekanik menunjukkan bahwa F3 memiliki nilai kuat tarik dan elongasi terbaik. Terdapat perbedaan yang signifikan secara statistik antara hasil uji evaluasi sebelum dan sesudah uji stabilitas. Hasil uji iritasi menunjukkan bahwa tidak ada formula yang menyebabkan iritasi. Sediaan masker gel *peel off* terbaik yang dibuat dengan ekstrak etanol biji kopi hijau Robusta adalah formula F3 yang menggunakan PVA 14%.

Kata kunci: Antioksidan, biji kopi hijau robusta, masker gel *peel-off*, polivinil alkohol

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INTRODUCTION

AGING is characterized by a progressive loss of the physiological integrity of cells, which leads to impaired function[1]. Signs of chronological aging in the skin include fine wrinkles, laxity, and benign neoplasm. Meanwhile signs of photoaging include deep, coarse wrinkles, mottled pigmentation, sallowness, dryness, laxity, leathery appearance, and elastosis[2]. The contribution of free radicals to the aging process occurs from the beginning of life and increases with age[3]. Free radicals in normal quantities are beneficial to health, while excessive amounts can cause oxidative stress. One plant that contains antioxidant compounds is coffee (*Coffea sp*.). Compounds in green coffee beans that have antioxidant activities include caffeine, chlorogenic acid, trigonelin, cafestol, and kahweol[4].

The Aceh province of Indonesia is among the top five coffee production centers in Indonesia with a coffee production contribution of 7.86%. So far coffee is only used as a drink, essence, and fragrance in air freshener, but the antioxidants in coffee beans have the potential to be used in cosmetic formulations.

Ethanol extract of Robusta coffee beans contains alkaloids, flavonoids, tannins, and saponins[5]. Chlorogenic acid is the main phenolic component in coffee[6]. Research shows that there is a significant correlation between the total content of chlorogenic acid in coffee and its ability to reduce free radicals[7]. Robusta green coffee beans have the highest concentration of chlorogenic acid when compared to Robusta roasted coffee beans and Colombian Arabica green, and roasted coffee beans[8].

Peel-off gel masks are known for their unique characteristics inherent to the use of film-forming polymers that, after complete drying, create a very cohesive plastic layer allowing for the manual removal of the product without leaving any residue[9]. Peel-off gel masks form layers on the skin that can increase the penetration of active substances. In addition, the peel-off gel mask also prevents the loss of natural lubricants on the skin, thereby keeping the skin moist[10].

The peel-off gel mask is formulated using polyvinyl alcohol (PVA). PVA in cosmetic preparations can be used for film forming and can produce a gel that dries quickly and forms a film layer that is transparent, strong, plastic and adheres well to the skin[11]. PVA concentration is an important factor that influences the drying time of the mask[12]. Based on the description above, the formulation and evaluation of the physical properties of the peel-off gel mask from ethanol extract of Robusta green coffee beans (*C. canephora*) were carried out with variations in the concentration of polyvinyl alcohol.

MATERIALS AND METHODS

MATERIALS. Robusta green coffee beans were collected from Coffee Plantation in Central Aceh District, Aceh Province, Indonesia. Beans were sorted, cleaned, dried, and grinded to make coffee powder.

METHODS. Extraction. Robusta green coffee beans were extracted by maceration. The dried beans were added to the maceration vessel, then n-hexane was added in a ratio of 1:10. The dried beans were soaked for 6 hours while being stirred occasionally, then were allowed to stand for 18 hours. This process was carried out for 3 days. The macerate was then filtered using a funnel coated with fabric so that the filtrate and residue could be obtained. The residue obtained was then macerated again using 95% ethanol solvent in a ratio of 1:10 identical to the procedure that had been done before. The filtrate from the ethanol extract was combined and evaporated using a rotary evaporator[13].

The concentrated extract obtained was characterized as well. Parameters tested were loss on drying, total ash content, water-soluble content, and ethanol-soluble content.

