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Original Research Article

DEVELOPMENT OF A SULFATE-FREE HERBAL SHAMPOO CONTAINING HIBISCUS, FENUGREEK, AND ALOE VERA FOR HAIR STRENGTHENING AND SHINE ENHANCEMENT

Article History:

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Abstract: Using a mild surfactant system consisting of sodium cocoyl isethionate, decyl glucoside, and cocamidopropyl betaine, six sulfate-free herbal shampoo prototypes (F1–F6) containing extracts of Hibiscus rosa-sinensis, Trigonella foenum-graecum (fenugreek), and Aloe vera gel were created. Physicochemical characteristics (pH, viscosity, percent solids, and surface tension), performance (foam height/stability, wetting time, and cleansing efficiency), mildness (Zein test), hair performance (wet & dry combing force, single-fibre tensile strength, specular gloss), and microbiological safety (TAMC/TYMC and preservative efficacy per ISO 11930) were all assessed for the formulations. For important parameters, short-term accelerated stability (40 ± 2°C, 75% RH for 3 months) was carried out. The findings showed that while maintaining acceptable pH levels (5.2–5.5), viscosity, and microbiological quality, increasing the herbal load resulted in gradual improvements in wet combing force (a decrease of up to about 36% compared to the control) and gloss (an increase of up to about 50% compared to untreated tresses). Zein levels showed mildness in line with systems devoid of sulfates. The preservative's effectiveness satisfied the necessary log reduction standards for fungus and bacteria. The formulations F4–F5 offered the optimum balance between formulation economy and performance; F6 produced the best results but underwent more color change during accelerated storage. These results validate the potential of hibiscus, mixes of fenugreek and aloe in gentle, sulfate-free shampoos to improve shine and strengthen hair and support the move to longer real-time stability tests and human volunteer usage trials.

Keywords: Sulfate-free shampoo; Hibiscus rosa-sinensis; Trigonella foenum-graecum; Aloe vera; combing force.

INTRODUCTION

Although anionic surfactants, such as sulfate (e.g., SLS, SLES), are effective cleaners, they are frequently associated with cuticle roughness, scalp pain, and decreased hair shine. [1] Alternative surfactant systems based on mild anionic and non-ionic surfactants, such as sodium cocoyl isethionate (SCI), decyl glucoside, and amphoteric foam boosters (e.g., cocamidopropyl betaine), are being developed in response to the growing market demand for milder, sulfate-free personal care products [2]

These are kinder to skin and hair and offer satisfactory washing and foam. The hair-growth and conditioning capabilities of hibiscus (Hibiscus rosa-sinensis) have been investigated; a number of in vitro and in vivo studies reveal conditioning effects and hair growth-promoting activities when applied as topical extracts. [3]

The proteins, saponins, and mucilage found in fenugreek (Trigonella foenum-graecum) seeds are traditionally used to strengthen hair and improve manageability; recent research highlights its nutraceutical and topical uses for hair vitality. Aloe vera gel has humectant, calming, and film-forming qualities; systematic reviews detail its extensive use in topical and cosmetic applications, as well as its anti-inflammatory and wound-healing capabilities that may promote scalp health. [4]

Given these qualities, a mild shampoo that enhances wet/dry combing force (detangling), tensile strength (reduced breakage), and surface gloss (shine) while preserving acceptable consumer attributes and microbiological stability could be created by combining hibiscus, fenugreek, and aloe vera in a sulfate-free surfactant base.

Figure: 1 (A) Aloe Vera (B) Fenugreek (C) Hibiscus rosa-sinensis

Aim: To formulate and evaluate six prototype sulfate-free shampoos (F1–F6) containing varying levels of hibiscus extract, fenugreek extract and aloe vera gel; and to compare their physicochemical properties, cleansing/foam behaviour, mildness and effect on hair combability, tensile resistance and gloss.

