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FORMULATION OF PEEL-OFF GEL MASK OF COCOA SKIN EXTRACT (*Theobroma cacao* L) WITH VARIED CONCENTRATION GELLING AGENT

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ABSTRACT

Cocoa shells are waste from the plantation industry, with a fairly good potential source of energy and protein. The ethanol extract of cocoa shells contains alkaloids, flavonoids, phenols, and tannins. Cocoa shell extract has strong antioxidant activity with an IC50 value of 17.21 ppm and has sunscreen activity. This study aimed to determine the formulation and physical test of a peel-off gel mask with cocoa shell extract (*Theobroma cacao* L) and to determine its effect. The research method used was an experimental method with variations in the concentration of a combination of HPMC and PVA of 2%:10%, 3%:9%, 4%:8%, 5%:7%, and 0%:12%. The physical tests used on the peel-off gel mask with cocoa shell extract (*Theobroma cacao* L) included organoleptic, homogeneity, pH, spreadability, drying time, adhesiveness, viscosity, and cycling tests. Based on the results of the research carried out, peel-off gel mask has the characteristics of a brown color, a distinctive extract smell and a thick texture, homogeneous for F1 and F5, and not homogeneous for F2, F3, and F4, sticking force 4.53±0.24 seconds; 5.45±0.36 seconds; 8.94±0.64 seconds; 11.23±0.72 seconds; 3.87±0.70 seconds; spreadability 3.99 ± 0.18 cm; 3.33±0.31 cm; 3.47±0.78 cm; 3.32±0.50 cm; 3.05±0.42 cm; pH 6.17 ± 0.10; 6.16±0.03; 6.15±0.10; 6.11±0.05; 6.42±0.03; viscosity 19481.10±201.10; 20165.50±1.05; 20165.73±2.02; 20182.53±9.47; 14618.40±158.48. The physical stability of the gel mask preparations after treatment showed that the results of the peel-off gel mask preparations, F1 and F5, were stable.

Keywords: *peel-off gel mask, cocoa shell, extract, gelling agent*

INTRODUCTION

The skin is the outermost organ of the human body and is the main defense system for humans. Skin damage disrupts human health and appearance; therefore, the health of the skin needs to be maintained and protected (Sari, 2015). Good skin care will make a person's skin appear healthy, look well-groomed, and radiate freshness (Haerani et al., 2018). Free radicals in the form of ultraviolet rays can cause skin damage. Under excessive conditions, UV rays can cause skin problems, including sunburn, redness of the skin (erythema), darkening of the skin (tanning), and can even cause skin cancer (Sari, 2015). Several alternative natural sources of antioxidants can be used to improve skin health. Among these are cocoa shells.

Indonesia is a cocoa producing country. The outer shell (mesocarp) is the largest part of the cocoa fruit, with up to 75% hard white placenta and brown seeds. The increased production of cocoa fruit resulted in an increase in cocoa pod shells. Cocoa shells are a plantation industry waste with the potential to be a good source of energy and protein. It contains 9-19% protein, 13-26% crude fiber, 2400 kcal/kg metabolic energy, 4.6% fat, 0.61% calcium, and 0.06% phosphorus (Sukatik et al., 2020).

The ethanol extract of cocoa shells contains alkaloids, flavonoids, phenols, and tannins (Khoiriyah et al., 2022). The results of this study showed that the tannin content of cocoa fruit skin in Toraja Regency was 12.679% (Pappa et al., 2019). Cocoa pod skin has antibacterial activity against Propionibacterium acnes and Staphylococcus epidermidis and is also antiparasitic (Adha & Ibrahim, 2021; Lestari et al., 2021; Pappa et al., 2019). Cocoa shell extract has strong antioxidant activity with an IC50 value of 17.21 ppm (Hidayah et al., 2021; Priani et al., 2021; Ulfa et al., 2019) and has sunscreen activity (Khoiriyah et al., 2022; Priani et al., 2021). Cocoa shell extract can be used as a potentially good raw material for medicine (Septriyanti et al., 2020). Cocoa shell extract has been widely used in cosmetic preparations, including emulgel preparations (Fahleni et al., 2021; Priani et al., 2021), gel mask preparations (Ulfa et al., 2019), serum preparations (Hidayah et al., 2021) and cream preparations (Huda et al., 2022).

