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Review Article

Herbal Shampoo: A Comprehensive Review of Ingredients, Formulation, Evaluation and Quality

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ABSTRACT

Shampoo is the most widely used hair-care product, and herbal shampoos, which rely wholly or partly on plant-derived materials, are attracting growing interest from consumers seeking gentle, natural and sustainable alternatives to conventional synthetic formulations. This review provides a structured account of herbal shampoos, beginning with the structure and physiology of hair and scalp, the classification and ideal properties of shampoos, and the cleansing mechanism and limitations of conventional ingredients. The principal herbal ingredients are discussed by function: saponin-based cleansers (reetha, shikakai, fenugreek), conditioners and mucilage providers (hibiscus, aloe vera, coconut oil), anti-dandruff plants (neem, tea tree, tulsi), hair-growth supporting plants (bhringraj, brahmi, rosemary, amla), and antioxidants and natural colourants. Extraction techniques, formulation development, excipient selection, preservation, manufacturing and scale-up are described, followed by evaluation methods including pH, viscosity, foaming, surface tension, wetting, detergency, safety, herbal marker analysis and stability studies. Safety, regulatory frameworks, labelling and claims, market trends, sustainability and packaging are also considered. Key challenges are batch-to-batch variability of plant materials, preservation of water-rich natural formulations, achieving performance equivalent to synthetic products and the limited availability of clinical evidence. Future directions include standardised and biotechnological actives, green extraction, microbiome-friendly formulations, data-driven development and circular packaging.

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INTRODUCTION

1.1 Background

Hair care is one of the largest segments of the personal-care industry, and shampoo is its most widely used product. The primary purpose of a shampoo is to cleanse the scalp and hair by removing sebum, dead skin cells, environmental dirt and residues of styling products, without leaving the hair dry, tangled or the scalp irritated [1,2]. Modern shampoos, however, are expected to do much more: they must foam attractively, rinse easily, condition the hair, suit different hair types, control dandruff, be mild enough for frequent use and remain stable on the shelf.

Most commercial shampoos are built on synthetic anionic surfactants such as sodium lauryl sulfate (SLS) and sodium laureth sulfate (SLES), combined with conditioning polymers, silicones, synthetic preservatives, colours and fragrances. These formulations are efficient and economical, but concerns about scalp dryness, irritation, environmental persistence of some ingredients and consumer preference for natural products have increased interest in alternatives [3,5].

1.2 The rise of herbal shampoos

Herbal shampoos use plant-derived materials (saponin-rich fruits and pods, mucilaginous flowers and seeds, essential oils and plant extracts) in place of, or in addition to, synthetic ingredients. Hair washing with plants such as reetha (soapnut), shikakai, amla and hibiscus has been practised for centuries in Ayurvedic and other traditional systems, and these materials are now being re-examined with modern analytical and formulation science [5,21,22]. The global market for natural and herbal personal-care products has grown steadily as consumers seek products perceived as safer, gentler and more sustainable.

The term 'herbal' is used loosely in the market. A shampoo may be labelled herbal because it contains a small amount of an extract added to a conventional synthetic base, or because it is made entirely from plant-derived materials. This review distinguishes between these cases wherever relevant, since performance, stability and safety differ considerably.

1.3 Objectives and scope

This review aims to provide an integrated account of herbal shampoos, covering the following topics:

- the structure and physiology of hair and scalp that determine shampoo requirements;
- the history, classification and ideal properties of shampoos;
- conventional ingredients, their functions and limitations;
- herbal ingredients, their phytochemistry and reported activities;
- extraction, formulation and manufacturing considerations;
- evaluation and quality-control tests, including stability studies;
- safety, regulatory aspects, market trends and sustainability; and
- challenges and future directions.

1.4 Methodology

The review is narrative. Literature was collected from PubMed, Scopus, ScienceDirect and Google Scholar using terms such as *herbal shampoo*, *natural surfactant*, *saponin*, *hair care*, *scalp*, *dandruff*, *hair growth*, *shampoo evaluation* and *cosmetic regulation*. Textbooks, pharmacopoeial and regulatory documents were also consulted. Figures labelled *illustrative*, *schematic* or *simulated* are conceptual aids prepared for this review and are not experimental results.

1.5 Organisation

Chapters 2 to 5 provide the scientific background on hair, shampoo types, conventional ingredients and the herbal concept. Chapters 6 to 9 deal with herbal ingredients, extraction, formulation and manufacture. Chapters 10 and 11 discuss evaluation and safety. Chapters 12 to 14 address regulation, market trends and the future, Chapter 15 summarises mechanistic insights, Chapter 16 provides practical experimental protocols and Chapter 17 concludes.

CHAPTER 2

2. Hair and Scalp: Structure, Physiology and Cleansing Needs

2.1 Structure of the hair fibre

A mature hair fibre is a dead, keratinised structure produced by the hair follicle. It consists of three concentric regions: the cuticle, the cortex and, in some hairs, the medulla (Figure 1). The cuticle is formed by overlapping flattened cells like roof tiles, and its integrity determines gloss, smoothness and protection of the interior. The cortex makes up most of the fibre mass, contains keratin macrofibrils embedded in a matrix and carries the melanin pigments responsible for colour. The medulla is a loosely packed central core which is often absent in fine hair [6,7].

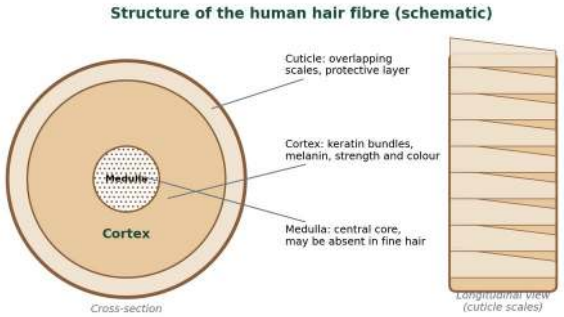


Figure 1: ***Schematic cross-section and longitudinal view of a hair fibre showing the cuticle, cortex and medulla.***

Table 1: ***Layers of the hair fibre and their relevance to shampooing.***

Layer	Composition	Function	Relevance to shampoo
Cuticle	Flattened keratinised cells, lipid-rich outer layer (18-MEA)	Protection, smoothness, gloss	Over-cleansing and alkalinity lift scales, causing roughness and tangling
Cortex	Keratin intermediate filaments, matrix proteins, melanin	Strength, elasticity, colour	Conditioners and proteins may deposit or penetrate; damage reduces strength
Medulla	Loosely packed cells and air spaces	Variable; may aid insulation	Little direct impact on shampoo performance
Cell membrane complex	Lipids and proteins between cells	Cohesion of layers	Surfactants can extract lipids if too harsh

2.2 The hair follicle and growth cycle

Each hair grows from a follicle that cycles through phases of growth (anagen), regression (catagen) and rest (telogen), followed by shedding (exogen) [8,9]. Anagen lasts several years on the scalp and determines hair length; catagen lasts a few weeks; telogen lasts about three months. Normally the majority of scalp hairs are in anagen at any time, and a daily loss of some tens of hairs is physiological. Disturbance of the cycle, caused by stress, nutritional deficiency, hormonal change, illness or medication, can increase shedding. Shampoos cannot alter follicle biology profoundly, but gentle cleansing and scalp-supporting actives are claimed to help maintain a favourable scalp environment.

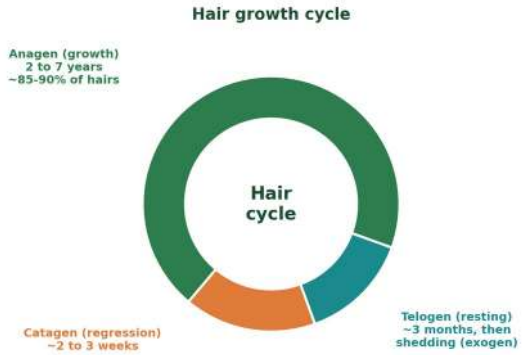


Figure 2: ***The hair growth cycle: anagen, catagen and telogen phases with approximate durations.***

2.3 The scalp surface

The scalp skin carries a high density of sebaceous glands. Sebum, a mixture of triglycerides, wax esters, squalene and free fatty acids, lubricates and waterproofs the hair but, with sweat, desquamated corneocytes and environmental particles, forms the soil that shampoo must remove. The scalp surface is mildly acidic, and a surface pH below about 5.5 supports the resident microbial flora and barrier function [10,11]. Cleansers that are strongly alkaline can disturb this acid mantle, increase swelling of the hair cuticle and promote dryness or irritation.

