Formulation, Optimization and Evaluation of Herbal Shampoo Containing Selected Medicinal Plant Extracts: A Quality by Design (QbD) Approach

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***Abstract*—The present study aimed to formulate, optimize, and evaluate a polyherbal shampoo using medicinal plant extracts with proven hair care benefits. Herbal ingredients including Amla (Phyllanthus emblica), Reetha (Sapindus mukorossi), Shikakai (Acacia concinna), Hibiscus (Hibiscus rosa-sinensis), Aloe vera (Aloe barbadensis), Neem (Azadirachta indica), and Fenugreek (Trigonella foenum-graecum) were selected based on their traditional use and reported pharmacological activities. Hydroalcoholic extracts of the selected plants were prepared and incorporated into shampoo formulations. Six formulations (F1–F6) were developed by varying the concentrations of Reetha, Shikakai, and Aloe vera using a Design of Experiments (DoE) approach within the Quality by Design (QbD) framework. The prepared formulations were evaluated for physicochemical and performance characteristics including appearance, homogeneity, pH, solid content, surface tension, wetting time, foamability, foam stability, viscosity, detergency power, conditioning performance, skin irritation, and stability. All formulations exhibited acceptable physicochemical properties and remained stable throughout the study period. The results demonstrated that increasing the concentration of natural surfactants enhanced cleansing and foaming characteristics, while Aloe vera improved viscosity and conditioning effects. Among the tested formulations, F6 showed superior performance with the highest foam volume, detergency power, viscosity, and conditioning properties. No skin irritation or instability was observed in any formulation. The findings indicate that the optimized polyherbal shampoo is safe, effective, and capable of providing satisfactory cleansing and conditioning effects. The study supports the potential application of herbal ingredients as sustainable and consumer-friendly alternatives to synthetic shampoo formulations.**

***Index Terms*—Herbal shampoo, Polyherbal formulation, Reetha, Shikakai, Aloe vera, Quality by Design, Optimization.**

I. INTRODUCTION

1. Hair and Scalp Physiology

Hair is a specialized epidermal appendage that plays a crucial role in protection, thermoregulation, sensory perception, and aesthetics. Healthy hair is often regarded as an indicator of overall well-being and personal grooming. The condition of the hair is closely associated with the physiological state of the scalp, as the scalp provides the necessary environment for hair follicle development, growth, and maintenance. Understanding the structure of hair, the biological mechanisms governing hair growth, and the functions of the scalp is fundamental for the development of effective hair care products, including herbal shampoos.

Structure of Hair

Human hair is a keratinized filamentous structure that originates from hair follicles embedded within the dermal layer of the skin. Hair consists primarily of keratin proteins, water, lipids, trace elements, and pigments. The average human scalp contains approximately 80,000 to 150,000 hair follicles, with variations depending on genetic and ethnic factors.

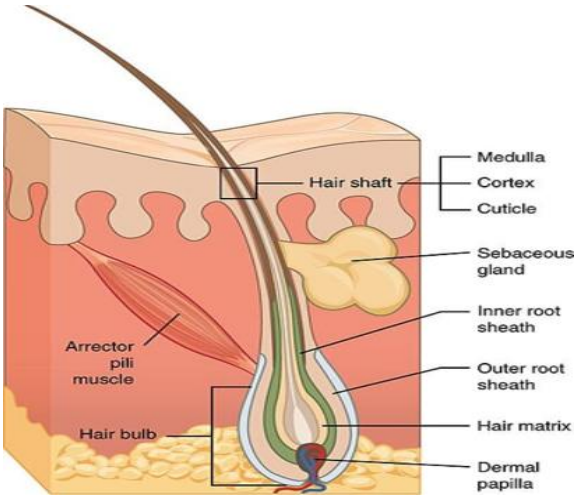

Fig.1 Structure of hair

Anatomical Components of Hair

Hair can be broadly divided into two major parts:

1. Hair Shaft

The hair shaft is the visible portion of the hair that extends above the skin surface. It is composed of dead, keratinized cells and consists of three distinct layers:

a) Cuticle: The cuticle is the outermost protective layer of the hair shaft. It comprises overlapping flattened cells arranged in a shingle-like pattern. These cells are rich in keratin and serve as a barrier against environmental damage, chemical treatments, ultraviolet radiation, and mechanical stress. The integrity of the cuticle determines the smoothness, shine, and manageability of the hair. Damage to the cuticle may lead to increased porosity, dryness, split ends, and reduced tensile strength.

b) Cortex: The cortex forms the major portion of the hair shaft and accounts for approximately 75–90% of its total mass. It contains elongated keratinized cells arranged in parallel bundles, providing strength, elasticity, and flexibility. The cortex also contains melanin granules responsible for hair color. Variations in melanin concentration and distribution contribute to differences in hair pigmentation among individuals.

c) Medulla: The medulla is the innermost core of the hair shaft. It consists of loosely arranged cells and air-filled spaces. Although its exact biological function remains unclear, the medulla may contribute to thermal insulation and structural support. The medulla is generally more prominent in thick hair and may be absent in fine hair fibers.

2. Hair Follicle

The hair follicle is a complex mini-organ located within the dermis and hypodermis. It is responsible for hair production and cyclical growth. The follicle consists of several specialized structures:

- Hair Bulb: The hair bulb is the enlarged base of the follicle where active cell division occurs. Matrix cells within the bulb proliferate rapidly and differentiate to form the hair shaft and inner root sheath.
- Dermal Papilla: The dermal papilla is a highly vascularized structure located at the base of the hair bulb. It supplies oxygen, nutrients, and signaling molecules necessary for follicular growth and development. Interactions between dermal papilla cells and epithelial cells regulate the hair growth cycle.
- Root Sheaths: The hair follicle contains both inner and outer root sheaths that provide structural support and guide the developing hair shaft toward the skin surface.
- Sebaceous Gland: Sebaceous glands are associated with hair follicles and secrete sebum, an oily substance composed of triglycerides, wax esters, and squalene. Sebum lubricates the hair and scalp, prevents excessive water loss, and contributes to the maintenance of scalp barrier function.
- Arrector Pili Muscle: This small smooth muscle is attached to the hair follicle and contracts in response to cold temperatures or emotional stimuli, causing hair to stand erect and producing the phenomenon commonly known as goosebumps.
- Hair Growth Cycle: Hair growth is not continuous but occurs through a highly regulated cyclical process consisting of growth, regression, rest, and shedding phases. This cycle ensures the renewal and maintenance of healthy hair throughout life. Various genetic, hormonal, nutritional, and environmental factors influence the duration and characteristics of each phase.
- Anagen Phase (Growth Phase): The anagen phase represents the active growth stage of the hair cycle. During this phase, matrix cells within the hair bulb undergo rapid mitotic activity, resulting in continuous hair shaft elongation. Approximately 85–90% of scalp hairs are normally in the anagen phase at any given time.

The duration of anagen varies significantly among individuals and can last between two and seven years. The length of this phase largely determines the maximum attainable hair length. Hair grows at an average rate of approximately 0.3–0.4 mm per day during anagen. Adequate nutrition, hormonal balance, and proper scalp health are essential for sustaining prolonged anagen activity.

Growth factors such as vascular endothelial growth factor (VEGF), insulin-like growth factor-1 (IGF-1), and fibroblast growth factors (FGFs) play important roles in promoting follicular proliferation and hair shaft production.

Catagen Phase (Regression Phase)

The catagen phase is a short transitional stage characterized by the cessation of active cell division. During this period, the lower portion of the follicle undergoes controlled involution through apoptosis-mediated processes.

The catagen phase typically lasts two to three weeks and involves structural remodeling of the follicle. The dermal papilla gradually separates from the hair matrix, leading to the termination of hair production. Only about 1–2% of scalp hairs are normally present in the catagen phase.

Telogen Phase (Resting Phase)

The telogen phase represents a period of relative inactivity during which hair growth ceases. The follicle remains dormant while the club hair remains anchored within the follicular canal. Approximately 10–15% of scalp hairs are in the telogen phase under normal physiological conditions.

