BULETIN VETERINER UDAYANA

pISSN 2085-2495
eISSN 2477-2712

Received: 21 June 2026; Accepted: 30 June 2026; Published: 30 June 2026

THE EFFECT OF VARYING PROPYLENE GLYCOL CONCENTRATIONS ON THE PHYSICAL AND CHEMICAL STABILITY OF *AVERRHOA BILIMBI* LEAF EXTRACT CREAM FORMULATION

Pengaruh Variasi Konsentrasi Propilen Glikol terhadap Stabilitas Fisik dan Kimia Sediaan Krim Ekstrak Daun Belimbing Wuluh (*Averrhoa bilimbi*)

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How to cite: Unique IGANP. 2026. The effect of varying propylene glycol concentrations on the physical and chemical stability of *Averrhoa bilimbi* leaf extract cream formulation. *Bul. Vet. Udayana.* 18(3): 736-743. DOI: https://doi.org/10.24843/bulvet.2026.v18.i03.p25

Abstract

Creams are one of the most widely used topical formulations for the treatment of acne because they are easy to apply, comfortable to use, and easy to wash off. *Averrhoa bilimbi* leaves are known to contain flavonoids and tannins that have potential as antibacterial agents against acne-causing bacteria; however, natural-based formulations generally have poor physical stability. This study aims to determine the effect of varying propylene glycol concentrations on the physical and chemical stability of *Averrhoa bilimbi* leaf extract cream formulations and to identify the most optimal propylene glycol concentration. This study is an experimental study using three formulations with varying propylene glycol concentrations, namely 8%, 10%, and 12%. Physical properties were evaluated through organoleptic testing, homogeneity, pH, spreadability, adhesion, and stability testing using the freeze-thaw cycling test method for 3 cycles or 6 days. The results showed that all formulations met the requirements for organoleptic testing, homogeneity, and pH. However, only formulation 3 with a 12% propylene glycol concentration met all physical evaluation parameters, including spreadability and adhesion. Formula 3 showed a spreadability value of 5.49 cm before the stability test and 6.10 cm after the stability test, as well as an adhesion value of 4.61 seconds before the stability test and 4.35 seconds after the stability test. Based on the research results, it can be concluded that variations in propylene glycol concentration affect the physical stability of the *Averrhoa bilimbi* leaf extract cream formulation, and a 12% propylene glycol concentration is the most optimal formula.

Keywords: *Averrhoa bilimbi* leaf extract, cream, humectant, physical stability, propylene glycol

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https://doi.org/10.24843/bulvet.2026.v18.i03.p25

Abstrak

Krim merupakan salah satu sediaan topikal yang banyak digunakan untuk pengobatan jerawat karena mudah diaplikasikan, nyaman digunakan, dan mudah dibersihkan. Daun belimbing wuluh (*Averrhoa bilimbi*) diketahui mengandung senyawa *flavonoid* dan *tanin* yang berpotensi sebagai antibakteri penyebab jerawat, namun sediaan berbahan alam umumnya memiliki stabilitas fisik yang kurang baik. Penelitian ini bertujuan untuk mengetahui pengaruh variasi konsentrasi propilen glikol terhadap stabilitas fisik dan kimia sediaan krim ekstrak daun belimbing wuluh serta menentukan konsentrasi propilenglikol yang paling optimal. Penelitian ini merupakan penelitian eksperimental menggunakan tiga formula dengan variasi konsentrasi propilen glikol yaitu 8%, 10%, dan 12%. Evaluasi sifat fisik dilakukan melalui uji organoleptis, homogenitas, pH, daya sebar, daya lekat, dan uji stabilitas menggunakan metode *freeze-thaw cycling test* selama tiga siklus atau enam hari. Hasil penelitian menunjukkan bahwa seluruh formula memenuhi persyaratan uji organoleptis, homogenitas, dan pH. Namun, hanya formula 3 dengan konsentrasi propilenglikol 12% yang memenuhi seluruh parameter evaluasi fisik, termasuk daya sebar dan daya lekat. Formula 3 menunjukkan nilai daya sebar sebesar 5,49 cm sebelum pengujian stabilitas dan 6,10 cm setelah pengujian stabilitas, serta nilai daya lekat sebesar 4,61 detik sebelum pengujian stabilitas dan 4,35 detik setelah pengujian stabilitas. Berdasarkan hasil penelitian dapat disimpulkan bahwa variasi konsentrasi propilenglikol berpengaruh terhadap stabilitas fisik sediaan krim ekstrak daun belimbing wuluh dan konsentrasi propilenglikol 12% merupakan formula yang paling optimal.