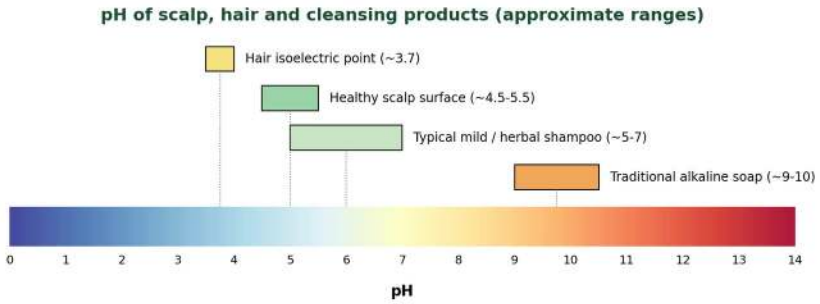


Figure 3: ***Approximate pH ranges of the scalp surface, the hair isoelectric point, mild shampoos and traditional alkaline soap. Ranges are indicative.***

2.4 Scalp microbiome and dandruff

The scalp hosts bacteria and fungi, including *Malassezia* yeasts, which are part of the normal flora [10]. Dandruff and seborrheic dermatitis are associated with *Malassezia* metabolism of sebum lipids into irritant fatty acids, together with individual susceptibility and a perturbed barrier [14]. Anti-dandruff shampoos therefore combine cleansing with an antifungal or keratolytic active. Plant materials such as neem, tea-tree oil and tulsi have reported antifungal or antimicrobial activity and are widely used in herbal products (Chapter 6).

2.5 Hair types and damage

Hair differs in diameter, curl, porosity and oiliness between individuals and ethnic groups. Chemical treatments (bleaching, colouring, perming), heat styling, ultraviolet radiation and mechanical grooming progressively damage the cuticle and cortex, increasing porosity and friction. Damaged hair requires milder cleansing and more conditioning, whereas oily scalps require more efficient sebum removal. A shampoo is therefore usually a compromise between cleansing and conditioning, tailored to the target hair type [1,6].

2.6 Requirements placed on a shampoo

From the biology above it follows that a shampoo should remove sebum and soil adequately, not strip the lipids of the cuticle and scalp, maintain a slightly acidic to neutral pH, produce a stable and pleasant foam, rinse easily, leave hair manageable and shiny, and not irritate the scalp or eyes. Herbal products must satisfy the same performance criteria as conventional ones while delivering any additional claimed benefits.

CHAPTER 3

3. Shampoo: History, Types and Ideal Properties

3.1 Historical perspective

Early cleansing of hair relied on natural materials such as clay, ash, plant saponins and fermented preparations. The word *shampoo* derives from the Hindi *champo*, meaning to press or massage, and was introduced into English in the eighteenth century. Soap-based shampoos appeared in the nineteenth century, but soap reacts with hard-water calcium and magnesium to form insoluble scum that dulls hair. Synthetic detergent-based products emerged in the 1930s, and the subsequent development of mild surfactants, conditioning agents, polymers and anti-dandruff actives produced the multifunctional shampoos of today [1,31].

3.2 Classification of shampoos

Shampoos can be classified in several ways (Figure 4). By physical form they may be liquids, gels, creams, powders, pastes, solid bars or aerosol foams. By function they include cleansing, conditioning ('2-in-1'), anti-dandruff, anti-hair-fall, clarifying, baby and colour-care shampoos. By surfactant chemistry they are anionic, cationic, amphoteric, non-ionic or natural saponin-based, and by origin they are synthetic, herbal or natural, organic or hybrid [2,23].

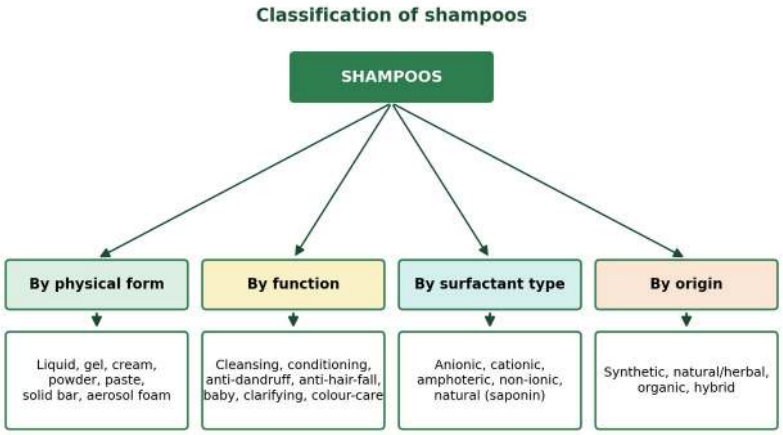


Figure 4: ***Classification of shampoos by physical form, function, surfactant type and origin.***

Table 2: ***Common types of shampoo and typical characteristics.***

Type	Typical features	Typical users / purpose
Clarifying	Higher detergency; removes product build-up	Oily scalp, swimmers, periodic deep clean
Conditioning (2-in-1)	Surfactant plus cationic polymer or silicone	Dry or damaged hair, ease of combing
Anti-dandruff	Antifungal / keratolytic active (zinc pyrithione, selenium sulfide, ketoconazole, or herbal actives)	Dandruff, seborrheic dermatitis
Baby / mild	Amphoteric and non-ionic surfactants, low irritation	Infants, sensitive scalps
Herbal / natural	Plant surfactants and extracts, minimal synthetics	Consumers seeking natural products
Powder / bar	Dry or solid form, water-activated	Travel, low-packaging, traditional use

3.3 Ideal properties of a shampoo

- Effective cleansing of sebum and soil without over-stripping.
- Good foam quantity and quality, with fine, creamy bubbles.
- Mildness to scalp and eyes, with a skin-compatible pH.
- Easy rinsing without residue; compatibility with hard water.
- Pleasant colour, fragrance and rheology (neither watery nor too thick).
- Conditioning benefits: wet and dry combing, softness, shine.
- Physical, chemical and microbiological stability throughout shelf life.
- Safe, non-toxic, and compliant with regulations; ideally environmentally acceptable.

3.4 Basic shampoo formulation approach

A shampoo is essentially an aqueous surfactant solution, to which performance modifiers and stabilisers are added. The formulator selects a primary surfactant for cleansing, secondary surfactants for foam and mildness, conditioning agents, a viscosity-building system, a preservative system, pH adjusters and aesthetic additives. Herbal formulators follow the same logic but substitute or supplement each category with plant-derived materials, which brings extra considerations of batch variability, colour, stability and microbial load [3,4,26].

CHAPTER 4

4. Conventional Shampoo Ingredients and Cleaning Mechanism

4.1 Mechanism of cleansing

Surfactant molecules are amphiphilic: a hydrophilic head group and a hydrophobic tail. At the hair surface the tails penetrate sebum and soil, lowering the interfacial tension, while the heads remain in the water. Mechanical action during massage rolls the oily soil into droplets that become surrounded by surfactant, and above the critical micelle concentration (CMC) micelles solubilise or emulsify the soil so that it can be rinsed away (Figure 5). Foam is not required for cleaning but is strongly associated by consumers with efficacy [1,23].

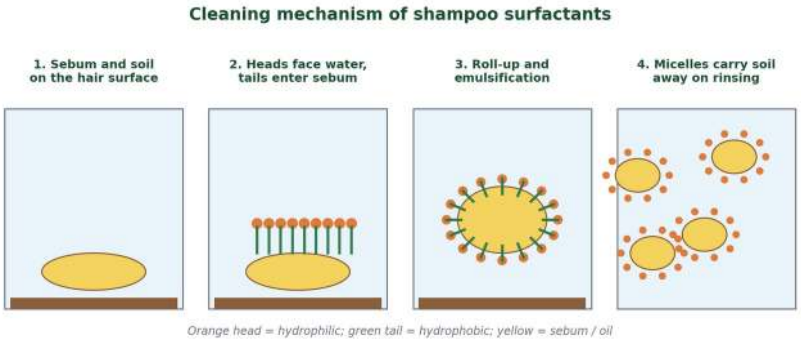


Figure 5: ***Schematic of the cleaning mechanism: adsorption of surfactant on soil, roll-up and emulsification, and removal of soil in micelles during rinsing.***

4.2 Surfactants

Anionic surfactants (SLS, SLES, ammonium lauryl sulfate, alpha-olefin sulfonates, sulfosuccinates) give strong detergency and foam but can be irritating and defat the scalp, particularly SLS. Ethoxylation (as in SLES) and the addition of amphoteric surfactants reduce irritation. **Amphoteric surfactants** (cocamidopropyl betaine, sodium cocoamphoacetate) improve mildness, foam and viscosity. **Non-ionic surfactants** (alkyl polyglucosides, fatty alkanolamides) are mild and enhance foam stability. **Cationic surfactants** and polymers are mainly used for conditioning rather than cleansing [1,2,23].

