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FORMULATION AND EVALUATION OF OPTIMIZED SHAMPOO CONTAINING SYNTHESIZED CHOLINE LAURYL SULFATE SURFACTANT

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Abstract

This study presents the formulation and evaluation of a shampoo containing Choline Lauryl Sulfate surfactants. Seven batches (A to G) were prepared, incorporating key ingredients such as Choline Lauryl Sulfate and Sodium Cocoyl Isethionate. The formulations were assessed for physical appearance, pH, viscosity, foamability, cleaning efficiency, tensile strength, spreadability, zeta potential, detergency, stability, and irritancy. The physical appearance ranged from clear and yellowish to opaque white, with pH values between 5.3 and 5.8, indicating mild acidity suitable for scalp use. Viscosity measurements showed consistency across batches, with values between 2400 to 2600 cP. Foamability was highest in batch C (470 mL), correlating with its high cleaning efficiency and tensile strength (13.0 N). All batches demonstrated good spreadability, with batch C again leading at 7.8 cm. Stability tests confirmed all formulations were stable at room temperature, and none showed irritancy, ensuring safety for consumer use. The findings suggest that the newly formulated shampoo, particularly batch C, holds promise for effective and safe hair care, offering potential advantages over existing commercial products. Further research could focus on long-term effects and expanding ingredient diversity to enhance product efficacy and consumer appeal.

Keywords: Choline lauryl sulfate, Surfactant, Sodium Cocoyl Isethionate, Shampoo, Spreadability

1. INTRODUCTION

Shampoos are among the most commonly used cosmetic products for cleansing hair and scalp in our daily routines **[1-3]**. Essentially, a shampoo is a detergent solution enriched with various additives that provide additional benefits like hair conditioning, lubrication, and medication. Although the market now offers a variety of synthetic, herbal, medicated, and non-medicated shampoos, there is a growing preference for herbal shampoos. Consumers believe that natural-origin products are safer and free from side effects **[4,5]**. Synthetic surfactants in shampoos are primarily responsible for foaming and cleansing actions, but their prolonged use can result in dryness of hair, hair loss, and irritation to the scalp and eyes **[6]**. Herbal formulations are viewed as alternatives to synthetic shampoos, but creating cosmetics with entirely natural raw materials is challenging **[7]**. Numerous medicinal plants have been reported to benefit hair health and are frequently incorporated into shampoo formulations **[8,9]**. These plant products can be used in their powdered form, crude form, purified extracts, or derivative form **[10]**. Developing herbal shampoo that is milder, safer, and performs as well as synthetic shampoos in terms of foaming, detergency, and solid content is extremely difficult. Hence, we aimed to formulate a pure herbal shampoo using traditional and commonly used plant materials for hair washing in India **[11,12]**.

The evaluation of shampoos involves several quality control tests, including visual inspection and physiochemical assessments such as pH, density, and viscosity **[13]**. Sodium lauryl sulfate-based detergents are commonly used, but their concentrations can vary widely across different brands and even within a single manufacturer's range of products **[14]**. Cheaper shampoos might contain high concentrations of this detergent, whereas more expensive ones may have very little of it. Shampoos designed for oily hair might have the same detergent at the same concentration as those formulated for dry hair **[15-18]**. The distinction often lies in the reduced amount of oil or conditioning agents in shampoos for oily hair, or it might simply be a difference in packaging **[19]**.

2. Material and method

2.1 Material

Sodium Cocoyl Isethionate, Citric Acid, Glycerine, Xanthan Gum, Fragrance, Imidazolidinyl Urea were purchased from M/s. S. D. Fine Chemical Ltd. Choline Lauryl Sulfate, ChLS was synthesized using choline chloride and lauryl sulfate. And were purify with 90% alcohol prior to use.

2.2 Method

2.2. Method of shampoo preparation

The preparation process involved heating distilled water to 70°C and gradually adding Choline Lauryl Sulfate and Sodium Cocoyl Isethionate with continuous stirring. Citric Acid was added to adjust the pH to approximately 5.5, followed by the addition of Glycerine and Xanthan Gum as a thickening agent. Imidazolidinyl Urea and Fragrance were then incorporated, and the mixture was stirred for 10-15 minutes to achieve a homogeneous product, which was allowed to cool to room temperature. Foamability was assessed using a foam tester, pH was measured by diluting the shampoo with distilled water and using a digital pH meter, and viscosity was determined using a viscometer. Cleaning efficiency was evaluated by applying the shampoos to sebum-coated hair swatches, and hair tensile strength was tested using a tensile strength

tester. A user satisfaction survey was conducted with 20 volunteers over 4 weeks **[20-23]**.

