



Investigation of sodium laurylglucosides hydroxypropyl sulfonate through response surface methodology – Effects of amphoteric surfactant and electrolyte

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ABSTRACT

The goal of this research was to investigate the effect of electrolyte and an amphoteric surfactant on the viscosity, foamability, and foam stability of sodium laurylglucosides hydroxypropyl sulfonate (SLHS), a new anionic surfactant. Different concentrations of cocamidopropyl betaine (CAPB, 0–6% w/w) and sodium chloride (NaCl, 0–3% w/w) were added into 10% w/w SLHS solution. Each formulation was evaluated for its viscosity using a viscometer, whereas the foamability and foam stability were measured using the cylinder shaking method and analyzed by response surface methodology. The result demonstrated that an increased viscosity of the mixture resulted with an increased NaCl concentration over the tested range, and with an increased CAPB concentration up to 2% w/w, after which the viscosity was reduced with higher CAPB levels. The CAPB and NaCl were observed to have a significant effect on the foamability, but not with foam stability. No significant interaction effect between the CAPB and NaCl concentration occurred. However, the response surface models for both foaming characteristics presented a satisfactory determination coefficient, thus ensuring the precision and reliability of the models. Thus, the influence of CAPB and NaCl concentrations on SLHS solutions was well predicted and similar to other anionic surfactants.

Keywords: Anionic surfactant, cocamidopropyl betaine, foaming characteristics, sodium chloride, viscosity

INTRODUCTION

Anionic surfactants are the main ingredient in many applications, ranging from industrial to household as well as personal care products. Within a personal care application area, cleansing products typically consist of anionic surfactants as the primary surfactant along with secondary surfactants, electrolytes, preservatives, and other additives, such as coloring agents, fragrances, and active compounds.^[1-3] However, when integrating surfactants into a product, the other ingredients need to be considered since the interactions of surfactant molecules with various other factors will occur, resulting in a change in the surfactant's physicochemical properties, and this seems to be a vital factor in developing suitable function-specific products.^[4-7] Secondary surfactants,

especially amphoteric surfactants, and electrolytes are among the most commonly investigated factors for studying the pattern of physicochemical properties of anionic surfactants as they are the two main components that usually have a great influence on the properties of anionic surfactants and are typically found in cleansing formulations.^[7-10]

Cocamidopropyl betaine (CAPB) is the most widely used amphoteric secondary surfactant due to its well-known viscosity building and foam boosting properties with the ability to increase mildness when combined with anionic surfactants.^[3,11] Meanwhile, sodium chloride (NaCl) is the most broadly used electrolyte due to its cheapness and availability.^[11]

Sodium laurylglucosides hydroxypropyl sulfonate (SLHS, D-Glucopyranose, oligomeric, C10-16-alkyl glycosides,

2-hydroxy-3-sulfopropyl ethers, and sodium salts), 100% naturally derived surfactant from corn and coconut, is one of the new mild anionic surfactants being used with good cleansing ability.^[12-14] Its structure is in Figure 1. However, its physicochemical properties have not been clearly studied yet. The aim of this study was, therefore, to investigate the effect of both CAPB and NaCl on the physicochemical properties of SLHS, including the viscosity, foamability, and foam stability. In addition, a systematic investigation was undertaken through implementing response surface model (RSM).

EXPERIMENTAL PROCEDURES

Materials

The SLHS solution, as 36% w/w SLHS in water (Suga® Nate 160NC), was kindly received as a gift from Colonial Chemical, Inc. (South Pittsburgh, USA) through Water Doctor Co., Ltd. (Bangkok, Thailand). The CAPB solution, as 30% w/w CAPB in water, and NaCl were obtained from the Aqua-Medi Products Pte. Ltd. (Singapore) and Thai Sanguanwat Chemical Co., Ltd. (Bangkok, Thailand), respectively. Both citric acid and potassium hydroxide (KOH) were purchased from Thai Sanguanwat Chemical Co., Ltd. (Bangkok, Thailand) and were used for pH adjustment. Deionized water was used in this study. All chemicals were cosmetic grade and used as received without further treatment.

Preparation of Test Solutions

Test solutions containing a constant 10% w/w SLHS with four different concentrations of CAPB (0, 2, 4, and 6% w/w) and four different concentrations of NaCl (0, 1, 2, and 3% w/w) were prepared in triplicate. All ingredients were weighed out on a mass basis. The SLHS was first dissolved in deionized water, followed by the stated amount of CAPB and then NaCl. After a homogeneous mixture was obtained, either citric acid or KOH was added to adjust the pH to 5.