Considering the potential of cocoa shell extract, the cocoa shell, which is usually just waste, can be developed into pharmaceutical preparations that will have added value. Cocoa shell extract can be developed into cosmetic preparations, such as a peel-off gel mask. A peel-off gel mask is a cosmetic dosage form that can be used in skin care in a gel dosage form that can be applied to the skin for a certain time until it dries. This preparation forms an elastic, transparent film layer that can be easily peeled off (Santoso et al., 2020). Peel-off facial masks have advantages in their use, that is, they are easy to lift or remove, provide a cooling effect, have good adhesion and dispersing power, can deliver active substances more optimally because of direct contact with the skin, and do not interfere with the physiological functions of the skin because they do not occur. The formation of a wax layer that coats the skin and does not cause blockage of skin pores. Peel-off gel masks can stimulate and repair active skin cells, remove dirt on the face, moisturize the skin, tighten the skin, and improve blood flow to the skin tissue on the face (Rahmawanty et al., 2015; Silvia & Dewi, 2022).

Peel-off gel mask formulations generally contain active ingredients, gelling agents, moisture retainers, preservatives, and water (Agoes 2013). The gelling agent makes the consistency of the mask gel-like, making it easy to apply (Silvia & Dewi, 2022). Polyvinyl alcohol (PVA), a gelling agent often used in making peel-off gel masks, also functions as a film-forming agent.

Film formation is one of the components added to peel-off gel mask preparation, where the preparation forms a thin and transparent film layer when applied to the skin after drying. The material that has optimum effectiveness as a film forming is polyvinyl alcohol (PVA). Polyvinyl alcohol (PVA) is widely used as a base for fabricating peel-off gel masks. PVA has a positive effect on the characteristics of peel-off gel masks and is easy to obtain. However, PVA has disadvantages: if the concentration used is too high, it will produce a stiff film layer with low flexibility (Silvia & Dewi, 2022). Preliminary tests were conducted on the use of PVA as a gelling agent. PVA is known to affect the adhesion, spreadability, viscosity, and drying time tests.

The results of a previous study showed that the optimal formula for peel-off gel mask preparations was obtained at a PVA concentration of 10% with a propylene glycol content of 10%, where a preparation was obtained that met good physical mask quality and produced a preparation that was stable in storage for each test parameter (Andini et al., 2017). The results of other studies show that variations in the concentration of PVA as a gelling agent influence the physical properties of peel-off gel masks, including pH, viscosity, spreadability, stickiness, and drying time, and the best results using PVA were obtained at a concentration of 12% (Anindhita et al., 2023). It is known that peel-off mask formulations for extracts derived from plant seeds (containing flavonoids as antioxidants) have good characteristics when the gelling agent variations PVA 12% and HPMC 2% are used (Rahmadiani & Nur Hasanah, 2019).

RESEARCH METHODS

Equipment and Materials

The tools used in this research included chemical measuring cups (Pyrex ®), beaker glass (Pyrex ®), glass funnels (Pyrex ®), clamps and statives, analytical scales (Durascale®), mortars and stampers, stirring rods, spoons, porcelain cups, filter paper, maceration vessels and evaporator (DLAB®), and mesh sieves. 40, glass object, pH meter (Ohaus®), viscometer (NDJ 8S®), oven (Memmert®), and a waterbath (Faithful®).

The materials used in this research included distilled water, nipagin, nipasol, HPMC (Hydroxyl Propyl methyl cellulose (HPMC), PVA, glycerin, propylene glycol, cocoa shell simplicia, 96% ethanol, and a mask container.

Research Procedure

1. Sample collection

Cocoa shells (*Theobroma cacao* L.) were collected in Curugsewu Village, Patean District, Kendal Regency. The extraction process was performed by maceration using 96% ethanol for 120 hours. The filtrate obtained was concentrated using a rotary evaporator at a temperature–50-60ºC and followed by evaporation in a water bath (60 °C) to obtain a thick extract.

2. Phytochemical Screening

Flavonoid

The extract (0.2 g) was dissolved in distilled water and magnesium powder and 2 N HCl solution were added. The solution was heated in a water bath for 5-10 minutes. After cooling, the mixture was filtered. The filtrate was then added to amyl alcohol and shaken vigorously. Positive results for flavonoids are indicated by the formation of a red-orange color on the amyl alcohol layer (Khoiriyah *et a*l., 2022).

