

Application of *Citrus maxima* Burm. Essential Oil in Shower Gel Products

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Nowadays, the trend of cosmetics containing natural ingredients is becoming one of the most popular trends in the beauty industries. More and more people are interested and looking for cosmetic products made from natural ingredients such as herbs, essential oils, fruits, vegetables, etc. Essential oils are widely incorporated in cosmetic, perfumery and related household products due to their diverse properties but mainly due to their pleasant aroma and biological activity. *Citrus maxima* essential oil contains D-limonene as the main compound (85-95%) and other compounds such as α -pinene, sabinene, β -myrcene, α -phellandrene, σ -cymene, β -ocimene, γ -terpinene, β -caryophyllen, β -copaene. These compounds further diversify the applications of the added product. In this study, the factors affecting the production process of *Citrus maxima* essential oil shower gel including main detergents, moisturizers, emulsifiers, preservatives were investigated to propose production formulas. The results showed that the main detergent accounted for 12%, co-detergent 2.0%, emulsifier 1.5%, foaming agent 7.0%, surfactant, emulsifier 3.0%, moisturizer 2.0%, preservative management 2.0% and *Citrus maxima* essential oil account 1.0% in the formula. The evaluations of the basic physico-chemical properties are in the permissible range. Thus, it shows that the shower gel products from *Citrus maxima* essential oil can be produced and marketed.

Keywords: *Citrus maxima*, Detergent, Essential oil, Shower gel.

INTRODUCTION

In recent years, with the conditions of society increasingly developed, the use of cleaning and personal care products such as shampoo, shower gel, soap, etc. as a daily necessity for the human activities [1,2]. It is obvious that customers engage in discussions with one another over the optimal selection for the product they intend to purchase in retail establishments such as stores and supermarkets. The method found is to open the product and smell the scent. Scent is the most important factor that directly affects the choice to buy or not to buy a product [3]. Therefore, in addition to the ingredients that make up the product, the scent also determines the quality and price of the product. Through many studies, it has been shown that essential oils are considered as a natural scent and the great benefits that it brings due to its own biological activities [4-6]. This contributes to increasing the preservation of the product as well as the use of the product.

Shower gel is a product in liquid form, with the basic purpose of cleaning the skin on the human body [7]. Shower gel is produced from synthetic substances derived from plants has a lower pH value than products made from the saponification process, which has a high pH value, in the range of pH 8-9 [8,9]. Therefore, shower gel is less drying to the skin than soap [10]. In the formula to produce shower gel, surfactants make up the majority, the rest is made up of ingredients to smooth, thicken, preserve, emulsify, anti-allergic, water, add fragrance and colour to product variety [11]. A product in general to survive in the market needs to meet the requirements of consumer tastes to some extent. Shower gel is no exception and must have a clear origin of synthetic substances and scents. This will definitely be the first condition required. The second factor is the ability to effectively clean and deodorize, besides providing the necessary moisture and nutrients to the skin. The equally important factor as mentioned above is that the

shower gel has a pleasant scent, is benign, natural-friendly and safe. Currently, many people tend to choose natural products to use. Therefore, shower gels containing essential oils extracted from orange, grapefruit, bitter melon, green tea, *etc.* become attractive.

Citrus maxima Burm. essential oil (*C. maxima*), which is extracted from the peels through hydrodistillation [12]. The composition of *C. maxima* essential oil can vary depending on the geographical location, climate and extraction process. However, the main components of *C. maxima* essential oil include D-limonene, accounting for about 80%. Limonene is a cyclic terpene that gives the oil having characteristic citrus aroma. It also contain other compounds such as α -pinene, myrcene, linalool, β -pinene, β -myrcene, γ -terpinene and geraniol, *etc.* [12-14]. The combination of these components gives *C. maxima* essential oil unique aroma and health benefits. The introduction of essential oils and their individual ingredients into the production of a wide range of cosmetic products has been encouraged by their well-recognized effects on health and beauty to the formula. Essential oils can affect the normal function of skin cells when applied topically, in anti-acne, anti-aging, skin lightening and sun protection formulations [15,16]. Furthermore, it can incorporate antibacterial, antifungal, anti-inflammatory and antioxidant properties into formulations that can benefit the skin [17,18]. Therefore, considering the cosmetic industry's interest in replacing traditional active ingredients with bioactive ingredients, more systematic trials need to be carried out to assess true efficacy of essential oils in the final formulations.