This phase generally lasts about two to four months. Various stressors, including illness, nutritional deficiencies, hormonal disturbances, and psychological stress, can prematurely induce telogen entry, resulting in increased hair shedding.

Exogen Phase (Shedding Phase)

The exogen phase refers to the release and shedding of the mature club hair from the follicle. Hair shedding is a natural physiological process, and healthy individuals typically lose between 50 and 100 hairs per day. Following shedding, a new anagen hair begins to emerge from the same follicle, initiating another growth cycle.

Regulation of Hair Growth

Hair follicle cycling is regulated by a complex network of molecular signaling pathways. Key pathways include:

- Wnt/β-catenin signaling
- Sonic Hedgehog signaling
- Transforming Growth Factor-β (TGF-β)
- Bone Morphogenetic Proteins (BMPs)
- Fibroblast Growth Factors (FGFs)[20]

Hormones such as androgens, estrogens, thyroid hormones, and cortisol also influence hair growth dynamics. Dysregulation of these pathways may contribute to conditions such as androgenetic alopecia, alopecia areata, and telogen effluvium.

Functions of the Scalp

The scalp is a specialized anatomical structure composed of skin, connective tissue, blood vessels, nerves, hair follicles, sebaceous glands, and sweat glands. It serves as the primary environment supporting hair growth and maintenance. The health of the scalp directly affects hair quality, density, and appearance.

Protective Function

One of the primary functions of the scalp is protection. The scalp acts as a physical barrier against environmental insults, pathogens, ultraviolet radiation, and mechanical injury. The stratum corneum and associated lipid matrix prevent excessive water loss while limiting the penetration of harmful substances.

Support of Hair Follicles

The scalp provides structural and nutritional support for hair follicles. Rich vascular networks deliver oxygen, nutrients, hormones, and growth factors necessary for follicular metabolism and hair production. Adequate blood circulation is essential for maintaining active hair growth and preventing follicular miniaturization.

Sebum Production

Sebaceous glands present within the scalp produce sebum, which plays an important role in maintaining hair and scalp health. Sebum lubricates hair fibers, enhances flexibility, reduces friction, and prevents excessive dryness. It also possesses antimicrobial properties that contribute to scalp defense mechanisms.

Thermoregulation

The scalp contributes to thermoregulation through sweat gland activity and hair coverage. Sweat evaporation facilitates heat dissipation, while hair provides insulation against temperature fluctuations. This thermoregulatory function helps maintain optimal conditions for scalp tissues and hair follicles.

Sensory Function

The scalp contains numerous sensory receptors capable of detecting touch, pressure, pain, temperature, and vibration. Hair follicles themselves function as sensory units, allowing rapid perception of external stimuli. These receptors contribute to protective reflexes and environmental awareness.

Immunological Function

The scalp participates in immune surveillance through resident immune cells, including Langerhans cells, dendritic cells, macrophages, and T lymphocytes. These cells recognize and respond to microbial pathogens and foreign antigens, thereby protecting scalp tissues from infection and inflammation.

Maintenance of Microbiome Balance

The scalp hosts a diverse microbiome consisting of bacteria, fungi, and other microorganisms. Common microbial inhabitants include species of Cutibacterium, Staphylococcus, and Malassezia. Under normal conditions, these microorganisms coexist in a balanced ecosystem that contributes to skin health. Disturbances in microbial balance may result in dandruff, seborrheic dermatitis, and scalp irritation.

Contribution to Hair Appearance

Scalp health significantly influences the cosmetic properties of hair. Proper hydration, balanced sebum secretion, and an intact skin barrier promote hair shine, softness, and manageability. Conversely, scalp disorders can lead to dryness, excessive oiliness, itching, inflammation, and hair loss.

1.2 Shampoo

Hair cleansing has been practiced for centuries using a variety of natural and synthetic substances. The development of shampoos revolutionized hair care by providing an effective and convenient means of removing dirt, excess sebum, sweat, dead skin cells, environmental pollutants, and cosmetic residues from the hair and scalp. In modern cosmetic science, shampoos are considered one of the most widely used personal care products because they not only cleanse the hair but also improve its aesthetic appearance, texture, manageability, and overall health.