4.3 Conditioning agents and polymers

Cationic polymers such as polyquaternium-10 and cationic guar form coacervates on dilution during rinsing, depositing on negatively charged damaged hair to improve wet combing. Silicones (dimethicone and derivatives) give smoothness and shine but may build up. Natural oils, proteins and humectants such as panthenol and glycerin are also used, and are frequently the entry point for herbal ingredients in conventional formulae [24,31].

4.4 Other functional ingredients

Table 3: ***Typical ingredients in conventional shampoos, their functions and concerns.***

Category	Examples	Function	Concerns
Primary surfactant	SLS, SLES, ammonium lauryl sulfate	Cleansing, foam	Irritation, dryness, eye stinging
Co-surfactant	Cocamidopropyl betaine, cocamide DEA / MEA	Mildness, foam, viscosity	Sensitisation (betaine impurities), nitrosamine concern for DEA
Conditioner	Polyquaternium-10, dimethicone, panthenol	Combing, softness, shine	Build-up, persistence of some silicones
Thickener	Sodium chloride, PEG derivatives, carbomer, xanthan gum	Viscosity, consumer feel	Salt curve limits; ionic sensitivity
Preservative	Parabens, phenoxyethanol, isothiazolinones, formaldehyde releasers	Microbial protection	Sensitisation, consumer rejection of some classes
Chelating agent	EDTA salts	Prevent hard-water effects, aid preservative	Environmental persistence
pH adjuster	Citric acid, sodium hydroxide	Skin-compatible pH	None at usual levels
Colour / fragrance	Synthetic dyes, perfume	Appearance, acceptance	Allergens, photosensitivity

4.5 Limitations that motivate herbal alternatives

Frequent use of strongly detersive formulations may cause scalp tightness, dryness or irritation in sensitive individuals; some preservatives and fragrances are recognised contact allergens; and persistent ingredients may accumulate in aquatic environments [1,2,5]. Importantly, most of these concerns are dose- and formulation-dependent, and modern mild sulfate-based shampoos are generally well tolerated. The motivation for herbal products is therefore a mixture of genuine dermatological benefit, consumer preference and sustainability, rather than a simple safety advantage.

CHAPTER 5

5. Concept of Herbal Shampoo

5.1 Definition and categories

A herbal shampoo is a hair-cleansing preparation in which the principal cleansing, conditioning or therapeutic functions are delivered fully or partly by plant-derived materials. In practice the following categories are encountered:

- **Fully herbal / traditional:** plant saponins (reetha, shikakai) and mucilages as the cleansing and conditioning system, with no synthetic surfactant.
- **Natural-origin surfactant based:** mild surfactants derived from plant oils and sugars, for example alkyl polyglucosides or coco-glucoside, plus herbal extracts.
- **Hybrid:** conventional mild surfactant base fortified with standardised herbal extracts and oils.
- **Powder and solid bar forms:** dried herbal powders or syndets that are mixed with water at use.

5.2 Rationale and potential advantages

Potential advantages claimed for herbal shampoos include milder cleansing from saponins and mucilages, conditioning from natural polysaccharides and oils, antioxidant and antimicrobial activity of plant polyphenols, a favourable biodegradability profile and lower use of synthetic additives [3,4,21]. Some plant extracts have in-vitro or animal evidence of hair-growth-promoting or anti-dandruff activity, although

evidence from controlled clinical trials is limited for many materials (Chapter 11).

5.3 Limitations and challenges

Herbal ingredients introduce challenges that must be managed: variation of constituents with species, season, soil and harvest; colour and odour that may change over time; poorer foam and sometimes lower viscosity; susceptibility to microbial growth because plant extracts are nutrient-rich; limited toxicological data for many extracts; and the potential for allergy to botanical constituents (for example essential-oil terpenes). 'Natural' does not imply safe, and herbal products must be assessed with the same rigour as synthetic ones [21,22].

Table 4: ***Comparison of herbal and conventional synthetic shampoos.***

Parameter	Herbal shampoo	Conventional synthetic shampoo
Cleansing agent	Saponins, natural-origin surfactants	Synthetic anionic / amphoteric surfactants
Foam	Often lower and coarser (saponins) unless boosted	High, fine, stable
Conditioning	Mucilage, oils, extracts	Cationic polymers, silicones
Consistency between batches	Variable; requires standardised extracts	High reproducibility
Preservation	More demanding; natural preservatives less broad-spectrum	Well-established preservative systems
Appearance / odour	Natural colour, plant odour, may darken	Controlled colour and fragrance
Environmental profile	Often readily biodegradable; sourcing matters	Variable; some persistent ingredients
Evidence base	Mostly in-vitro / small studies for actives	Extensive performance and safety data
Consumer perception	Natural, gentle, traditional	Efficient, familiar

5.4 Key design principles

1. Define the target claim (cleansing, anti-dandruff, hair care) before choosing ingredients.
2. Use authenticated plant material and standardised extracts with documented marker compounds.
3. Ensure physical and microbiological stability, since natural systems are more vulnerable.
4. Select a surfactant system that delivers acceptable foam and mildness; do not rely on saponins alone for consumer acceptance.
5. Substantiate claims with appropriate in-vitro, instrumental and clinical or sensory evidence.
6. Comply with the relevant cosmetic regulation of each market.

CHAPTER 6

6. Herbal Ingredients Used in Shampoos

Plants contribute to shampoos in several functional roles (Figure 6). Many are multifunctional: amla, for example, is both a conditioner and an antioxidant, and fenugreek contributes mucilage, protein and saponins. The following sections group the most commonly used materials by principal function; Table 5 summarises botanical details and reported constituents [15,21,22,27].

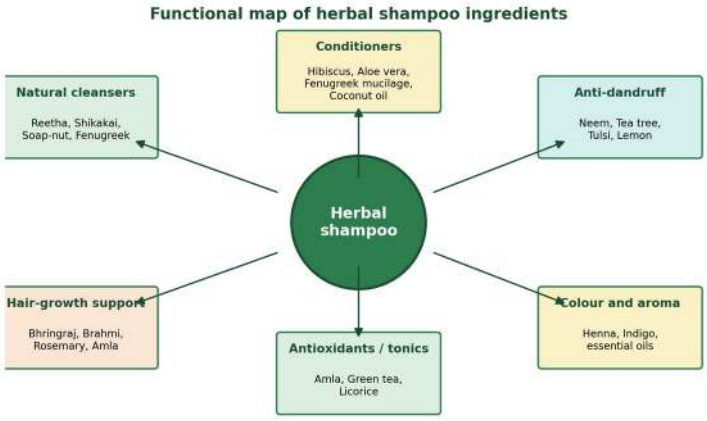


Figure 6: ***Functional map of herbal shampoo ingredients: cleansers, conditioners, anti-dandruff agents, hair-growth supporting plants, antioxidants and colour/aroma materials.***

6.1 Natural cleansers (saponin-containing plants)

Saponins are glycosides with a lipophilic aglycone (triterpenoid or steroid) and hydrophilic sugar chains, giving surface activity, foaming and emulsifying properties. **Reetha / soapnut** (*Sapindus mukorossi*) pericarp is among the richest sources and has been used for centuries as a hair and fabric cleanser. **Shikakai** (*Acacia concinna*) pods contain saponins and are valued for mild cleansing with a lower tendency to strip hair, and traditionally it is combined with reetha and amla. **Fenugreek** seeds contain steroidal saponins and mucilage and offer both cleansing and conditioning. Saponin solutions foam well but may be slightly alkaline and darken on storage; their pH typically requires adjustment for shampoo use [3,4,21].

6.2 Conditioners and mucilage providers

Hibiscus (*Hibiscus rosa-sinensis*) flowers and leaves contain mucilage, flavonoids and anthocyanins; the mucilage gives slip and detangling, and extracts have shown hair-growth-promoting activity in animal studies [19]. **Aloe vera** gel contains polysaccharides (acemannan), amino acids and vitamins that moisturise and soothe the scalp. **Coconut oil** rich in lauric acid is one of the few oils shown to reduce protein loss from hair when used before washing [12,13]. **Fenugreek**, **flaxseed** and **marshmallow root** mucilages also improve wet combing.

