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Optimization of Liquid Shampoo Formula from Chia Seed Oil (*Salvia hispanica* L) Using the Simplex Lattice Design Method

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ABSTRACT

Hair loss is a common concern affecting both men and women, with various factors contributing to this condition. Chia plants are known to contain agents that stimulate sebaceous glands, which are crucial for promoting hair follicle growth and preventing hair loss, due to their keratin protein concentration of 23%. This study aimed to determine the optimal formulation of a liquid shampoo using Design Expert software, focusing on the physical characteristics and stability of the product. The optimization process involved two key components: Hydroxypropyl Methylcellulose (HPMC) and Sodium Lauryl Sulfate (SLS), with viscosity and foam height as the primary responses. The optimal formulation was identified through the software, followed by a series of characteristic and stability tests. Stability was assessed using a cycling test, where the shampoo was stored at 4°C and 40°C for 48 hours. The results indicated an optimal formula with component concentrations of X1 at 0.589307% and X2 at 9.41069%. Statistical analysis using a one-sample t-test revealed no significant differences in viscosity and foam height ($p > 0.05$). The physical characteristics of the optimal formula met acceptable standards, exhibiting organoleptic properties, homogeneity, viscosity of 414.80 ± 9.94 cPs, foam height of 7.060 ± 0.11 cm, and a pH of 7.39 ± 0. These findings suggest that the optimized shampoo formulation can effectively support hair health and prevent hair loss, providing a promising option for consumers seeking effective hair care solutions.

1. Introduction

Hair is part of the epidermis layer that grows in almost all parts of the human body. Hair is an important part of the body to look after and care for because it is considered a crown for both men and women. There are various problems with hair, ranging from thin and brittle texture, difficulty growing, to hair that falls out easily. Hair loss is actually a normal cycle, but when the loss occurs in large quantities it will become a problem for the hair growth period itself. The normal number of hair follicles is around 100,000, it is said to be an abnormality if the number is not normal, namely around 50,000 strands. The normal range for hair loss on the scalp is 80-120 strands/day (Sari & Wibowo, 2016). This issue can be addressed with appropriate hair care products. The hair care product that is commonly used is shampoo.

Shampoo is a cosmetic cleansing product that is useful for cleaning the scalp and hair from various adhering dirt such as oil, fat and sweat (Iskandar et al., 2023). The term 'shampoo' is believed to have originated in colonial India. However, since the 19th century, the use of soap began to appear to clean hair (Rantika, 2017). Technological advances to overcome hair problems and research that continues to be studied increase knowledge and innovation in the use of herbal plants, extending to cosmetic preparations. One of the plants that can be used as a cosmetics in hair care specifications is the chia plant.

The chia plant (*Salvia hispanica* L) is included in the group of flowering plants. This plant comes from Central America, namely Mexico and Guatemala (Kusnandar et al., 2020). Physically, chia is an herbaceous plant that has a height of more than one meter with stems that are square and covered with fine hairs. The plant has a simple leaf shape and there are fine hairs on the leaf blade. The flowers are purple, blue, or white in colour and have a monopetalus hermaphrodite flower type (plants with two fully functional sex organs). The flowering process of chia plants takes 15 to 25 days (Brandán et al., 2019). The seeds have an oval shape with a smooth and glossy texture. Most of the chia seeds are brown, white, grey, and dark in colour (Kusnandar et al., 2020). The linolenic acid content in chia seed oil is known to be a sebaceous gland stimulating agent that stimulates hair growth. Research conducted by (Fitri, 2022) stated that the safety of hair emulsion derived from Chia seed oil has been demonstrated and could accelerate hair growth in local male rabbits. Supported by the presence of high amounts of copper and zinc and 23% keratin protein in it, it can also make hair roots stronger and provide a shiny effect (Fitri, 2022). Research conducted by (Rahmayanti et al., 2023) on the use of chia seed oil as a hair emulsion contained phase separation after stability. So it is necessary to optimize the formula to become a shampoo dosage form that is more stable against temperature changes.