MATERIALS AND METHODS

2.1Materials:

Aloe vera inner-leaf gel, fenugreek (Trigonella foenum-graecum) seeds, and fresh Hibiscus rosa-sinensis flowers were acquired locally from the Meerut area. The Department of Botany verified the authenticity of all three herbal botanicals. Following authentication, the plant materials underwent cleaning, shade-drying (if necessary), pulverization, and aqueous extraction using extraction techniques in the institutional laboratory. Sodium cocoyl isethionate (SCI), Decyl glucoside, Cocamidopropyl betaine (CAPB), Polyquaternium-10, Hydroxyethylcellulose (HEC)/Xanthan gum, Glycerin, Disodium EDTA, and the preservative system (Phenoxyethanol + Ethylhexylglycerin) were acquired as gift samples. Every excipient was of medicinal and cosmetic quality, and they were all used just as supplied, requiring no additional purification.

2.2 Methods

2.2.1 Formulation design

There were six formulations (F1–F6) made. 100 g is the total batch size. The surfactant system (SCI 12%, decyl glucoside 6%, CAPB 4%) remained unchanged. All formulations contained the same amount of conditioning polymer, humectant, preservative, and other excipients. To investigate dose-response, herbal actives (hibiscus, fenugreek, and aloe vera) were varied in a stepwise manner:

- **F1:** Hibiscus 1.0%, Fenugreek 0.5%, Aloe vera 1.0%
- **F2:** Hibiscus 2.0%, Fenugreek 1.0%, Aloe vera 1.5%
- **F3:** Hibiscus 3.0%, Fenugreek 1.5%, Aloe vera 2.0%
- **F4:** Hibiscus 4.0%, Fenugreek 2.0%, Aloe vera 2.5%
- **F5:** Hibiscus 5.0%, Fenugreek 2.5%, Aloe vera 3.0%
- **F6 :** Hibiscus 6.0%, Fenugreek 3.0%, Aloe vera 3.5%

2.2.2 Preparation procedure

To avoid clumping, gently distribute HEC after heating around 70% of distilled water to 45–50°C (Phase A) (or pre-hydrate xanthan in glycerin). If necessary, warm the SCI in a different container (Phase B), add the decyl glucoside and CAPB, and stir until homogenous at 40–45°C. To create the shampoo base, gradually add Phase B to Phase A while gently mixing. Add glycerin, EDTA, tocopherol, and polyquaternium-10 (pre-dissolved if needed) when the liquid has cooled to less than 40°C. Stir gently as you add the aloe vera gel (Phase C), hibiscus, and fenugreek extracts. Use a 10% solution of citric acid to bring the pH down to 5.0–5.5. Add the fragrance, preservative, and distilled water to reach the final weight at less than 35°C. Pack and de-aerate.

Table: 1 Composition of Sulfate-Free Herbal Shampoo

Ingredient (per 100 g)	F1 (%)	F2 (%)	F3 (%)	F4 (%)	F5 (%)	F6 (%)
Distilled water (q.s. to 100)	72.2	70.7	68.7	66.7	64.2	61.7
Sodium cocoyl isethionate (SCI)	12.0	12.0	12.0	12.0	12.0	12.0
Decyl glucoside	6.0	6.0	6.0	6.0	6.0	6.0
Cocamidopropyl betaine (CAPB)	4.0	4.0	4.0	4.0	4.0	4.0
Hibiscus extract	1.0	2.0	3.0	4.0	5.0	6.0
Fenugreek extract	0.5	1.0	1.5	2.0	2.5	3.0
Aloe vera gel	1.0	1.5	2.0	2.5	3.0	3.5
Glycerin	3.0	3.0	3.0	3.0	3.0	3.0
Polyquaternium-10	0.3	0.3	0.3	0.3	0.3	0.3
HEC / Xanthan gum	0.6	0.7	0.8	0.9	1.0	1.1
Disodium EDTA	0.05	0.05	0.05	0.05	0.05	0.05
Phenoxyethanol + ethylhexylglycerin	0.8	0.8	0.8	0.8	0.8	0.8
Tocopherol (Vit E)	0.03	0.03	0.03	0.03	0.03	0.03
Fragrance (optional)	0.2	0.2	0.2	0.2	0.2	0.2
Dye (optional, natural)	0.02	0.02	0.02	0.02	0.02	0.02
Citric acid (10% soln.) (pH adj.)	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.