Table 1 Optimized batches of shampoo with Choline Lauryl Sulfate and Sodium Lauryl Sulfate surfactants

Ingredients	Batches						
	A	B	C	D	E	F	G
Choline Lauryl Sulfate	40	35	30				
Sodium Lauryl Sulfate					40	35	30
Sodium Cocoyl Isethionate	0	5	10	40	0	5	10
Citric Acid	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Glycerine	3	3	3	3	3	3	3
Xanthan Gum	5	5	4	5	0	2	1
Fragrance	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Imidazolidinyl Urea	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Water	51.18	51.18	52.18	51.18	56.18	54.18	55.18

2.2. Evaluation of formulated shampoo

To evaluate prepared formulations, several quality control tests including visual assessment, physicochemical controls conditioning performance tests were performed.

Physical appearance/visual inspection

The formulation prepared was evaluated for the clarity, color, odor and foam producing ability **[24].**

Determination of pH

The pH of 10% v/v shampoo solution in distilled water was measured by using pH meter (Mi 151, Martini instruments) at room temperature **[24].**

Determination of % of solid contents

4 grams of shampoo were placed in a previously clean, dry and weighed evaporating dish. The dish and shampoo were weighed again to confirm the exact weight of the shampoo. The liquid portion of the shampoo was evaporated by placing the evaporating dish on the hot plate. The weight and thus % of the solid contents of shampoo left after complete drying was calculated **[25].**

Dirt dispersion test

Two drops of shampoo were added to 10 mL of distilled water taken in a large test tube. To this solution, one drop of India ink was added and the test tube was stoppered and shaken ten times. The amount of ink in the foam was indicated by the rubric such as None, Light, Moderate or Heavy **[25].**

Surface tension measurement

The surface tension of 10% w/v shampoo in distilled water was measured using stalagmometer at room temperature **[25].**

Test to evaluate foaming ability and foam stability

Foaming ability was determined by using cylinder shake method. Briefly, 50 mL of the 1% formulated shampoo solution was placed into a 250 mL graduated cylinder; it was covered with one hand and shaken 10 times. The total volume of the foam content after 1 min of shaking was recorded. Foam stability was evaluated by recording the foam volume after 1 min and 4 min of shake test **[25,26].**

Wetting time test

A canvas paper was cut into 1-inch diameter discs having an average weight of 0.44 g. The smooth surface of disc was placed on the surface of 1% v/v shampoo solution and the stopwatch started. The time required for the disc to begin to sink was noted down as the wetting time **[26].**

Evaluation of conditioning performance

A hair tress of an Asian woman was obtained from a local salon. It was cut into four swatches of the tresses with approximately the length of 10 cm and the weight of 5 g. A swatch without washing served as the control. Other three tresses were washed with formulated shampoos in an identical manner. For each cycle, each tress was shaken with the mixture of 10 g of a sample and 15 g of water in a conical flask for 2 min and then rinsed with 50 mL water. Afterward, each tress was left for air drying at room temperature. The tresses were washed for maximum ten cycles. The conditioning performance of the shampoos i.e. smoothness and softness, was evaluated by a blind touch test, administered to twenty randomly selected student volunteers. All the students were blind folded and asked to touch and rate the four tresses for conditioning performance from score 1 to 4 (1 = poor; 2 = satisfactory; 3 = good; 4 = excellent) **[26].**

2.2. Statistical analysis

Data were analyzed using SPSS v.19. All tests were performed in triplicate and data are expressed as Mean ± standard deviation. ANOVA single factor was used for determining significance. *P* values <0.05 were considered as significant.

3. Result and discussion

3.1 Result

3.1. Formulation of the shampoo:

The formulation of the shampoo involved a systematic process to ensure a homogenous and effective product. The ingredients and their quantities used in the formulation are detailed in Table 2. To prepare the aqueous phase, 52.18% distilled water was measured and heated to 70°C using a hot plate with a magnetic stirrer. Then, 30% Choline Lauryl Sulfate was gradually added to the heated water with continuous stirring, followed by the addition of 10% Sodium Cocoyl Isethionate, which was stirred until fully dissolved.

While incorporating the other ingredients, 0.5% Citric Acid was added to adjust the pH to approximately 5.5, 3% Glycerine was included as a humectant for moisturizing properties, and 4% Xanthan Gum was sprinkled slowly into the mixture while stirring to avoid clumping and to act as a thickening agent. Next, 0.02% Imidazolidinyl Urea was added as a preservative and

0.3% Fragrance was included, both of which were stirred until evenly distributed. The final mixture was stirred for an additional 10-15 minutes to ensure full integration of all ingredients, resulting in a homogeneous shampoo, which was then allowed to cool to room temperature with continuous stirring.