Regarding formulation, technological developments present a new method for formula optimization, namely by using Design Expert software. One option that can be used for research is optimization. Optimization can be done using several methods, one of which is mixture. This method can be used for components with variable proportions that are differentiated in a formulation. For this reason, optimization of the liquid shampoo preparation formula from chia seed oil was carried out using the simplex lattice design method.

2. Materials and Methods

The equipment utilized in this study includes Design Expert 13 software for optimization. The tools for formulating the shampoo preparations from chia seed oil comprised a refrigerator (Samsung: South Korea), an oven (Memmert), an analytical balance (Shimadzu: Japan), a Brookfield viscometer (LV DV3T), and a set of equipment including a hot plate, magnetic stirrer (Heidolph: Germany), benchtop pH meter (Mettler Toledo: USA), test tubes, stir bars, watch glasses, beakers, measuring cups (Iwaki), thermometers (GEA Medical), rulers (Butterfly), and droppers.

The formulation included chia seed oil (Salvia hispanica L) sourced from CV Happy Green in Indonesia as the active ingredient. The excipients used in the shampoo formulation were of analytical grade and included hydroxypropyl methylcellulose (HPMC) (Bratachem), potassium hydroxide (Bratachem), sodium lauryl sulfate (SLS) (Bratachem), propylene glycol (Bratachem), glacial acetic acid (Bratachem), methylparaben (Bratachem), and distilled water (Brataco).

2.1. Preparation of samples

The research method begins with formula optimization. The liquid shampoo preparation formula was optimized using Design Expert software. This application has three research direction options: screening, characterization, and optimization. One method in mixture design is simplex lattice design (SLD). This optimization method is used to determine the best formula for a mixture of ingredients selected, with the proportion of the total amount of different elements having to be 1 (Hidayat et al., 2020). Weigh all the ingredients needed in the formulation, then heat 30 mL of distilled water at a temperature of 60-70°C. Then, develop the HPMC by adding it little by little and stirring it with a magnetic stirrer (Mixture 1). Dissolve SLS with propylene glycol in a magnetic stirrer (Mixture 2). We heated chia seed oil in a different container at a temperature of 70°C. We heated potassium hydroxide in a separate container at a temperature of 35°C. Mix wild chia seed oil and potassium hydroxide and stir until mixed (Mixture 3). Mix mixture one and mixture two until homogeneous. Added a solution of methylparaben, mixture 3, glacial acetic acid, and the remaining distilled water. After obtaining the preparation, validation is carried out. Confirmation of the shampoo preparation was carried out to ensure that the practice complies with the optimum formula results from the Design Expert software by repeating the shampoo in triplicate (3 times).

2.2. Preparation Evaluation

2.2.1 Organoleptic Test

The organoleptic test was carried out by visual observation of the liquid shampoo preparation. This test is conducted to observe the quality of the preparation before it falls into the hands of consumers. Good organoleptic results are stable preparations that are signalled by the surfactant content used (Pradigdo et al., 2022). Observations in this organoleptic test include colour, odour, and dosage form.

2.2.2 pH Test

The pH test is carried out so that the pH balance of the shampoo can be known so that it can stabilise the ecological balance of the scalp and minimise irritation to the eyes. The pH range of acceptable shampoo according to SNI 06-2692-1992 guidelines is 5-9 (Salsabila et al., 2022). Shampoo pH measurement uses a tool in the form of a pH meter benchtop.

2.2.3 Homogeneity Test

The homogeneity test was carried out with to determine whether there were any inhomogeneous or coarse particles in the shampoo preparation. Homogeneity testing uses two beakers that are glued together. Weighed 1 gram of shampoo preparation sample and placed in the middle of the two glass preparations. Furthermore, observations were made whether there were clumps of coarse particles in the preparation. A good shampoo has a homogeneous dosage form and no scattered coarse particles are found (Tee & Badia, 2019).