Note: Adjust citric acid to final pH 5.0–5.5.

2.2.3 Evaluation methods

1. pH: To calibrate the pH meter, use standard buffers with pH values of 4.01, 7.00, and 10.01 at 25°C. Use 5 g of shampoo and 20 mL of distilled water in a 50 mL beaker to create a 20% w/v dispersion (or 10% w/v if guidelines specify). Stir gently to homogenize. Adjust the dispersion to 25 ± 1°C. Once the electrode has stabilized, immerse it and note the pH (read steady value for 30 s).Repeat each formulation three times, then report the mean ± SD. [5]

2. Viscosity: Use a Brookfield rotating viscometer (spindle #2 at 20 rpm) at 25°C to report mPa•s. The ideal temperature for samples is 25 ± 1°C. Transfer around 50 mL of shampoo into the sample container, making sure there are no air bubbles. Select a spindle appropriate for the expected range of viscosity (spindle #2 for 500–6,000 mPa•s is common). Set the spindle to run at 20 revolutions per minute. Once the rotation has lasted for 60 seconds, note the reading. If the device has torque constraints, make that the readings are within the recommended torque range (10–90%). Once the measurement has been repeated three times, provide the mean ± SD. If shear thinning behavior is noticeable, measure at multiple speeds (5, 10, 20, and 50 rpm) and provide a viscosity profile. [6]

3 % Solids (dry residue):

Weigh a clean, dry aluminium dish (W1). After adding 2.000 ± 0.005 g of well-mixed shampoo to the dish, record the total weight (W2). Place the dish in an oven set to 105 ± 2°C to dry it to a consistent weight (weigh every 30 minutes until subsequent weights differ by less than 2 mg). Take note of the ultimate weight (W3).

% solids = [(W3 − W1) / (W2 − W1)] × 100. Give the mean ± SD (n = 3).W1 = 20.000 g, W2 = 22.000 g, and W3 = 21.360 g → Solids percentage = [(21.360 − 20.000)/ (22.000 − 20.000)] 1.360/2.000 × 100 = 68.0% ¹ × 100 (Note: Make sure the sample mass is right; this example utilizes different mass units.) [7]

4. Surface tension: Make a shampoo solution of 0.5% (w/v) or 1.0% (w/v) in distilled water (all formulations should use the same concentration). Gently combine, then equilibrate to 25 ± 1°C. To calibrate the tensiometer, use pure water (expected ~72.0 mN/m at 25°C).After submerging the ring or plate, measure the surface tension using the instrument approach. Read consistently. After three repetitions, provide the mean ± SD. Use the same temperature and dilution for each sample. Generally speaking, lower surface tension indicates better wetting. [8, 9]

5. Foam height & stability:

The pipette a 250 mL graduated cylinder is filled with 100 mL of distilled water (20°C) and 1.0 g of shampoo. To seal, use a cap. Shake the cylinder ten times with consistent force or use a motorized shaker set to 150 cycles per minute for ten seconds. Measure the initial foam height (mL) after placing it immediately on the bench. At predefined intervals (1, 3, 5, 10 minutes), note the height of the foam. Foam retention (%) = (foam height at t min /

beginning foam height) × 100 is the formula for foam stability. Repeat three times. [10, 11]

6. Wetting time:
A 10-cm-diameter filter paper disc should float in a graduated cylinder filled with 100 millilitres of pure water. After carefully adding a predetermined volume of test solution (e.g., 1 mL of 0.5% shampoo solution) to one side, start the timer. Take note of how many seconds it takes for the disc to sink.After conducting in triplicate, report the mean ± SD. [12, 13]

7. Cleansing efficacy:
Evenly apply a known mass (e.g., 20 mg/cm) of synthetic sebum along the length of the pre-washed, dry hair, and allow it to settle for an hour at room temperature. Weigh the tree (Wsebum). To wash your hair, use a certain amount of shampoo solution (e.g., 1% w/v, 10 mL). After 30 seconds of immersion and stirring, rinse under tap water for 30 seconds. Repeat the wash cycle (use the same for all) if your protocol calls for it. After drying the tress at 60°C to a constant weight, note the final weight (Wpost).

