

Formulation, Scale-Up and Physicochemical Characterization of a Foaming Exfoliating Face and Body Scrub Containing 5% Alpha-Hydroxy Acid

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SUMMARY

In this study, a foaming exfoliating face and body scrub formulation containing 5% lactic acid (alpha-hydroxy acid, AHA) was developed, and its physicochemical, microbiological, and antimicrobial properties were evaluated at different production scales. The formulation contains lactic acid (5%, pH 3.5–4.0) as a chemical exfoliant, fine crystalline sucrose (30%) as a physical exfoliant, a combination of sodium cocoyl isethionate (SCI) and cocamidopropyl betaine as the surfactant system, glycerin (10%) as a humectant, yellow iron oxide (CI 77492) as a colorant, and lemon peel essential oil (Citrus Limon Peel Oil) as a fragrance ingredient. The formulation was successfully scaled up from laboratory scale (100 mL) to semi-industrial scale (10,000 mL) without any modification to the original composition. Physicochemical characterization revealed that pH values remained within the target range (3.885–3.997) across all production scales, homogeneous appearance and organoleptic properties were maintained, and no phase separation was observed following centrifugal testing. Microbiological quality assessment demonstrated that total aerobic mesophilic microorganism and yeast/mould counts were below 10 CFU/g, and the absence criterion was met for all specified pathogenic microorganisms. Challenge testing conducted in accordance with TS EN ISO 11930:2019 confirmed that the preservative system reduced all five standard challenge organisms below the detection limit by day 7, maintaining efficacy through day 28. Three-month stability evaluation confirmed that pH, color, odor, appearance, and microbiological safety parameters remained stable under all tested storage conditions. These findings demonstrate the physicochemical stability, microbiological safety, and suitability of the developed formulation for commercial cosmetic product development.

Keywords: Alpha-hydroxy acid, exfoliating scrub, lactic acid, cosmetic formulation, scale-up, surfactant

%5 Alfa-Hidroksi Asit İçeren Köpüren Eksfoliyen Yüz ve Vücut Scrub'ının Formülasyonu, Ölçek Büyütme ve Fizikokimyasal Karakterizasyonu

ÖZ

Bu çalışmada, %5 laktik asit (alfa-hidroksi asit, AHA) içeren köpüren eksfoliyen bir yüz ve vücut scrub formülasyonu geliştirilmiş; fizikokimyasal, mikrobiyolojik ve antimikrobiyal özellikleri farklı üretim ölçeklerinde değerlendirilmiştir. Formülasyon, kimyasal eksfoliyen olarak laktik asit (%5, pH 3,5–4,0), fiziksel eksfoliyen olarak ince kristal şeker (sakkaroz, %30), sürfaktan sistemi olarak sodyum kokil izetinat (SCI) ve kokamidopropil betain kombinasyonu, humektan olarak gliserin (%10), renklendirici olarak sarı demir oksit (CI 77492) ve koku bileşeni olarak limon kabuğu uçucu yağını (Citrus Limon Peel Oil) içermektedir. Formülasyon, laboratuvar ölçeğinden (100 mL) yarı-endüstriyel ölçeğe (10.000 mL) herhangi bir kompozisyon değişikliği yapılmaksızın başarıyla büyütülmüştür. Fizikokimyasal karakterizasyon sonuçları, tüm üretim ölçeklerinde pH değerlerinin hedef aralıkta (3,885–3,997) kaldığını, homojen görünüm ve organoleptik özelliklerin korunduğunu ve santrifüj testi sonucunda faz ayrışması gözlemlenmediğini ortaya koymuştur. Mikrobiyolojik kalite değerlendirmesi kapsamında toplam aerobik mezofilik mikroorganizma ve maya-küf sayıları 10 CFU/g'ın altında tespit edilmiş; tüm belirtilen patojen mikroorganizmalar için yokluk kriteri sağlanmıştır. TS EN ISO 11930:2019 standardına göre gerçekleştirilen antimikrobiyal etkinlik (challenge) testi, koruyucu sisteminin beş standart mikroorganizmaya karşı 7. günde tespit sınırının altına inerek 28. güne kadar etkinliğini koruduğunu göstermiştir. Üç aylık stabilite analizinde pH, renk, koku, görünüm ve mikrobiyolojik güvenlik parametrelerinin tüm depolama koşullarında stabil kaldığı belirlenmiştir. Bu bulgular, geliştirilen formülasyonun fizikokimyasal kararlılığını, mikrobiyolojik güvenliğini ve ticari üretim için uygulanabilirliğini ortaya koymaktadır.

