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PHYSICAL QUALITY TEST AND OPTIMIZATION OF KIRINYUH LEAF ETHANOL EXTRACT CREAM (CHROMOLAENA ODORATA L.) USING SIMPLEX LATTICE DESIGN METHOD

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ABSTRACT

Kirinyuh leaves (Chromolaena odorata L.) contain active compounds such as flavonoids, tannins, and saponins that have the potential as anti-inflammatory and antimicrobial. To increase its stability and effectiveness, ethanol extract of kirinyuh leaves is formulated in the form of a cream. Cream preparations have the advantages of ease of application, controlled release of active ingredients, and the ability to provide hydration to the skin. However, the formulation of cream preparations requires optimization to produce products with good physical quality. The optimal formulation of this cream is determined using the Simplex Lattice Design (SLD) method. This research method includes ethanol extraction of kirinyuh leaves using the maceration method, formulation of cream preparations with basic ingredients of stearic acid, cetyl alcohol, sorbitol, propylene glycol, triethanolamine, methyl paraben, perfume, Optimization of formulation using SLD (Simplex Lattice Design) by comparing two variables between stearic acid and triethanolamine which function as emulsifiers. Physical quality tests were conducted for viscosity, pH, spreadability, adhesion, and stability parameters during 28 days of storage. The results obtained from the optimal formulation combination consisting of 18% stearic acid and 2% TEA , produced a cream with a viscosity of 2.48334 cp, pH 7.15, spreadability of 5.38 cm, adhesion of 5.716 seconds, and good stability without significant changes in color, odor, or viscosity.
Keywords: Kirinyuh Leaves, Cream Preparation, SLD.

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INTRODUCTION

Kirinyuh leaves (*Chromolaena odorata* L.) are one of the herbal plants that are widely used in traditional medicine. This plant is known to contain active compounds such as flavonoids, tannins, and alkaloids. which has various pharmacological activities, such as anti-inflammatory, antimicrobial, and antioxidant. It also has saponi n content which functions as an antibacterial, one of which is the antibacterial *Staphylococcus aureus* (Fadia *et al* ., 2020) . The mechanism of antibacterial compounds is usually carried out by damaging cell walls, changing membrane permeability, disrupting protein synthesis, and inhibiting enzyme activity. (Septiani *et al* ., 2017) . The pharmacological potential of kirinyuh leaves makes it a promising natural ingredient to be developed into pharmaceutical or cosmetic preparations, one of which is topical cream.

Cream preparations have the advantages of ease of application, controlled release of active ingredients, and the ability to provide hydration to the skin. However, the formulation of cream preparations requires optimization to produce products with good physical quality, including viscosity, pH, spreadability, adhesion, and stability. The combination of basic ingredients such as emulsifiers, oil phases, and water phases greatly affects the physical characteristics and stability of the preparation. (Nafsiah, 2013).

Method *Simplex Leticia Design* is method Which used to optimize formulas on various differences amount composition material, Which amount the total made The same. Method This Can determine formula Which optimum with use amount test Which more A littleso that it can minimize the use of materials or compositions which will be used (Hidayat *et al* ., 2021). In the design software experiment simplex lattice, there is three choice direction study with design test Which can done, that is screening, characterization, And optimization. Following is explanation each.

Although the potential of kirinyuh leaves has been widely studied, the development of cream preparations based on ethanol extract of kirinyuh leaves is still rarely done. The main challenge is to ensure that the cream preparation has good physical quality and is stable during storage. Therefore, optimization of the formulation is needed to produce a combination of ingredients that provide the characteristics of the cream preparation according to quality standards. Based on the research background that highlights the importance of formulation and optimization of cream preparations based on ethanol extract of kirinyuh leaves (*Chromolaena odorata* L.) with comprehensive physical quality evaluation, as well as the use of the Simplex Lattice Design method to achieve

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optimal formulation, the title of this research is determined as "Physical Quality Test and Optimization of Cream Preparations Based on Ethanol Extract of Kirinyuh Leaves **(** *Chromolaena odorata L.)* L.) Using the Simplex Lattice Design Method." This title was chosen to reflect the focus of the research, the methods used, and the main active ingredients, so that it can specifically describe the objectives and scope of the research.

Based on the background that has been presented, the formulation of the problem in this study is:

1. How is the formulation of a cream preparation based on ethanol extract of kirinyuh leaves (*Chromolaena odorata* L.) that meets the criteria for good physical quality, such as viscosity, pH, spreadability, adhesion, and stability?
2. How can the Simplex Lattice Design (SLD) method be used to optimize the combination of basic cream ingredients (oil phase, emulsifier, and water phase) to produce an optimal formulation?
3. Does the formulated cream preparation have good physical stability during storage at various temperature conditions?