Phytochemical Screening and Dried Bean and Extract Characterization. Phytochemical screening and extract characterization was done to the extract obtained. Phytochemical screening included testing for the presence of alkaloids, saponins, tannins, phenolics, and flavonoids groups. Meanwhile parameters tested for extract characterization were loss on drying, total ash content, water-soluble content, and ethanol-soluble content.

Formulation. Robusta green coffee bean ethanolic extract was formulated into peel-off gel masks using various concentrations of Polyvinyl Alcohol (PVA). The formula is modification from formula used in previous study[14]. The concentrations of PVA used were 10% (F1), 12% (F2), and 14% (F3). The rest of formulation consisted of 2% HPMC, 10% propylene glycol, 0.18% methyl paraben, 0.02% propylparaben, and coffee essential oil. The formula can be seen in Table 1.

Formula Evaluation. All formulations obtained were evaluated before and after a cycling test, in which the parameters were: an organoleptic test, pH, spreadability, tackiness, drying time, cycling, viscosity, and mechanical properties tests. All formulations were also tested using irritation and preference tests.

Table 1. Peel off-gel mask from Takengon robusta green coffee bean formula.

No.	Ingredient	Concentration (%)					
		F0-1	F1	F0-2	F2	F0-3	F3
1.	Ethanolic extract robusta green coffee bean	-	0.014	-	0.014	-	0.014
2.	Polyvinyl alcohol	10	10	12	12	14	14
3.	HPMC	2	2	2	2	2	2
4.	Propylene glycol	10	10	10	10	10	10
5.	Methylparaben	0.18	0.18	0.18	0.18	0.18	0.18
6.	Propilparaben	0.02	0.02	0.02	0.02	0.02	0.02
7.	*Coffee essential oil*	-	qs.	-	qs.	-	qs.
8.	Aquadest	ad 100	ad 100	ad 100	ad 100	ad 100	ad 100

RESULTS AND DISCUSSION

Phytochemical Screening and Dried Bean and Extract Characterization. Characterization of both dried beans and extract, and phytochemical screening are shown in Table 2. Phytochemical screening of green coffee bean ethanolic extract showed that it contained alkaloids, saponins, tannins, phenolics, and flavonoids (flavonon, flavonol, and xanton group).

Table 2. Characterization and phytochemical screening results.

Test Parameters	Dried Green coffee bean	Green coffee bean ethanolic extract
	Mean (%)±SD	Mean(%)±SD
Loss on Drying	7.09 ± 1.40	16.24 ± 0.74
Total ash content	2.13 ± 1.44	7.89 ± 3.94
Water-soluble content	23.29 ± 1.37	66.71 ± 1.98
Ethanol-soluble content	16.59 ± 3.83	35.43 ± 2.97
Phytochemical screening for Green coffee bean ethanolic extract		
Alkaloids, saponins, phenolics, flavonoids		

Evaluation Results Before Cycling Test. The concentration of Robusta green coffee beans that were used in this research was based on the IC50 on the research on ethanolic extract of Thai Robusta green coffee from n-hexane residue that was 70 ppm[13].

Based on the results of organoleptic observations (Table 3), it can be seen that prior to the cycling test, the bases (F0-1, F0-2 and F0-3) and preparations (F1, F2, and F3) had the same yellowish white color and the same shape in gel form. Both F0-3 and F3 had a clearer white color than F-01, F1, F0-2, and F2. This was due to the concentration of PVA solution which has a clear white color. Thus, the higher the concentration of PVA, the whiter the color of the preparation.

The pH test was conducted to determine the acidity of the preparation and determine the suitability of the pH of the preparation with skin pH, which is between 4.5-6.5. The results of the pH test showed that the pH of the preparation before the cycling test ranged between 6.17 and 6.33 and met the pH criteria of the topical preparation. Since the preparations made will be used on facial skin, the pH of the preparations must meet the requirements for safety during use. Preparations that are too acidic can irritate the skin and preparations that are too alkaline can cause dry skin.