Anahtar Kelimeler: Alfa-hidroksi asit, eksfoliyen scrub, laktik asit, kozmetik formülasyon, ölçek büyütme, sürfaktan

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INTRODUCTION

The global market for exfoliating scrubs and personal care products incorporating exfoliating agents has expanded substantially in recent years, driven by increasing consumer awareness of skincare, growing demand for multifunctional formulations, and the rising influence of beauty and wellness trends. The use of alpha-hydroxy acids (AHAs) in cosmetic formulations has grown considerably over the past two decades, with lactic acid and glycolic acid being the most widely incorporated AHAs in both consumer and professional skin care products (Babilas, Knie, & Abels, 2012; Karwal & Mukovozov, 2023). Within this context, foaming scrubs that combine cleansing, physical exfoliation, and chemical exfoliation represent a particularly innovative and growing subcategory, as they address multiple skin concerns including dull texture, uneven tone, and superficial hyperpigmentation in a single step.

AHAs are a group of organic acids widely used in cosmetic and dermatological formulations due to their well-documented exfoliating and skin-renewing properties. AHAs, including glycolic acid, lactic acid, malic acid, tartaric acid, and citric acid, act by disrupting the cohesion of corneocytes in the stratum corneum, promoting desquamation, and stimulating epidermal cell turnover; their effectiveness is closely dependent on pH, concentration, and exposure time (Karwal & Mukovozov, 2023; Tang & Yang, 2018). In the present study, lactic acid — one of the most widely incorporated and best-characterized members of the AHA class (Babilas et al., 2012) — was selected as the representative chemical exfoliant; the term “alpha-hydroxy acid” is therefore retained in the title to reflect the formulation’s alignment with this established ingredient category, consistent with common usage in the cosmetic and regulatory literature (Karwal & Mukovozov, 2023). It moreover occupies a distinctive position among AHAs: unlike glycolic acid, which penetrates more deeply due to its smaller molecular size, lactic acid penetrates less aggressively, making it more

suitable for sensitive and dry skin types. Critically, lactic acid also functions as a humectant, drawing moisture from the environment into the stratum corneum and thereby providing dual chemical exfoliation and skin hydration in a single ingredient (Andersen, 1998; Karwal & Mukovozov, 2023). Additionally, lactic acid has been reported to elevate ceramide levels in the stratum corneum, enhance epidermal desquamation, disperse basal layer melanin, and stimulate collagen synthesis in the dermis (Wang, Lin, & Hong, 2024).

The physical exfoliating component of the present formulation is fine crystalline sucrose, selected to complement the chemical exfoliation provided by lactic acid through gentle mechanical action.

The cleansing and foaming system is based on a combination of sodium cocoyl isethionate (SCI), whose safety in rinse-off cosmetics has been affirmed by the CIR at concentrations up to 50% (Cosmetic Ingredient Review, 1993), and cocamidopropyl betaine (CAPB), for which a margin of safety greater than 100 has been reported at concentrations up to 30% (Burnett et al., 2012). Glycerin was incorporated as a humectant, consistent with its established role as one of the most extensively used humectants in cosmetic formulations (Becker et al., 2020).

Despite the growing commercial interest and widespread use of foaming exfoliating scrubs incorporating AHAs, the peer-reviewed literature contains a limited number of studies reporting comprehensive formulation development supported by systematic physicochemical characterization, regulatory-compliant microbiological safety testing, and scale-up validation across multiple production scales. Published studies on scrub formulations predominantly focus on basic parameters such as spreadability, pH, and sensory attributes, while neglecting critical aspects including preservative efficacy testing according to international standards and reproducibility under different manufacturing conditions. Scale-up of cosmetic formulations involves complex challenges related to heat transfer, mixing dynamics, and ingredient

homogeneity, all of which can significantly influence product quality and safety. However, there remains a limited number of studies documenting systematic scale-up validation from laboratory to semi-industrial production. In addition, recent developments in cosmetic science emphasize the importance of integrating formulation design with comprehensive analytical evaluation; nevertheless, such integrated approaches are still insufficiently applied in rinse-off exfoliating systems. In this context, the present study provides a valuable contribution to the field by combining: (i) the development of a dual-action exfoliating system integrating chemical (AHA) and physical exfoliation in a foaming surfactant-based matrix, (ii) comprehensive physicochemical, microbiological, and preservative efficacy evaluation in accordance with international standards (TS EN ISO 11930), and (iii) systematic scale-up validation from laboratory to semi-industrial production without formulation modification.