Tannin

The extract (0.2 g) was then dissolved in distilled water. Then heated. Subsequently, 1% FeCl3 was added to the solution. The presence of tannin is indicated by the formation of a blue-black color (Khoiriyah *et a*l., 2022).

Saponin

0.2 gram of the extract was dissolved in sufficient water. The mixture was then heated in a water bath for 5 minutes. Once cooled, strain and shake vigorously. Samples positive for tannin were indicated by the formation of a stable foam 1 cm in height within 30 minutes (Khoiriyah *et a*l., 2022).

Phenol

The extract (0.2 g) was then dissolved in methanol. Then, for 30 seconds the solution was then heated in boiling water. Next, H2SO4 was gradually added. Subsequently, 10% NaOH was added to the mixture. The phenol content was indicated by the formation of a red-brown precipitate (Khoiriyah *et a*l., 2022).

Alkaloid

0.3 grams of the extract was dissolved in distilled water, and 10% ammonia was added to make it alkaline. Chloroform was then added to extract alkaloids. Next, 1 N HCl was added to the mixture. Dragendorf and Meyer reagents were then added to the solutions. Positive results on the Meyer test are indicated by the presence of a white precipitate. Positive Dragendorf test results are indicated by the formation of an orange-red precipitate (Khoiriyah *et a*l., 2022).

Table I. Peel off Gel Mask Formula

Material	Content (%) b/b					Function
	F1	F2	F3	F4	F5	
Extract	1	1	1	1	1	Active substance
HPMC	2	3	4	5	-	Gelling Agent
PVA	10	9	8	7	12	Film Forming
Methyl Paraben	0,2	0,2	0,2	0,2	0,2	Preservative
Propyl Paraben	0,1	0,1	0,1	0,1	0,1	Preservative
Glyserin	10	10	10	10	10	Humectant
Ethanol	15	15	15	15	15	Solvent
Propylene glycol	15	15	15	15	15	Humectant
Aquadest	ad 100	ad 100	ad 100	ad 100	ad 100	Solvent

3. Procedure for Making Peel off Gel Mask

Making a peel-off gel mask from the cocoa shell extract began by weighing each ingredient. Aquadest was heated in two separate containers to a temperature of 90 °C to dissolve the PVA and HPMC so that they expanded quickly while stirring at a constant speed. After the PVA and HPMC were expanded, HPMC was placed in a PVA container. After stirring until homogeneity, the mixture was cooled. Methyl paraben and propylparaben were dissolved in glycerin and propylene glycol, respectively, and stirred until homogeneous. The extract was dissolved in ethanol, stirred until homogeneous, added to a mixture of PVA and HPMC, and stirred until homogeneous. Dissolved methyl paraben and propylparaben were added to the HPMC and PVA base mixture while stirring until homogeneous. Distilled water was added up to 100 grams. They were then packaged in tightly closed containers (Amaliah *et al.* 2018).

4. Physical Evaluation

Organoleptic Test

Organoleptic testing is carried out by visually observing changes in the consistency, color, and aroma of the gel mask preparation (Ariana, 2016).

Homogeneity Test

Homogeneity testing was performed by applying the gel to a glass object. A certain amount of the preparation when applied to a piece of glass or other suitable transparent material, the preparation must show a homogeneous composition and no visible coarse grains (Ariana, 2016).

pH test

The pH test of the gel mask was performed using a pH meter. The instrument was first calibrated using a neutral standard solution (pH 7.01) and an acidic buffer solution (pH 4.01) until the instrument shows the pH value was determined. The electrode was then washed with distilled water and dried with tissue. The electrodes were then dipped in the mask preparation. The tool shows the price of the pH meter, which is the pH preparation. The pH of the gel mask preparation must match the pH of the skin, that is 4.5 – 6.5 (Ariana, 2016).

Spreadability Test

0.5 gram of the gel mask was placed on a glass scale. Then, it was covered with another glass and weights of 50, 100, 150, 200, and 250 grams were used on top until the preparation stopped spreading. The optimum spreadability criterion is 5.0 – 7.0 cm (Nafisa & Salsabilla, 2021).