6.3 Anti-dandruff and antimicrobial plants

Neem (*Azadirachta indica*) leaf and seed extracts contain limonoids such as nimbin and azadirachtin with antifungal and antibacterial properties. **Tea-tree oil** (*Melaleuca alternifolia*) contains terpinen-4-ol with antifungal activity against *Malassezia* and is used at low concentrations because it may cause contact allergy when oxidised. **Tulsi** (*Ocimum sanctum*) and **lemon** (citric acid and flavonoids) are common adjuncts. **Licorice** (*Glycyrrhiza glabra*) glycyrrhizin and flavonoids show anti-inflammatory activity that may soothe the scalp [14,21,22].

6.4 Hair-growth supporting and tonic plants

Bhringraj (*Eclipta alba*) is traditionally the 'king of hair herbs'; the coumestan wedelolactone and other constituents have been reported to promote hair growth in animal models [20]. **Brahmi** (*Bacopa monnieri*) contains triterpenoid saponins (bacosides) and is used as a scalp tonic. **Rosemary** (*Rosmarinus officinalis*) oil and extract, rich in carnosic acid and 1,8-cineole, is a popular modern ingredient for scalp stimulation. **Amla** (*Phyllanthus emblica*) is rich in ascorbic acid and tannins, and in hydrolysable tannins with antioxidant activity. Most evidence is preclinical, so claims should be worded cautiously and supported by data [19-22].

6.5 Antioxidants, colour and fragrance

Green tea (*Camellia sinensis*) catechins such as epigallocatechin gallate (EGCG) offer antioxidant activity but are sensitive to oxidation and pH. **Henna**

(*Lawsonia inermis*) contains lawsone, which binds keratin and gives red-orange tint and some conditioning. **Indigo** (*Indigofera tinctoria*) is used with henna for darker shades. Essential oils (lavender, sandalwood, citrus) supply fragrance but also potential allergens, so their levels must follow regulatory and IFRA limits [22,27].

Table 5: ***Selected herbal materials used in shampoos.***

Common name / botanical name	Part used	Main constituents	Principal role
Reetha / *Sapindus mukorossi*	Pericarp	Triterpenoid saponins	Cleanser, foaming agent
Shikakai / *Acacia concinna*	Pods	Saponins, tannins	Mild cleanser, conditioner
Amla / *Phyllanthus emblica*	Fruit	Ascorbic acid, tannins, flavonoids	Antioxidant, conditioner
Hibiscus / *Hibiscus rosa-sinensis*	Flowers, leaves	Mucilage, flavonoids, anthocyanins	Conditioner, hair-growth support
Neem / *Azadirachta indica*	Leaves, seed oil	Nimbin, azadirachtin, flavonoids	Anti-dandruff, antimicrobial
Aloe vera / *Aloe barbadensis*	Leaf gel	Polysaccharides, amino acids	Moisturiser, soothing
Bhringraj / *Eclipta alba*	Whole herb, leaves	Wedelolactone, flavonoids	Hair-growth support, tonic
Brahmi / *Bacopa monnieri*	Aerial parts	Bacosides (saponins)	Scalp tonic
Fenugreek / *Trigonella foenum-graecum*	Seeds	Steroidal saponins, mucilage, protein	Cleanser, conditioner
Tea tree / *Melaleuca alternifolia*	Leaf oil	Terpinen-4-ol	Antifungal
Rosemary / *Rosmarinus officinalis*	Leaf extract, oil	Carnosic acid, 1,8-cineole	Scalp stimulation, antioxidant
Green tea / *Camellia sinensis*	Leaf	Catechins (EGCG)	Antioxidant
Henna / *Lawsonia inermis*	Leaf powder	Lawsone	Colour, conditioning
Licorice / *Glycyrrhiza glabra*	Root	Glycyrrhizin, flavonoids	Anti-inflammatory
Coconut / *Cocos nucifera*	Oil	Lauric acid, triglycerides	Pre-wash conditioning, emollient

6.6 Quality of herbal raw materials

Plant materials must be correctly identified and free from adulterants, contaminants and excess microbial load. Quality evaluation follows pharmacognostic principles: macroscopic and microscopic identification, determination of foreign matter, ash values, extractive values, moisture content, phytochemical fingerprinting (TLC, HPTLC, HPLC) and tests for heavy metals, pesticide residues, aflatoxins and microbial limits [28,29]. Good agricultural and collection practices (GACP) reduce variability at source [30].

CHAPTER 7

7. Extraction and Preparation of Herbal Actives

7.1 General scheme

Herbal actives are normally incorporated as aqueous or hydro-alcoholic extracts, powders, glycerites or oils. The general sequence (Figure 7) comprises authentication and collection, washing and drying, size reduction, extraction, filtration, concentration, and standardisation with quality control [16,17,18].

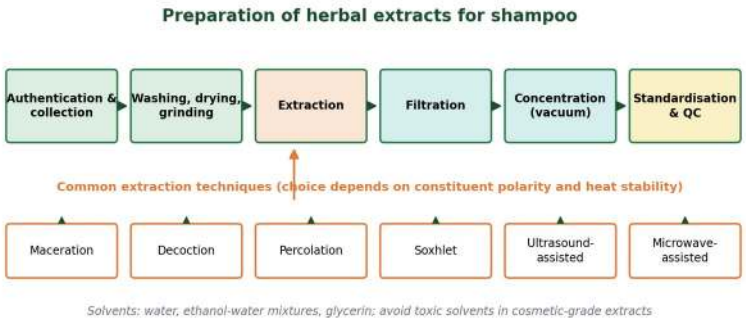


Figure 7: ***Preparation of herbal extracts for shampoo: processing sequence and common extraction techniques.***

7.2 Pre-processing

Fresh material is washed to remove soil, then dried in shade or at low temperature (typically below about 50 C for heat-sensitive constituents) to a moisture level that prevents mould growth. Drying and grinding to an appropriate particle size increase the surface area for extraction, but too fine a powder can hamper filtration. Dried material should be stored in airtight, light-protected containers.

7.3 Extraction techniques

- **Maceration:** soaking the powdered drug in solvent at room temperature for several days with occasional shaking; simple and suitable for thermolabile constituents, but slow.
- **Decoction and infusion:** extraction in boiling or hot water; the traditional method for saponin-rich and mucilaginous materials, and the most relevant to aqueous herbal shampoos.
- **Percolation:** continuous passage of fresh solvent through a packed bed; more complete extraction than maceration.
- **Soxhlet extraction:** continuous hot-solvent extraction, efficient but unsuitable for heat-labile compounds.
- **Ultrasound-assisted extraction (UAE):** cavitation disrupts cell walls, reducing time and temperature.
- **Microwave-assisted extraction (MAE):** rapid heating improves yield and shortens extraction time, requiring control to avoid degradation.
- **Supercritical CO2 extraction:** used for essential oils and lipophilic fractions without solvent residues, at higher capital cost.

The choice of solvent follows the 'like dissolves like' principle: water extracts saponins, mucilage and polar phenolics; ethanol-water mixtures extract flavonoids and tannins more fully; non-polar solvents or CO2 extract essential oils and lipids. For cosmetics, only solvents with acceptable residual limits (water, ethanol, glycerin, propylene glycol) are generally used [17,18].

7.4 Concentration, standardisation and preservation of extracts

Extracts are filtered and concentrated under reduced pressure at a low temperature, or spray- or freeze-dried to powders. Standardisation, meaning adjustment to a defined content of a marker compound (for example total saponins, total phenolics or a specific marker such as wedelolactone), reduces batch-to-batch variability and allows meaningful dosing in the final product [28]. Aqueous extracts are highly susceptible to microbial growth and need preservation or sterilisation, for example by preservative addition, glycerin or propylene glycol dilution, or filtration.

7.5 Critical factors affecting extract quality

Extract yield and composition depend on plant part, harvest time, drying conditions, solvent polarity, solvent-to-drug ratio, temperature, time, particle size and method. Each of these should be specified in a documented process to ensure reproducibility. Modern 'green' approaches (water-based, UAE, MAE) align with sustainability expectations for herbal cosmetics.

CHAPTER 8

8. Formulation Development of Herbal Shampoo

8.1 Pre-formulation considerations

Pre-formulation includes characterising the herbal extract (solubility, pH, colour, odour, saponin and phenolic content, microbial load, compatibility with other ingredients) and defining the product profile: hair type, form, claims, shelf life and target market. Compatibility screening is particularly important because saponins and polyphenols can interact with cationic conditioners (loss of foam or precipitation), with electrolytes (viscosity changes) and with metal ions (colour change).

