

ENHANCING THE PHYSICAL CHARACTERISTICS AND SHELF LIFE OF RICE WATER (*ORYZA SATIVA* L.) GEL SHAMPOO: THE ROLE OF PROPYLENE GLYCOL CONCENTRATION

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ABSTRACT

Objective: This study aims to determine the effect of variations in propylene glycol concentration on the physical properties and physical stability of rice water shampoo gel and the physical strength of rice water shampoo gel preparation.

Methods: The active ingredient was rice water, which was prepared by washing rice with water at the proportion of 1:1. The rice water then was applied to HPMC K4M-based gel preparations and further designed to prepare shampoo gel with varying concentrations of propylene glycol 8%, 10%, and 12%. The rice water shampoo gel was evaluated for its organoleptic properties, pH, viscosity, spreadability, flowability test, softness test, and physical stability test.

Results: The results of the physical properties test on the three formulas produced an organoleptic clear pale yellow color, lemon odor, and semi-solid shape. The pH test results obtained by F1 was 7.24 ± 0.08 , F2 was 5.89 ± 0.01 , and F3 was 6.02 ± 0.01 . The viscosity test results obtained by F1 amounted to 2082 ± 80.07 cP, F2 amounted to 4987 ± 883.88 cP, and F3 amounted to 6531 ± 232.79 cP. The foam height test results obtained by F1 amounted to 4.6 ± 2.11 cm, F2 amounted to 1.46 ± 0.05 cm, and F3 amounted to 1.7 ± 0.17 cm. Variations in propylene glycol levels affect the physical properties of shampoo gel preparations at pH, viscosity, and foam height but do not affect the organoleptic practice. The stability test that was conducted at weeks 0, 1, 2, 3, and 4 with varying levels of propylene glycol showed significant changes on viscosity and foam height stability.

Conclusion: Variations in the concentrations of propylene glycol in the three formulations affect the physical but have no effects on the pH stability of the rice water shampoo gel preparation.

Keywords: Rice water, Shampoo gel, Propylene glycol, Physical properties, Stability

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INTRODUCTION

Rice (*Oryza sativa* L.) cultivation is an essential commodity worldwide, as it is the staple food for more than half of the world's population. Rice is cultivated in over 100 countries and provides over 20% of the calories consumed worldwide [1]. Rice is rich in carbohydrates and contains proteins, fats [2], as well as vitamins and minerals, such as vitamin B1, vitamin B2, vitamin B3, vitamin B5, vitamin B6, vitamin B9, biotin, inositol, choline, vitamin B12, vitamin E, calcium, iron, phosphorus, glycemic index, and retinol if consumed directly [3]. Milled rice must be washed to remove bran, dust, and dirt before cooking. However, the rice-washing process can remove many water-soluble nutrients from the rice grains. The water from washed rice containing these nutrients is often thrown into the environment. This wastewater is called rice water (RW) [4]. Cloudy RW is known to have benefits for hair care because of its vitamin B8 or inositol content, which can improve hair elasticity and soften hair [5], and the antioxidants in its vitamin E content can keep the hair cuticles from opening or being damaged [6].

These benefits make RW a highly potential ingredient to be developed into a shampoo preparation. Shampoo has become very important as a routine practice in daily life. Hair shampoo has the dual purpose of maintaining personal hygiene and cleansing, promoting beautiful hair and a healthy scalp. More consumers prefer to use plant-based hair shampoos, which are natural and sustainable. Recent reports show that formulated herbal shampoos can also be equivalent to commercially available ones [7]. Compared to other preparations, the formulation of shampoo preparation in the gel is preferable due to some advantages, such as the high viscosity and adhesion that prevent the gel from flowing too quickly on the skin surface, the pseudoplastic property that makes it easy to spread when applied, no marks left, the film-

like thin layer when applied, the easily-washed property, and the cool sensation after use [8, 9]. A gel preparation should have adequate carrier stability to maintain its condition during storage. However, a gel preparation can undergo a process that damages stability. One of the phenomena that can damage gel preparation's strength is syneresis [10, 11]. Syneresis is a phenomenon in which the solvent separates from the gel matrix, thus reducing the gel volume [12]. Syneresis is associated with an imbalance of strength between the two sides of the gel-liquid interface and is associated with gel stiffness [12].