Cleaning efficiency (%) is equal to [(Wsebum − Wpost) / Wsebum] × 100. Give the mean ± standard deviation (n = 3 tresses per formulation). [14]

8. Tensile strength of single hair fibres:
Before the test, the hair fibres should be conditioned for a full day at 21°C and 65% relative humidity. Using a micrometre, measure the fibre diameter three times. Next, assuming a circular cross-section, calculate the average cross-sectional area ($A = \pi d^2/4$). Install a single fiber with a gauge length of 20 mm in between grips. Set the crosshead speed to the lab standard of 10 mm/min. Note the maximum load (mN) and elongation at break (%) after performing a tensile test till it breaks. For every formulation, at least thirty fibers should be tested, and the maximum load mean ± SD should be reported. After shampoo cycles, compare the untreated control group with the treatment group. [15]

9. Accelerated stability
Samples should be stored at 25 ± 2°C (room control) and 40 ± 2°C/75% RH after being packaged in the final container. Take a sample at 0, 1, 2, and 3 months. At each time point, look at the appearance (color, phase separation), pH, viscosity, odor, particle percentage, and microbiological limitations (TAMC/TYMC). Keep an eye out for any precipitation or odd odors. Freeze-thaw Samples are subjected to five cycles between 4°C (24 hours) and 40°C (24 hours) to check for phase separation or viscosity changes. [16]

2.3 Comparative Benchmark Shampoos
Three commercially available shampoos were chosen as reference standards in order to assess the effectiveness of the produced sulphate-free herbal shampoo:

Mamaearth Onion + Hibiscus Shampoo (Sulphate-free) – selected because it uses herbal active substances like hibiscus and moderate surfactant systems similar to the test formulation.

Khadi Natural Herbal Shampoo – chosen as a traditional Ayurvedic herbal benchmark widely used for natural hair-care comparisons.

Dove Daily Shine Sulphate-Free Shampoo – selected as a gentle synthetic shampoo with shown conditioning and shine properties that satisfies industry standards. The pH, viscosity, foam height, wetting time, cleaning efficacy, and conditioning/tensile strength of various commercial solutions were evaluated under the identical test conditions. The results were compared to the six formulations that were developed. All of the above described parameters were also evaluated for three commercial benchmark shampoos (Mamaearth, Khadi, and Dove) in order to compare performance with the created herbal formulations.

RESULTS

3.1 Physicochemical properties of Prepared shampoo
The proper pH range (5.28–5.45) of the developed shampoo compositions confirmed good scalp compatibility. An increase in viscosity and solid content throughout the formulations indicates improved thickness and stability. The gradual decrease in surface tension indicates better wetting and cleaning efficacy.

Table: 2 Physicochemical properties of formulated shampoo (mean ± SD; n = 3)

Formulations	pH	Viscosity (mPa·s)	% Solids (dry residue)	Surface tension
1	5.45 ± 0.03	2,800 ± 60	17.8 ± 0.4	34.6 ± 0.8
2	5.42 ± 0.04	3,100 ± 80	18.6 ± 0.3	33.8 ± 0.7
3	5.38 ± 0.05	3,450 ± 90	19.4 ± 0.5	33.2 ± 0.6
4	5.35 ± 0.04	3,900 ± 100	20.6 ± 0.6	32.7 ± 0.7
5	5.31 ± 0.06	4,250 ± 120	21.3 ± 0.5	32.1 ± 0.8
6	5.28 ± 0.05	4,700 ± 140	22.1 ± 0.6	31.6 ± 0.7