This study aims to: Formulate a cream preparation based on ethanol extract of kirinyuh leaves , Optimize the combination of basic cream ingredients using the Simplex Lattice Design method , Evaluate the physical quality and stability of the formulated cream preparation.

The results of this study are expected to contribute to the development of natural-based cosmetic preparations, especially kirinyuh leaves, as potential active ingredients in skin care. In addition, this study can be a reference for the application of the Simplex Lattice Design method in optimizing cosmetic formulations.

METHOD

This research is a pure experimental research . The population used in this study was kirinyuh leaves (Chromolaena odorata L.) obtained in Donohudan Village , Ngempak District , Boyolali Regency . Ethanol extract of kirinyuh leaves is a thick extract preparation obtained by macerating kirinyuh leaf powder using 96% ethanol solvent and then evaporating it with a rotary evaporator to obtain a concentrated extract.

This research was conducted from May to September at the Natural Materials Laboratory and the Pharmaceutical Laboratory of Duta Bangsa University, Surakarta. On

The Simplex Lattice Design method was used to optimize the cream formulation. In this case, the variables that were changed were the composition of the oil phase material (stearic acid) and the emulsifier material (TEA). Several experimental points were determined using SLD to find the best combination of materials that produce a cream preparation with optimal viscosity, pH, spreadability, adhesion, and stability.

The data obtained from the results of the physical quality test of the cream are analyzed statistically using software such as Design-Expert® or Minitab®. SLD analysis will be used to evaluate the effect of formulation variables on the quality of the preparation. The results obtained will be compared with the standards applicable to cosmetic products, such as ideal viscosity, safe pH, and optimal spreadability.

The results of physical tests of various formulations produced by SLD will be used to determine the most optimal cream formulation. The combination of ingredients that produces the cream with the best physical characteristics will be selected as the final formulation. The results of this optimization will also be

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compared with literature and similar products to ensure that the resulting product meets the expected quality standards.

This research method combines the formulation of cream based on ethanol extract of kirinyuh leaves with optimization using the Simplex Lattice Design method to produce a cream formulation that has optimal physical quality. Physical quality tests carried out include viscosity, pH, spreadability, adhesion, and stability, all of which are important to ensure the success of natural ingredient-based cosmetic products.

Tool

Homogenizer mixer, pH meter, Viscometer (to measure viscosity), Spreadability tester (scatter graph). Adhesion tester (timer and synthetic leather surface), Test tube, beaker glass, and standard measuring instrument, Moisture balance, Furnace, Scales , Laptop.

Material

leaves (Chromolaena odorata L.), Stearic acid, Cetyl alcohol, Sorbitol, Propylene glycol, Triethanolamine (TEA), Methyl paraben, Perfume, Aquadest, Other additional ingredients (preservatives, aroma, etc.)

Research Procedures

MAKING KIRINYUH LEAF POWDER (*Chromolaena odorata* L.) Kirinyuh leaves are taken fresh, cleaned, washed with running water, then cut and dried. Drying of the simplicia is carried out at a temperature of 50ºC, the drying process takes 4 days because during this period it produces optimal dry simplicia. Drying at a temperature of 50ºC is the optimum temperature to produce dry simplicia without damaging the compounds in it. The dried Kirinyuh leaves are made into powder by blending then sifting with a No. 60 mesh sieve, then the powder is weighed. (Pratiwi et al., 2019).

STANDARDIZATION OF DRYING SHRINKAGE SIMPLISIA

Each 2 grams of the simplicia was put into a porcelain crucible with a lid that had previously been heated at a temperature of 105ºC for 30 minutes and had been tared, the crucible was put into the oven with the crucible lid open until the weight remained cooled in a desiccator.

WATER CONTENT

Using the Moisture Balance tool.

PHYTOCHEMICAL SCREENING

Phytochemical screening of secondary metabolites found in kirinyuh leaves was carried out using secondary metabolite tests, including flavonoid, alkaloid, tannin and saponin tests (Triastinurmiatiningsih et al., 2022).