The components of gel preparation that affect the physical quality and stability are the gelling agent and humectant. A gelling agent will form a gel matrix. At the same time, a humectant will maintain the stability of gel preparation by absorbing moisture and reducing water evaporation in the gel to overcome syneresis [13, 14]. One of the humectants frequently used for gel preparation is propylene glycol (PG). Under the IUPAC name propane-1,2-diol, PG is a colorless, viscous, almost odorless, slightly sweet liquid. With its chemical structure illustrated in fig. 1, PG contains two alcohol groups and is classified as a diol [15]. PG is widely used as an excipient, humectant, co-solvent, and penetration enhancer [16, 17]. In addition, PG can facilitate the dissolution of chemicals and, at certain levels, can affect drug release [18]. This ingredient is used in various pharmaceutical dosage forms since it is generally known as a relatively non-toxic material [15].

This study aims to determine the effect of variations in PG concentration as a humectant on RW shampoo gel's physical properties and stability. The physical properties of the gel preparation evaluated in this study include viscosity, pH, and foam height. In addition, physical stability testing was also carried out at weeks 0, 1, 2, 3, and 4.

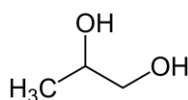


Fig. 1: Chemical structure of PG (propylene glycol)

MATERIALS AND METHODS

Materials

Rice water (RW) was obtained from washing at the Pharmaceutical Technology Laboratory of Universitas Islam Indonesia, Yogyakarta, Indonesia. Genuine hair knots with such criteria as straight, black, and thick were obtained from a salon in Sleman Regency, Yogyakarta, Indonesia. SLS (Sodium Lauryl Sulfate) (Brataco), HPMC K4M (Colorcon), propylene glycol (PG) (Brataco), methylparaben (Brataco), propylparaben (Brataco), lemon fragrance (Lansida), and distilled water were obtained from the Pharmaceutical Technology Laboratory of Universitas Islam Indonesia. The equipment consisted of a pH meter (Inolab WTW Series), a Brookfield Viscometer (DV-1 Prime), a hotplate stirrer (Thermolyne-Cimarec), and a set of glassware (Phyrex).

Extraction of active substance

The active substance used was the RW from the washing process using distilled water with a ratio of rice and water of 1:1. The washing was carried out using a 1000 ml beaker glass by kneading the rice for 3 min. The RW from the first washing was filtered using Whatman paper No. 42, and 20 ml of the filtrate was collected.

Preparation of gel base

The RW shampoo gel was prepared from 4 grams of HPMC K4M gel base for all formulations. HPMC K4M was developed using 100 ml of distilled water, stirred using a magnetic stirrer until it became homogeneous, formed a semisolid mass, and then allowed to stand overnight.

Preparation of shampoo gel

The finished gel base was added with different concentrations of PG for each formulation, 0.01 g of methylparaben, and 0.1 g of propylparaben dissolved with PG to obtain a homogeneous mixture. Then, 5 g of sodium lauryl sulfate dissolved in distilled water was added gradually and stirred until it became homogeneous. As much as 20 ml of RW was added to the mixture, followed by adding lemon fragrance as needed, and the mixture was stirred to obtain a homogeneous mix until gel was formed. The formulations of the RW shampoo gel preparation are presented in table 1.

Table 1: Formulations (F) of the RW shampoo gel

Name of ingredient	F1	F2	F3
Rice Water	20%	20%	20%
Sodium Lauryl Sulfate	5%	5%	5%
HPMC K4M	4%	4%	4%
Propylene Glycol	8%	10%	12%
Methyl Paraben	0.01%	0.01%	0.01%
Propyl Paraben	0.1%	0.1%	0.1%
Lemon Fragrance	Qs	Qs	Qs
Distilled Water	100 ml	100 ml	100 ml

The ingredients used in the RW shampoo gel preparation have their respective functions. RW acts as an active substance to soften hair [5]. Sodium lauryl sulfate is an anionic surfactant with a solid ability to clean in hard water [19]. HPMC K4M is a gelling agent not affected by electrolytes, can be mixed with preservatives, and has a wide pH range. PG functions as a humectant that binds water from humid air while retaining the water of the preparation [20]. The maximum concentration of PG as a humectant for topical preparations is 15% [21]. Meanwhile, methyl paraben and propyl paraben act as preservatives that prevent microbial contamination due to the high water content in the preparation. Combining preservatives will produce a preservative with a robust antimicrobial activity [21]. In addition, lemon fragrance has a role as a deodorizer, and distilled water acts as a solvent in the preparation.