The aim of this study was to develop and systematically evaluate a foaming exfoliating face and body scrub formulation containing 5% lactic acid (AHA) as a chemical exfoliant and sucrose as a physical exfoliant, supported by a mild SCI / CAPB surfactant system, glycerin as a humectant, yellow iron oxide (CI 77492) as a colorant, and lemon peel essential oil (Citrus Limon Peel Oil) as an active fragrance ingredient. The physicochemical and microbiological properties of the formulation were evaluated across two production scales (100 mL and 10,000 mL). The study also documents the scale-up process from laboratory to semi-industrial manufacturing conditions, thereby contributing to the limited published data on scale-up validation of AHA-containing cosmetic scrub formulations.

MATERIALS AND METHODS

Preliminary formulation optimization

Prior to finalizing the formulation presented in this study, a series of preliminary trials was conducted at laboratory scale to identify an appropriate surfactant-to-exfoliant ratio and to evaluate the resulting sensory and rheological characteristics of the base. Four candidate formulations were prepared, varying the concentrations of sodium cocoyl isethionate (5–11%), cocamidopropyl betaine (5–9%), stearic acid (1–4%), and sucrose (20–35%), while keeping the lactic acid concentration fixed at 5%. Formulations containing lower surfactant and sucrose concentrations exhibited low viscosity and insufficient foaming, whereas formulations with higher surfactant and stearic acid concentrations resulted in excessively high viscosity and poor spreadability. The intermediate formulation containing 9% sodium cocoyl isethionate, 9% cocamidopropyl betaine, 3% stearic acid, and 30% sucrose was selected as the final composition, as it provided the optimum balance of consistency, foam generation, and exfoliating texture among the trialed formulations. This composition was subsequently used for all further scale-up and characterization studies described below.

Formulation development

A foaming exfoliating scrub formulation containing 5% lactic acid (AHA) was developed through a two-phase manufacturing process. The complete formulation is presented in Table 1. All ingredients were weighed using a Weightlab Instruments WL-303L precision balance (max 300 g, d = 0.001 g, Weightlab Instruments, China).

Table 1. Formulation of the foaming exfoliating face and body scrub (%5 AHA)

Phase	Function	Component/INCI Name	%
A	Solvent	Aqua	27
A	Surfactant	Sodium Cocoyl Isethionate	9
A	Surfactant / Foam Booster	Cocamidopropyl Betaine	9
A	Viscosity Enhancer	Stearic Acid	3
A	Humectant	Glycerin	10
A	Emollient / Carrier Oil	<i>Simmondsia Chinensis</i> Seed Oil	5
A	Chemical Exfoliant (AHA)	Lactic Acid	5
A	Physical Exfoliant	Sucrose	30
A	Colorant	Iron Oxides (CI 77492)	1
B	Preservative	Phenoxyethanol, Ethylhexylglycerin	0.5
B	Active / Fragrance	<i>Citrus Limon</i> Peel Oil	0.5

Phase A ingredients — distilled water, SCI, cocamidopropyl betaine, stearic acid, glycerin, jojoba oil, lactic acid (85%), fine crystalline sucrose, and yellow iron oxide (CI 77492) — were weighed and transferred into a suitable vessel. The mixture was placed in a Mipro Lab MCS shaking water bath and heated to 60 °C until a uniform, viscous consistency was achieved, with continuous mixing for approximately 10–15 minutes. Following removal from the water bath, the mixture was processed using a Wiggins D-500 homogenizer to ensure particle homogeneity and dispersion of yellow iron oxide throughout the base. The formulation was then allowed to cool to below 40 °C under intermittent overhead mechanical stirring. Phase B ingredients — the preservative system (K 705: phenoxyethanol and ethylhexylglycerin) and lemon peel essential oil (*Citrus Limon* Peel Oil) — were added at this stage and incorporated by gentle mechanical mixing. The pH of the final formulation was measured using a Thermo Scientific Orion Star A215 pH/conductivity meter (Thermo Fisher Scientific, USA) and adjusted to the target range of 3.5–4.0 using lactic acid solution as required.

Scale-up process

The formulation was developed progressively across three production scales to evaluate reproducibility and homogeneity. The same manufacturing procedure was applied at each scale with equipment appropriate to batch volume, as summarized in Table 2.

At laboratory scale (100 mL), heating was performed using the Mipro Lab MCS water bath and mixing was carried out with the Wiggins D-500 homogenizer and an overhead mechanical stirrer. At intermediate scale (1,000 mL), an Emsan planetary stand mixer was employed for mixing following the same heating procedure. At semi-industrial scale (10,000 mL), production was conducted in a jacketed stainless steel mixing tank (İstay Endüstriyel Paslanmaz, Turkey) equipped with an anchor impeller driven by an industrial motor. Temperature control was maintained via the tank's jacket system. Following production, the semi-industrial batch was filled into primary packaging using a manual piston-type stainless steel filling unit. Physicochemical and microbiological characterization was performed on samples taken from the 100 mL and 10,000 mL batches.