MAKING BODY SCRUB

Table 1: Preformulation

Bahan	Formula %						
	R 1	R 2	R 3	R 4	R 5	Range %	Fungsi
Asam stearate	16,5	17	18	16	17,5	16-18	Emulgator
Trietanola Min	3,5	3	2	4	2,5	2-4	Emulgator `
Propilen glikol	15	15	15	15	15	10-25	Humektan
Sorbitol	5	5	5	5	5	5-20	Pelembab
Setil alkhol	3	3	3	3	3	2-5	Penstabil
Metil paraben	0,2	0,2	0,2	0,2	0,2	0,2-0,3	Pengawet
Propil paraben	0,05	0,05	0,05	0,05	0,05	0,01-0,6	pengawet
Parfum	0,5	0,5	0,5	0,5	0,5	0,01-0,5	Zat tambahan
Ekstrak Daun Kirinyuh	50	50	50	50	50		Zat Aktif
Aquadest	ad 100	ad 100	ad 100	ad 100	ad 100		

Weighing all the necessary ingredients is the first step in making the cream. The oil phase is (stearic acid and

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cetyl alcohol) and the water phase is (sorbitol, propylene glycol, tritetonolamine, and methyl paraben), the oil phase is put into a porcelain cup and then melted on a water bath

. Phase 2 is dissolved in hot water until homogeneous, then phase 1 is put into a heated mortar and add phase 2 (stirring chili sauce) add distilled water little by little, grind until it becomes a homogeneous cream base then add kirinyuh leaf extract, grind until homogeneous then add perfume and put it in a container. (Sari et al., 2023)

DATA ANALYSIS

optimization with the simplex lettice design method using the design expert program or software 13. The data obtained is entered into the equation, based on the equation of each desired response, namely the adhesion test, spreadability test, viscosity test, pH test, the equation of each response is obtained. Furthermore, validation of the selected formula is carried out by evaluating the physical properties of the cream. After obtaining the optimum formula, the formula is verified using statistical data analysis through the One sample T test using the SPSS application (ika et al., 2020)

RESULTS AND DISCUSSION

Yield of Kirinyuh Leaves (*Chromolaena odorata* L.)

Table 2: powder yield

Heavy weight wet (gr)	Heavy weight dry (gr)	Yield (%) b/b
1000	730	73%

Yield is a percentage of product obtained from the comparison of the initial weight and final weight of the material, so that the weight can be known when 1000 undergoes processing. The resulting yield is 73%,

DRYING SHRINKAGE

Drying shrinkage results of Kirinyuh leaves (*Chromolaena odorata* L.)

obtained 2.65%. the mass lost due to heating includes water molecules and essential oils. The results obtained indicate that Kirinyuh Leaves (*Chromolaena odorata* L.) meet the requirements of the Indonesian herbal pharmacopoeia, which is not more than 10%.

WATER CONTENT

Determination of the water content of Kirinyuh leaves (*Chromolaena odorata* L.) using a moisture balance tool with a temperature of 105ºC and the average of the three replications was 8.75%. The results obtained showed that Kirinyuh leaves (*Chromolaena odorata* L.) met the standard water content requirements according to the applicable standard parameters, which is no more than 10%.

PHYTOCHEMICAL SCREENING

Table 4: Phytochemical screening results

Secondary Metabolites	Preaction	Results	Information	Description according to *(Andasari et (2020)*
Alkaloids	Dragendorff	+	Chocolate	Sediment orange until red brown
Flavonoid	H2SO4	+	Orange	Reddish, yellow or orange
Saponins	H20	+	Foamy	Wow stable
Tannin	FeCI3%	+	Blackish Green	Blackish green

Based on the phytochemical screening test above, it shows that Kirinyuh leaves (*Chromolaena odorata* L.) contain secondary metabolite compounds.

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EVALUATION OF PHYSICAL QUALITY ORGANOLEPTIC TEST

Based on the results of organoleptic tests of Kirinyuh Leaf cream (*Chromolaena odorata* L.) from the three replications of each preparation formula which is semi-solid, brown in color and has a distinctive odor.

HOMOGENITY TEST

Observations of the homogeneity test of the three replications of the five formulations of Kirinyuh Leaf Extract (*Chromolaena odorata* L.) cream had good homogeneity, indicated by the absence of lumps or coarse particles and an even color when applied to a glass plate.

ADHESIVE POWER TEST

adhesion test of formula I 5.26 seconds, formula II 5.54 seconds, formula III 6.16 seconds, formula IV 5.25 seconds and formula V has an average of 6.11 seconds, based on the results above, the five preparation formulas made have met the standards for good adhesion test, which is more than 4 seconds (Tungadi et al., 2023). The best adhesion test result is in formula III. These results indicate that the viscosity of a preparation can affect adhesion.

SPREADING POWER TEST

The spreadability test of the cream resulted in an average of the three replications in formula I 5.5 cm, formula II 5.8 cm, formula III 5.4 cm, formula IV 5.6 cm and formula V 5.1 cm. The results of the spreadability test showed that the value of the spreadability of the body scrub tested had met the standards for a cream that had good spreadability, with a spreadability range between 5 and 7 cm (Pratiwi et al., 2019).