Physical properties testing

The physical properties testing was conducted by observing the preparation made on the first day of the prepared shampoo gel. Physical properties of evaluation of the formulas included organoleptic testing, pH testing, viscosity testing, and foam height testing.

Organoleptic testing

Organoleptic testing is a test that includes observation of the form, fragrance, and color of the obtained shampoo gel. The test was performed by observing the sample in a 100 ml beaker glass by involving the human senses [22].

pH testing

A pH test is carried out using a digital pH meter. The sample was put in a 100 ml beaker glass, and then a pH meter was placed in the model, and the pH value would appear on the monitor [23].

Viscosity testing and flow properties testing

The viscosity and flow properties of the preparation were tested using a Brookfield Viscometer (DV-1 Prime) with spindle No. 64. The

viscosity testing was carried out at 50 rpm. In comparison, the flow properties were tested at 10 rpm, 20 rpm, and 50 rpm. As much as 100 ml of shampoo gel preparation was gradually poured into a 100 ml beaker glass and placed in the viscometer. The equipment was started, and the spindle was lowered into the practice up to the specified limit. Then, the viscosity was obtained by observing the cP value displayed on the equipment monitor [24].

Foam height testing

The foam height testing was performed using the Ultra-Turrax dispersing machine at 1000 rpm. As much as 20 ml of RW shampoo gel preparation was mixed with 120 ml of distilled water in a 250 ml measuring cup. The sample was tested in Ultra-Turrax for 3 min and then photographed, and the height of the foam produced was measured at 0 min and 5 min. The test was replicated three times [9].

Softness testing

A test of the softness level of the preparation was done on hair samples by preparing three hair knots, each weighing 2 grams and having a length of 12 cm. The three hair knots were placed in different Erlenmeyer flasks. Next, three samples of 10-gram shampoo gel, each with 8%, 10%, and 12% PG concentrations, were put into the Erlenmeyer flasks containing hair knots, mixed with 30 ml of tap water, and then shaken for 2 min. The hair was removed, rinsed under running tap water, and then left to dry at room temperature. This test also involved 20 volunteers who assessed the softness level of the three hair knots [24]. The selected volunteers touched the three hair knots for 2 min [25]. After that, all hair samples were scored between 1 and 4 (1 = not smooth; 2 = relatively smooth; 3 = smooth; 4 = very smooth) [26].

Physical stability testing

The physical stability test of the shampoo gel was done by observing changes in the preparation's color, form, and fragrance. This was followed by observation of changes in pH, viscosity, and foam height in weeks 0, 1, 2, 3, and 4.

Data analysis

The test results were analyzed descriptively. The data obtained from the test results, including the form, color, fragrance, viscosity, pH, and foam height of the shampoo gel, were compared with the references. The analysis of physical properties testing and physical stability testing results was done using the One-Way ANOVA statistical test method.

RESULTS AND DISCUSSION

Physical properties testing of the RW shampoo gel

Organoleptic testing

Organoleptic testing includes observation of the color, form, and fragrance of the prepared shampoo gel. Organoleptic testing is a method used to test the quality of a material or product using the five senses [22]. The organoleptic testing of the physical properties observed in the preparation showed that the F1, F2, and F3 shampoo gel had a clear pale yellow color, semisolid form, and distinctive lemon fragrance. The addition of PG as a humectant to the RW

shampoo gel preparation did not affect the organoleptic properties of the three formulations. PG binds water fairly strongly in practice [27]; hence, the RW shampoo gel produced for the three formulations shows a constant result in which the form remains semisolid (gel). The formulations of RW shampoo gel preparation can be seen in fig. 2A.

pH testing

pH test is performed to determine the acidity degree of a material. The more acidic or alkaline the pH of the preparation, the more it will affect the practice or the user [23]. The pH test results in this study can be seen in fig. 4A. The pH test results showed that the variation of PG concentration in the three RW shampoo gel formulations had a significant effect on the pH of the preparation. This indicates that the variation in PG concentration added to the shampoo gel affects the pH of the practice. However, the change in pH does not reduce the safety of the RW shampoo gel preparation because the pH values of F1, F2, and F3 are remain in the pH range applied by the Indonesian National Standard (SNI), which is 5.0-9.0 [28].