Viscosity Measurement

The relative viscosity of the test solutions was measured at $25 \pm 2^\circ\text{C}$ using a Brookfield viscometer (Model LVDV-II, Brookfield Engineering Laboratories, MA, USA) attached with an appropriate spindle (cylindrical or disk spindle). Cylindrical spindle (spindle No. 1) was applicable for solutions with a low viscosity and here was used for the 6% w/w CAPB with all tested NaCl concentrations and the 4% w/w CAPB without NaCl. All the other solutions were considered as viscous solutions and so were tested with disk spindle (spindle No. 2). The experimental conditions, including the temperature and container sizes, were kept constant during this study.

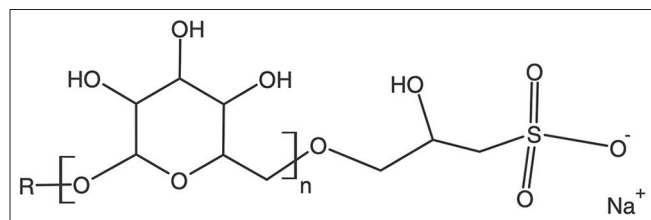


Figure 1: Structure of SLHS^[14]

Foaming Characteristics

Determination of the foaming properties was performed by employing a modification of the Bartsch (shaking test) method.^[15-17] To generate foam, the test solution was further diluted to 1:100 with deionized water and 20 mL of that diluted solution was then placed into a 100 mL graduated cylinder and sealed. The cylinder was then inverted vertically for 30 s at a constant rate of 1 revolution/s to generate the foam. The cylinder was then placed upright, left undisturbed and the volume of the foam column above the liquid phase was measured as a function of the elapsed time. The maximum foam volume (immediately after foam generation) was expressed as the foamability value of the test solution and the microscopic images of foam were also recorded by a Nikon microscope (Model Eclipse E200, Nikon Inc., Tokyo, Japan).

The foam volume monitored at 1, 5, 10, 15, and 30 min after foam generation was used to calculate the percentage of foam remaining through by comparing the subsequent foam volume (mL) at a specific period of time (T ; min) to the original foam volume (mL) reached by the immediate generated one, as shown in Equation (1),

$$\% \text{ Foam remaining } (T) = \frac{\text{Foam volume at time } T}{\text{Initial foam volume}} \times 100 \quad (1)$$

The graphs were then plotted as the percentage of foam remaining (for zero-order kinetic) and the natural logarithm of the percentage of residual foam (for first-order kinetic) overtime to determine an appropriate model for the foam stability. The slopes obtained from the graphs giving the lowest P -value from statistical analysis represent the rate of foam collapse and were expressed as the foam stability value of the test solutions. All experiments were conducted at $25 \pm 2^\circ\text{C}$.

Statistical Analysis

Statistical significance was analyzed with the SPSS Statistics program version 22 (IBM, Armonk, NY, USA) using analysis of variance (ANOVA) and accepting significance at $P < 0.05$ level. For better visualization of the effect of the mixture composition on the foaming characteristics, RSM was then applied using the RSM package (version 2.10) in the R program (version 3.6.1).

RESULTS AND DISCUSSION

Over the years, new surfactants have been developed that are less irritating and less damaging to health and the environment. Mild surfactants have received increasing attention due to consumer awareness on irritation.^[18,19] However, despite its mildness, this type of surfactant has traditionally been limited by their difficulty in building viscosity and boosting foam. Thus, the pattern of the physicochemical properties of individual surfactants is needed since this can be useful information for product development.