Table 2. Equipment used at each production scale

Scale	Volume	Weighing	Heating	Mixing	Filling
Laboratory scale	100 ml	Weightlab WL-303L	Mipro Lab MCS water bath (60 °C)	Wiggins D-500 homogenizer; overhead mechanical stirrer	-
Intermediate scale	1,000 ml	Weightlab WL-303L	Mipro Lab MCS water bath (60 °C)	Emsan planetary stand mixer	-
Semi-industrial scale	10,000 ml	Platform scale	Jacketed stainless steel mixing tank (İstay)	İstay industrial mixer with anchor impeller	Manual piston filling unit (stainless steel)

Physicochemical characterization

The organoleptic properties of each batch — including color, odor, and overall appearance — were evaluated visually and sensorially by trained observers under standardized laboratory lighting conditions. The pH of each batch was measured at room temperature (25 ± 1 °C) using the Thermo Scientific Orion Star A215 pH/conductivity meter, calibrated with standard buffer solutions prior to each measurement session. Measurements were performed in triplicate, and mean values were recorded. Viscosity was measured using a Brookfield-type rotational viscometer equipped with Spindle 4 at 50 rpm under constant temperature conditions (25 ± 1 °C). Measurements were performed in triplicate for each production scale.

Phase separation was assessed by centrifugation using a Nuve NF 1200R refrigerated centrifuge (Nuve, Turkey). Samples were centrifuged at 3,000 rpm for 30 minutes at 25 °C. Following centrifugation, samples were visually examined for phase separation, sedimentation, or any change in homogeneity.

Microbiological quality assessment

Microbiological quality testing was conducted in

accordance with the guidelines of the Turkish Medicines and Medical Devices Agency (TİTCK) for cosmetic products (Turkish Medicines and Medical Devices Agency, 2016). Analyses were performed exclusively on the semi-industrial scale batch (10,000 mL), as this represents the final production scale intended for commercial application. The total count of aerobic mesophilic microorganisms was determined according to TSE EN ISO 21149:2017. Detection of specified microorganisms — *Staphylococcus aureus* (ATCC 6538), *Pseudomonas aeruginosa* (ATCC 9027), *Escherichia coli* (ATCC 8739), *Candida albicans* (ATCC 10231), and moulds/yeasts — was performed using the corresponding TSE EN ISO reference standards (Orth, Kabara, Denyer, & Tan, 2006). All microbial reference strains were initially cultivated in Brain Heart Infusion Broth for 18–24 hours at the appropriate incubation temperature. Bacterial suspensions were turbidimetrically standardized to 0.5 McFarland (equivalent to approximately 1×10^8 CFU/mL) and subsequently diluted in sterile physiological saline to a working concentration of 5×10^5 CFU/mL prior to use. The culture media and methods employed for each microorganism are detailed in Table 3.

Table 3. Microbiological methods applied for cosmetic quality assessment

Analysis	Method	Medium
<i>Staphylococcus aureus</i> ATCC 6538	Enrichment & Inoculation of Culture Media	Baird Parker Agar Medium
<i>Pseudomonas aeruginosa</i> ATCC 9027	Enrichment & Inoculation of Culture Media	Cetrimide Agar Medium
<i>Escherichia coli</i> ATCC 8739	Enrichment & Inoculation of Culture Media	MacConkey Agar Medium + Levine Eosin - Methylene Blue Agar Medium
<i>Candida albicans</i> ATCC 10231	Enrichment & Inoculation of Culture Media	Sabouraud 4% Dextrose Agar + Supplement
Moulds - Yeast	Pour Plate or the Spread Plate Technique	Sabouraud 4% Dextrose Agar + Supplement
Total Aerobic Mesophilic Microorganisms	Pour Plate or the Spread Plate Technique	Tryptic soy Agar with Polysorbate 80 and Lecithin

Antimicrobial efficacy (challenge test)

The efficacy of the preservative system was evaluated by challenge testing (preservative efficacy testing) in accordance with TS EN ISO 11930:2019. Challenge testing was conducted on the semi-industrial scale batch (10,000 mL). Results were interpreted against the acceptance criteria defined in European Pharmacopoeia 5.1.3 (Effectiveness of Antimicrobial Preservation) (Russell, 2003). Five standard microorganisms were used as challenge strains: *Staphylococcus aureus* (ATCC 6538), *Pseudomonas aeruginosa*

(ATCC 9027), *Escherichia coli* (ATCC 8739), *Candida albicans* (ATCC 10231), and *Aspergillus brasiliensis* (NCPF 2275). Each formulation sample was inoculated with a standardized microbial suspension and stored under controlled conditions. Viable microbial counts were determined on days 7, 14, and 28 post-inoculation, and logarithmic reductions relative to the initial inoculum were calculated for each strain. All tests were performed in triplicate. The culture media and incubation conditions for each organism are presented in Table 4.