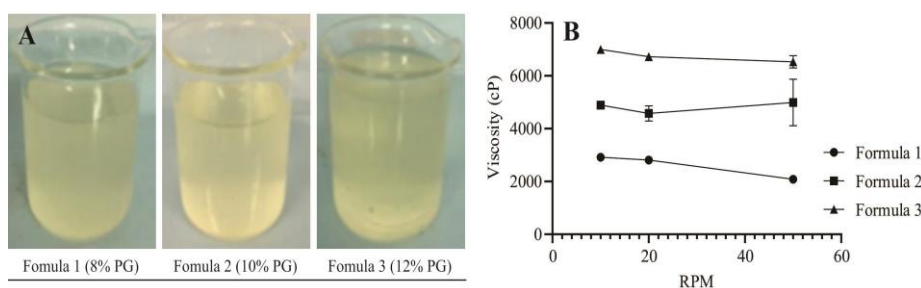


Fig. 2: Formulations of the RW shampoo gel (A), Graph of the flow properties of the RW shampoo gel foam height testing (B)

Evaluation of viscosity and flow properties

Viscosity evaluation describes a preparation's resistance to flow. Practices with high viscosity will have high resistance to flow [29]. The results of the viscosity testing in this study can be seen in fig. 4B. Viscosity testing showed that increasing PG concentration in the three formulations significantly affected the viscosity of RW shampoo gel. A higher concentration of PG will improve the consistency of a preparation. This is because, as a humectant, PG binds water by forming hydrogen bonds. An increase in viscosity will affect a preparation's ease of flow out of the container and comfort of application [30]. The formulation of RW shampoo gel preparation that shows the best viscosity is F1, with a viscosity range within the acceptance criteria of 2000-4000 Cp [28].

Viscosity test results at 10 rpm of RW shampoo gel for F1 was 2915 cP±77.11; F2 was 4887 cP±80.30; and F3 was 6998 cP±43.88. Viscosity at 20 rpm of RW shampoo gel for F1 was 2810 cP±18.82; F2 was 4575 cP±285.51; and F3 was 6724 cP±95.87. While the viscosity at 50 rpm of RW shampoo gel for F1 was 2082 cP±80.07, F2 was 4987 cP±883.88, and F3 was 6531 cP±232.79. The flow properties of the three preparation formulations are categorized into the non-Newtonian pseudoplastic type, in which the viscosity will decrease as the shear rate increases. This occurs in such long-chain molecules as polymers, including in gums, tragacanth, Na-alginate, methylcellulose, and carboxymethylcellulose [29]. In preparing RW shampoo gel, HPMC functions as a gelling agent and simultaneously affects the preparation's flow properties. The pseudoplastic system is also known as the shear-thinning system since increasing the shear stress decreases viscosity [29]. The graph of the flow properties of the RW shampoo gel can be seen in fig. 2B.

Foam height testing reflects the ability of a detergent to produce foam. Testing foam height is one of the methods used to control the quality of a detergent product to ensure the expected capacity of a preparation to produce foam [31]. The results of the foam height testing in this study can be seen in fig. 4C. The effects of physical

properties testing on the foam height of RW shampoo gel preparation showed that the variation of PG concentration added to the shampoo gel preparation significantly affected the foam height in formulation one versus formulation 2. Meanwhile, formulation one versus formulation three and formulation two versus formulation 3 showed no significant difference. The data showed that the foam height obtained from the three RW shampoo gel formulations showed good results because it was in the specified foam height range of 1.3-22 cm [32].

Softness testing

The softness testing in this study aims to prove that the RW shampoo gel preparation can provide good efficacy in softening hair due to its inositol compound content. The test was evidenced by an assessment from 20 volunteers who gave a softness score to the three hair knots washed with the three formulations of the shampoo gel preparation. The results of the softness testing in this study are shown in fig. 5.