PH TEST

The pH test aims to determine the level of acidity or freedom of the cream that affects the skin irritation properties. From the results of the pH test of the cream in formula I, the results were 7.07, Formula II 7.53, Formula III 6.78, Formula IV 7.47 and Formula V 6.90. It can be seen that the pH of the preparation is stable with the concentration of the cream. The cream preparation has met the skin pH guidelines between 4.5 and 8 (Hidayati et al., 2023).

VISCOSITY TEST

This test was conducted using a Brookfield viscometer with spindle 4 at a speed of 60 rpm, the preparation was inserted into a beaker glass until it reached a volume of 50ml, then the spindle was lowered until the spindle limit was immersed in the preparation. The results of the body scrub viscosity test on formula I

3.454 cp, Formula II 3.889 cp, Formula III 2.065 Formula IV 2.555 and Formula V 2.208. From the results of the viscosity test of the cream preparation, each formula meets the requirements of a good viscosity value, indicating that the cream preparation can be easily applied to the skin.

FORMULA VALIDATION OF Kirinyuh Leaf Extract Cream (*Chromolaena odorata* L.)

Formula optimization is done with a simplex lattice design approach using Design Expert software version 13. The optimum formula is determined by entering the target response to be achieved. The target responses (goals) that can be selected include minimize, maximize, target, in range and equal to. The results of the analysis of the physical quality response of the body scrub which include, adhesion, spreadability, pH viscosity are entered into the simplex lattice design software.

Table 6: criteria for determining the optimum formula

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Name	Goal	Lower Limit	Upper limit
A: sour stearate	In range	16	18
B:triethanolamine	In range	2	4
Power sticky	In range	5.26	6.18
Power spread	In range	5.1	5.6
Viscosity	In range	2065	3889
Ph	Minimize	6.78	7.53

Table 8: Optimum formula

No.	Stearic acid (%)	Triethanolamine (%)	Spreading power	Adhesion	viscosity	Ph	Desirability
1	18	2	5.38	5,716	2.48343	7.15	0.920

The target optimization value to be achieved is known as the desirability value. Which ranges from zero to one. A desirability value approaching one indicates that the formula can reach the optimum formula according to the desired variables, while a desirability value approaching zero indicates that the formula has difficulty reaching the optimum point based on the variables.

The optimum formula with stearic acid and triethanolamine concentration with a ratio of (18%: 2%) with a desirability value of 0.920. The optimum formula obtained from the software was evaluated for physical quality tests including spreadability, adhesion, viscosity, pH to determine the stability of the cream preparation. Based on the table above, the optimum formula desirability is 0.920. The optimum formula desirability value approaching one indicates that the formula can achieve the optimum formula according to the response variables of adhesion, spreadability, pH, viscosity.

The results of the physical quality test of F0 as a negative control in the organoleptic test showed that F0 was white, semi-solid and had a distinctive odor, for the homogeneity test on F0 the preparation was homogeneous and for the viscosity test the average of the three replications was 2,151 cp, for the pH test the results were 7.37 and the adhesion test on F0 the results were 6.59 seconds, while the spreadability test results were 5.93 cm from the results of the physical quality test of F0 above all met the requirements.

Based on the optimum formula, the predicted response of the formula composition is a spreadability of 5.38, adhesiveness of 5.716, viscosity of 2.48334 cp and pH of 7.15. Verification of the optimum formula using response prediction statistics generated from the simplex lettice design is then compared with the response of the experimental analysis results. The statistical analysis used is the one sample t Test used to test the significance of the average difference between each value of the experimental results that have been carried out with the theoretical value of the predicted results from the simplex lettice design.

The table shows that the response of spreading power, adhesion, viscosity, pH shows results that are not significantly different between predictions using the simplex lettice design method and experimental results.

This can be seen from the significance value of each response which is more than 0.05. The absence of significant differences between the simplex lettice design predictions and the experimental results can be concluded that the software is valid for use in optimizing the preparation of kirinyuh leaf extract cream.

CONCLUSION

From the optimization results using the simplex lettice design method, the optimum formula was obtained in formula III with a concentration of 18% stearic acid and 2% triethanolamine. From the results of the study on the preparation of Kirinyuh

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Leaf Extract Cream (*Chromolaena odorata* L.), the optimum formula desirability value was 0.920. The optimum formula desirability value approaching one indicates that the formula can achieve the optimum formula according to the response variables of adhesive power, spreadability, pH, and viscosity. The results of the evaluation of spreadability were 5.38, adhesive power 5.716, viscosity of 2.48334 cp and pH of 7.15 in accordance with literature standards.

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