Table 4. Challenge test conditions for antimicrobial efficacy assessment

Analysis	Method	Culture Media	Incubation Condition
<i>Staphylococcus aureus</i> ATCC 6538	Pour Plate	Tryptic Soy Agar	30 °C - 35 °C
<i>Pseudomonas aeruginosa</i> ATCC 9027	Pour Plate	Tryptic Soy Agar	30 °C - 35 °C
<i>Escherichia coli</i> ATCC 8739	Pour Plate	Tryptic Soy Agar	30 °C - 35 °C
<i>Candida albicans</i> ATCC 10231	Pour Plate	Sabouraud 4% Dextrose Agar	30 °C - 35 °C
<i>Aspergillus brasiliensis</i> NCPF 2275	Pour Plate	Potato Dextrose Agar	30 °C - 35 °C

Stability analysis

The physicochemical stability of the formulation was evaluated under accelerated and long-term stress conditions to predict shelf-life performance. Stability analyses were performed on the semi-industrial scale batch (10,000 mL). It was conducted using a programmable stability cabinet (JSDS-300C, South Korea) ca-

pable of precise temperature control throughout all storage conditions.

Samples were subjected to the following storage protocols: (i) freeze-thaw stress, in which samples were held at -24 °C for 24 hours followed by thawing and evaluation; (ii) cold storage at -5 °C for one week; (iii) room temperature storage at 25 °C/60% RH

in the dark for four months, with evaluations performed weekly during the first month and monthly thereafter; and (iv) elevated temperature storage at 45 °C/75% RH for three months, with assessments at day 3, weekly during the first month, and at months 2 and 3. Relative humidity was not controlled or monitored for the freeze-thaw and cold storage conditions, as humidity is not considered a critical parameter at sub-zero temperatures. Cyclic temperature stress was applied over six weeks by alternating 24-hour periods at –5 °C and 25 °C per cycle (ICH, 1995; ICH, 2003).

At each evaluation time point, color, odor, and overall appearance were recorded by trained observers, and pH was measured using the Thermo Scientific Orion Star A215 pH meter as described above.

RESULTS AND DISCUSSION

Formulation development

The foaming exfoliating scrub formulation was developed by combining a mild surfactant system with dual-action exfoliating agents and supporting functional ingredients. SCI and cocamidopropyl betaine were selected as the primary surfactant system due to their well-documented skin mildness and foaming capacity, which are particularly important in a rinse-off product intended for both facial and body use (Ghosh, Kim, So, & Bhav, 2007; Kim et al., 2024). Lactic acid was incorporated at 5% (w/w) as the chemical exfoliant, a concentration within the safety limits established by both the CIR Expert Panel and the FDA for consumer cosmetic products (Andersen, 1998; FDA, 2002). Fine crystalline sucrose was included at 30% as the physical exfoliant, providing mechanical abrasion to complement the chemical exfoliation of lactic acid (Cosmetic Ingredient Review, 2012). Glycerin at 10% was incorporated to support skin hydration during the cleansing process (Chen et al., 2022). Jojoba oil was added as an emollient carrier to improve skin feel and reduce potential irritation associated with the acidic pH environment. Yellow iron oxide (CI 77492) was dispersed in glycerin prior to incorporation to ensure uniform colorant distribution

throughout the formulation. The preservative system K 705 (phenoxyethanol and ethylhexylglycerin) and lemon peel essential oil (Citrus Limon Peel Oil) were added in Phase B at below 40 °C to prevent thermal degradation of volatile and heat-sensitive components.



Figure 1. Photograph of the developed foaming exfoliating face and body scrub formulation (%5 AHA)

The target pH of 3.5–4.0 was established based on two primary considerations: the requirement for lactic acid to remain predominantly in its undissociated free acid form to exert effective exfoliating activity, and compliance with the minimum pH threshold of 3.5 stipulated by the CIR Expert Panel for AHA-containing consumer cosmetic products (Andersen, 1998; FDA, 2002; Karwal & Mukovozov, 2023). The final formulation presented a homogeneous, yellow-toned, foaming scrub texture with a characteristic lemon fragrance (Figure 1.).