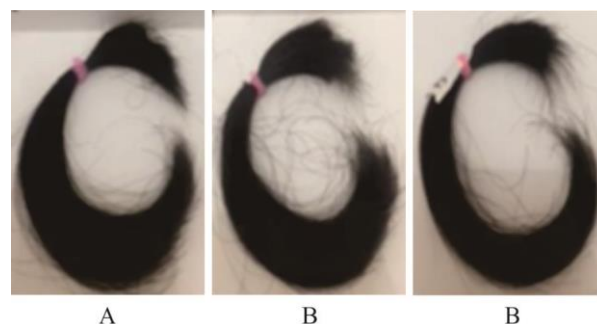


Fig. 3: Appearance of the hair washed with the shampoo gel. F1 volumized and smooth (A); F2 volumized but not smooth (B); F3 not volumized but smooth (C)

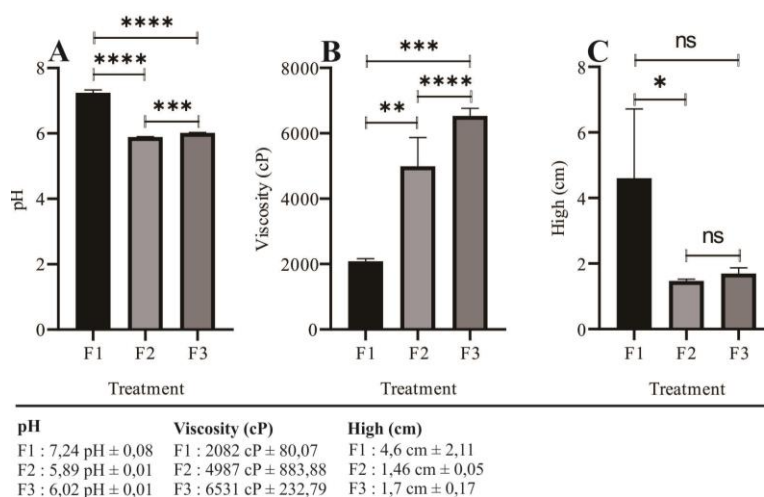


Fig. 4: Results of the physical properties testing on the RW shampoo gel. pH Test (A), Viscosity Test (B) and Foam height Test (C). Samples were analyzed by one-way ANOVA at 95% confidence interval, followed by post hoc comparison test. n: 3; *p<0.05, **p<0.01, ***p<0.001, ****p<0,0001

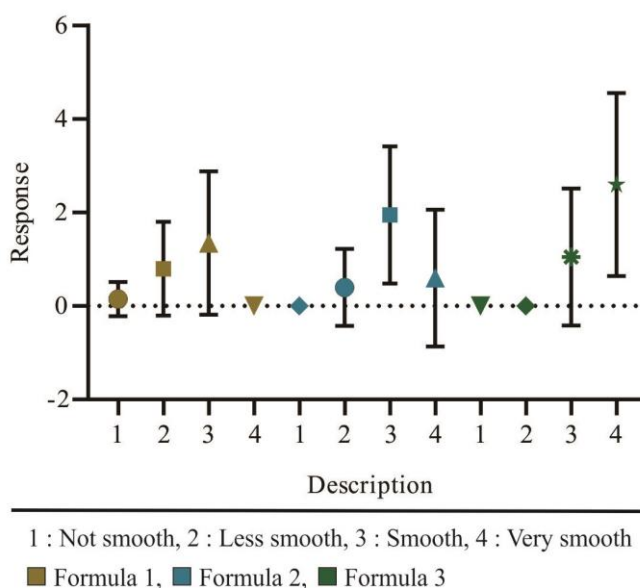


Fig. 5: Softness test results on RW shampoo gel. The x-axis is the description score, while the y-axis is the indicated response value score

The results of the softness testing showed that F3 of RW shampoo gel gave the impression of softening the hair, with the average hair softness score of F3 being higher than that of F1 and F2. The results indicate that the higher the PG concentration, the more influential the RW will be in softening hair. The appearance of the hair washed with the preparation can be seen in fig. 3.