8.2 Typical composition

A typical liquid herbal shampoo is mainly water, with roughly 10 to 20 percent active surfactant, a few percent of conditioning/foam-boosting agents, a viscosity-building system, a preservative system, pH adjuster and fragrance (Figure 8). Herbal extracts may be used at roughly 1 to 10 percent depending on concentration and claim, subject to stability and regulatory constraints [3,4,41]. Table 6 gives a representative formula, which must be adapted and validated experimentally.

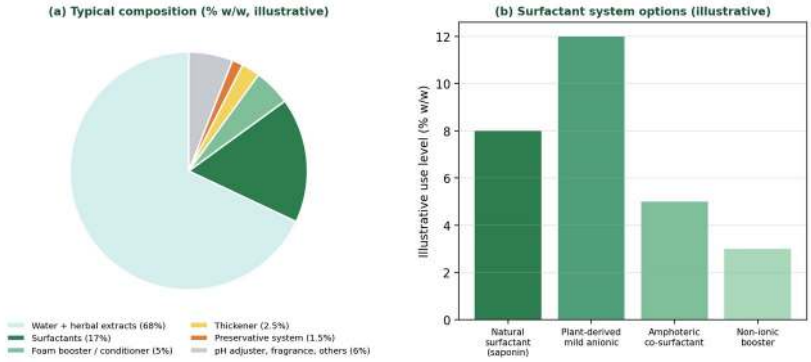


Figure 8: ***(a) Illustrative composition of a herbal shampoo; (b) options for the surfactant system. Values are indicative only.***

Table 6: ***Representative formulation of a liquid herbal shampoo (illustrative).***

Ingredient	Function	Approx. % w/w
Purified water	Vehicle	q.s. to 100
Herbal extract blend (reetha, shikakai, amla, hibiscus)	Cleansing, conditioning, antioxidant	5-10
Mild natural-origin surfactant (e.g., coco-glucoside / sodium cocoyl isethionate)	Primary cleanser	8-12
Amphoteric co-surfactant (e.g., cocamidopropyl betaine)	Foam, mildness	3-5
Glycerin	Humectant	1-3
Xanthan gum / guar derivative	Thickener	0.3-1.0
Aloe vera gel	Soothing agent	1-3
Essential oil (e.g., rosemary or tea tree)	Fragrance, activity	0.1-0.5
Preservative system (per regulation)	Microbial protection	as required
Citric acid / sodium citrate	pH adjustment, buffer	q.s. to pH 5-6.5

8.3 Selection of excipients

Surfactants

Fully saponin-based systems give a low viscosity and strong foam but pH and stability need control. Natural-origin surfactants such as alkyl polyglucosides, sodium cocoyl isethionate, sodium lauroyl sarcosinate and sodium cocoyl glutamate offer mildness and acceptable foam. Blending anionic and amphoteric/non-ionic surfactants enhances mildness through mixed-micelle formation and increases viscosity [23,26].

Thickeners and rheology modifiers

Natural polymers (xanthan gum, guar, hydroxypropyl guar, cellulose derivatives) provide structure and suspend particles. Electrolyte-based thickening of surfactant systems works at low salt levels but is less effective with natural-origin surfactants and highly loaded extracts.

Preservation

Water-rich herbal formulations favour microbial growth and are among the most demanding to preserve. Options include benzoate/sorbate systems at acidic pH, phenoxyethanol with ethylhexylglycerin, organic acids (levulinic, anisic) and glycols. The system must be validated by a preservative efficacy (challenge) test and comply with the permitted list of the target market. Essential oils and some extracts contribute some antimicrobial effect but are not sufficient alone [26,36].

pH and buffering

The target pH is generally between about 5 and 6.5, compatible with the scalp and hair. Saponin extracts tend to be alkaline and may require citric or lactic acid; buffering with citrate keeps the pH stable during storage [11].

Colour, fragrance and antioxidants

Plant extracts often darken through oxidation. Antioxidants (tocopherol, ascorbic acid), chelating agents and opaque packaging reduce colour change. Fragrance selection should consider allergen labelling and the stability of the essential oil in the base.

8.4 Optimisation using experimental design

Formulation variables (surfactant ratio, extract level, thickener level, pH) interact, so structured experimentation such as factorial or mixture designs provides an efficient route to optimise foam volume, viscosity, pH stability and sensory score simultaneously [4]. Regression models and response-surface plots can then identify a robust formulation region and support quality-by-design practices.

CHAPTER 9

9. Manufacturing Process and Scale-up

9.1 Process flow

Herbal shampoo is manufactured by batch mixing in jacketed stainless-steel vessels fitted with an agitator and, where required, a homogeniser. The principal stages are shown in Figure 9: raw-material dispensing, preparation of the herbal extract, dispersion of the thickener, heating of the water phase, addition of surfactants with gentle mixing to avoid air entrapment, cooling, addition of heat-sensitive actives, preservatives and fragrance, pH and viscosity adjustment, deaeration, filtration, in-process testing and filling [3,26].

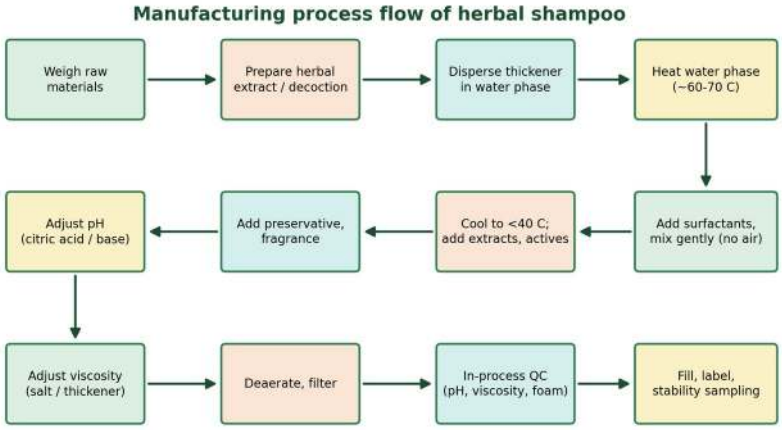


Figure 9: ***Manufacturing process flow for a herbal liquid shampoo from raw-material weighing to filling and stability sampling.***

9.2 Laboratory-scale procedure

1. Prepare the herbal extract or decoction and filter; adjust to the specified marker content.
2. Disperse the thickener in part of the water with stirring until fully hydrated.
3. Heat the water phase gently (about 60 to 70 C) if required, then add surfactants slowly with low-shear mixing.
4. Cool below about 40 C; add the herbal extracts, glycerin, aloe gel and other heat-sensitive materials.
5. Add the preservative system and fragrance; adjust the pH with citric acid or a base.
6. Adjust viscosity with a thickener, filter, check the in-process parameters and fill into containers.

9.3 Equipment and facility requirements

Typical equipment includes dispensing balances, extraction and concentration units, mixing tanks, homogeniser, transfer pumps, filter, filling and capping machines, and laboratory instruments for QC. Facility design follows cosmetic GMP according to ISO 22716 with controlled hygiene, cleaning validation, calibrated instruments, documentation and traceability [36]. Because botanical materials increase the microbial load, raw-material quarantine, supplier qualification and water-system control are particularly important.

9.4 Scale-up considerations

On scale-up, mixing time, shear rate, heat-transfer and the rate of ingredient addition change. Foaming and air entrapment become more significant in large tanks, and viscosity-building systems may behave differently. Scale-up is best carried out stepwise (laboratory to pilot to commercial) with geometric similarity where possible, constant tip speed or power per volume as scaling criteria, and verification of critical quality attributes at each stage.

9.5 Common manufacturing problems

Table 7: ***Common problems in herbal shampoo manufacture and corrective measures.***

Problem	Likely cause	Corrective action
Excess foam / air bubbles	High-speed mixing, adding surfactant too fast	Reduce shear, add surfactant slowly, deaerate under vacuum
Low viscosity	Low electrolyte or thickener, saponin-rich base	Add xanthan / guar, optimise salt, adjust surfactant ratio
Haziness or sediment	Insoluble plant matter, incompatible ingredients	Filter extracts, use solubiliser, adjust pH
Darkening on storage	Oxidation of polyphenols, high pH	Add antioxidant and chelator, lower pH, use opaque packaging
Microbial growth	Nutrient-rich extracts, weak preservation	Improve hygiene, use validated preservative system, adjust pH
Fragrance fading / off-odour	Volatile loss, oxidation	Use fixative, protect from heat and light, change fragrance

CHAPTER 10

10. Evaluation and Quality Control of Herbal Shampoo

Evaluation confirms that the shampoo meets its specifications for appearance, performance, safety and stability. Parameters can be grouped as shown in Figure 10 and are summarised in Table 8 [3,4,25,28].