In this study, factors affecting the production process of shower gel and taking advantage of the source of accessories and essential oils extracted from *C. maxima* peel to create a scent for the production of gel shower products, through evaluation factors such as foaming level and time of emulsion.

EXPERIMENTAL

Citrus maxima essential oil was purchased by Evodia Vietnam Co., Ltd., Ho Chi Minh City, Vietnam. Other chemicals included EDTA, glycerin, PEG-7 glycerin cocoate, sodium lactate, sodium methyl cocoyl taurate, cocamidopropyl betaine (CAPB), citric acid, cocodiethanolamide (CDE), phenoxyethanol ethyl-hexylglycerin (EHGP), cocamide monoethanolamine (CMEA) dimethylol dimethyl hydantoin (DMDMH), disodium cocoamphodiacetate, plantacare 1200, D-panthenol and NH_4Cl were purchased by Nguyen Ba Trading and Production Co., Ltd. Ho Chi Minh City, Vietnam.

Instrumentation: The pH values were measured using a benchtop pH meter (HI2210-02, Hanna Instruments) and the

The viscosity measurements were conducted using a viscometer (model: DV1M model, Brookfield, USA).

Preparation of base formula for shower gel products:

The details of the ingredients used in the shower gel products at different concentrations and the other selected added contents are given in Table-1. Sodium methyl cocoyl taurate is the main detergent in the basic shower gel formula and additives need to be added such as emollients (glycerin, sodium lactate, *etc.*), foaming agents (CAPB, *etc.*), emulsifiers (PEG 120, *etc.*) were The ingredients were combined in accordance with the analysis of the impact of content within the formulation. Then, *C. maxima* essential oil was added to increase the aesthetics of the product. Finished products were then analyzed for viscosity, pH value, foaming level, *etc.* and user perception.

Analytical methods

Foaming level: In order to measure the foaming, this study employs the shaking test. After being diluted 1100 times, 5 mL of solution was poured in a sealed tube and shaken until the maximum amount of foam is produced (foam volume does not fluctuate). The level of foaming can be calculated according to eqn. 1:

$$\epsilon_f = \frac{V_{\text{foam}} - V_{\text{liquid}}}{V_{\text{foam}}} \quad (1)$$

where ϵ_f : foaming level, V_{foam} : foam column volume; V_{liquid} : initial volume of solution.

Dry matter content: Calculated by formula (2)

$$\text{Dry matter content (\%)} = 100\% - \text{Moisture} \quad (2)$$

Emulsion time: The cleaning effect is expressed in terms of emulsion time, *i.e.* the ability of the product to emulsify with a selected paraffin oil (simulated for dirt). A volume of 2 mL of diluent was added to 2 g of paraffin oil and then shaken to form an emulsion. The emulsion stability time of the system can be calculated by using a stopwatch and observing the point at which 1 mL of oil separates into discrete layers.

Assessing the appearance of products: The methods of evaluating the appearance of products are presented in Table-2.

Sensory evaluation method: Survey of 20 people (10 men + 10 women), evaluated factors include:

- (i) Before use (homogeneity, colour, thickness, sample ability, odor, fragrant, turbidity)
- (ii) When to use (foaming ability, skin irritation (itching), fragrant, cleaning ability (fast or slow when washed with water)
- (iii) After use (save incense, dry skin, hissing degree, skin irritation (itching, redness), cleanliness, skin smoothness).

The evaluation factors were expressed on a scale according to the method of taste perception. In which, the highest score

TABLE-1
FORMULA OF SHOWER GEL WAS SURVEYED

Composition	Content (%)	Composition	Content (%)	Composition	Content (%)
Cocamide monoethanolamine (CMEA)	1.0	Sodium methyl cocoyl taurate	8.0-16.0	Essential oil	1.0
EDTA	0.1	Disodium cocoamphodiacetate	0.5-2.5	EHGP	0.1
Hot water	Remaining	Plantacare 1200	1.0-5.0	DMDMH	0.4
Glycerin	1.0-4.0	D-Panthenol	0.3	CAPB	1.0-9.0
PEG-7 glycerin cocoate	0.5-2.5	CDE	1.5	NH_4Cl 25%	0.5
Sodium lactate	0.5-2.5	Citric acid 30%	0.5		

TABLE-2
VISUAL CRITERIA ACCORDING TO
VIETNAMESE STANDARDS TCVN 6971:2001

Target	Requirement
Status	Viscous, homogeneous, non-layered liquid
Colour	Uniform and according to the registration form
Fragrant	Odorless or pleasant smell

is 5 points for the most favourite product and the scale of decreasing from favourite, like, good, good, average and lowest is dislike with a scale of 0.