Considering that both the viscosity and foaming characteristics of a cleansing product are important for the consumers' perception of the cleansing ability, then these two properties are among the most commonly investigated types of physicochemical properties. Even though foaming properties

are an esthetic attribute that is rarely actually related to the cleansing ability of the product, it is a crucial criterion for the consumers' acceptance, in terms of a perceived signal of product efficacy.^[2,20] Meanwhile, the viscosity, another critical factor for formulators, also needs to be adjusted to obtain a suitable value for product spreading and product dispensing from the container without stinginess.^[21]

Many researchers have investigated the physicochemical properties of various anionic surfactants after the addition of other compounds.^[7,8,22-25] Unlike traditional anionic surfactants, such as sodium lauryl sulfate (SLS) or sodium laureth sulfate (SLES), many of the mild surfactants, such as amino acid-based surfactants, do not increase their viscosity or foamability through the traditional methods of adding salt or amphoteric surfactants, respectively.^[18,21,26]

The influences of CAPB and NaCl on the viscosity and foamability of a 10% w/w SLHS solution were investigated here since they are both common ingredients in cleansing products. The concentration range of each ingredient was selected from the commonly used concentrations in commercially available products and recommendations from surfactant's manufacturers.^[1,2,11,27,28] The concentration of SLHS, as the primary anionic surfactant, was kept constant, while the concentration of the cosurfactant and electrolyte was varied. Regarding the study of viscosity, analysis of variance (ANOVA) was carried out to compare the results between each factor together with an illustration of a statistical difference in viscosity at each level. As for foaming characteristics study, response surface methodology was taken into account with the purpose of determining whether any interaction between factors could occur and generating the prediction equations.

Effect of the Mixture Composition on the Viscosity

For a given NaCl concentration, the viscosity of mixture increased with increasing CAPB concentrations up to 2% w/w CAPB, after which a reduction in the viscosity with higher CAPB concentrations was observed [Figure 2a]. To obtain the precise peak (maximal viscosity), additional investigations on varying the CAPB concentration between 1.5 and 4% w/w are recommended. On the other hand, increasing the NaCl concentration over the investigated range was associated with a corresponding increase in the viscosity of the mixture at a given CAPB concentration [Figure 2b]. These differences in the viscosity were statistically significant at all levels (ANOVA, $P < 0.01$).

Overall, the pattern of these findings were in accordance with the previous studies on anionic surfactants, where the variation in the viscosity of anionic surfactant solutions was mainly due to the transformation of their micellar morphology and structure.^[4,5,8-10,29] Firstly, anionic surfactant molecules assemble to form individual spherical micelles by themselves. With the addition of amphoteric surfactants, CAPB (the isoelectric point = 6.25), at pH 5, inserts their slightly positively charged molecules between the negatively charged head groups of the anionic surfactant and this promotes the more tightly packing of the monomers.^[5,30] Likewise, adding electrolyte helps to minimize the electrostatic repulsion between the charged head groups of the surfactant and so allows a closer packing of the surfactant molecules.^[4,8,29] As the concentration

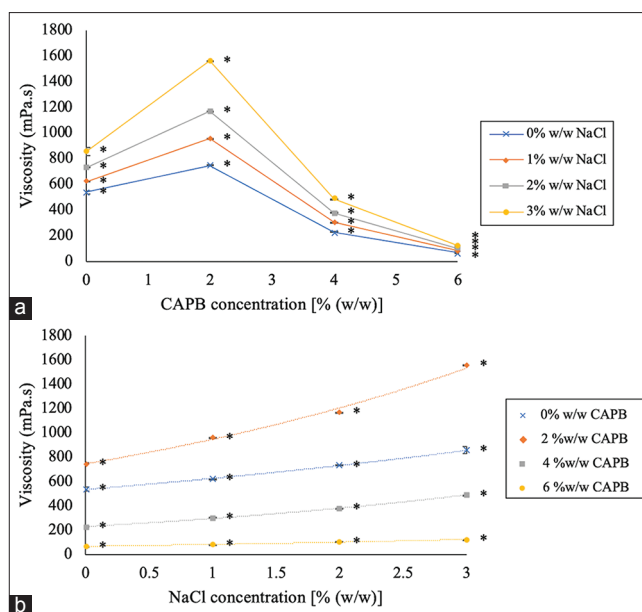


Figure 2: Viscosity of a SLHS/CAPB/NaCl mixture as a function of the (a) CAPB concentration with different NaCl concentrations and (b) NaCl concentration with different CAPB concentrations. *Indicates significant difference ($P < 0.05$) from the viscosity of 10% w/w SLHS solution without CAPB and NaCl

of either the amphoteric surfactant or electrolyte increases, the structure of the surfactant aggregates begins to transform leading to the changes in the packing parameters. Up to a certain concentration value, the transition from spherical to rod-like and then to worm-like micelles begins to take place accordingly. With further addition of those compounds, the worm-like micelles continue to grow and overlap leading to a transient entangled network. Within this step, each micellar transformation contributes to a correspondingly higher viscosity. However, this corresponding increase in the viscosity will continue only up to a certain concentration threshold, after which the viscosity will decrease with further increases in the CAPB, for example, due to different mechanisms, such as a transition into branched micellar networks or reversing back to either spherical or rod-like micelles instead.^[4,29]