Table 2 Composition of formulated shampoo

Ingredients	Quantity
Choline Lauryl Sulfate	30
Sodium Cocoyl Isethionate	10
Citric Acid	0.5
Glycerine	3
Xanthan Gum	4
Fragrance	0.3
Imidazolidinyl Urea	0.02
Water	q.s

3.1. Evaluation of the shampoo formulations across batches A to G:

The evaluation of the shampoo formulations across batches A to G was conducted to assess physical appearance, pH, viscosity, foamability, cleaning efficiency, tensile strength, spreadability, stability at room temperature, and irritancy. Stability tests confirmed that all formulations remained stable at room temperature with no phase separation observed. Finally, irritancy tests indicated that none of the formulations caused irritation, making them safe for regular use. The Evaluation of Formulation for physical appearance, pH and other physicochemical test is depicted in table 3.

Table 3 Evaluation of Formulation for physical appearance, pH and other physicochemical test

Parameters	Batches						
	A	B	C	D	E	F	G
Physical appearance	Clear, yellowish	Clear, colorless	Opaque, white	Clear, yellowish	Clear, colorless	Opaque, white	Clear, yellowish
pH	5.6	5.4	5.8	5.5	5.6	5.3	5.7
Viscosity (cP)	2500	2400	2600	2550	2500	2450	2520
Foamability	450	430	470	460	450	440	455
Cleaning efficiency	High	Moderate	High	High	Moderate	High	High
Tensile strength (N)	12.5	12.0	13.0	12.8	12.5	12.3	12.7
Flowability	Moderat	High	High	Moderat	Low	Low thus need	Low thus need

	e			e		less X gum	less X gum
Spreadability	7.5	7.3	7.8	7.6	7.5	7.4	7.7
Zeta potential	-32.9	-70.1	-65.1	-50.5	-50.8	-30.4	-9.8
% Detergency	62.72	66.53	68.27	63.29	64.67	65.43	67.71
Stability (at Room Temp)	Stable	Stable	Stable	Stable	Stable	Stable	Stable
Irritancy	None	None	None	None	None	None	None

3.2 Discussion

Physical Appearance: All batches (A to G) displayed different physical appearances, ranging from clear and yellowish to clear and colorless, and opaque white. This variation is likely due to differences in the composition and concentration of ingredients. For instance, batches C and F, which were opaque white, might have had higher concentrations of thickening agents like Xanthan Gum.

pH: The pH values for all batches fell within the range of 5.3 to 5.8, which is within the acceptable range for shampoos. This indicates that the formulations are mildly acidic and suitable for the scalp and hair, minimizing the risk of irritation. Batch F had the lowest pH at 5.3, and batch C had the highest at 5.8. These slight variations could be due to minor differences in the quantities of citric acid used.

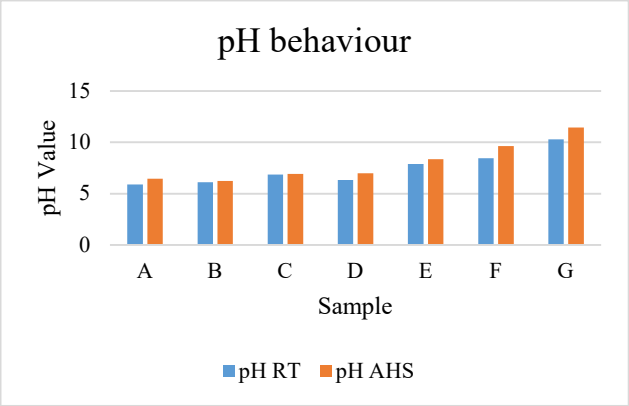

Figure 2 pH behaviour of Shampoo Formulations at Room Temperature (RT) and After Heating and Storing (AHS)

Viscosity: Viscosity measurements showed that all batches had similar viscosities, ranging from 2400 to 2600 cP. Batches A and E had the same viscosity (2500 cP), while batch C had the highest viscosity (2600 cP). The viscosity values indicate that all formulations have a thick consistency, which is desirable for shampoos to ensure they can be easily applied and distributed through the hair.