2.2.4 Foam Height Test

The foam height measurement test was carried out using a test tube filled with shampoo samples. Then the mouth of the test tube was closed and shaken vigorously for 20 seconds. The foam height that appears in the test tube is then measured using a ruler and observed for 5 minutes. A good shampoo preparation has a foam height range of 1.3-22 cm (Hidayat et al., 2020; Salsabila et al., 2022; Tee & Badia, 2019).

2.2.5 Viscosity Test

The viscosity test was carried out with the aim of knowing the level of viscosity of the resulting preparation. the viscosity of the shampoo preparation was measured using a Brookfield viscometer at a speed of 1 rpm. The viscosity range of a good shampoo preparation according to SNI guidelines is between 400-4000 cps (Hidayat et al., 2020).

2.2.6 Stability Test (Cycling Test)

The stability test of the shampoo preparation was kept in a refrigerator at a cold temperature of 4°C for 24 hours, and then transferred to an oven at a high temperature of 40°C for 24 hours. The process is counted as 1 cycle. This test can be carried out as many as 6 cycles (Auliah, 2020). Stability test observations include viscosity test, pH test, and foam height test. The parameters assessed are the level of viscosity of the preparation, the level of acidity or basicity of the shampoo preparation, and the level of foam height obtained from testing before the stability test is carried out.

2.3. Data Analysis

Data obtained from observations of the physical characteristics test of liquid shampoo preparations are descriptive and quantitative. Descriptive data was obtained from the results of organoleptic tests, homogeneity, pH, viscosity, and foam height adjusted to the acceptance range. Then, quantitative data was obtained from the optimization formula response, which was analyzed using a one-sample t-test. The stability test data obtained was then examined statistically using a paired sample t-test.

3. Results and Discussion

Formula rationalization aims to determine the right ingredients to be used in the liquid shampoo formulation. The results of the formula design are as listed in Table 1.

Rationalization, in this case, means designing the ingredients used in the dosage formulation based on valid and relevant literature (Illu, 2020). Chia seed oil is an active ingredient with a concentration range of 7.5% w/v. Hydroxypropyl methylcellulose (HPMC) is used as a thickening agent or preparation thickener. HPMC, known to be inert to many substances, is compatible with packaging components and can affect the viscosity value (Dewi & Saptarini, 2016). HPMC was chosen in the shampoo making component because it is able to stabilise the foam so that the physical stability of the preparation is maintained (Salsabila et al., 2022). The foaming agent used is Sodium Lauryl Sulfate (SLS). SLS is a solid surfactant type commonly used in products for cleaning oil stains and dirt (Chasani et al., 2022). The combination of HPMC and SLS in shampoo is considered to be able to produce optimum foam. Another important component in toiletries preparations is potassium hydroxide. Potassium hydroxide (KOH) which functions in the saponification process. The saponification process converts fats into fatty acids and glycerol by adding KOH at a specific concentration until it is wholly hydrolyzed at a certain temperature (Purwati, 2021).

The simplex lattice design method requires researchers to enter the material components to be optimized by determining the upper and lower limits for each material. The mixed designs can contain anywhere from 2 to 30 materials or components. Optimization results are listed in Table 2.

Table 1. Formula rationalization result

Raw Materials	Function	Concentration (% w/v)
Chia seed oil	Active ingredient	7.5
Hydroxypropylmethyl cellulose	Thickening agent	Optimized
Sodium Lauryl Sulfate	Surfactant	Optimized
Potassium hydroxide	Saponification agent	2.5
Propylene glycol	Humectan	15
Methylparaben	Preservative	0.1
Distilled water	Solven	Ad to 100

Table 2. Formula optimization result

Run	X1	X2	Y1	Y2
	HPMC	SLS	Viscosity (cP)	Foam Height (cm)
1	0.8625	9.1375	409	5.0
2	0.7250	9.2750	396	6.0
3	0.4500	9.5500	344	5.5
4	0.7250	9.2750	396	6.0
5	0.4500	9.5500	344	5.5
6	1.0000	9.0000	728	4.7
7	1.0000	9.0000	728	4.7
8	0.5875	9.4125	383	7.0