Scale-up process

The formulation was successfully scaled up from laboratory scale (100 mL) through intermediate scale (1,000 mL) to semi-industrial scale (10,000 mL) without any modification to the original formulation composition or manufacturing procedure. At each production scale, the formulation maintained its characteristic homogeneous appearance, uniform

colorant distribution, and consistent foaming properties, confirming the robustness of the developed process (Table 2.).

At laboratory scale, heating was performed in the Mipro Lab MCS water bath at 60 °C, and mixing was carried out using the Wiggins D-500 homogenizer combined with an overhead mechanical stirrer, which provided effective dispersion of ingredients including yellow iron oxide. At intermediate scale, the Emsan planetary stand mixer enabled adequate mixing of the increased batch volume while preserving formulation homogeneity. At semi-industrial scale, the jacketed stainless steel mixing tank (İstay Endüstriyel Paslanmaz, Turkey) provided uniform heat distribution and sufficient shear force through its anchor impeller system, ensuring product quality comparable to smaller-scale batches. The final semi-industrial batch

was filled into primary packaging using a manual piston-type stainless steel filling unit. Representative photographs of the formulation at each production scale are presented in Figure 2.

The absence of phase separation, color inconsistency, or textural changes across all three production scales demonstrates that the key process parameters — particularly heating temperature, ingredient addition sequence, and mixing conditions — were appropriate for scale-up without requiring process optimization. These findings are consistent with established principles of cosmetic scale-up, whereby maintaining critical process parameters across increasing batch volumes is essential to ensure batch-to-batch consistency and product quality (Durgun & Algin Yapar, 2021).



Figure 2. Photographs of the foaming exfoliating scrub formulation at (A) laboratory scale (100 mL), (B) intermediate scale (1,000 mL), and (C) semi-industrial scale (10,000 mL)

Physicochemical characterization

The physicochemical properties of the formulation were evaluated at all three production scales — laboratory (100 mL), intermediate (1,000 mL), and

semi-industrial (10,000 mL) — to confirm consistency throughout the scale-up process. The results are summarized in Table 5.

Table 5. Physicochemical characterization of the foaming exfoliating scrub at each production scale

Scale	Color	Odor	Appearance	pH $\pm$ SD	Viscosity (cP) $\pm$ SD	Centrifuge
Laboratory scale (100 ml)	Light yellow	Characteristic lemon scent	Homogeneous, semi-solid paste with visible sucrose granules	3.885 $\pm$ 0.036	138,000 $\pm$ 3,900	No phase separation
Intermediate scale (1,000 ml)	Light yellow	Characteristic lemon scent	Homogeneous, semi-solid paste with visible sucrose granules	3.997 $\pm$ 0.026	144,000 $\pm$ 4,200	No phase separation
Semi-industrial scale (10,000 ml)	Light yellow	Characteristic lemon scent	Homogeneous, semi-solid paste with visible sucrose granules	3.871 $\pm$ 0.033	145,000 $\pm$ 4,400	No phase separation

Organoleptic evaluation revealed that all three batches presented a uniform light yellow color, a characteristic lemon scent, and a homogeneous semi-solid paste appearance with visible sucrose granules. No differences in color intensity, odor strength, or textural consistency were observed between scales, indicating that the yellow iron oxide colorant was uniformly dispersed and that the volatile fragrance component was retained without thermal degradation across all production conditions.

The pH values measured at laboratory, intermediate, and semi-industrial scales were 3.885 ± 0.036 , 3.997 ± 0.026 , and 3.871 ± 0.033 , respectively, all within the target range of 3.5–4.0. This range ensures that lactic acid remains predominantly in its undissociated free acid form, thereby maintaining its exfoliating efficacy, while complying with the minimum pH threshold of 3.5 established by the CIR Expert Panel for AHA-containing consumer cosmetic products (Andersen, 1998; FDA, 2002). The consistency of pH values across scales confirms that the acid-base equilibrium of the formulation was not affected by changes in batch volume or equipment geometry. Viscosity values increased slightly with batch volume ($138,000 \pm 3,900$, $144,000 \pm 4,200$, and $145,000 \pm 4,400$ cP at laboratory, intermediate, and semi-industrial scales, respectively), which may be attributed to minor differences in shear input and cooling rate between mixing systems; nonetheless, all batches exhibited a comparably thick, semi-solid consistency, confirming that the rheological properties were not adversely affected by scale-up.

Phase separation assessment by centrifugation at 3,000 rpm for 30 minutes yielded no evidence of phase separation, sedimentation, or homogeneity loss in any of the tested batches. This outcome demonstrates the physical stability of the surfactant-based formulation system and confirms that the SCI/CAPB surfactant combination provided adequate structural integrity to resist centrifugal stress at both laboratory and semi-industrial scales (Ghosh et al., 2007; Kim et al., 2024).