RESULTS AND DISCUSSION

Influence of parameters in the production process of shower gel products: In this study, the evaluation parameters like pH value, foaming level, emulsion stability time, viscosity, colour, aroma, appearance, sensory, moisture and dry matter contents of 7 samples were evaluated. The results show that the pH value is in the range of pH 5-6, the foaming level is in the range from 0.69 to 0.904, the foam stability time is in the range of 32-38.5 min with a viscosity of about 3.3-25.44 Pa.s, most products have transparent form, high and low consistency depending on the product, all have scents from synthetic fragrances, various colours according to the characteristics of each product. This is the basic condition to conduct to evaluate the influence of the parameters in the production process of shower gel products.

Effect of main detergent content: Sodium methyl cocoyl taurate (SMCT) is a sulfate-free surfactant, a sodium salt of the coconut fatty acid amide *N*-methyltaurine, is a mild foaming agent, commonly used in shampoos, shower gels and cleaning products [19]. Iwata & Shimada [20] described the cosmetic formulations and provide samples of authentic formulations, and the SMCT content of shampoo and shower gel products ranges from 6.0 to 18.0%. Therefore, this study conducted a survey on the concentration in the range of 8.0-16.0% as show in Fig. 1a. The results showed that as the SMCT content developed, so did the foaming strength and emulsion stability time; at 8% concentration, the lowest foaming level reached 0.677 ± 0.020 and the shortest emulsion stability time was 30 min.

When increasing the content to 12-16%, the foaming level increased insignificantly, at 12% content reached 0.800 ± 0.020 , 14% content reached 0.810 ± 0.010 , 16% content reached 0.818 ± 0.010 . Therefore, to optimize the production costs, 12% content is considered as optimal choice.

Effect of copper detergent content: Disodium cocoamphodiacetate is a surfactant based on fatty acids derived from coconut oil. They are a soft, mild cleanser with an amphoteric structure. Disodium cocoamphodiacetate is also capable of foaming by increasing the surface viscosity of liquid surrounding the individual bubbles in the foam. They help clean skin/hair by allowing water to mix with oil and dirt particles, thereby allowing water to wash them away easily. This ingredient is appreciated for cleansing the skin/hair without stripping the natural oils [21,22]. In this study, the content of disodium cocoamphodiacetate was investigated from 0.5 to 2.5%. The results show that at the content of 2.5% foaming and the emulsion stability time reached the highest values, respectively 0.850 ± 0.020 and 34 min, 0.5% content for these values was the lowest, accounting for 0.500 ± 0.010 , 26 min (Fig. 1b). However, at 2.0% foaming and emulsion stability time reached quite high values, there was no difference, accounting for 0.810 ± 0.020 compared to 0.850 ± 0.020 and 33 min compared to 34 min. Therefore, this work chooses the content of 2.0% to be fixed for the next parameters.

Effect of emulsifier content: PEG-7 glyceryl cocoate is a synthetic polymer of glyceryl cocoate, a monomer of glycerin and coconut oil fatty acids, both of which are highly beneficial for the skin. PEG-7 has a wide range of uses including emulsifying, providing moisture to the skin/hair, besides PEG-7 also has the ability to thicken, increase skin surface wetting and increase foam [21-24]. The safety assessment of pegylated alkyl glycerides used in cosmetics by Fiume *et al.* [25] showed that the highest concentration used in skin cleansing products was 10%. In this study, the PEG-7 content was investigated from 0.5 to 2.5%. As the content increases, the foaming level of the product increases, but when it comes to the content of 1.5%, this value does not increase significantly to 0.810 ± 0.020 and the emulsion stability time maximum is 34 min. This value