Keywords: Ekstrak daun *Averrhoa bilimbi*, humektan, krim, propilen glikol, stabilitas fisik

INTRODUCTION

Indonesia is a tropical country with abundant biodiversity, including various plant species that have the potential to be used as ingredients in traditional medicine. One plant widely used by the community is *Averrhoa bilimbi* leaf or commonly known in Indonesia as *belimbing wuluh*. This plant is known to have various health benefits, such as helping to treat coughs, lower blood pressure, brighten the skin, and relieve joint pain. In addition to the fruit, the leaves of the *Averrhoa bilimbi* are also frequently used to treat itching, acne, and coughs. The use of this natural ingredient has since been developed into a health product in the form of a cream because it is practical, comfortable to apply to the skin, non-sticky, and easy to wash off with water (Insan *et al.*, 2019).

Skin problems such as acne are still commonly experienced by people, especially teenagers and young adults. Acne, or acne vulgaris, is a skin condition characterized by clogged pores, leading to inflammation in the form of red spots or even pus-filled abscesses. One of the causes of acne is the growth of the bacterium *Staphylococcus aureus*. The leaves of the *Averrhoa bilimbi* are known to possess antibacterial activity due to their content of active compounds such as flavonoids, tannins, saponins, triterpenoids, and citric acid. Flavonoids and tannins act as antibacterial agents capable of inhibiting the growth of acne-causing bacteria (Wintariani *et al.*, 2021). Previous research has shown that a 10.5% *Averrhoa bilimbi* leaf extract is capable of inhibiting the growth of *Staphylococcus aureus* (Rachmawaty *et al.*, 2018). Therefore, *Averrhoa bilimbi* leaves have the potential to be developed as a natural active ingredient in acne treatments.

One of the most commonly used topical formulations in acne treatment is cream. Cream is a semisolid formulation in the form of an oil-in-water (O/W) or water-in-oil (W/O) emulsion that has a relatively soft consistency and is easy to apply to the skin. Cream formulations offer several advantages, such as being comfortable to use, non-sticky, easy to spread, and easy to wash off—especially in the oil-in-water (O/W) type (Annisa, 2022). However, natural-based

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formulations containing extracts generally have poor physical and chemical stability due to the influence of particle size, solvent type, temperature, pH, and storage duration (Rosa *et al.*, 2022). Therefore, the addition of humectants is necessary to help maintain water content and improve formulation stability. One commonly used humectant is propylene glycol, as it helps maintain moisture and improve the physical characteristics of the cream during storage (Anita *et al.*, 2017).

Based on the above description, this study was conducted to develop a cream formulation containing *Averrhoa bilimbi* leaf extract with varying concentrations of propylene glycol as a humectant in order to obtain a cream formulation with good physical stability. Stability evaluation was performed through physical quality testing, including organoleptic assessment, homogeneity, pH, spreadability, and adhesion. This study is expected to provide scientific information regarding the effect of varying propylene glycol concentrations on the physical and chemical stability of the *Averrhoa bilimbi* leaf extract cream formulation as a natural acne treatment.

This study aims to determine the effect of varying propylene glycol concentrations on the physical properties of *Averrhoa bilimbi* leaf extract cream, as well as to identify the optimal propylene glycol concentration in the cream formulation to ensure good physical stability during storage. In addition, this study also aims to analyze the effect of varying propylene glycol concentrations on the physical stability of the *Averrhoa bilimbi* leaf extract cream through physical quality testing, including organoleptic testing, homogeneity, pH, spreadability, and adhesion. This study is expected to determine a formulation with the optimal propylene glycol concentration, resulting in a *Averrhoa bilimbi* leaf extract cream formulation that possesses physical characteristics meeting quality requirements and remains stable during storage.