Adhesion Test

0.5 grams of the gel was placed on the top and left for 5 minutes. The glass object was placed on the adhesion test equipment, a load of 80 grams was released, and the time until the glass object was released was recorded (Ariana, 2016).

Dry Time Test

The drying time test was carried out by scratching several samples, such as when applying a mask on the back of the palm of one of the probands and calculating the time needed for the preparation to dry until it could be peeled off. The optimum drying time is 15-30 minutes (Zhelsiana *et al*., 2016).

Viscosity Test

Viscosity tests were performed using a viscometer. The mask gel was placed in the cup, and the spindle was installed. The rotor is run at a speed of 30 rpm (Natalia & Hosea, 2020).

Cycling Test

The cycling test method was used for the physical stability test. The mask gel was stored at 4 ± 2ºC for 24 hours, after which it was placed in an oven at 40 ± 2ºC for 24 hours. This treatment is referred to as a single cycle. This treatment was carried out for 12 days (6 cycles), and physical changes were observed in the peel-off gel mask preparation (pH, viscosity, organoleptic, and homogeneity). Subsequently, the physical conditions of the preparation were compared during the experiment with the initial condition of the preparation (Amaliah *et al*., 2018).

Data Analysis

In this study, the researchers used data by comparing the research results with those in the literature. To determine the effect of variations in gelling agent concentration on the physical properties of peel-off gels, SPSS (Statistical Package for the Social Science) software was used.

RESULTS AND DISCUSSION

Table II. Results of Phytochemical Screening of Cocoa Peel Extract

Test	Requirement	Result	Explanation
Flavonoids	Red, yellow or orange in color	+	An orange layer is formed
Tannin	The color of the solution is blue-black	+	The color of the solution is black
Saponin	Forms stable foam	+	Forms stable foam
Phenol	Red-brown precipitate	-	There is no brown precipitate
Alkaloids	Dragendorf test: A red-orange precipitate is formed	+	An orange precipitate is formed

Organoleptic Test

Based on the results of organoleptic testing, the peel-off gel mask has the characteristics of a light brown color, distinctive extract smell, and thick texture. The five formulas showed that the texture of each formula was different; the higher the HPMC concentration, the thicker the texture obtained. This increase in texture is caused by HPMC, which can increase the

number of polymer fibers; the higher the HPMC concentration, the more liquid will be retained so that the gel concentration increases (Silvia *et al*., 2015).

Table III. Organoleptics Test Result

Formula	Organoleptic Test		
	Smell	**Colour**	**Tekstur**
Formula 1	Typical Extract	Light Brown	**
Formula 2	Typical Extract	Light Brown	***
Formula 3	Typical Extract	Light Brown	****
Formula 4	Typical Extract	Light Brown	*****
Formula 5	Typical Extract	Light Brown	*

Information :
(*) Viscosity level
Formula 1 = PVA 10% HPMC 2%
Formula 2 = PVA 9% HPMC 3%
Formula 3 = PVA 8% HPMC 4%
Formula 4 = PVA 7% HPMC 5%
Formula 5 = PVA 12% HPMC 0%

Homogeneity Test

Homogeneity testing aims to evenly and homogeneously determine the mixture of the preparations. An indication that a preparation is declared homogeneous is that the preparation does not contain coarse grains (Ambari & Suena, 2019). Based on the results of research on antioxidant mop-off gel mask preparations, the results showed that the peel-off gel mask preparations in formula 1 and formula 5 had a homogeneous physique, as evidenced by the absence of coarse grains in the preparation when applied to the watch glass. Formula 2, formula 3 and formula 4 have a non-homogeneous physique as evidenced by the presence of coarse grains in the preparation when applied to the watch glass. This was influenced by the water factor not optimally dissolving HPMC and PVA, causing insoluble coarse grains to appear.

Table IV. Homogeneity Test Result

Formula	Homogeneity
Formula 1	Homogeneous
Formula 2	Inhomogeneous
Formula 3	Inhomogeneous
Formula 4	Inhomogeneous
Formula 5	Homogeneous

pH Test

The pH test aims to determine the pH value of a preparation that falls into the acid or basic category. This needs to be known because if the pH of a preparation has a high level of acidity, an irritation or injury reaction will occur on the surface area of the skin, whereas, if the pH of a preparation is too alkaline, there will be dryness on the surface area of the skin. The pH value range of the preparation is 4.5-6.5 (Silvia *et al*., 2015).