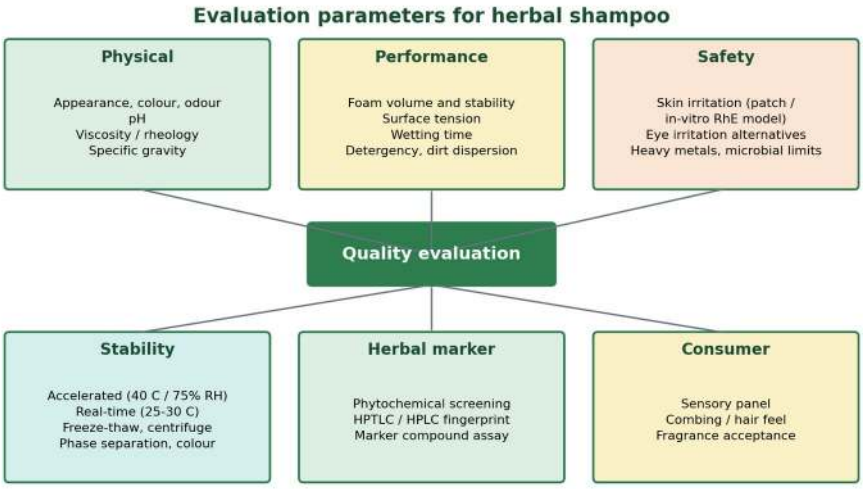


Figure 10: ***Groups of evaluation parameters for herbal shampoos: physical, performance, safety, stability, herbal marker and consumer tests.***

10.1 Organoleptic and physical evaluation

Colour, odour, clarity and homogeneity are assessed visually and by smell against a reference standard. The pH of a 10 percent aqueous solution is measured with a calibrated pH meter. Viscosity is measured with a rotational (Brookfield-type) viscometer at defined spindle and speed, and flow behaviour is typically shear-thinning. Specific gravity is determined with a pycnometer or density bottle. Solid or dry content and moisture content are determined by oven drying.

10.2 Performance tests

- **Foaming ability and foam stability:** the cylinder shake method (a shampoo solution is shaken a fixed number of times and the foam height recorded initially and after 1 to 5 minutes) or the standardised Ross-Miles apparatus [25].
- **Surface tension:** measured with a stalagmometer, du Nouy ring or Wilhelmy plate tensiometer; lower values indicate better wetting and cleansing.
- **Wetting time:** the time for a standard canvas disc to sink in the shampoo solution; shorter times indicate better wetting.
- **Dirt dispersion:** the ability to keep carbon black or soot suspended in the solution rather than depositing in the foam.
- **Detergency / cleaning action:** removal of standard soil or sebum from hair swatches or wool yarn, assessed gravimetrically or by colorimetry.
- **Conditioning performance:** wet and dry combing force, hair-tress softness and shine, and sensory panel scoring.

10.3 Safety evaluation

Safety tests include skin compatibility through human repeat-insult patch tests or in-vitro reconstructed human epidermis models (OECD TG 439) [33], and assessment of eye irritation potential using validated in-vitro methods where possible (for example OECD TG 437 and TG 492), while TG 405 describes the traditional in-vivo test [34]. Microbial limits (total aerobic count, yeasts and moulds, absence of specified organisms such as *Pseudomonas aeruginosa*, *Staphylococcus aureus*, *Candida albicans*), heavy metals and, for plant materials, pesticide residues are also controlled [29,32]. Ingredient-level safety evaluation follows the principles of the Scientific Committee on Consumer Safety notes of guidance [32].

10.4 Herbal-specific tests

Phytochemical screening (tests for saponins, flavonoids, tannins, phenolics), TLC/HPTLC fingerprinting, and HPLC quantification of marker compounds demonstrate that the declared herbal ingredients are present in the finished product and

remain stable. Antioxidant assays (DPPH, FRAP) and antimicrobial tests (agar diffusion, minimum inhibitory concentration against *Malassezia*, *Candida*, *Staphylococcus*) may be used to support claims, with the caution that in-vitro activity does not guarantee in-use efficacy [28].

Table 8: ***Evaluation parameters, methods and indicative acceptance criteria for a herbal shampoo.***

Parameter	Method	Indicative acceptance
Appearance, colour, odour	Visual / olfactory vs standard	Uniform, no separation, characteristic odour
pH (10% solution)	Calibrated pH meter at 25 C	About 5.0-7.0
Viscosity	Rotational viscometer	Within specified range (e.g., 2,000-8,000 mPa.s)
Foam volume / stability	Cylinder shake or Ross-Miles	Adequate initial height, stable over 5 min
Surface tension	Du Nouy ring / stalagmometer	Lower than water, consistent between batches
Wetting time	Canvas disc test	Shorter time preferred; compare with reference
Dirt dispersion	Carbon-black dispersion test	Low carbon in foam layer
Microbial limits	Plate count, specified organisms	Within pharmacopoeial / ISO cosmetic limits
Preservative efficacy	Challenge test	Meets recognised criteria
Marker compound content	HPLC / HPTLC	Within declared range
Skin irritation	Patch test / RhE model	Non-irritant or mild

10.5 Stability testing

Stability studies verify that physical, chemical and microbiological properties remain acceptable through shelf life. Samples in final packaging are stored at controlled conditions, for example long-term 25 C/60% RH or 30 C/65% RH, accelerated 40 C/75% RH, refrigerated 4 C and also exposed to freeze-thaw cycles and light, using the approach of ICH Q1A (R2) as a framework [40]. Appearance, colour, odour, pH, viscosity, foam, marker content and microbial quality are checked at intervals (for example 0, 1, 2, 3 and 6 months). Centrifugation (for example 3,000 rpm for 30 minutes) is a quick test for phase separation. Figure 11 illustrates typical outputs of such tests using simulated data.

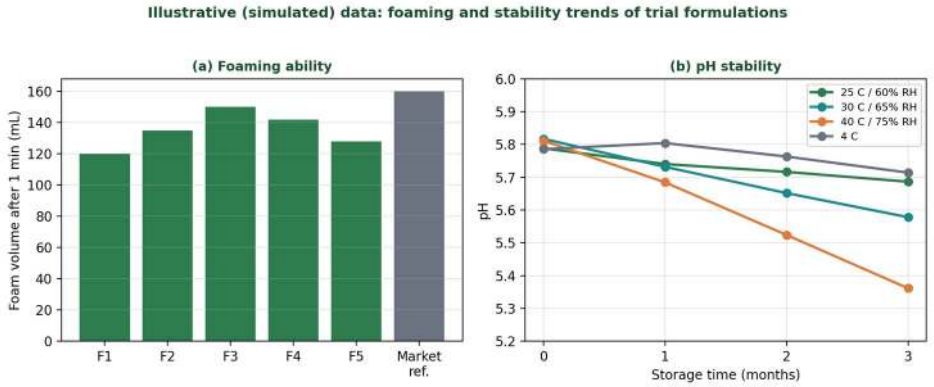


Figure 11: ***Illustrative (simulated) data: (a) foaming ability of trial formulations F1 to F5 compared with a market reference; (b) pH trend over three months at different storage conditions.***

CHAPTER 11

11. Safety, Toxicology and Clinical Evidence

11.1 Dermal safety of botanical ingredients

Botanical ingredients are complex mixtures, and each constituent may have its own toxicological profile. Contact allergy is the most common adverse reaction: essential oils (tea tree, lavender, citrus oils), some plant resins and fragrance terpenes can sensitise, especially when oxidised. Phototoxic furocoumarins occur in some citrus and umbelliferous plants. Henna-based products sometimes contain adulterants such as *p*-phenylenediamine, a strong sensitiser, which underlines the need for supplier control [21,22,32].

11.2 Contaminants in plant materials

Plants may accumulate heavy metals (lead, arsenic, cadmium, mercury) from soil and may carry pesticide residues, mycotoxins and microbial contamination. Limits should be specified for raw materials and finished products, and verified by validated analytical methods. Sourcing from controlled cultivation under GACP, certificate-of-analysis checks and periodic independent testing are recommended [28-30].

11.3 Eye and mucosal safety

Shampoo is likely to contact the eyes. Saponins can cause eye irritation at high concentrations, and traditional soapnut solutions are not tear-free. Formulation with amphoteric and non-ionic surfactants reduces ocular irritation; the finished product must be tested using validated methods [33,34].