Next, optimize the formula using Design Expert 13 software using the Simplex Lattice Design (SLD) method. Components that can be included according to the researcher's wishes range from 2 to 30 materials or components. Where all members must have the same range. The material components used in this research are component A, HPMC, with a lower limit of 0.45% and an upper limit of 1% (Rowe et al., 2009). Component B has a lower limit of 0% and an upper limit of 10%. The total used for these two materials is 10. The determination of the total of these materials is adjusted to the researcher's wishes. Every two ingredients or components entered will produce eight runs of the prediction formula. The response entered will be coded with the letter Y. The viscosity and foam height responses were chosen because these two parameters are important in shampoo products. Viscosity shows the viscosity of the product so that it can be known the resistance of the preparation in flowing when used. Foam height shows the cleanliness of the product in cleaning the dirt on the hair and can affect the user's psychology.The response obtained is obtained from the results of experiments in the laboratory. Based on the results of the optimum formula, it was found that differences in HPMC concentration significantly influenced the viscosity response. Where the concentration of HPMC is greater, the viscosity value will also increase. HPMC can increase viscosity by absorbing solvents, retaining the liquid, and forming a compact liquid mass. The more HPMC concentration is used in preparation, the more fluid will be retained and bound by HPMC (Dewi & Saptarini, 2016). The Simplex Lattice Design equation obtained in ANOVA for the viscosity response has a p-value <0.05, which means the modeling is significant for the viscosity response. The equation obtained is as follows.

Y= 725.255A + 341.255B – 614.902AB .... (Equation 1)

Information:

Y = Viscosity

A = HPMC Factor

B = SLS Factor

Equation 1 shows that HPMC has a dominant influence on increasing the viscosity value. This is inversely proportional to the SLS component. The interaction of the two material components is marked with a negative sign, which interprets that the exchange affects reducing the viscosity value.

Then, the difference in SLS concentration also has a significant effect on the foam height response. The more SLS given, the more the viscosity value will increase. SLS is a solid surfactant commonly used in products for cleaning oil stains and dirt. Foam formation occurs when the surfactant at the water-air interface with hydrophobic groups extends into the gas phase. Foam is a colloidal system with a gas-dispersed phase and a liquid dispersion medium. Foam stability is obtained from the presence of foaming agents or what is usually called surfactants. The surfactant is adsorbed into the interphase area and binds the gas bubbles to bring stability (Chasani et al., 2022). The Simplex Lattice Design equation obtained in ANOVA for the foam height response has a p-value <0.05, which means the modeling is significant for the foam height response. The equation obtained is as follows.

Y= 4.717A + 5.517B + 3.952AB – 8.53 (A-B) .... (Equation 2)

Information:

Y = Foam Height

A = HPMC Factor

B = SLS Factor

Equation 2 shows that SLS has a dominant influence on increasing the foam height value. This is inversely proportional to HPMC components. The interaction of the two material components is marked with a negative sign, which interprets that this interaction affects reducing the foam height value. The optimum formula results were obtained after the selected model was significant and analysis was carried out on each response (Table 3).

The optimum formula results were obtained after the selected model was significant, and analysis was carried out on each response. Determining the optimum formula is based on the formula that has the highest desirability value. Desirability is a function value used for optimisation purposes. It indicates the ability of the programme to fulfil desires based on parameters set by the researcher. A desirability value that is close to 1 indicates that the procedure is increasingly optimal (Ramadhani et al., 2017). The resulting prediction formula has a desirability value of 0.939, which suggests that closer to 1 means it is more optimal (Figure 1). The desirability value is measured in the range 0-1. Values 0 and 1 represent undesirable and highly desirable, respectively. This range scale is used to find global functions that must be strengthened by effective variable selection and optimization (Iftikhar et al., 2020).