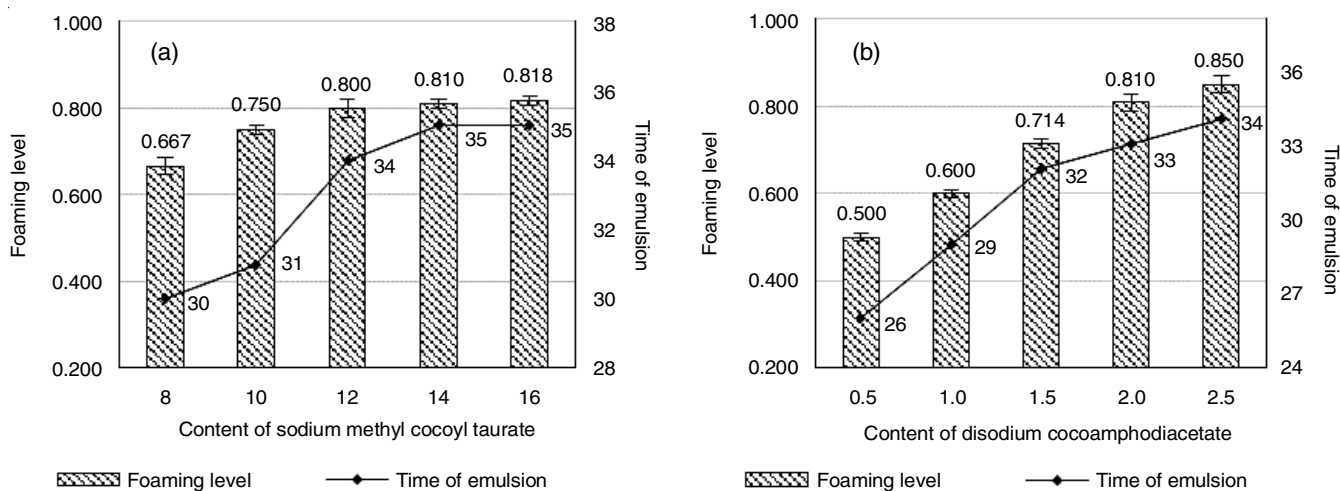


Fig. 1. Effect of main (a) and copper detergent (b) content

is higher than 0.5 and 1.0% content (0.714 ± 0.020 and 0.733 ± 0.010) (Fig. 2a). Therefore, PEG-7 glyceryl cocoate content of 1.5% was selected for further experiments.

Effect of foaming agent: As a surfactant derived from coconut oil, cocamidopropyl betaine (CAPB) is typically sold as 25-37% w/w aqueous solution [26]. The use of CAPB can produce better and longer-lasting foam and potentially improve viscosity upon dilution. Besides, the use of CAPB makes the soap thicker and improves the foaming ability. The optimal CAPB concentration for shampoos as stated by Iwata & Shimada [20], is somewhere between 8% and 12%. In this study, the CAPB content was investigated from 1.0 to 9.0%. The results show that the higher the content, the more foaming and the longer the emulsion time. At 9.0%, the highest foaming strength and emulsifying time are 0.833 ± 0.020 and 38 min, respectively, but these values increased insignificantly when compared to the content of 7.0% (0.826 and 37 min) (Fig. 2b). To ensure the economic value of production costs, the selected content is 7.0%.

Effect of moisture content: Glycerine is probably one of the most commonly used ingredients in all the personal care products, after water. It is used as a humectant (attracts water

to the skin) in everything from skin creams to shampoos and toothpastes [27-29]. According to Ngan *et al.* [9], glycerine level in shampoo formulas ranged from 1.0 to 5.0%, whereas it is about 2% in facial cleanser formulas. In this study, the glycerine content was also investigated from 1.0 to 5.0%. Increasing the glycerine concentration has a significant impact on the foaming, with the maximum levels observed at 3% glycerine. However, even at 2% glycerine, the foaming and emulsifying times are still rather high (0.818 ± 0.010 ; 36 min) (Fig. 3a). Therefore, the amount of glycerine used in this formula is optimized at 2%.