The observed result of the viscosity test before the cycling test ranged from 6317.33 to 22753.33 cPs and met the viscosity criteria of semi-solid preparations. Good viscosity of semi-solid preparations ranges from 4,000 to 40,000 cPs[15]. The higher the concentration of PVA used, the higher the viscosity of the resulting preparation. This is due to one function of PVA which is as a viscosity enhancing agent[16].

The spreadability test aims to determine the ability of the preparation to spread when it is applied to the skin and its ability to be removed from the container. The higher the spreadability value, the wider the skin area that can be reached by the preparation and the easier it is to remove it from the container. The expected spread value for topical preparations is 5.0-7.0 cm[17]. The observations of the scatter power test showed that prior to the cycling test, only F0-3 and F3 met the spread test criteria, at 6.95 cm and 6.93 cm respectively, while the other formulations did not meet the criteria. This is due to the low viscosity of the preparations which are easily spread. Viscosity of the dosage is inversely proportional to the value of the dispersion, where the lower the viscosity of the preparation, the higher the value of the spreadability[18].

Table 3. Evaluation results of peel-off gel mask before cycling test.

No	Parameter	PVA 10%		PVA 12%		PVA 14%	
		F0-1	F1	F0-2	F2	F0-3	F3
1	Organoleptic						
	Color	Clear, White yellowish (+++)	Clear, white yellowish (+++)	Clear, white yellowish (++)	Clear, white yellowish (++)	Clear, white yellowish (+)	Clear, whit yellowish (+)
	Form	Gel	Gel	Gel	Gel	Gel	Gel
2	pH	6.33	6.17	6.27	6.27	6.33	6.27
3	Viscosity (cPs)	6457.09	6317.33	1050.33	10523.33	22753.33	22660.00
4	Spreadability (cm)	7.48	7.57	7.42	7.51	6.95	6.93
5	Adhesion (second)	3.48	3.59	4.01	4.02	4.28	4.28
6	Drying Time (minute)	14.00	14.33	12.00	11.67	10.67	11.33
7	Mechanical properties						
	Tensile strength (kgf/mm^2)	11.27	11.25	11.33	13.29	14.98	14.95
	Elongation (%)	10.28	10.20	11.67	11.65	12.38	12.30

The adhesion test aims to determine the ability of the preparation to adhere to the surface of the skin when used. The longer the adhesion time, the longer the active substances in the preparation will stick on the surface of the skin and provide maximum effect[19]. The expected value of adhesion is no less than 4 seconds[20]. The results of the adhesion test before the cycling test showed that the base adhesion that met the criteria were F0-2 and F0-3, and the adhesion of the preparations that met the criteria was F2 and F3, whereas F0-1 and F1 did not meet the adhesion criteria for topical preparations. This is due to the lowest PVA concentration in F0-1 and F1, which is 10%. The low concentration of PVA causes the low viscosity of the preparation, so that the sticking time of the preparation is short. Viscosity of the preparation is directly proportional to the length of adhesion, where the higher the viscosity of the preparation, the longer it adhered to the surface[21].

The drying time test aims to find out how long the preparation takes to form a film layer on the surface of the skin. The expected drying time of a peel-off gel mask preparation is 10-30 minutes[22]. The observation results of the drying time test showed the drying time of the preparation before the cycling test ranged from 10.67 to 14.33 minutes and met the criteria for drying time of the gel peel-off mask preparation. F0-1 and F1 had the longest drying time, while F0-3 and F3 had the shortest drying time. The higher the concentration of PVA used, the faster the drying time of the preparation.

Mechanical properties tests of the preparation included a measurement of tensile strength and elongation. The tensile strength measurement aims to determine the maximum pressure that can be applied until the film breaks. The elongation measurement aims to determine the maximum length the film can stretch before breaking.