Likewise, studies on the effect of electrolytes on anionic surfactants, such as SLS and SLES, revealed a viscosity peak at 4.5% and 5.5% w/w NaCl, respectively. However, when SLS or SLES was combined with CAPB, the NaCl concentration required to create the viscosity peak was reduced in accord with the CAPB concentration.^[8,29,31] Thus, the NaCl concentration used in this study was limited to 3% w/w, since higher NaCl concentrations were out of the commonly used range of NaCl concentrations, which is beyond the scope of this present study. As a consequence, further investigations on the micelle characterization of these mixtures are required to correlate the changes in the viscosity with the micelle microstructure to determine the precise mechanism underlying this rheological behavior.

Effect of Mixture Composition on Foaming Characteristics

The microscopic images of foam from different solutions are demonstrated in Figure 3. For different concentration of CAPB,

no trend of bubble size and its size distribution was obviously detected, but it has been reported that an increase of CAPB concentration resulted in a decrease of bubble size.^[21] On the other hand, when the NaCl concentration was increased, the bubble size seemed smaller. This observation is similar to the result from the study by Xu *et al.* on the effect of electrolyte (NaCl) on SLS solution and, additionally, they found that NaCl concentration barely had an impact on size distribution of bubble.^[32]

As far as the physicochemical properties of foam are concerned, the foamability and foam stability are the

two main important foaming properties that need to be considered. Within this study, an increase in either the CAPB or NaCl concentration resulted in a corresponding significant ($P < 0.01$) increase in the foamability. Since this study of foam stability was mainly focused on foam degradation over a period of time, then the rate of the foam collapse as a function of time was the most suitable variable to represent the foam stability. From Table 1, both kinetic models demonstrated the significant regression, however, the first-order model exhibited the lowest p-value from all test solutions. Thus, the first order was the most suitable

Table 1: R^2 and P -value for each test solution from different models

% CAPB	% NaCl	Zero-order kinetic (% foam remaining versus time)		First-order kinetic (ln % foam remaining versus time)	
		R^2 from regression	P -value from ANOVA	R^2 from regression	P -value from ANOVA
2	1	0.391	0.006	0.416	0.004
4	1	0.423	0.003	0.450	0.002
6	1	0.431	0.003	0.452	0.002
2	2	0.392	0.005	0.417	0.004
4	2	0.407	0.004	0.431	0.003
6	2	0.420	0.004	0.441	0.003
2	3	0.329	0.013	0.344	0.010
4	3	0.412	0.004	0.434	0.003
6	3	0.447	0.002	0.471	0.002

Bold numbers indicate the lowest P -value among two models.