Microbiological quality assessment

The microbiological quality of the semi-industrial scale batch (10,000 mL) was evaluated in accordance with TITCK guidelines for cosmetic products. The results of triplicate analyses are presented in Table 6.

All microbiological parameters were within the specified tolerance limits. The total aerobic mesophilic microorganism count and total yeast and mould count were both below 10 CFU/g across all three samples, well within the acceptable limit of less than 10^2 CFU/g. None of the specified pathogenic microorganisms — *Staphylococcus aureus*, *Pseudomonas aeruginosa*, *Escherichia coli*, and *Candida albicans* — were detected in any of the tested samples, satisfying the absence criterion required for cosmetic products. These results confirm that the formulation was produced under appropriate hygienic conditions and that the preservative system (K 705: phenoxyethanol and ethylhexylglycerin) effectively controls microbial contamination at the semi-industrial production scale (Orth et al., 2006).

Table 6. Microbiological analysis results of the foaming exfoliating scrub

Parameter	Unit	Microbiological Analysis Results			Tolerances
		Sample 1	Sample 2	Sample 3	
Total Aerobic Mesophilic Microorganisms	CFU/g	< 10	< 10	< 10	< 10 ²
<i>Staphylococcus aureus</i>	/1g-mL	Absence	Absence	Absence	Absence/1g-mL
<i>Pseudomonas aeruginosa</i>	/1g-mL	Absence	Absence	Absence	Absence/1g-mL
<i>Escherichia coli</i>	/1g-mL	Absence	Absence	Absence	Absence/1g-mL
<i>Candida albicans</i>	/1g-mL	Absence	Absence	Absence	Absence/1g-mL
Total Yeast and Moulds	CFU/g	< 10	< 10	< 10	< 10 ²

*CFU: Colony forming unit

Antimicrobial efficacy (challenge test)

The preservative efficacy of the formulation was assessed by challenge testing in accordance with TS EN ISO 11930:2019, with results interpreted against the criteria of European Pharmacopoeia 5.1.3. The microbial counts recorded at days 0, 7, 14, and 28 post-inoculation are presented in Table 7. In Table 7,

N denotes the initial concentration of the standardized microbial suspension prior to inoculation, N₀ represents the viable count immediately after inoculation into the formulation (time zero), and N₇, N₁₄, and N₂₈ denote the viable counts recorded at days 7, 14, and 28 post-inoculation, respectively.

Table 7. Antimicrobial efficacy (challenge test) results of the foaming exfoliating scrub

Microorganism	Day 0		Day 7	Day 14	Day 28
	N	N ₀	N ₇	N ₁₄	N ₂₈
	(cfu/g)	(cfu/g)	(cfu/g)	(cfu/g)	(cfu/g)
<i>Staphylococcus aureus</i> ATCC 6538	2.2 x 10 ⁸	8.5 x 10 ⁵	< 10	< 10	< 10
<i>Pseudomonas aeruginosa</i> ATCC 9027	3.5 x 10 ⁸	1.7 x 10 ⁴	< 10	< 10	< 10
<i>Escherichia coli</i> ATCC 8739	1.8 x 10 ⁸	5.2 x 10 ⁵	< 10	< 10	< 10
<i>Candida albicans</i> ATCC 10231	2.2 x 10 ⁶	3.7 x 10 ³	< 10	< 10	< 10
<i>Aspergillus brasiliensis</i> NCPF 2275	2.5 x 10 ⁶	5.3 x 10 ³	< 10	< 10	< 10

All five challenge microorganisms demonstrated a progressive and substantial reduction in viable counts following inoculation. For the bacterial strains, initial inocula ranged from 1.8×10^8 to 3.5×10^8 CFU/g, which were reduced to below 10 CFU/g by day 7 and remained below the detection limit through day 28. For the fungal strains, *Candida albicans* and *Aspergillus brasiliensis* showed initial counts of 2.2×10^6 and 2.5×10^6 CFU/g, respectively, both reduced to below 10 CFU/g by day 7 and maintained at this level

through day 28. These logarithmic reductions are consistent with Category A criteria as defined in European Pharmacopoeia 5.1.3, demonstrating that the preservative system provided effective antimicrobial protection against all tested microorganisms within the required timeframe (Orth et al., 2006; Russell, 2003). Counts at days 7, 14, and 28 are reported as below the limit of quantification (<10 CFU/g) rather than as exact values, since viable colonies fell below the detectable threshold of the enumeration method

at these time points; this is consistent with the Category A requirement of European Pharmacopoeia 5.1.3 for reduction below the detection limit within the specified timeframe.