Effect of surfactant content: Plantacare 1200 UP is a non-ionic surfactant made from plant-based, renewable and sustainable certified RSPO-MB. They have the most skin friendly profile, creating the perfect synergy of gentleness, foaming and effective cleansing. Due to its invaluable mildness, this surfactant is also perfectly suited for sensitive skin and baby cleansing ideas. It is a gentle and effective alternative to PEG/sulfate formulations and is therefore used in this formulation. In this work, plantacare 1200 UP was evaluated for foaming and emulsion time through the content from 1.0 to 5.0%. The results revealed that the foaming strength is unaffected by the

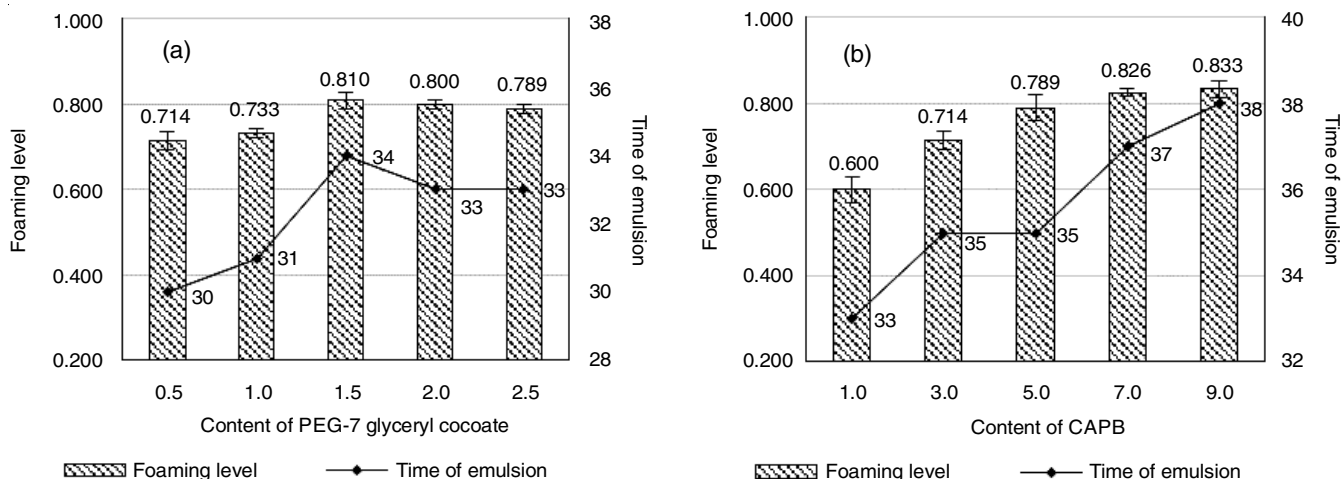


Fig. 2. Effect of emulsifier (a) and foaming agent (b) content

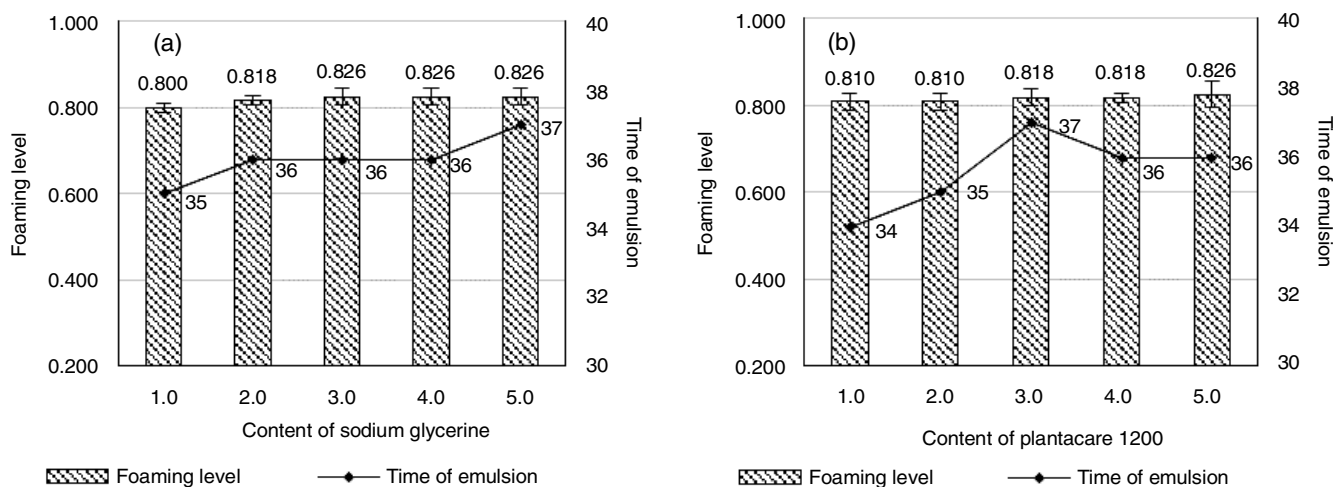


Fig. 3. Effect of moisture (a) and surfactant (b) content

content adjustment but that the emulsion stability time is dramatically affected. Foaming rates of 0.818 ± 0.020 were achieved at 3.0% and 4.0% concentrations, but 3% will save more than 4% of production materials and its emulsion time value is higher, accounting for 37 min. Consequently, the 3% content was chosen for the subsequent investigations.

Effect of preservative content: Sodium lactate is a water-soluble additive that has been successfully used to inhibit a number of microorganisms, such as *Salmonella*, *Pseudomonas* spp. species or lactic acid bacteria. According to Ricci *et al.* [30], at the concentration of 2.5%, sodium lactate exhibited inhibitory effects on some bacteria, including *Salmonella* spp., *L. monocytogenes* and *B. cereus*, during their pathogenic activities. To maximize the efficacy of citronella essential oil, 2% content was added into the mixture of preserving shampoo and shower gel products [31]. In this work, the results showed that the foaming level increased when the content increased from 0.5 to 2.5%, when the optimal value of foaming level was reached 0.810 ± 0.010 and emulsion stability time 34 min at 2.0% content (Fig. 4a). Hence, 2.0% sodium lactate is employed in the formulation of shower gel.