Table 3. Optimum Formula Recommendation Results

XI (HPMC)	X2 (SLS)	Viscosity	Foam Height	*Desirability*	
0.5893	9.4106	404.8410	6.8590	0.9390	Suggested
0.7316	9.2683	378.9130	6.0440	0.5840	

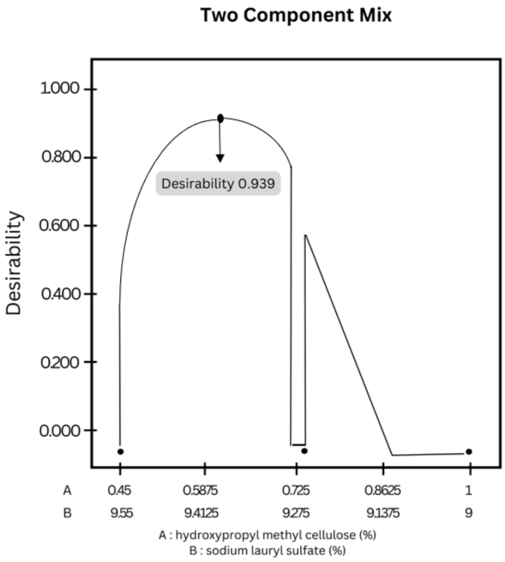

Fig. 1. Desirability graphic

The prediction formula produced by the software is then verified and validated. Verification is a test of the correctness of the optimum formula whose physical properties have been predicted using the simplex lattice design method.

Table 4. Prediction Formula Verification Results with Experiments

Parameter	Prediction	Experiment Results ($\bar{x}$ ± SD)	p-value	Significance
Viscosity	404.843 cPs	414.866±9.94 cPs	0.500	Not difference different
Foam Height	6.859 cm	7.060±0.11 cm	0.503	Not difference different

Verification tests the correctness of the optimum formula whose physical properties have been predicted using the simplex lattice design method. Verification is done by re-making the preparation using the same approach when optimizing the procedure and carrying out physical testing. The test results obtained were carried out with statistical analysis using the one-sample t-test method. Statistical testing is carried out to find out the difference between research results and the predictions given. The test results show a p-value >0.05, meaning the optimum formula is not significantly different (not very different) from the predicted value. This can mean that the results provided through predictions by the simplex lattice design method are correct and reliable.

Physical characteristics were carried out to compare the material parameter values obtained from the optimum formula of chia seed oil liquid shampoo with the predetermined acceptance range. Based on characteristic testing, the optimum procedure and its replication meet the good acceptance range. The preparation has a slightly thick liquid form; this is the state of liquid shampoo, which has a viscosity value of around 400-4000 cPs (Figure 2) and (Table 5).

Fig. 2. Optimum formula 3 replications

Table 5. Optimum Formula Characteristic Results

Parameter	Optimum Formula Replication			Acceptance
	R1	R2	R3	
Organoleptic	The liquid is slightly thick, white, with a distinctive chia oil odor	The liquid is slightly thick, white, with a distinctive chia oil odor	The liquid is slightly thick, white, with a distinctive chia oil odor	-
Homogeneity	Homogenous	Homogenous	Homogenous	-
Viscosity (cPs)	404.1	416.8	423.7	400-4000 cPs
Foam Height (cm)	7.0	7.0	7.2	1.3-22 cm
pH	6.62	7.90	6.19	4-9

Information: R1, R2, and R3 are replications

Stability testing is one of the quality parameters. It is carried out to determine the ability of a product to survive within specified specification limits throughout the storage and use period (Table 6). Stability testing was carried out using the cycling test method at temperatures of 4°C and 40°C for 48 hours for six cycles.