RESEARCH METHODOLOGY

Ethical Approval for Laboratory Animals

This study does not require ethical approval because it does not use laboratory animals or involve interventions on live animals. The study was conducted using a natural ingredient, i.e., *Averrhoa bilimbi* leaves formulated into a cream preparation.

Research Subjects

The subjects of this study were cream formulations of *Averrhoa bilimbi* leaf extract with varying concentrations of propylene glycol as a humectant. The *Averrhoa bilimbi* leaves were obtained from Bangbang Village, Bangli Regency, Bali Province, Indonesia and extracted using the maceration method with 70% ethanol as the solvent. The cream formulations were prepared in three treatments with varying propylene glycol concentrations: 8%, 10%, and 12%.

Research Design

This study is an experimental study conducted at the Pharmacology and Pharmacy Laboratory, Faculty of Veterinary Medicine, Udayana University. The study was carried out in March 2026. The study aimed to determine the effect of varying propylene glycol concentrations on the physical and chemical stability of a cream formulation containing *Averrhoa bilimbi* leaf extract.

Research Variables

The independent variable in this study was the concentration of propylene glycol as a humectant in the cream formulation. The dependent variables included the physical properties of the *Averrhoa bilimbi* leaf extract cream preparation, consisting of organoleptic testing, homogeneity, spreadability, adhesion, pH, and formulation stability. The controlled variables

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in this study included the extraction temperature, mixing technique during cream preparation, and temperature during stability testing.

Data Collection Method

A total of 2.85 kg of *Averrhoa bilimbi* leaves were selected from green leaves, then washed under running water, drained, finely chopped, and dried in an oven at 50°C for 90 minutes. The dried crude drug was then ground into a powder using a blender. Extraction was performed using the maceration method with 70% ethanol at a 1:10 ratio for 3 × 24 hours, with stirring every 24 hours. The macerate was then filtered and concentrated using a vacuum rotary evaporator at 40°C until a concentrated extract was obtained (Masaenah *et al.*, 2019).

The cream formulation was prepared using an oil-in-water (O/W) cream base, as it is suitable for polar active compounds such as flavonoids and tannins. The oil phase, consisting of cera alba, stearic acid, and white petrolatum, was melted at 75°C. The aqueous phase, consisting of triethanolamine (TEA) and propylene glycol, was also heated to the same temperature. The two phases were combined and stirred until homogeneous, then hot distilled water and methylparaben were added. After the cream cooled, the *Averrhoa bilimbi* leaf extract was added to the cream base and homogenized.

The evaluation of the physical quality of the preparation includes organoleptic testing, homogeneity, spreadability, adhesion, pH, and stability. Organoleptic testing is performed by observing the shape, color, and odor of the preparation. The homogeneity test is performed by applying the cream to a microscope slide to assess particle uniformity. The spreadability test is conducted using graduated weights of 50 grams, 100 grams, and 150 grams. The pH test is performed using universal pH paper within the optimal skin pH range of 4.5–6.5 (Jufri *et al.*, 2020). The adhesion test was performed using two glass slides with a 100-gram load for 5 minutes, and the time it took for the two glass slides to separate was recorded. Stability testing was conducted using a freeze-thaw cycling test over 3 cycles, with storage at 1–2°C and room temperature (29–30°C) for 24 hours each (Wiguna, 2016).

Data Analysis

The data from the physical quality testing of the cream formulation were analyzed descriptively and presented in tabular form and as a narrative description. The analysis was conducted to compare the effects of varying propylene glycol concentrations on the physical characteristics and stability of the *Averrhoa bilimbi* leaf extract cream formulation.

RESULTS AND DISCUSSION

Results

Organoleptic Test Results

An organoleptic test was conducted to visually assess the physical characteristics of the cream formulation, including its form, color, and odor. Based on the test results before and after the stability test using the freeze-thaw method for 3 cycles, all formulations exhibited a semi-solid form, were brown in color, and were odorless. The observations showed that there were no changes in form, color, or odor in any of the formulations after the stability test. The results of the organoleptic test are shown in Table 1.