Table V. pH Test Result

Formula	pH
Formula 1	6,17 ± 0,10
Formula 2	6,16 ± 0,03
Formula 3	6,15 ± 0,10
Formula 4	6,11 ± 0,05
Formula 5	6,42 ± 0,03

Based on the pH value data obtained, the pH value of the peel-off mask preparation still met the requirements for a good pH value.

Viscosity

The viscosity test was used to determine the viscosity level of the preparation. Viscosity describes the viscosity of a preparation, which is related to spreadability and stickiness (Thomas et al., 2023). The viscosity of a preparation affects other physical properties of the preparation, such as spreadability, stickiness, and flow properties. The viscosity of the preparation affects the comfort and effectiveness of the preparation. Based on the test parameters, the viscosity of peel-off mask preparations ranges from to 5,000-50,000 cPs (Silvia *et al*., 2015).

Table VI. Viscosity Test Result

Formula	Viscosity (cPs)
Formula 1	19481,10 ± 201,10
Formula 2	20165,70 ± 1,05
Formula 3	20165,73 ± 2,02
Formula 4	20182,53 ± 9,47
Formula 5	14618,40 ± 158,48

HPMC is a hydrogel-forming material that can expand in water. The use of HPMC in peel-off gel masks because HPMC can increase the number of polymer fibers, which will increase the amount of liquid retained and the gelling agent will bind the water. Mechanism of gel formation by HPMC occurs because the polymer-solvent interacts, causing more distance between the particlessmall and causes intermolecular molecules to form cross-links which can cause reduction mobility or movement of the solvent so that a gel mass will form, and the active substance will be trapped inside gel matrix that will be released upon application (Silvia & Dewi, 2022)

Spreadability Test

The spreadability test is a preparation test that aims to determine the ability of a preparation to spread on a surface. To test the spreadability of peel-off mask preparations using a test tool, namely a petri dish with a millimeter block scale, the test is still carried out manually without using a machine tool. The testing parameter for the adhesive strength of peel-off mask preparations is 5-7 cm (Thomas et al., 2023).

Table VII. Spreadability Test Result

Formula	Spreadability (cm)
Formula 1	3,99 ± 0,18
Formula 2	3,33 ± 0,31
Formula 3	3,47 ± 0,78
Formula 4	3,32 ± 0,50
Formula 5	3,05 ± 0,42

Spreadability in shape preparation is inversely proportional to viscosity. The lower the viscosity, the higher is the spreading power (Syam et al., 2021).

According to Ningrum (2018)The higher the HPMC concentration, the the spreadability of the gel will decrease or on the contrary. The higher the concentration of the gelling agent, the lower the spreadability value. The decrease in the value of spreading power occurs through an increase in the size of the molecular unit, as it absorbs the solvent so that the liquid restrains and increases the resistance to flow and spread (Aprilianti et al., 2020).

Adhesion Test

The adhesion test was carried out with the aim of determining the ability of a preparation to adhere to a surface. Peel-off mask adhesion testing was performed to measure the ability of the mask to adhere when applied and function, as well as its ability to perform its action during the drying process (Syam et al., 2021).

Table VIII. Adhesion Test Result

Formula	Daya Lekat (Second)
Formula 1	4,53 ± 0,24
Formula 2	5,45 ± 0,36
Formula 3	8,94 ± 0,64
Formula 4	11,23 ± 0,72
Formula 5	3,87 ± 0,70

Based on the results of measuring the adhesive power of the peel-off gel mask preparation, it can be explained that the peel-off gel mask preparation has a good adhesive value. The adhesion results of the peel-off gel mask preparation met the test parameter requirements, that is, more than 1 second (Syam et al., 2021).

Drying Time Test

A drying time test was carried out to determine how long it took for the preparation to dry when applied to the skin. Test time This drying is expected to obtain a good film coating formulation for application, apart from that it is also related to comfort when used. Expected drying time for a peel-off gel mask produced is between 15-30 minutes (Zubaydah et al., 2020).