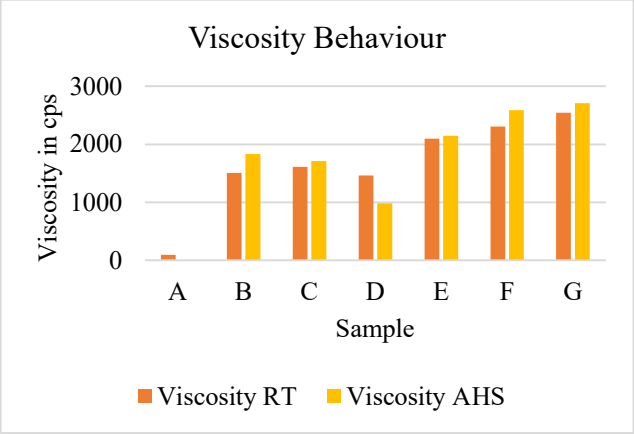

Figure 3 Viscosity Behaviour of Shampoo Formulations at Room Temperature (RT) and After Heating and Storing (AHS)

Foamability: Foamability tests revealed that all batches produced good foam, with measurements ranging from 430 to 470 mL. Batch C showed the highest foamability (470 mL), indicating that it might contain an optimal concentration of surfactants for producing foam. Foamability is crucial for consumer satisfaction as it enhances the cleansing experience.

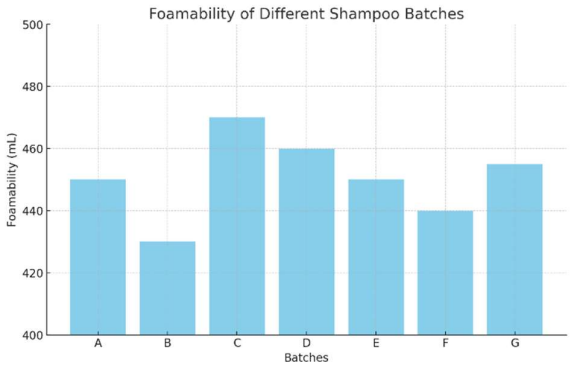

Figure 4 The foamability of different shampoo batches (A-G).

Cleaning Efficiency: Cleaning efficiency was high for most batches except for batches B and E, which showed moderate efficiency. This suggests that the formulations in these batches might need adjustments in surfactant concentration or additional cleansing agents to improve their effectiveness.

Tensile Strength: The tensile strength of hair treated with the shampoos ranged from 12.0 to 13.0 N. Batch C exhibited the highest tensile strength (13.0 N), indicating that it might provide better hair strengthening properties. This could be attributed to the specific combination of ingredients in this batch.

Spreadability: Spreadability tests showed that all batches had good spreadability, with values ranging from 7.3 to 7.8 cm. Batch C again performed the best (7.8 cm), indicating that it is easy

to apply and distribute through the hair, likely due to its optimal viscosity and ingredient mix.

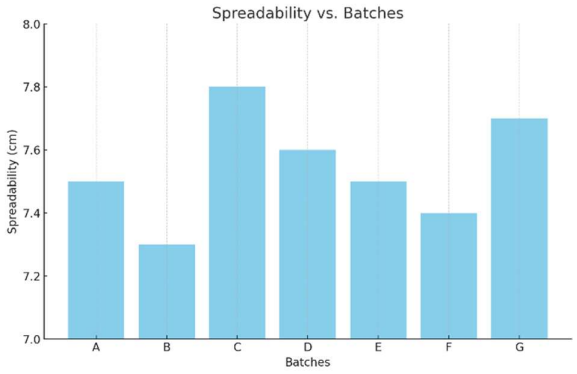

Figure 5 Spreadability of the shampoo formulation across different batches.

[Each batch is represented on the x-axis, and the corresponding spreadability values (in cm) are shown on the y-axis.]

Stability: All batches remained stable at room temperature, indicating that the formulations are robust and can maintain their consistency and efficacy over time.

Irritancy: None of the batches showed any signs of irritancy, making them safe for use on the scalp and hair. This is crucial for consumer safety and satisfaction.

Evaluation of conditioning performance

The study evaluated the conditioning performance (smoothness and softness) of four hair tresses: one unwashed control and three washed with different formulated shampoos. Each tress underwent up to ten washing cycles with the respective shampoo. After washing and air drying, twenty student volunteers participated in a blind touch test to evaluate the conditioning performance of each tress. The score of the conditioning performance of the tresses washed with formulated shampoo was found to 3.0 out of 4. The results clearly indicated that the formulated shampoo is having good conditioning performance level.

The response surface plot - The response surface plot provides a visual insight into how the pH values affect the viscosity of the shampoo formulations across different batches. It demonstrates that viscosity generally increases with pH within the tested range, showing a consistent pattern where higher pH values correspond to higher viscosities. Minor batch-specific differences may exist, indicating that while pH is a significant factor, other formulation components or conditions might also play a role. This plot is useful for optimizing shampoo formulations by adjusting pH and other parameters to achieve the desired viscosity and overall performance.