Homogeneity Test Results

Homogeneity testing was conducted to determine whether all ingredients were evenly mixed in the cream formulation. Based on the test results before and after the freeze-thaw stability test, all formulations were found to be homogeneous, and no coarse particles were detected in the

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cream formulations. The color distribution in all formulations also appeared uniform, indicating that all formulations met the homogeneity requirements. The homogeneity test results are shown in the table below.

pH Test Results

pH testing was conducted to determine the safety of the cream formulation when applied to the skin. Based on the results of tests conducted before and after the freeze-thaw stability test, all formulations showed a pH value of 6.0 and did not change during storage. These pH values remain within the normal skin pH range of 4.5–6.5, meaning all formulations meet the requirements for topical preparations. The pH test results are shown in Table 2.

Spreadability Test Results

Spreadability testing was conducted to determine the cream's ability to spread across the skin's surface. Test results prior to the freeze-thaw stability test showed that Formula 1 had a spreadability of 7.63 cm, Formula 2 of 5.80 cm, and Formula 3 of 5.49 cm. After the stability test, the spreadability of Formula 1 increased to 8.03 cm, Formula 2 to 7.12 cm, and Formula 3 to 6.10 cm. Based on these results, Formula 3 met the requirement for good spreadability (5–7 cm), while Formulas 1 and 2 did not meet the requirement after the stability test. The spreadability test results are shown in Table 3.

Adhesion Test Results

Adhesion testing was conducted to determine the ability of the cream formulations to adhere to the skin's surface. Based on the test results prior to the freeze-thaw stability test, Formula 1 had an adhesion time of 1.27 seconds, Formula 2 had 4.09 seconds, and Formula 3 had 4.61 seconds. After the stability test, the adhesion time for Formula 1 was 1.00 seconds, for Formula 2 was 3.87 seconds, and for Formula 3 was 4.35 seconds. Based on these results, Formula 3 met the requirement for good adhesion—more than 4 seconds—while Formulas 1 and 2 did not meet the requirement after the stability test. The adhesion test results are shown in Table 4.

Discussion

The results of the organoleptic testing showed that all formulations exhibited good physical stability during storage. The absence of changes in form, color, and odor after freeze-thaw testing indicates that the cream formulations were able to maintain their physical characteristics despite changes in storage temperature. Organoleptic stability is influenced by the stability of the formula's ingredients, the homogeneity of the mixture, and the ability of propylene glycol, as a humectant, to maintain the formulation's moisture content. According to Yuli *et al.* (2022), organoleptic changes in cream formulations can be influenced by emulsion instability, oxidation of active ingredients, and temperature fluctuations during storage.

Homogeneity is a critical parameter in topical formulations because it affects the distribution of active ingredients on the skin's surface. The study results indicate that variations in propylene glycol concentration do not affect the homogeneity of the cream formulation. The absence of coarse particles indicates that the mixing of the oil phase and water phase proceeded effectively, resulting in a stable emulsion system. According to Safitri *et al.* (2023), a homogeneous cream formulation is characterized by uniform mixing of ingredients and the absence of aggregates or coarse particles in the formulation.

Stable pH values indicate that variations in propylene glycol concentration do not affect the acidity of the cream formulation. pH stability during storage indicates that the formulation has good chemical stability and is safe for use on the skin. Topical formulations with a pH that is too acidic or too alkaline can cause skin irritation. According to Jufri *et al.* (2020), the pH of a

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good topical formulation must match the skin's physiological pH to avoid causing irritation and damage to the skin barrier. The pH stability in this study was also influenced by propylene glycol's ability to maintain moisture and the stability of the emulsion system.

Spreadability is influenced by the viscosity and consistency of the cream formulation. The lower the viscosity of a formulation, the wider the resulting spreadability. The study results show that an increase in propylene glycol concentration affects the spreadability stability of the cream during storage. Formula 3, with a 12% propylene glycol concentration, exhibited the best spreadability stability compared to the other formulas. Changes in spreadability can be influenced by mixing temperature, stirring time, particle size, and storage temperature. According to Baskara *et al.* (2020), smaller particle sizes can improve homogeneity and affect the extent of the formulation's spread across the skin's surface.