The term "shampoo" originates from the Hindi word chāmpo, meaning "to massage" or "knead." Historically, herbal preparations made from soapnuts, plant extracts, and aromatic herbs were employed for cleansing hair in many cultures. With advances in surfactant technology and cosmetic chemistry, modern shampoos have evolved into sophisticated formulations containing cleansing agents, conditioning ingredients, preservatives, fragrances, thickeners, and therapeutic compounds.

An ideal shampoo should effectively remove impurities without causing irritation to the scalp or damaging the hair shaft. It should generate adequate foam, possess acceptable viscosity and stability, maintain physiological scalp pH, and leave the hair soft, shiny, and manageable after washing. The increasing awareness regarding adverse effects associated with synthetic chemicals has stimulated interest in herbal and medicated shampoos that offer both cleansing and therapeutic benefits.

Definition of Shampoo

A shampoo can be defined as a cosmetic preparation designed primarily for cleansing the hair and scalp through the action of surfactants. It functions by emulsifying and removing sebum, dirt, particulate matter, microorganisms, and cosmetic residues accumulated on the scalp and hair fibers. Beyond cleansing, modern shampoos are formulated to provide additional benefits such as conditioning, moisturizing, strengthening, dandruff control, hair growth promotion, and scalp protection.

The effectiveness of a shampoo depends largely on its surfactant system. Surfactants reduce surface tension and facilitate the removal of oily substances through micelle formation. Besides surfactants, shampoos may contain conditioning polymers, botanical extracts, vitamins, proteins, fragrances, preservatives, pH adjusters, and active pharmaceutical ingredients depending on their intended use.

From a formulation perspective, shampoos are commonly classified based on their composition, mechanism of action, and intended therapeutic

purpose. The major categories include synthetic shampoos, herbal shampoos, and medicated shampoos.

1.2.1 Synthetic Shampoos

Synthetic shampoos are formulations that primarily contain chemically synthesized surfactants as cleansing agents. These products dominate the global shampoo market because they provide excellent detergency, superior foaming properties, long shelf life, and cost-effective manufacturing.

The principal cleansing activity of synthetic shampoos is derived from anionic surfactants such as sodium lauryl sulfate (SLS), sodium laureth sulfate (SLES), ammonium lauryl sulfate, and ammonium laureth sulfate. These surfactants effectively solubilize oils and particulate contaminants from the scalp and hair.

Composition of Synthetic Shampoos

Synthetic shampoos typically contain:

- Primary surfactants
- Secondary surfactants
- Foam boosters
- Conditioning agents
- Preservatives
- Chelating agents
- Fragrances
- Colorants
- pH modifiers
- Viscosity enhancers.

The primary surfactant system is responsible for cleansing, while conditioning agents such as silicones, quaternary ammonium compounds, and cationic polymers improve hair softness and reduce static electricity.

Advantages of Synthetic Shampoos

Synthetic shampoos possess several advantages:

- Efficient removal of dirt and sebum
- Rich foam production
- Excellent cleansing performance
- Consistent quality
- Long-term stability
- Ease of large-scale manufacturing.

The widespread availability and affordability of synthetic shampoos have contributed significantly to their commercial success. Modern formulations are often optimized to provide specialized functions such as volumizing, moisturizing, smoothing, color protection, and repair of damaged hair.

Limitations of Synthetic Shampoos

Despite their effectiveness, synthetic shampoos have several drawbacks. Frequent use of strong surfactants such as SLS may strip natural lipids from the scalp, resulting in dryness, irritation, itching, and increased hair fragility.

Certain preservatives, fragrances, and synthetic additives have been associated with allergic reactions and contact dermatitis in susceptible individuals. Environmental concerns have also emerged regarding the biodegradability and ecological impact of some synthetic ingredients.

Consumer demand for safer, eco-friendly, and naturally derived alternatives has therefore accelerated research into plant-based cleansing systems and herbal cosmetic formulations.

1.2.2 Herbal Shampoos

Herbal shampoos are cosmetic formulations prepared using plant-derived ingredients that provide cleansing, conditioning, nourishing, and therapeutic benefits to the hair and scalp. Unlike conventional synthetic shampoos, herbal shampoos emphasize the use of botanical extracts, natural surfactants, essential oils, and phytoconstituents with minimal reliance on harsh synthetic chemicals.