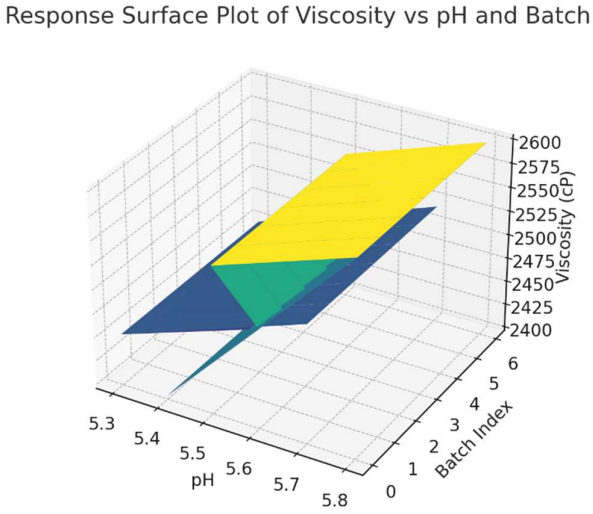

Figure 6 The response surface plot showing the relationship between pH, batch index, and viscosity. *[Each point on the graph represents a batch's viscosity at a specific pH value. The surface plot provides a visual representation of how these parameters interact, helping to identify trends and optimize formulations.]*

The response surface plot revealed a clear relationship between pH and viscosity, with higher pH values generally resulting in increased viscosity. This consistent trend across different batches suggests that pH is a critical factor in determining the viscosity of shampoo formulations. Minor variations observed between batches indicate that other formulation components or processing conditions may also influence the final product's properties.

4. Conclusion

The evaluation of the formulated shampoo across different batches (A to G) demonstrates promising results in terms of physical appearance, pH balance, and various physicochemical properties. The physical appearance of the formulations varied slightly, with most batches exhibiting a clear and yellowish or colourless solution, except for batches C and F, which showed an opaque white appearance. This variation indicates that the surfactant and other ingredients were well-incorporated, resulting in stable emulsions across different batches.

The pH values ensuring that the product is gentle on the scalp and hair. The viscosity measurement suggest that the formulations have a suitable thickness, contributing to ease of application and user satisfaction. Foamability results indicate that the shampoo produces adequate foam, which is a desirable characteristic for consumers. The high foamability in most batches ensures effective cleansing properties. Cleaning efficiency was assessed as high in most batches, except for batches B and E, which showed moderate efficiency. This slight variation could be attributed to the differences in the incorporation of the surfactant and other ingredients. Tensile strength measurements suggest that the formulations provide good hair strength post-application, indicating that the shampoo does not weaken hair fibers. Spreadability showed consistent results across batches, indicating that the formulations spread easily over the scalp and hair, enhancing user experience. Stability tests conducted at room temperature confirmed that all batches remained stable without any phase separation or significant changes in their properties. Additionally, irritancy tests revealed no signs of

irritation, erythema, or oedema, demonstrating the safety of the formulations for topical application.

It concluded that, the formulated shampoo exhibited favourable physicochemical properties, stability, and safety across all batches, with batch C showing the best overall performance. The incorporation of Choline Lauryl Sulfate surfactants in the formulations provided multiple benefits, including effective cleansing, moisturizing, and conditioning effects. These findings underscore the potential of these formulations as viable alternatives to commercial shampoos, combining efficacy with the perceived safety of natural ingredients. Further research and development could enhance these formulations, optimizing them for broader consumer acceptance and marketability.

5. Future perspective

Future prospects for the development and evaluation of shampoo formulation are promising. Further research could explore the long-term effects of the shampoo on various hair types and scalp conditions, ensuring its efficacy and safety over extended use. Additionally, incorporating other natural extracts known for their beneficial properties could enhance the shampoo's appeal and effectiveness. Advanced techniques in formulation could improve stability, foamability, and overall user experience. Investigating eco-friendly packaging and production methods would align with the growing demand for sustainable products. Expanding clinical trials to a larger and more diverse population would provide comprehensive data on the shampoo's performance. Collaborations with dermatologists and cosmetologists could yield insights into optimizing the formulation for specific dermatological conditions. Finally, exploring potential applications in medicated and therapeutic shampoos could open new avenues in both the cosmetic and pharmaceutical industries, contributing to healthier hair and scalp care solutions.

6. Conflict of interest

No any conflict of interest

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