Effect of grapefruit essential oil content: In this study, the content of *Citrus maxima* essential oil was investigated from 0.5 to 2.5%. The addition of *C. maxima* essential oil at varying concentrations (ranging from 0.5% to 2.5%) does not result in the substantial changes in the foaming level and emulsion time of the product (Fig. 4b). However, there is an apparent change in both colour and odour; higher the concentration of smell, the more unpleasant strong, and uncomfortable the experience when it is utilized. In terms of colour of the product, a higher

concentration results in a more yellowish colour, which produces an appearance which is inappropriate for clear items. If the content of essential oil is low (0.5%), the scent will not radiate and retain much, moreover, if the content is too high (over 1%), it will be costly to produce. Hence, for the purposes of this investigation, the concentration of essential oil was chosen to be 1%.

Optimization of the formulation and the assessment of product: By conducting a comprehensive survey on the various aspects that influence the technological process involved in the production of shower gel using essential oils, this study has put forth a comprehensive formula for the aforementioned procedure, which is provided in Table-3, whereas the basic parameters of shower gel products are presented in Table-4. Accordingly, the shower gel product has a light yellow colour due to the content of essential oil, transparent appearance, no air bubbles and especially the scent of essential oils. Other basic parameters such as viscosity 3.338 Pa.s are within the range investigated, pH value is about pH 6-6.5, foaming is quite high about 0.833 with foam stability after 30 min is 0.155 in the emulsion time of 35 min. In addition, the structure of the system before and after use is identical, without layer separation. The findings of this study indicate that the prepared shower gel items examined successfully fulfilled the established criteria and fell within the parameters of the sample product survey.

Product evaluation by consumers: This rating is made based on 20 consumers (10 men + 10 women) based on 3 periods including before use, when to use and after use with a scale of 1 to 5, respectively. The results show that in general, the conditions of use are highly appreciated with a score of over 4.0.