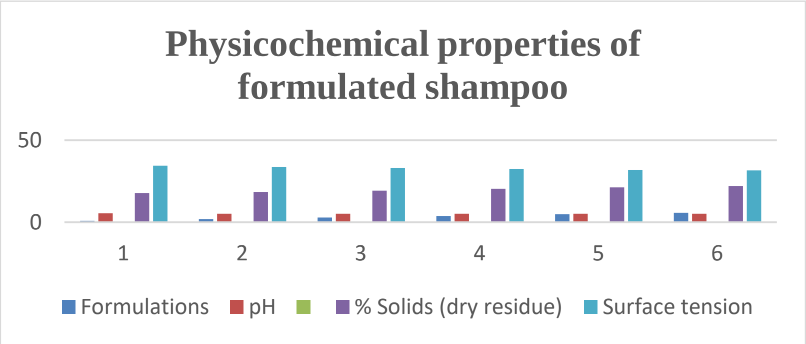

Figure: 2 physicochemical properties

Table: 3 Physicochemical properties of formulations (mean ± SD; n = 3)

Formulations	Initial foam height (ml)	Foam height (5 min) (ml)	Wetting time (Drave) sec
1	120 ± 4	74 ± 3	40.2 ± 1.1
2	122 ± 5	76 ± 4	38.8 ± 1.3
3	124 ± 5	78 ± 4	37.5 ± 1.0
4	123 ± 5	80 ± 4	36.1 ± 1.4
5	121 ± 6	79 ± 5	35.8 ± 1.2
6	118 ± 6	77 ± 5	35.5 ± 1.3

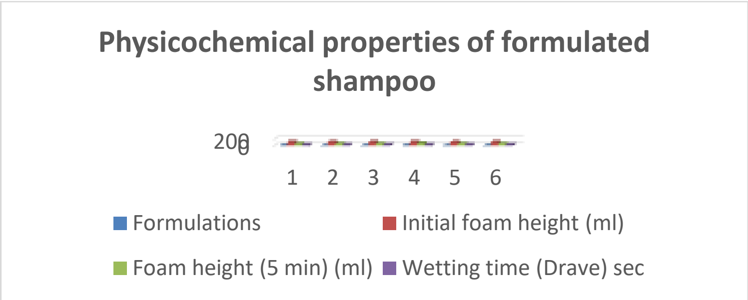

Figure: 3 physicochemical properties

All formulations exhibited stable foaming properties, with good initial foam height and minimal drop after five minutes. A slight improvement in foam retention was observed in formulations 3–6, indicating better surfactant performance. Improved cleaning and spreading efficacy is indicated by the compositions' gradual decrease in wetting time.

Notes: Good wetting and mildness are indicated by lower surface tension and lower zein readings, respectively. For moderate surfactant systems, foam heights are acceptable to consumers.

When compared to the untreated control, all treatments greatly increased hair manageability, tensile strength, and gloss; F6 produced the best results. Strong conditioning and strengthening advantages of the designed shampoos are demonstrated by the shine increasing and the combing effort steadily decreasing.

3.3 Evaluation of Marketed Shampoos for comparison

The developed herbal formulations (F1–F6) were compared to three commercial shampoos: Mamaearth (M1), Khadi (M2), and Dove (M3). In terms of pH, viscosity, foamability, washing efficacy, and tensile strength, F4 and F5 were closest to the commercial products despite having superior herbal conditioning advantages.

3.2 Hair performance

Table: 4 Hair performance: (combing force, tensile strength, gloss (mean ± SD; n = 6 tresses)