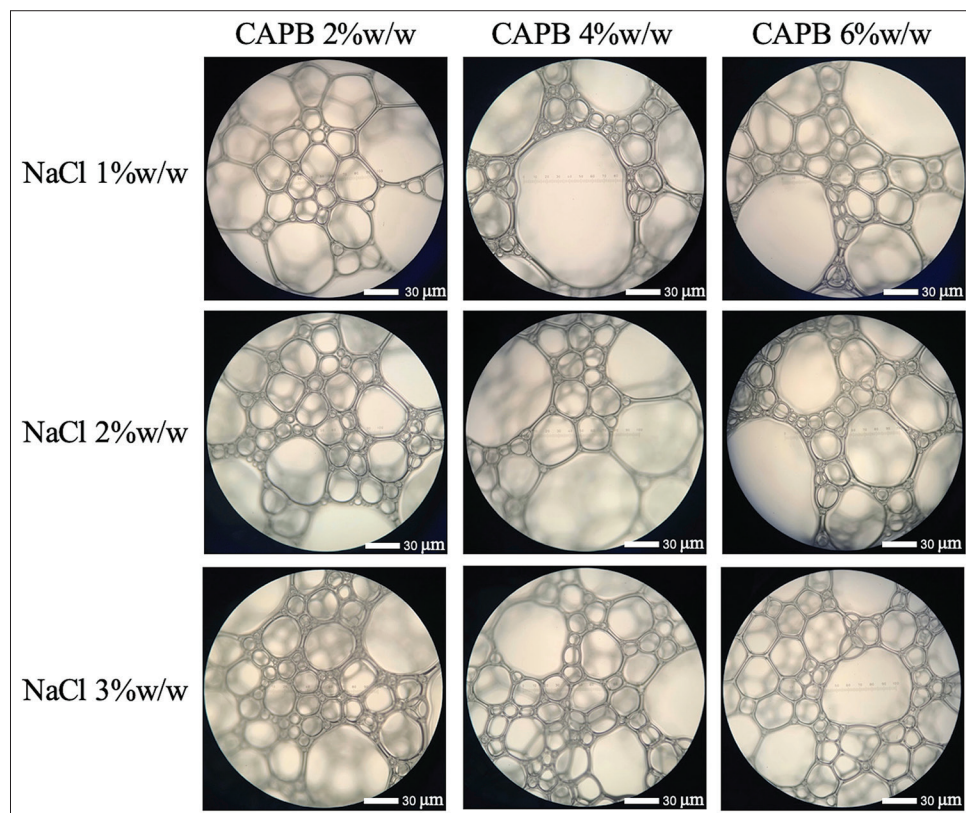


Figure 3: Microscopic images of foam from SLHS/CAPB/NaCl mixtures

model for the foam collapse, and the slopes from the plot between the natural logarithm of the percentage of residual foam versus time were generated and used as the foam stability value for further RSM analysis.

To further examine the influence of these factors on foaming, RSM for the foaming characteristics was applied. A two-factor response surface design with three levels of each variable was performed using the CAPB concentration (X_1) and NaCl concentration (X_2) as the independent factors and the foamability (Y_1) and foam stability (Y_2) as the response variables. All data were analyzed using the R program over coded variables (-1, 0, and +1), in which each of the actual values were coded according to Equation (2):

$$X_i = \frac{U_i - U_i^0}{\Delta U_i} \quad (2)$$

Where, X_i is the coded value of the independent variable, U_i is the actual value of the independent variable; U_i^0 is the actual value on the center point of the independent variable, and ΔU_i is the step change value.^[33-35] The actual and corresponding coded concentrations of each ingredient are summarized in Table 2.

The quadratic response surface from the second-order model was adopted to predict the variation of response variables as a function of the independent variables, as shown in Equation (3);

Table 2: Concentration of each ingredient in coded values and actual values

Ingredient	Concentration		
	Coded values		
	-1	0	1
Actual values (% w/w)			
CAPB (X_1)	2	4	6
NaCl (X_2)	1	2	3

Table 3: Validation of the RSM for foamability and foam stability

Statistical estimators	Condition for good fit	Foamability	Foam stability
Regression p-value	< 0.05	0.01304	0.03525
R ²	Close to 1	0.9067	0.8579
Adjusted R ²	In agreement with R ²	0.8133	0.7157

$$Y_i = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{12} X_1 X_2 \quad (3)$$

Where, Y_i is the predicted response; β_0 , β_1 , β_2 , and β_{12} are the constant regression coefficients of the model, and X_1 and X_2 are the independent variables.^[33,35,36] According to the multiple regression analysis, the quadratic RSM for the foamability and foam stability of the mixture in terms of both NaCl and CAPB concentrations is presented in Equations (4) and (5), respectively:

$$\text{Foamability} = 94.737 + 3.6667X_1 + 1.6667X_2 - 1.8421X_1^2 - 0.8421X_2^2 + 1.7211 \times 10^{-14} X_1 X_2 \quad (4)$$

$$\text{Foam stability} = -3.0316 \times 10^{-3} - 1.5000 \times 10^{-4} X_1 + 8.3333 \times 10^{-5} X_2 + 2.2895 \times 10^{-4} X_1^2 - 7.1053 \times 10^{-5} X_2^2 - 7.5000 \times 10^{-5} X_1 X_2 \quad (5)$$

To interpret the precision and reliability of the RSM, the data were analyzed by ANOVA. Table 3 presents the R², adjusted R², and p-value of both the foamability and foam stability RSM, while the experimental and predicted values of both responses are compared in Figure 4. It was observed that the RSM for foamability was statistically significant with a determination coefficient (R²) of 0.9067, explaining 90.7% of the variance in the response. The ANOVA for the RSM used to estimate the foam stability was less correlated, and showed a lower determination coefficient (R² of 0.8579), explaining 85.8% of the variance in the response. However, both second-order regression models were statistically significant (P-value = 0.01304 and 0.03525 for foamability and foam stability, respectively), and presented a satisfactory determination coefficient, indicating that this RSM was applicable for the prediction of responses with the required combination of parameters.