Physical stability testing of the RW shampoo gel

Physical stability testing aims to determine preparation stability when stored at a specific temperature in an appropriate container at a predetermined time. Stability testing in this study includes organoleptic testing, pH testing, viscosity testing, and foam height testing, described in tables and graphs that are statistically analyzed. Physical stability testing of organoleptic properties was carried out in weeks 0, 1, 2, 3, and 4 by storing the preparation in a clear, tightly closed bottle at 25 °C, and the results obtained are shown in table 2.

The results of the organoleptic stability test of the RW shampoo gel in F1, F2, and F3 observed in weeks 0, 1, 2, 3, and 4 showed that the preparation in F1 underwent a form change in week 2. This is because,

in F1 preparation, the concentration of PG tends to be lower than that of F2 and F3. According to the literature, the higher the concentration of propylene glycol, the stronger the ability of PG to bind water with hydrogen bonds, resulting in a more stable consistency of shampoo gel preparation during prolonged storage [33].

The results of the pH stability test of RW shampoo gel in F1, F2, and F3 observed for one month showed that the variation of PG concentration in F1 showed a significant difference in week 0 compared to weeks 1 and 2, while in weeks 3 and 4 it did not show a significant difference. The results of the pH stability test of F2 showed no significant difference between week 0 versus weeks 1, 2, 3, and 4. The results of the pH stability test of F3 showed a substantial difference in week 0 versus week 3, while weeks 1, 2, and 4 showed no significant difference compared to the control results in week 0. The results of the pH stability test of RW shampoo gel in F1, F2, and F3 observed for one month show that the variation in PG concentration does not significantly affect the pH of the three formulations. The results of pH stability testing in this study can be seen in fig. 6.

Table 2: Results of the organoleptic stability testing (color, form, and fragrance) of the RW shampoo gel in week 0, 1, 2, 3, and 4

Week	0	1	2	3	4
F1	Pale Yellow	Pale Yellow	Pale Yellow	Pale Yellow	Pale Yellow
	Clear	Clear	Clear	Clear	Clear
	Semisolid (Gel)	Semisolid (Gel)	Semisolid (Gel)	Semisolid (Gel)	Semisolid (Gel)
F2	Distinctive Lemon	Distinctive Lemon	Distinctive Lemon	Distinctive Lemon	Distinctive Lemon
	Pale Yellow	Pale Yellow	Pale Yellow	Pale Yellow	Pale Yellow
	Clear	Clear	Clear	Clear	Clear
F3	Semisolid (Gel)	Semisolid (Gel)	Semisolid (Gel)	Semisolid (Gel)	Semisolid (Gel)
	Distinctive Lemon	Distinctive Lemon	Distinctive Lemon	Distinctive Lemon	Distinctive Lemon
	Pale Yellow	Pale Yellow	Pale Yellow	Pale Yellow	Pale Yellow
	Clear	Clear	Clear	Clear	Clear
	Semisolid (Gel)	Semisolid (Gel)	Semisolid (Gel)	Semisolid (Gel)	Semisolid (Gel)
	Distinctive Lemon	Distinctive Lemon	Distinctive Lemon	Distinctive Lemon	Distinctive Lemon