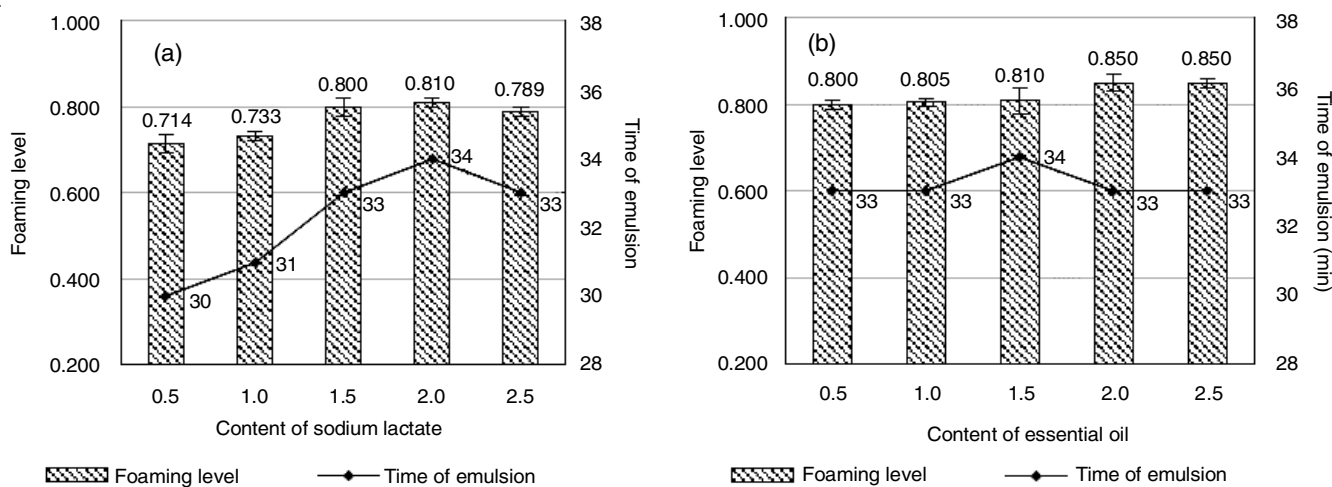


Fig. 4. Effect of preservative (a) and essential oil (b) content

TABLE-3
FORMULA FOR SHOWER GEL PRODUCTS

Composition	Content (%)	Composition	Content (%)	Composition	Content (%)
C MEA	1.0	Sodium methyl cocoyl taurate	12	Essential oil	1.0
EDTA	0.1	Disodium cocoamphodiacetate	2.0	EHGP	0.1
Hot water	Remaining	Plantacare 1200	3.0	DMDMH	0.4
Glycerin	2.0	D-Panthenol	0.3	CAPB	7.0
PEG-7 glycerin cocoate	1.5	CDE	1.5	NH ₄ Cl 25%	0.5
Sodium lactate	2.0	Acid citric 30%	0.5		

TABLE-4
BASIC PARAMETERS OF SHOWER GEL PRODUCTS

Parameters	Shower gel products	Parameters	Shower gel products
External inspection	Transparent, no air bubbles, uniform	Emulsion time (min)	35
Dry matter content (%)	27.04	Colour	Light yellow
pH value	6-6.5	Fragrant	Scent of <i>C. maxima</i> essential oil
Viscosity (Pa.s)	3.338	System structure	Uniformity
Foaming level	0.833	Feeling when using	Small foam, smooth, soft skin
Foam strength (foam volume after 30 min)	0.155	Structure of the system after centrifugation	Uniformity

The evaluation factors of the product before use (consistency, turbidity, clarity, scent, colour, sample ability) achieved an average score of 4.0 or higher, in which clarity and homogeneity accounted for the highest scores of 4.82 and 4.70, respectively. For the assessment while using the evaluation factors (foaming ability, skin irritation, fragrance, cleansing ability), an average score of 4.25 with the highest rated foaming ability was 4.43. Condition after use (fragrance, dry skin, hiss, skin irritation, cleanliness, skin smoothness) is also highly appreciated with an average score of all factors 4.22 (Fig. 5). This demonstrates

that the essential oil shower gel was well received and popular, and that the majority of consumers valued its clarity and fragrance.

Conclusion

This study aimed to examine the various aspects that influence the production process of grapefruit (*Citrus maxima* Burm.) essential oil based shower gel. These factors involved the selection of primary detergents, moisturizers, emulsifiers and preservatives, with the objective of formulating optimal production formulas. The product that has been developed is