As the next step, each of the foaming characteristics was discussed through their empirical models and response surface graphs. Within each quadratic regression model, only some of the terms were statistically significant, as shown in Table 4. For the foamability, the contour plot and response surface graph are presented in Figures 5a and 6a, respectively. The main effects of the changes in the CAPB or the NaCl concentration were significant ($P = 0.0019$ and 0.0426 , respectively) on the foamability but with no interaction between them. Foamability was maximized when both the CAPB and NaCl concentrations were at a higher level.

Theseresultsagreewellwithpreviousstudiesonotheranionic surfactants, such as SLS and sodium dodecylpolyoxyethylene sulfate, where the addition of either amphoteric surfactants or

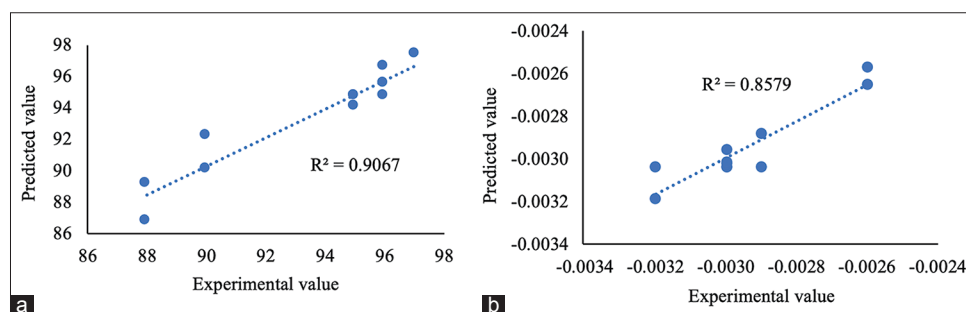


Figure 4: Comparisons of the responses observed from the experiments with those predicted by the developed RSM for the (a) foamability and (b) foam stability

electrolytes promoted the foamability of the mixture.^[7,37,38] As for an amphoteric surfactant, the increased foamability could be interpreted as being a result of lowering the surface tension by the secondary surfactant. Considering that foam generation requires expenditure of energy against the surface tension, then the presence of the electrolyte will minimize the electrostatic repulsion between charged head groups, which, in turn, facilitates the surface adsorption, resulting in a higher rate of surface tension lowering and promoting a higher foamability as a consequence.^[6,7] In addition, when compared to the viscosity, which is considered as one of the main factors influencing foamability, it was observed that, throughout a concentration range of foam studied, the increased foamability was in accordance with the lower range of bulk viscosity.

Meanwhile, the contour plot and response surface graph for the foam stability are presented in Figures 5b and 6b,

respectively. At a constant NaCl concentration, the results demonstrated that both the main effect and quadratic effect of the CAPB concentration had a significant ($P = 0.0172$ and 0.0177 , respectively) influence on the foam stability. Since the mechanism of foam stabilization is complex, several studies have proposed various factors affecting the foam stability of mixtures; for example, foam stability will be increased as the bulk viscosity and surface viscosity of mixtures increase, while it will be decreased as the fluidity of foam film and gravitational force become greater.^[39-41] Among these factors, the observed decrease in the foam stability at a lower CAPB concentration of 2–4% w/w could be attributed to the reduced viscosity and higher weight that would eventually contribute to a gravitational force on the foam rupture.^[39,42] Nevertheless, this trend was reversed at a higher CAPB concentration of 4–6% w/w. The reason for this rather contradictory result is

Table 4: ANOVA of the developed RSM for the foamability and foam stability

Term		Foamability		Foam stability	
		Coefficient	P-value	Coefficient	P-value
Main effects:	X_1	3.6667	0.0019*	-1.5000×10^{-4}	0.0172*
	X_2	1.6667	0.0426*	8.3333×10^{-5}	0.1093
Quadratic effects:	X_1^2	-1.8421	0.1098	2.2895×10^{-4}	0.0177*
	X_2^2	-0.8421	0.4154	-7.1053×10^{-5}	0.3303
Interaction effect:	X_1X_2	1.7211×10^{-14}	1.0000	-7.5000×10^{-5}	0.2122