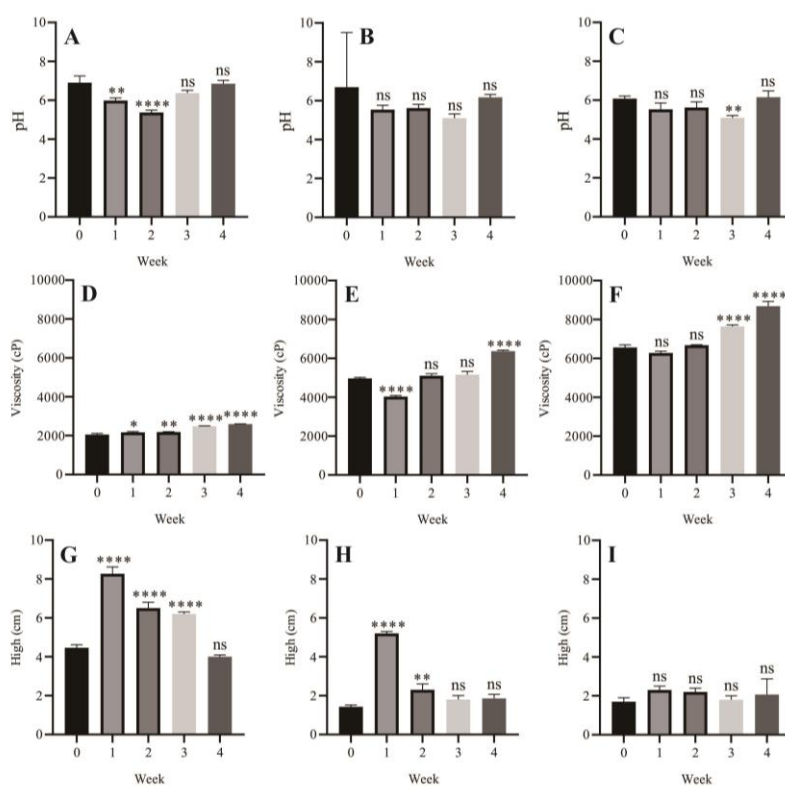


Fig. 6: Graph of the stability testing of the RW shampoo gel in week 0, 1, 2, 3, and 4. pH Test Formula 1 (A), pH Test Formula 2 (B), pH Test Formula 3 (C), Viscosity Test Formula 1 (D), Viscosity Test Formula 2 (E), Viscosity Test Formula 3 (F), Foam Height Test Formula 1 (G), Foam Height Test Formula 2 (H) dan Foam Height Test Formula 3 (i). Samples were analyzed by one-way ANOVA at 95% confidence interval, followed by post hoc comparison test. n: 3; * $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$, **** $p < 0.0001$

The results of testing the viscosity stability of the preparation in F1 showed a significant difference at week 0 compared to weeks 1, 2, 3, and 4. Meanwhile, F2 showed a significant difference at week 0 compared to weeks 1 and 4, while weeks 2 and 3 showed no significant difference. As for F3 showed a significant difference at week 0 compared to weeks 3 and 4, while weeks 1 and 2 showed no significant difference compared to the control results at week 0. It can be concluded that the results of the viscosity stability test of RW shampoo gel preparations in F1, F2, and F3 observed for one month show that the variation in PG concentration significantly affects the viscosity stability of RW shampoo gel preparations in F1, F2, and F3 as shown in fig. 6. An increase in PG concentration will increase the ability of PG as a humectant to bind water in the gel structure, thereby increasing the viscosity and stability of the gel during storage [33].

The results of testing the stability of the foam height of the preparation in F1 showed a significant difference in week 0 compared to weeks 1, 2, and 3. while week 4 showed no significant difference. Meanwhile, F2 showed a substantial difference in week 0

compared to weeks 1 and 2, while weeks 3 and 4 showed no significant difference. As for F3 showed no significant difference at week 0 compared to weeks 1, 2, 3, and 4, which were compared to the control results at week 0. It can be concluded that the results of the foam height stability test conducted at weeks 1, 2, 3, and 4 on F1, F2, and F3 show that increasing PG concentration affects the stability of RW shampoo gel foam height. The results of the foam height stability test in this study can be seen in fig. 6. Sodium Lauryl Sulfate, which acts as a foam generator in RW shampoo gel preparations, functions as a cleanser and an emulsifying agent that can stabilize the shampoo gel dosage form [34]. The higher the concentration of PG, the lower the ability of the shampoo to foam because PG binds water in the gel preparation structure, making it difficult for surfactants to produce foam [32].

CONCLUSION

Variations in the concentrations of PG in the three formulations affect the physical properties of the shampoo gel preparation in

terms of the pH, viscosity, and foam height but do not affect the organoleptic properties of the RW shampoo gel. The varied concentrations of PG also affect the organoleptic stability, consistency, and foam height but do not affect the pH stability of the RW shampoo gel preparation.

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AUTHORS CONTRIBUTIONS

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Data analysis/interpretation- Lutfi Chabib, Arman Suryani, Siti Z. Munawiroh, Siti Mariyam, Marlyn Dian Laksitorini

Critical revision of manuscript- Lutfi Chabib, Arman Suryani, Siti Z. Munawiroh, Marlyn Dian Laksitorini

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Final approval- Lutfi Chabib, Arman Suryani, Siti Z. Munawiroh, Marlyn Dian Laksitorini

CONFLICT OF INTERESTS

Declared none

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