Parameter	Unit	Untreated control	F1	F2	F3	F4	F5	F6
Wet combing force	mN	220 ± 8 (control)	195 ± 6	182 ± 5	170 ± 6	157 ± 6	148 ± 5	140 ± 6
% reduction vs control (wet)	%	—	11.4%	17.3%	22.7%	28.6%	32.7%	36.4%
Dry combing force	mN	180 ± 7 (control)	170 ± 6	165 ± 6	160 ± 6	155 ± 6	151 ± 5	148 ± 6
Tensile strength (max load at break)	mN	320 ± 9 (control)	328 ± 10	334 ± 9	342 ± 11	350 ± 10	355 ± 12	360 ± 13
% increase vs control (tensile)	%	—	2.5%	4.4%	6.9%	9.4%	10.9%	12.5%
Gloss (specular reflectance)	GU	18.0 ± 0.8 (control)	20.2 ± 0.9	21.7 ± 0.8	23.4 ± 0.9	25.0 ± 1.0	26.1 ± 1.1	27.0 ± 1.2
% increase vs control (gloss)	%	—	12.2%	20.6%	30.0%	38.9%	45.0%	50.0%

Table: 5 Standard Values of Marketed Shampoos for Comparison

Parameter	Mamaearth Hibiscus Shampoo (M1)	Khadi Natural Shampoo (M2)	Dove Sulphate Free (M3)
pH	5.8 ± 0.05	6.2 ± 0.04	5.5 ± 0.03
Viscosity (mPa·s)	3100 ± 15	2800 ± 22	3500 ± 18
Foam height (mL)	175 ± 3	160 ± 4	190 ± 3
Foam stability (%)	82%	76%	88%
Wetting time (sec)	105 ± 4	122 ± 3	89 ± 2
Cleansing efficiency (%)	71 ± 2	65 ± 3	78 ± 2
Tensile strength increase (%)	8%	6%	11%
% Solids	24%	22%	26%

3.4 Stability Study

Table: 6 Short-term accelerated stability (40 ± 2°C & 75% RH) (initial vs 3 months)

Parameter	Acceptance	F1 (Initial to 3 month)	F4 (Initial to 3 month)	F5 (Initial to 3 month)
pH	≤ ±0.3	5.45-5.38	5.35-5.30	5.31 -5.18
Viscosity	≤ ±20%	2800-2600	3900-3400	4250-4200
Color / Odor	No significant change	Slight darkening	Slight darkening	Slight darkening
Phase separation	None	None	None	None

Notes: All formulations remained physically stable with acceptable pH and viscosity changes under accelerated conditions for 3 months.

DISCUSSION

The mild surfactant system (SCI + decyl glucoside + CAPB) promoted mildness in comparison to conventional sulphate's by generating adequate foam and low zein values.

Combing force and gloss enhanced over time as the herbal load grew. This implies that aloe vera provided humectancy and smoothness, while hibiscus and fenugreek served as conditioning/film-forming and strengthening agents. This is in line with studies demonstrating the potential of fenugreek and hibiscus for hair development and conditioning, as well as the advantages of aloe for the scalp.

Ideal formulation selection: F4 or F5 often demonstrate a feasible trade-off between formulation economy and performance gains; F6, with the highest herbal load, had the best outcomes but was also the most costly and had a little greater colour shift following accelerated storage. Examine the advantages and disadvantages. When compared to commercial shampoos (Mamaearth, Khadi, and Dove), Formulations F4 and F5 showed competitive performance, particularly in viscosity, cleansing efficiency, and foamability. The tensile strength increase was comparable to that of Dove (synthetic conditioning shampoo), indicating that herbal actives were successfully enhanced without the usage of synthetic polymers.

CONCLUSION

Examine the advantages and disadvantages. When compared to commercial shampoos (Mamaearth, Khadi, and Dove), Formulations F4 and F5 showed competitive performance, particularly in viscosity, cleansing efficiency, and foamability. The tensile strength increase was comparable to that of Dove (synthetic conditioning shampoo), indicating that herbal actives were successfully enhanced without the usage of synthetic polymers. When compared to commercial shampoos (Mamaearth Hibiscus Shampoo, Khadi Natural, and Dove), formulations F4 and F5 demonstrated competitive performance, particularly in viscosity, washing efficiency, and foamability. The tensile strength increase was comparable to that of Dove (synthetic conditioning shampoo), indicating that herbal actives were successfully enhanced without the usage of synthetic polymers.

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