11.4 Evidence for efficacy claims

The evidence base for herbal shampoo claims is heterogeneous. Foaming, detergency and conditioning claims can be substantiated by instrumental and sensory testing in vitro or on hair tresses. Antifungal and antioxidant activity can be supported by laboratory assays. Hair-growth claims for plants such as hibiscus and bhringraj rest mainly on animal and in-vitro studies [19,20]; well-designed human trials are scarce, and a rinse-off product has limited contact time with the follicle. Anti-dandruff claims should ideally be supported by clinical scoring of flaking and itching against a vehicle or reference product. Reviews of herbal cosmetics repeatedly call for standardised extracts and controlled studies [5,21,22].

11.5 Pre-wash oil treatments

Coconut oil applied before shampooing has been shown in laboratory studies to reduce protein loss from hair during washing, because its lauric acid, a small straight-chain triglyceride component, can penetrate the hair fibre, whereas mineral oil does not offer the same effect [12,13]. This supports the use of coconut oil in pre-wash or leave-on herbal hair care, and as a conditioning ingredient in shampoos, although the benefit from a rinse-off shampoo is smaller.

11.6 Safety assessment framework

1. Identify each ingredient and its plant part, extract type and composition.
2. Review the literature and regulatory restrictions (prohibited and restricted substances, concentration limits).
3. Evaluate toxicological endpoints: sensitisation, irritation, repeated-dose, genotoxicity, phototoxicity where relevant.
4. Calculate exposure for a rinse-off product and determine the margin of safety.
5. Perform safety testing on the finished formulation, including stability and challenge tests.
6. Document in a product safety report signed by a qualified safety assessor, as required in some jurisdictions.

CHAPTER 12

12. Regulatory Aspects and Standards

12.1 Overview

In most markets shampoos are regulated as cosmetics, not as drugs, unless therapeutic claims are made (for example 'treats dandruff', 'prevents hair loss'), in which case they may be regulated as drugs or over-the-counter products. Herbal shampoos are subject to the same general rules, with additional attention to botanical identity, contaminants and claim substantiation. The key frameworks are summarised in Table 9; readers should consult current versions and local requirements before commercialisation, since regulations change [35-39].

Table 9: ***Selected regulatory frameworks and standards relevant to herbal shampoos.***

Region / body	Instrument	Key points
European Union	Regulation (EC) No 1223/2009 on cosmetic products	Responsible person, product safety report, notification (CPNP), restricted / prohibited substance lists, labelling and allergen declaration [35]

Region / body	Instrument	Key points
United States	FD&C Act and the Modernization of Cosmetics Regulation Act of 2022 (MoCRA)	Facility registration, product listing, safety substantiation, adverse-event reporting, GMP requirements being developed [39]
India	Drugs and Cosmetics Act, 1940 and Rules, 1945; Bureau of Indian Standards standards for cosmetics	Manufacturing licence, ingredient and label rules, standards for specified cosmetics [38]
International	ISO 22716:2007	Good manufacturing practice guidelines for cosmetics [36]
International	ISO 16128-1:2016	Definitions and criteria for natural and organic cosmetic ingredients and products [37]
WHO	Quality control methods and GACP for medicinal plants	Quality of herbal raw materials [29,30]
OECD	Test guidelines	Standardised irritation and toxicity methods [33,34]

12.2 Labelling and claims

Labels must list ingredients according to the applicable nomenclature (INCI in many markets, with botanical names and extract types), declare mandatory allergens in fragrance, state batch number, net content, manufacturer details and precautions. Claims such as 'natural', 'herbal', 'organic' or 'free from' should be truthful and substantiated; ISO 16128 provides a technical basis for calculating natural-origin content [37]. Unsubstantiated therapeutic claims can cause the product to be treated as an unapproved drug.

12.3 Animal testing

Animal testing of cosmetic products and ingredients is banned in the European Union and several other regions, so safety substantiation relies on non-animal methods, existing data and human studies [32,35]. Formulators targeting multiple markets should plan their testing strategy accordingly.

CHAPTER 13

13. Market Trends, Sustainability and Packaging

13.1 Market trends

Natural, clean-label and ayurvedic products continue to gain a share of the hair-care market as consumers become more ingredient-conscious and look for sulfate-free, paraben-free and 'free-from' claims. Online sales, social media and personalised hair-care have accelerated product launches. At the same time, regulators and consumer groups are scrutinising 'greenwashing', where a product's marketing implies greater natural content than its formula justifies [5,37].

13.2 Sustainability of ingredients

Sustainability concerns include over-harvesting of wild plants, land and water use for cultivation, fair trade and biodiversity access and benefit-sharing (for example under the Nagoya Protocol). Cultivation under GACP, traceability and upcycling of agricultural by-products (for example fruit peels and seed cakes) can lower the environmental footprint. Biodegradability and aquatic toxicity of surfactants are also evaluated, noting that 'natural' does not guarantee a favourable environmental profile [5].

13.3 Waterless and solid formats

Shampoo bars, powders and concentrates reduce water content, transport weight and plastic packaging, and are easier to preserve because of low water activity. Traditional herbal powders (reetha, shikakai, amla) are naturally suited to this format, and modern syndet bars based on mild surfactants offer improved lather. Formulation challenges include hardness, solubility, hard-water compatibility and consumer familiarity.

13.4 Packaging

Packaging must protect the product from light, oxygen and microbial ingress, and must be compatible with the formulation. Recyclable, refillable and post-consumer-recycled plastics, glass and aluminium are being adopted. Stability studies must be run in the final pack, since herbal products are particularly sensitive to oxidation and light.

CHAPTER 14

14. Challenges and Future Perspectives

14.1 Current challenges

- **Standardisation:** variation in plant composition leads to variation in performance; marker-based specifications and fingerprinting remain unevenly applied.
- **Preservation:** safe, effective, natural-claim-compatible preservatives are limited.
- **Performance gap:** achieving foam, viscosity and conditioning equivalent to conventional shampoos with natural ingredients only.
- **Evidence:** few robust clinical studies; many claims depend on traditional use.
- **Regulatory heterogeneity:** definitions of 'natural' and permitted claims differ between countries.
- **Supply chain:** seasonal availability, adulteration and contamination risk.

14.2 Future directions



Figure 12: ***Conceptual roadmap for the future development of herbal shampoos, from current practice to personalised and circular formulations.***

- **Standardised and biotechnological actives:** cell-culture-derived plant actives, fermentation-derived saponins and biosurfactants (such as rhamnolipids and sophorolipids) offer consistent quality and lower land use.
- **Green extraction:** ultrasound, microwave, supercritical CO2 and natural deep eutectic solvents.
- **Microbiome-friendly products:** pre- and probiotic-supported formulations aiming to maintain scalp flora.
- **Data-driven development:** statistical design, machine learning and high-throughput screening to optimise complex natural formulations and predict stability.
- **Personalisation:** formulations tailored to hair type, scalp condition and climate using diagnostics.
- **Circular design:** upcycled raw materials, concentrates, refills and biodegradable packaging.
- **Rigorous clinical validation** of hair and scalp claims, including controlled dandruff and hair-fall trials.

CHAPTER 15

15. Mechanistic Insights into Herbal Actives

Understanding why herbal ingredients work helps in selecting them rationally and in designing claims that can be defended. Figure 13 summarises the principal constituent classes, their proposed mechanisms and the resulting benefits. Mechanistic evidence varies from well-established physical chemistry (saponins) to hypothesis-level (hair growth).

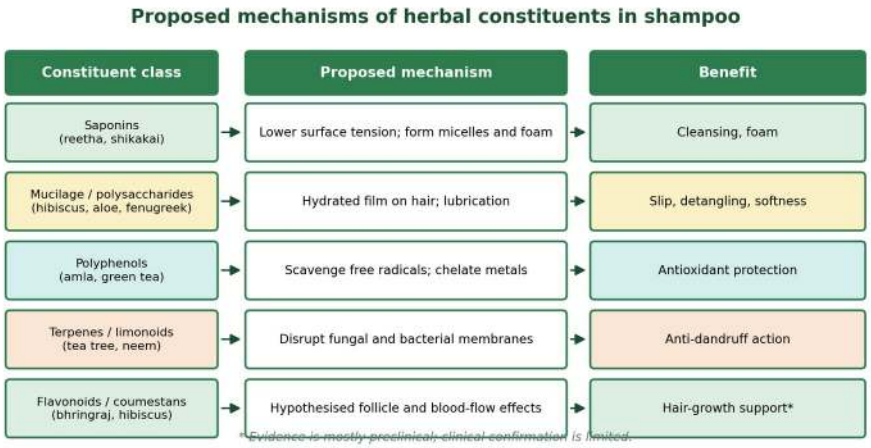


Figure 13: ***Constituent classes of herbal shampoo ingredients, proposed mechanisms of action and resulting benefits.***

15.1 Saponins: surface activity and cleansing

Saponins adsorb at the air-water and oil-water interfaces, lowering surface and interfacial tension and stabilising foam through a viscoelastic interfacial film. They form micelles at relatively higher concentrations than synthetic surfactants and are comparatively mild. Triterpenoid saponins from *Sapindus* and *Acacia* have been evaluated for foaming and detergency in natural shampoos, although performance varies between sources and pH [3,4,21]. Because saponin foam is often coarse and drains faster, it is typically combined with mild co-surfactants to meet consumer expectations.