X_1 , CAPB concentration (%); X_2 , NaCl concentration (%). * $P \leq 0.05$

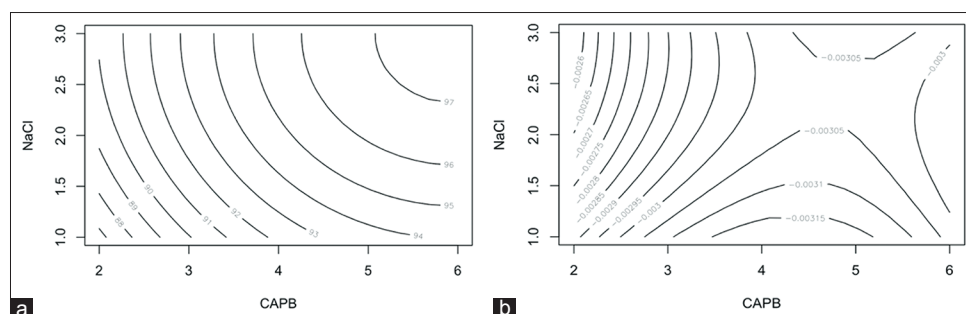


Figure 5: Contour plots of the (a) foamability and (b) foam stability

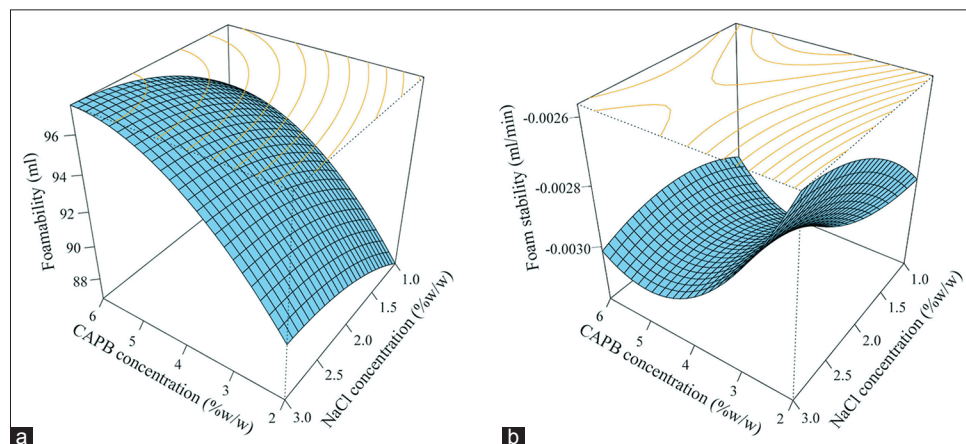


Figure 6: Variation of the (a) foamability and (b) foam stability of a SLHS/CAPB/NaCl mixture with different CAPB and NaCl concentrations and a fixed SLHS concentration of 10% w/w

still not completely understood. However, the results of this study appeared to be consistent with those of studies with a wide range of surfactant concentrations.^[42-45] Thus, future study about foaming mechanism might help extend the explanations of this optimum point.

The variation in foam stability with increasing NaCl concentration was marginal compared to that with changes in the CAPB concentration, and the effect of the NaCl concentration was insignificant in the foam stability regression model. However, this retardation in drainage could be attributed to the more rigid film fluidity together with its higher viscosity.^[7,41] According to the above results, the foam stability was maximized when the CAPB and NaCl concentrations were at lower and higher levels, respectively, but there was no significant interaction ($P = 0.2122$) between the CAPB and NaCl concentrations as far as foam stability was concerned.

CONCLUSIONS

This study evaluated whether the physicochemical properties of SLHS, a mild surfactant, would be affected by the addition of NaCl or CAPB, as an amphoteric surfactant, under their commonly used concentrations in commercially available products. It appeared that there was an optimal CAPB and NaCl concentrations for the viscosity, foamability, and foam stability. Using RSM, the foamability and foam stability could be predicted by second-order equations. These findings confirmed that SLHS, a mild surfactant, was compatible with the commonly used CAPB and NaCl, and could be used with them to obtain the desired physicochemical properties, the same as with other anionic surfactants.

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