The growing popularity of herbal cosmetics is driven by increasing consumer preference for natural products, concerns regarding chemical toxicity, and the perception that herbal ingredients are safer and environmentally sustainable.

Historical Perspective

The use of herbs for hair care dates back thousands of years. Traditional systems of medicine such as Ayurveda have extensively employed plants including Amla (Phyllanthus emblica), Reetha (Sapindus mukorossi), Shikakai (Acacia concinna), Neem (Azadirachta indica), Aloe vera (Aloe barbadensis), and Hibiscus (Hibiscus rosa-sinensis) for cleansing and maintaining healthy hair.

These plants contain bioactive compounds such as saponins, flavonoids, alkaloids, tannins, terpenoids, vitamins, and antioxidants that contribute to scalp health and hair nourishment.

Composition of Herbal Shampoos

Herbal shampoos commonly contain:

- Plant extracts
- Natural surfactants
- Essential oils
- Natural conditioning agents
- Herbal preservatives
- Natural fragrances.

Among these ingredients, saponin-rich plants such as Reetha and Shikakai serve as natural cleansing agents. Saponins possess surface-active properties that facilitate dirt removal while being comparatively milder than many synthetic surfactants.

Benefits of Herbal Shampoos

Herbal shampoos offer numerous advantages:

- Gentle cleansing action
- Reduced scalp irritation
- Improved hair conditioning
- Antioxidant protection
- Promotion of hair growth
- Dandruff control
- Eco-friendly formulation.

Amla is rich in vitamin C and polyphenols that support follicular health and provide antioxidant protection. Neem exhibits antimicrobial activity against scalp pathogens, while Aloe vera contributes moisturizing and soothing effects. Hibiscus has been reported to improve hair strength and reduce hair breakage.

Challenges Associated with Herbal Shampoos

Although herbal shampoos provide several benefits, they also present formulation challenges. Variability in plant composition, limited foam generation, microbial stability concerns, and shorter shelf life may affect product performance. Standardization of herbal raw materials is therefore essential to ensure consistency and reproducibility.

Advances in phytochemical characterization, extraction technology, and formulation optimization have significantly improved the quality and efficacy of modern herbal shampoos.

1.2.3 Medicated Shampoos

Medicated shampoos are specialized formulations designed not only to cleanse the hair and scalp but also to treat specific dermatological conditions. These products contain active pharmaceutical ingredients (APIs) capable of preventing or managing scalp disorders such as dandruff, seborrheic dermatitis, psoriasis, fungal infections, and scalp inflammation.

Unlike cosmetic shampoos, medicated shampoos are often classified as therapeutic products because their primary function extends beyond cosmetic cleansing.

Therapeutic Applications

Medicated shampoos are widely used in the management of:

- Dandruff
- Seborrheic dermatitis
- Psoriasis
- Tinea capitis
- Scalp folliculitis
- Excessive scalp oiliness.

Common Active Ingredients

Several pharmacologically active substances are incorporated into medicated shampoos:

Ketoconazole: Ketoconazole is an antifungal agent effective against Malassezia species implicated in dandruff and seborrheic dermatitis.

Zinc Pyrithione: Zinc pyrithione exhibits antifungal, antibacterial, and anti-inflammatory properties. It remains one of the most commonly used anti-dandruff agents worldwide.

Selenium Sulfide: Selenium sulfide reduces fungal proliferation and decreases epidermal cell turnover associated with dandruff formation.

Salicylic Acid: Salicylic acid acts as a keratolytic agent that promotes the removal of scales and dead skin cells from the scalp.

Coal Tar: Coal tar helps slow excessive epidermal proliferation and is frequently used in psoriasis management.

Advantages of Medicated Shampoos

The primary advantage of medicated shampoos lies in their ability to simultaneously cleanse and treat scalp disorders. Regular use can reduce itching, inflammation, flaking, microbial colonization, and discomfort associated with scalp diseases.