If the tensile strength and elongation values are high, the gel film is not easily broken. The tensile strength test results before the cycling test ranged between 11.25-14.98 kgf/mm2 and the results of the elongation test before the cycling test ranged from 10.20 to 12.38%. F03 and F3 had the highest tensile and elongation strength values. The higher the concentration of PVA, the greater the tensile strength and elongation of the film material.

Differences in evaluation results between bases and preparations (F0-1 and F1, F0-2 and F2, F0-3 and F3) showed insignificant results ($P>0.05$) for pH, viscosity, dispersion, adhesion, dry time, and mechanical properties evaluation. These results indicate that the addition of extracts in formulations F1, F2, and F3 had no significant effect on the results of the evaluation.

However, differences in the evaluation results between preparations (F1, F2, and F3) showed significant results ($P<0.05$) on the evaluation of viscosity, dispersion, adhesion, drying time, and mechanical properties. These results indicate that different PVA concentrations affect the results of the evaluation of the preparations. The higher the concentration of PVA used, the higher the viscosity, adhesion and mechanical properties, and the lower the spreadability and drying time. The results of evaluations between preparations (F1, F2, and F3) showed insignificant results ($P>0.05$) on the

evaluation of pH. These results indicate that different PVA concentrations do not affect the pH of the preparation.

Evaluation Result After Cycling Test. Organoleptic observations show that all formulas were a clear yellow color in the fourth cycle of the cycling test. While the shape of the base and preparation did not change, and there was no syneresis. Discoloration was caused by the process of degradation of the material used. Possible materials that degraded were PVA and extracts, because PVA can be degraded at 100°C[16].

The pH test results show a significant difference (P<0.05) between the results before and after the cycling test (Table 4). The pH of the whole formula after the cycling test ranged from 5.27 to 5.53 and still met the pH criteria for topical preparations. The decrease in pH after the cycling test could be caused by the hydrolysis reaction that occurs in the preparation[23].

The spread test results show a significant difference (P<0.05) between the results before and after the cycling test. Only F1 met the parameter criteria of the spreadability test. The decrease in spreadability after the sixth cycle cycling test could be caused by the increased viscosity of the preparation after the cycling test, so that the ability of the preparation to spread has decreased. The spreadability of the stocks in each cycle fluctuated allegedly because the viscosity of the preparations of each cycle also fluctuated due to stretching and formation of hydrogen bonds during the cycling test.

The adhesive test results show a significant difference (P<0.05) between the results before and after the cycling test. The adhesion of all formulas after the cycling test ranged from 4.01 to 5.26 seconds and still met the criteria for adhesion of topical preparations. Increased adhesion time after the cycling test could have been caused by the increased viscosity of the preparation after the cycling test, which causes the adhesion of the preparation to increase. The adhesion of the preparations in each cycle fluctuated allegedly because the viscosity of the preparations of each cycle also fluctuated due to stretching and formation of hydrogen bonds during the cycling test.

Viscosity test results (Table 5) show a significant difference (P<0.05) between the results before and after the cycling test. The viscosity of all formulas after the cycling test ranged from 6,671 to 29,693 cPs and still met the criteria for viscosity of semi-solid preparations. The increase in viscosity occurred

Table 4. Evaluation of pH, spreadability, thickness, and drying after cycling test.