Table 6. Optimum Formula Physical Stability Test Results

Parameter	0th cycle ($\bar{x}$ ± SD)	6th cycle ($\bar{x}$ ± SD)
Organoleptic	The liquid is slightly thick, white, with a distinctive chia oil odor	The liquid is slightly thick, white, with a distinctive chia oil odor
Homogeneity	Homogenous	Homogenous
Viscosity	414.80±9.94 cPs	424.80±2.02 cPs
Foam Height	7.06±0.11 cm	6.30±0.36 cm
pH	7.39±0	7.58±0.02

Stability testing is one of the quality parameters. It is carried out to determine the ability of a product to survive within specified specification limits throughout the storage and use period (Primadiamanti et al., 2017). Several factors can influence the stability of a product, namely temperature and time. In this research, stability tests were carried out at temperatures of 4°C and 40°C for six cycles, one cycle consisting of 48 hours (Auliah, 2020). Based on stability testing, between cycle 0 and cycle six, there was no change in the organoleptic test; the dosage form remained a slightly thick liquid, white in color, and had a distinctive odor of chia seed oil. This is a desirable outcome since the visual and sensory properties are critical in consumer product appeal. This can be caused by the use of HPMC, which can provide stability even if stored for a long time; it is a non-toxic and non-irritating material. These results align with research conducted by (Auliah et al., 2020), who stated that anti-dandruff shampoo preparation with HPMC and SLS ingredients there were no organoleptic changes in shampoo after 12 days of storage.

Then, in the homogeneity test, no changes were found between the 0th and 6th cycles. The shampoo preparation should be homogeneous so that it is evenly distributed and does not cause irritation to the skin. A homogeneous shampoo will also give good results because the components are evenly dispersed in the base material. Thus, each part of the shampoo will contain the same amount of ingredients. The preparation remains homogeneous, characterized by the practice being evenly distributed. The stirring process influences this during the trial. Stirring can cause the particles to become smaller (Baskara et al., 2020). These results align with research conducted by (Babu et al., 2021), who found that shampoo preparations have good homogeneity after the storage period. In the viscosity test, there was an increase in the value after the 6th cycle, however, this increase is still within the range of shampoo viscosity. This is because HPMC has increased thermo-plasticity; namely, HPMC will change and form a gel so that the viscosity increases (Deshmukh et al., 2017). The same result was also stated by (Auliah et al., 2020) who stated that the shampoo preparation experienced an increase in viscosity after stability from 37.50 mPas to 40 mPas.

Meanwhile, the foam height test decreased after the 6th cycle. The foam height parameter indicates the ability of the preparation to produce foam and provide a cleansing effect. The material that has the most influence on foam is SLS, which has high biodegradability. Long storage causes biodegradation of SLS into sulfate and lauryl alcohol. This results in a loss of surface active properties and biochemical changes (Freitas et al., 2023). Similar results were also obtained by (Sari et al., 2019) research, showing a decrease in foam after heating temperature treatment.

Furthermore, the pH test showed an increase after the 6th cycle. The pH test is an evaluation intended to determine the pH balance of the preparation so as to stabilize the ecological balance of the scalp and minimize irritation. The increase in pH after stability was influenced by the increased thermo-plasticity properties of HPMC in the preparation (Setiani & Endriyatno, 2023). (Cahyaningsih et al., 2019) also stated an increase in pH from week 1 to week 12.

4. Conclusion

The optimum composition of the thickening agent (hydroxypropyl methylcellulose) in the optimum formula is 0.589307% with a combination of surfactant (sodium lauryl sulfate) of 9.41069% and a desirability value of 0.939 obtained. The desirability value obtained shows close to 1, which means that the value better fulfils the researcher's wishes. The physical characteristics and stability of liquid shampoo preparations from chia seed oil (*Salvia hispanica* L) meet the requirements for good practice. Based on the results of formula optimization, physical characteristics, and stability tests that fulfil the product criteria, this shampoo product has marketing potential. Using the advantages of the research conducted, the marketing strategy can focus on consumer education, communication of the product's added value, and delivery of reliable test results. This ensures the product is well received in the market and builds consumer loyalty.

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Competing Interests

The authors declare no conflict of interest.

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