These findings confirm that the K 705 preservative system — comprising phenoxyethanol and ethylhexylglycerin — maintains its efficacy at semi-industrial production scale, supporting the microbiological safety of the final formulation for consumer use.

Stability analysis

The physicochemical stability of the semi-industrial scale batch was monitored over a three-month period under various stress conditions. The results of the stability evaluation are presented in Table 8.

The color, odor, and overall appearance of the formulation remained unchanged throughout the entire observation period, with all batches retaining their characteristic light yellow color and lemon scent at each evaluation time point. No alterations in packaging integrity or phase separation were observed under

any of the applied storage conditions, confirming the physical robustness of the formulation.

The pH values recorded at weeks 1 and months 1, 2, and 3 were 3.81, 3.77, 3.89, and 3.80, respectively, all remaining within the target range of 3.5–4.0 throughout the study period. The minor fluctuations observed between time points are within acceptable analytical variation and do not indicate any systematic drift or chemical instability of the formulation. The maintenance of pH within the target range is particularly significant, as it ensures the continued exfoliating efficacy of lactic acid throughout the intended shelf life of the product (Andersen, 1998; Karwal & Mukovozov, 2023).

No microbiological growth was detected at any evaluation time point, confirming that the preservative system remained effective under all tested storage conditions and that the formulation maintained its microbiological safety throughout the stability study period (Orth et al., 2006; Russell, 2003).

Table 8. Physicochemical and microbiological stability results of the foaming exfoliating scrub formulation under various storage conditions

Parameters	1 st Week	1 st Month	2 nd Month	3 rd Month
	09.06.2025	02.07.2025	02.08.2025	02.09.2025
Color	Light yellow	Light yellow	Light yellow	Light yellow
Odor	Lemon scent	Lemon scent	Lemon scent	Lemon scent
Appearance	No change	No change	No change	No change
pH	3.81	3.77	3.89	3.80
Package (glass jar)	No change	No change	No change	No change
Phase Separation	No change	No change	No change	No change
Microbiological growth	No	No	No	No

CONCLUSION

In this study, a novel foaming exfoliating face and body scrub formulation containing 5% lactic acid as a chemical exfoliant and 30% sucrose as a physical exfoliant was successfully developed and characterized. The formulation was designed to combine dual-action exfoliation with mild cleansing properties through

the use of a sodium cocoyl isethionate and cocamidopropyl betaine surfactant system, while incorporating glycerin as a humectant, yellow iron oxide (CI 77492) as a colorant, and lemon peel essential oil as an active fragrance ingredient.

The formulation was successfully scaled up from laboratory scale (100 mL) to semi-industrial scale

(10,000 mL) without any modification to the original composition or manufacturing procedure. Physicochemical characterization demonstrated that all three production scales yielded a homogeneous, light yellow, semi-solid paste with consistent organoleptic properties. pH values at all scales remained within the target range of 3.5–4.0, ensuring the exfoliating efficacy of lactic acid and compliance with CIR and FDA safety guidelines. No phase separation was observed following centrifugal stress testing at any scale.

Microbiological quality assessment confirmed that the semi-industrial batch met TITCK cosmetic product safety requirements, with total aerobic mesophilic microorganism and yeast/mould counts below 10 CFU/g, and complete absence of all specified pathogenic microorganisms. Challenge testing in accordance with TS EN ISO 11930:2019 demonstrated that the K 705 preservative system provided effective antimicrobial protection against all five challenge organisms, with microbial counts reduced to below the detection limit by day 7 and maintained through day 28, consistent with Category A criteria of European Pharmacopoeia 5.1.3.

Stability evaluation over three months confirmed that the formulation maintained its physicochemical and microbiological characteristics under all tested storage conditions, with pH values remaining stable within the target range and no microbiological growth detected at any time point.

This study has certain limitations. Particle size distribution and polydispersity index of the dispersed solid components, detailed rheological characterization (e.g., flow curves, thixotropic behavior), and instrumental texture analysis (adhesiveness, spreadability) were not investigated in the present work; these parameters would provide valuable additional insight into the formulation's physical behavior and are recommended for future studies.

Overall, these findings demonstrate that the developed foaming AHA scrub formulation is physicochemically stable, microbiologically safe, and suc-

cessfully scalable to semi-industrial manufacturing conditions, supporting its potential for commercial cosmetic product development.

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AUTHOR CONTRIBUTION STATEMENT

S.D.K.: Developing hypothesis, experimenting, preparing the study text, reviewing the text, analysis and interpretation of the data, literature research.

CONFLICT OF INTEREST

The author declares that there is no conflict of interest.

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