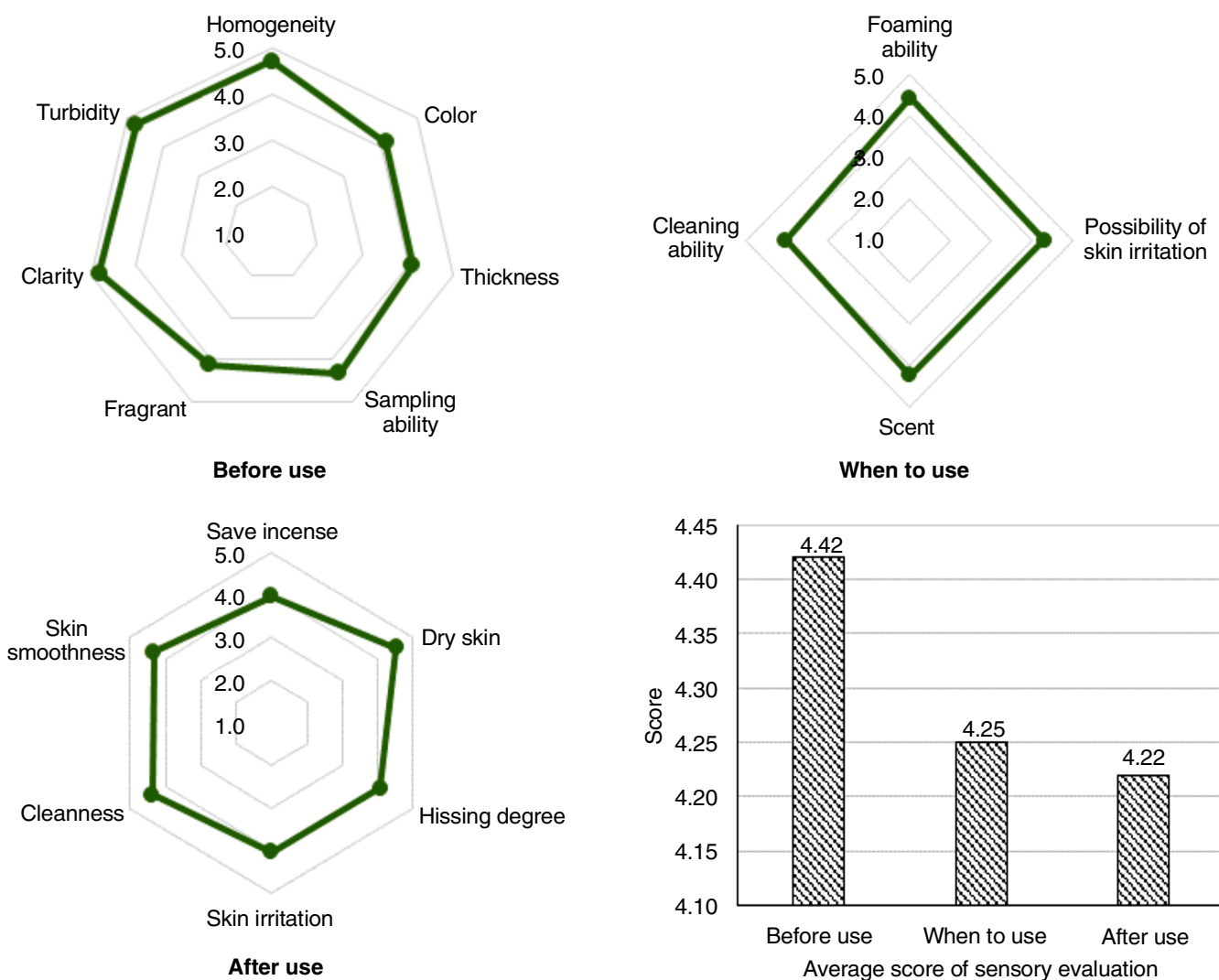


Fig. 5. Consumer perception of the shower gel product

subjected to an assessment of its fundamental physico-chemical characteristics and consumer preferences through the process of sensory evaluation. The results show that the shower gel products made from *C. maxima* essential oil has a homogeneous structure, transparent, no air bubbles, the typical scent of *C. maxima*, the dry matter content accounts for 27.04%, the pH value is in the range of pH 6-6.5, the viscosity is 3.338 Pa.s, the foaming is about 0.833, the emulsion time is 35 min, when using the product has small, smooth, soft foam. However, it is important to assess the finalized product for other characteristics too, including sensitization, skin allergy potential, antibacterial qualities and changes in skin texture. This evaluation should be conducted both prior to and during the use of the product in subsequent research endeavors.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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