Table IX. Drying Time Test Result

Formula	Drying Time (Minute)
Formula 1	25,67 ± 2,08
Formula 2	49,33 ± 2,31
Formula 3	>60 ± 1,00
Formula 4	>60 ± 1,00
Formula 5	17,33 ± 1,15

The drying time is very important for peel-off gel masks because formulations with fast drying times allow for a fast peeling process (Syam et al., 2021).

Cycling Test

Table X. pH test Result

Formula	Before *Cycling Test*	After *Cycling Test*
Formula 1	6,17 ± 0,10	4,88 ± 0,04
Formula 2	6,16 ± 0,03	4,79 ± 0,01
Formula 3	6,15 ± 0,10	4,83 ± 0,02
Formula 4	6,11 ± 0,05	4,89 ± 0,02
Formula 5	6,42 ± 0,03	4,94 ± 0,03

The results of the pH test after the cycling test were carried out, and all formulas experienced a decrease in pH because storage at different temperatures would cause changes in pH due to the influence of CO2 because CO2 reacts with the water phase so that it becomes acidic (Septiani *et al*., 2011). The pH values obtained before and after the cycling test still show that the preparation falls within the parameters of a good pH value, namely 4.5-6.5 (Ariana, 2016).

Table XI. Viscosity Test Result

Formula	Before *Cycling Test* (cPs)	After *Cycling Test* (cPs)
Formula 1	19481,10 ± 201,10	20168,87 ± 4,88
Formula 2	20165,70 ± 1,05	20170,03 ± 2,71
Formula 3	20165,73 ± 2,02	20169,23 ± 2,35
Formula 4	20182,53 ± 9,47	20171,57 ± 9,14
Formula 5	14618,40 ± 158,48	20165,33 ± 0,46

The results of the cycling test showed that all formulas experienced an increase in viscosity values caused by high temperature treatment when the cycling test was carried out and the addition of ethanol to the evaporated formula, so that the preparations would become thicker, as indicated by the higher viscosity values (Sri Agustini et al., 2021).

Table XII. Organoleptics Test Result

Formula	Before *Cycling Test*			After *Cycling Test*		
	Smell	Colour	Tekstur	Smell	Colour	Tekstur
Formula 1	Typical Extract	Light Brown	**	Typical Extract	Light Brown	**
Formula 2	Typical Extract	Light Brown	***	Typical Extract	Light Brown	***
Formula 3	Typical Extract	Light Brown	****	Typical Extract	Light Brown	****
Formula 4	Typical Extract	Light Brown	*****	Typical Extract	Light Brown	*****
Formula 5	Typical Extract	Light Brown	*	Typical Extract	Light Brown	*

Organoleptic test results showed no changes in smell, color, and texture before and after the cycling test. Thus, the peel-off gel mask was organoleptically stable.

Table XIII. Homogeneity

Formula	Before Cycling Test	After Cycling Test
Formula 1	Homogeneous	Homogeneous
Formula 2	Inhomogeneous	Inhomogeneous
Formula 3	Inhomogeneous	Inhomogeneous
Formula 4	Inhomogeneous	Inhomogeneous
Formula 5	Homogeneous	Homogeneous

The homogeneity test results showed no change before and after the cycling test. Formulas 1 and 5 remained homogeneous, while formulas 2, 3, and 4 before the cycling test were carried out were no longer homogeneous owing to the lack of solvent used.

Data Analysis

The results of the SPSS test showed that variations in PVA and HPMC concentrations had a significant effect on the spreadability, adhesion, and drying times of the peel-off gel mask preparation of the cocoa husk extract. The effect of treatment on the pH test before and after the cycling test showed that after the cycling test treatment was carried out on the peel-off mask, there was a significant change in pH during the peel off mask preparation. Meanwhile, the viscosity test before and after cycling showed that after the cycling test treatment was carried out on the peel-off gel mask, there was no significant change in viscosity in the peel-off mask preparation.

CONCLUSION

Peel off gel masks that meet the requirements as a good peel off gel mask are formula 1 (with a gelling agent concentration of HPMC 2% and PVA 10%) and formula 5 (with a gelling agent concentration of PVA 12%). Varying the gelling agent concentration had a significant effect on stickiness, spreadability, drying time, and pH, but did not have a significant effect on the viscosity of the peel-off gel mask preparation.

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