Parameter	Cycle	Formula					
		F0-1	F1	F0-2	F2	F0-3	F3
pH	0	6.33	6.17	6.27	6.27	6.33	6.27
	1	6.33	6.30	6.30	6.23	6.43	6.37
	2	6.20	6.23	6.30	6.30	6.33	6.37
	3	6.27	6.23	6.30	6.20	6.27	6.30
	4	6.27	6.27	6.20	6.20	6.13	6.07
	5	6.10	6.00	6.03	5.93	5.70	5.57
	6	5.53	5.43*	5.47	5.30*	5.37	5.27*
Spreadability (cm)	0	7.48	7.57	7.42	7.51	6.95	6.93
	1	6.56	6.41	5.89	6.00	5.18	4.93
	2	6.33	6.31	5.89	5.96	4.84	4.70
	3	6.65	6.79	5.60	5.80	5.12	4.83
	4	6.43	6.51	5.15	5.00	4.97	4.84
	5	6.24	6.37	5.24	5.07	4.67	4.23
	6	6.02	6.11*	4.41	4.35*	4.35	4.13*
Stickiness (second)	0	3.48	3.59	4.01	4.02	4.28	4.28
	1	3.79	3.68	4.60	4.67	4.93	4.70
	2	3.92	3.67	4.84	4.68	4.93	4.95
	3	4.02	3.83	4.74	4.50	5.01	4.97
	4	4.01	3.71	4.36	4.42	4.80	4.97
	5	4.00	3.90	4.82	4.77	4.86	4.90
	6	4.19	4.01*	5.06	4.98*	5.26	5.11*
Drying time (minute)	0	14.00	14.33	12.00	11.67	10.67	11.33
	1	13.67	14.00	11.00	12.00	10.33	11.33
	2	13.00	13.33	10.67	11.67	10.33	10.33
	3	13.33	13.67	11.33	12.33	10.33	11.33
	4	13.67	13.33	12.67	12.67	10.67	11.00
	5	13.00	13.33	11.67	12.00	10.00	10.33
	6	13.00	13.00*	10.33	11.33*	10.00	10.00*

Table 5. Viscosity evaluation result of peel-off gel mask.

Cycling Test	PVA 10%		PVA 12%		PVA 14%	
	F0-1	F1	F0-2	F2	F0-3	F3
Before cycling test (cPs)	6457.09	6317.33	10503.33	10523.33	22753.33	22660.00
After cycling test (cPs)	6671.36	6604.78	14353.33	14403.33	29693.33	29503.33

Table 6. Preparation mechanical properties evaluation result of peel-off gel mask.

Formula		Tensile Strength (kg/cm^2)		Elongation (%)	
		Before Cycling Test	After Cycling Test	Before Cycling Test	After Cycling Test
PVA 10%	F0-1	11.27	11.56	10.28	10.35
	F1	11.25	11.55	10.20	10.33
PVA 12%	F0-2	13.33	13.70	11.67	11.84
	F2	13.29	13.65	11.65	11.82
PVA 14%	F0-3	14.98	15.08	12.38	12.61
	F3	14.95	15.02	12.30	12.57

because it is assumed that after cycling tests more and more hydrogen bonds are formed, namely hydrogen bonds between PVA molecules and between PVA molecules and the water molecules[24].

Tensile strength and elongation after the cycling test increased in all formulas (Table 6). Increased tensile strength and elongation after cycling tests could be influenced by increased viscosity. The higher the viscosity of the preparation, the greater the tensile strength and elongation produced.

Evaluation of Irritation and Preference Test. The irritation test aims to determine, if any, irritating effects on the skin are due to an ingredient. The irritation test was conducted on 30 respondents. The irritation test results showed that the three peel-off gel mask formulations showed no signs of irritation including redness, itching, and swelling.

Preference test results that have been performed are calculated using the average preference value interval listed in Indonesia National Standard 01-2346-2006[25]. Based on the results of these calculations, the three formulations are preferred for the parameters of the form and aroma of the preparation. As for the color parameters, F2 and F3 preparations are preferred over F1. This difference was due to the fact that F3 has a more clear and white color than the other formulations. PVA solution which has a clear white color will make the dosage color clearer. Thus, F1 with the lowest PVA concentration of 10% has a more yellowish color than F2 and F3.

CONCLUSION

From the results of this study, it can be concluded that the F3 formulation, with 14% PVA concentration, produced the best peel off gel mask that meets general semisolid preparation standards.

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