Adhesion is related to the duration of contact between the formulation and the skin surface, thereby affecting the effectiveness of active ingredient absorption. The longer a formulation's adhesion, the better the active ingredient's ability to come into contact with the skin. The results of the study show that Formula 3, with a 12% propylene glycol concentration, has the best adhesion stability compared to the other formulas. Differences in adhesion are influenced by the propylene glycol concentration, mixing temperature, and stirring duration during the formulation process. According to Baskara *et al.* (2020), an optimal mixing process can produce smaller particle sizes, thereby improving the homogeneity and stability of the cream formulation. Additionally, the storage temperature during freeze-thaw testing can also affect the consistency and adhesion of the formulation.

CONCLUSIONS AND SUGGESTIONS

Conclusion

Based on the results of the study on the effect of varying propylene glycol concentrations on the physical and chemical stability of *Averrhoa bilimbi* leaf extract cream formulations using a 6-day freeze-thaw cycling test, it can be concluded that all cream formulations met the physical test requirements, including organoleptic properties, homogeneity, and pH. However, in the spreadability and adhesion tests, some formulations did not meet the requirements, namely Formulation 1 and Formulation 2. Formulation 3 showed the best results because it met all physical test requirements both before and after the stability testing. The most optimal cream formulation containing *Averrhoa bilimbi* leaf extract is Formula 3, with a propylene glycol concentration of 12%. This formulation exhibited the best physical characteristics and stability during storage, as determined by the results of the organoleptic, homogeneity, pH, spreadability, and adhesion tests.

Suggestions

Based on the results of this study, it is recommended that future research develop formulations of *Averrhoa bilimbi* leaf extract creams with varying concentrations of propylene glycol or other types of humectants to achieve more optimal product characteristics. In addition, viscosity testing and quantitative data analysis should be conducted to support a more in-depth evaluation of the physical and chemical stability of the cream formulations.

ACKNOWLEDGMENTS

The author would like to thank Udayana University for providing facilities and support throughout the research process. The author also extends gratitude to all parties who assisted in conducting this study on the formulation and stability evaluation of *Averrhoa bilimbi* leaf extract cream, enabling the successful completion of this research.

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Tables

Table 1. Organoleptic Test Results

Formula	Shape		Color		Odor	
	Before Freeze-thaw	After Freeze-thaw	Before Freeze-thaw	After Freeze-thaw	Before Freeze-thaw	After Freeze-thaw
F1	Semisolid	Semisolid	Brown	Brown	negative	negative
F2	Semisolid	Semisolid	Brown	Brown	negative	negative
F3	Semisolid	Semisolid	Brown	Brown	negative	negative

Table 2. Homogeneity Test Results

Formula	Before Freeze-thaw	After Freeze-thaw	Note
F1	Homogenous	Homogenous	Q
F2	Homogenous	Homogenous	Q
F3	Homogenous	Homogenous	Q

Abbreviations: Q=Qualified; NQ=Not Qualified

Table 3. pH Test

Formula	Mean Before *Freeze-thaw* ± SD	Mean after *Freeze-thaw* ± SD	Note
F1	6.0 ± 0.000	6.0 ± 0.000	Q
F2	6.0 ± 0.000	6.0 ± 0.000	Q
F3	6.0 ± 0.000	6.0 ± 0.000	Q

Abbreviations: Q=Qualified; NQ=Not Qualified

Table 4. Spreadability Test Results

Formula	mean Before *Freeze-thaw* (cm) ± SD	Mean after *Freeze-thaw* (cm) ± SD	Note
F1	7.63 ± 1.525	8.03 ± 1.269	NQ
F2	5.80 ± 0.883	7.12 ± 1.073	NQ
F3	5.49 ± 0.883	6.10 ± 0.525	Q

Abbreviations: Q=Qualified; NQ=Not Qualified. Note: It qualifies if it has a diameter of 5–7 cm (Genatrika *et al.*, 2020).

Table 5. Adhesion Test Results

Formula	Mean Before *Freeze-thaw* (detik) ± SD	Mean after *Freeze-thaw* (detik) ± SD	Note
F1	1.27 ± 0.454	1.00 ± 0.240	NQ
F2	4.09 ± 0.572	3.87 ± 0.316	NQ
F3	4.61 ± 0.548	4.35 ± 0.250	Q

Abbreviations: Q=Qualified; NQ=Not Qualified

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