15.2 Mucilage and polysaccharides: hydration and lubrication

Mucilages are branched, hydrophilic polysaccharides that swell in water to produce viscous gels. On hair they form a thin, hydrated film that lowers friction between fibres, which reduces tangling and improves wet combing. They also increase viscosity of the shampoo and help suspend particles. Acemannan in aloe vera and galactomannan in fenugreek are typical examples [21,22].

15.3 Polyphenols and antioxidant action

Tannins, flavonoids and catechins scavenge reactive oxygen species and can chelate transition metals, which may protect scalp lipids and hair proteins from oxidative damage by ultraviolet light and pollution. In formulation they also act as natural antioxidants protecting the product itself, although polyphenols can themselves oxidise and discolour. Their in-vitro antioxidant capacity (DPPH, FRAP) does not necessarily translate into benefit from a rinse-off product with short contact time [22].

15.4 Antimicrobial and anti-dandruff mechanisms

Terpinen-4-ol from tea-tree oil and other lipophilic terpenes partition into fungal membranes, increasing permeability and disrupting function. Neem constituents are reported to inhibit fungal growth and have anti-inflammatory activity. Because *Malassezia* species are lipid-dependent, reducing excess sebum by effective cleansing also decreases substrate for their growth. Clinical evidence exists for tea-tree oil shampoo in mild-to-moderate dandruff, but products need concentration and stability control because the oil oxidises [14,21].

15.5 Hair-growth hypotheses

Proposed mechanisms for hair-growth promotion include prolongation of the anagen phase, stimulation of dermal papilla cells and perifollicular blood flow, inhibition of 5-alpha-reductase and anti-inflammatory or antioxidant effects on the follicle. Hibiscus and *Eclipta alba* extracts increased the number of follicles in anagen or hair length in rodent models [19,20]. These findings are promising but preclinical, and the contact time of a shampoo is short; leave-on tonics or serums are more likely to deliver actives to the follicle than rinse-off products [1,6].

15.6 Hair-fibre interactions of colourants and oils

Lawsone from henna binds covalently to keratin amino groups, providing a semi-permanent colour that is slowly lost on repeated washing. Coconut oil, with its low molecular weight and high affinity for hair proteins, penetrates the cortex and reduces water uptake and swelling, which decreases hygral fatigue and protein loss [12,13].

Table 10: ***Strength of evidence for selected herbal claims in shampoos (qualitative summary).***

Claim	Typical ingredients	Nature of evidence	Confidence
Cleansing and foaming	Reetha, shikakai, fenugreek	Instrumental and in-vitro studies, long traditional use	Moderate to high
Conditioning, detan-gling	Hibiscus, aloe, mucilages, co-conut oil	Combing studies, in-vitro and sensory tests	Moderate
Antioxidant effect	Amla, green tea	In-vitro assays	Moderate (in-vitro only)
Anti-dandruff	Tea tree, neem	In-vitro antifungal data; limited clinical studies	Low to moderate
Hair-growth support	Bhringraj, hibiscus, rosemary	Animal and cell studies; few human trials	Low
Hair colour	Henna, indigo	Well-understood dye chemistry	High

CHAPTER 16

16. Practical Experimental Protocols

This chapter provides a practical, laboratory-level guide to preparing and evaluating trial herbal shampoos so that the concepts of Chapters 8 to 10 can be applied. All procedures must be adapted to local safety rules, and new formulations should be assessed by a qualified safety assessor before any human use.

16.1 Preparation of an aqueous herbal extract

1. Weigh equal parts of dried, powdered reetha peri-carp, shikakai pods and amla fruit (for example 10 g each).
2. Add about 300 mL purified water and heat gently (not boiling vigorously) for 30 to 45 minutes with stirring, then macerate overnight if desired.
3. Cool, filter through muslin and then filter paper, and record the volume, colour, odour and pH.
4. Determine the dry-residue content by evaporating a measured volume to constant weight.
5. Preserve the extract promptly (for example with a suitable preservative) and store refrigerated for no more than the validated period.

16.2 Preparation of trial formulations

Five trial formulations (F1 to F5) can be prepared by varying the extract level and surfactant system as in Table 11, following the manufacturing procedure in Chapter 9. Each batch of about 100 g is made in a beaker with an overhead stirrer, adjusted to pH 5.5 to 6.0 with citric acid solution, and stored in labelled, closed containers.

Table 11: ***Example design of five trial formulations (illustrative % w/w).***

Ingredient	F1	F2	F3	F4	F5
Herbal extract (reetha, shikakai, amla)	2	5	5	8	10
Coco-glucoside	10	10	8	8	6
Cocamidopropyl betaine	3	3	4	4	5
Xanthan gum	0.4	0.5	0.6	0.7	0.8
Glycerin	2	2	2	2	2
Aloe vera gel	1	1	2	2	3
Preservative system, fragrance	q.s.	q.s.	q.s.	q.s.	q.s.
Purified water	to 100	to 100	to 100	to 100	to 100

16.3 Evaluation protocols

pH and viscosity

Dilute 1 g of shampoo with 9 mL distilled water, equilibrate at 25 C and measure pH using a calibrated meter. Measure viscosity of the undiluted shampoo with a rotational viscometer using a defined spindle and speed (for example 20 rpm) after allowing the sample to rest and eliminating air bubbles; report in mPa.s.

Foaming ability (cylinder shake method)

Place 50 mL of a 1 percent shampoo solution in a 250 mL graduated cylinder, shake for 10 strokes, and record the total foam volume after 1 minute and after 5 minutes. Repeat in triplicate and report the mean and standard deviation. Foam stability is the percentage of the initial foam retained after 5 minutes.

Surface tension and wetting time

Measure the surface tension of a 1 percent solution with a stalagmometer or tensiometer at constant temperature. For wetting time, drop a standard canvas disc onto the surface of a 1 percent solution in a beaker and record the time taken for it to sink.

Dirt dispersion

Add 10 mL of a 1 percent shampoo solution and a drop of ink or about 0.1 g of carbon black to 10 mL of water in a test tube, shake ten times and observe the amount of colour in the foam; the less colour in the foam, the better the dirt dispersion.

Accelerated stability

Fill samples into the final container and store at 4 C, 25 C/60% RH and 40 C/75% RH. Evaluate colour, odour, pH, viscosity, foam height and microbial count at 0, 1, 2 and 3 months, and examine after three freeze-thaw cycles (for example 24 hours at -10 C then 24 hours at 25 C). A change of pH or viscosity beyond a predefined limit (for example 10 to 20 percent) or visible separation indicates instability [40].

Preservative efficacy (challenge) test

Inoculate separate portions of product with defined test organisms (bacteria, yeast and mould), sample at defined intervals (for example 7, 14 and 28 days) and enumerate survivors; the log reduction should meet the criteria of the chosen standard. This test must be performed by a trained microbiologist under suitable containment.

16.4 Data recording and interpretation

Results should be recorded in a controlled worksheet with batch number, date, analyst, instrument identification, raw readings and calculations. Statistical comparison of formulations (analysis of variance with post-hoc tests) allows selection of the best formulation, and the data can be combined into a desirability function to balance foam, viscosity, pH stability and sensory score. Deviations, out-of-range results and corrective actions should be documented to ensure data integrity.

CHAPTER 17

CONCLUSION

Herbal shampoos draw on a long tradition of plant-based hair care and are supported by a growing body of scientific knowledge on saponins, mucilages, essential oils and polyphenols. Plants such as reetha, shikakai, amla, hibiscus, neem and fenugreek can provide mild cleansing, conditioning, antimicrobial and antioxidant functions, while good formulation, appropriate preservation and standardised extracts are essential to achieve products that are stable, safe and pleasant to use.

Evaluation should combine physical, performance, safety, stability and herbal marker tests, and claims must be substantiated by suitable evidence. Regulatory compliance, safety assessment, sustainability and transparent labelling are as important as efficacy. Future progress will depend on standardised and biotechnologically produced actives, green extraction, improved natural preservation systems and well-designed clinical studies, so that herbal shampoos can combine tradition, science and sustainability.

CHAPTER 18

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