Limitations of Medicated Shampoos

Long-term use of medicated shampoos may be associated with side effects such as scalp dryness, irritation, unpleasant odor, discoloration of hair, or

reduced patient compliance. Additionally, certain therapeutic agents require careful monitoring to avoid adverse reactions.

Recent research has focused on integrating herbal extracts with therapeutic ingredients to develop multifunctional formulations that combine efficacy with improved safety and patient acceptance.

1.3 Advantages of Herbal Shampoos

Herbal shampoos have gained considerable attention in recent years due to the increasing demand for natural, safe, and environmentally friendly hair care products. Unlike conventional shampoos that rely heavily on synthetic surfactants and chemical additives, herbal shampoos utilize plant-derived ingredients rich in bioactive phytoconstituents that provide cleansing, conditioning, and therapeutic benefits.

The major advantages of herbal shampoos are summarized below.

1. Reduced Risk of Scalp Irritation: Herbal ingredients are generally milder than synthetic detergents and are less likely to cause irritation, itching, or redness of the scalp.
2. Lower Incidence of Allergic Reactions: Plant-based formulations contain fewer harsh chemicals, reducing the chances of contact dermatitis and hypersensitivity reactions.
3. Free from Harsh Synthetic Chemicals: Most herbal shampoos avoid excessive use of sulfates, parabens, and artificial colorants that may damage hair and scalp health.
4. Suitable for Frequent Use: Due to their gentle cleansing action, herbal shampoos can be used regularly without excessive stripping of natural scalp oils.
5. Compatibility with Hair and Scalp Physiology: Herbal ingredients work harmoniously with the natural structure and function of hair and scalp tissues, minimizing adverse effects.
6. Maintenance of Natural Scalp pH: Many herbal formulations help preserve the physiological pH of the scalp, supporting barrier integrity and microbial balance.
7. Biodegradable and Eco-Friendly: Plant-derived ingredients are generally biodegradable and exert less environmental impact compared to synthetic surfactants.
8. Improves Hair Softness and Smoothness: Herbal constituents such as Aloe vera, Hibiscus, and Fenugreek naturally condition hair fibers and improve texture.
9. Enhances Hair Strength and Shine: Phytochemicals including flavonoids, vitamins, and antioxidants nourish hair shafts, resulting in stronger and shinier hair.
10. Supports Healthy Hair Growth: Many medicinal plants used in herbal shampoos promote scalp circulation, nourish hair follicles, and help reduce hair fall.

1.4 Medicinal Plants Used in Herbal Shampoo

Amla (Phyllanthus emblica Linn.)

Amla, commonly known as Indian gooseberry, is one of the most widely utilized medicinal plants in herbal hair care formulations. The biological source of Amla consists of the fresh or dried fruits of Phyllanthus emblica Linn. belonging to the family Phyllanthaceae. The fruits are rich in vitamin C, gallic acid, ellagic acid, emblicanin A, emblicanin B, flavonoids, tannins, and polyphenolic compounds. These bioactive constituents exhibit potent antioxidant activity that protects hair follicles from oxidative stress and premature aging. In herbal shampoos, Amla is incorporated as a natural hair tonic to strengthen hair roots, reduce hair fall, improve hair pigmentation, and enhance hair growth. Furthermore, its antioxidant and antimicrobial properties contribute to maintaining scalp health and preventing dandruff formation.

Reetha (Sapindus mukorossi Gaertn.)

Reetha, commonly referred to as soapnut, is obtained from the dried pericarp of the fruits of Sapindus mukorossi Gaertn., a member of the family Sapindaceae. The fruit contains high concentrations of triterpenoid saponins, sugars, mucilage, flavonoids, and fatty acids. Saponins possess excellent surface-active properties and function as natural surfactants capable of producing foam and removing dirt, oil, and impurities from the scalp and hair. Because of its cleansing efficiency, Reetha serves as a primary ingredient in many herbal shampoo formulations. Unlike synthetic detergents, Reetha provides gentle cleansing while preserving the natural lipid balance of the scalp. It also exhibits antimicrobial activity that assists in controlling scalp infections and dandruff.

Shikakai (Acacia concinna (Willd.) DC.)
Shikakai is derived from the dried pods, leaves, and bark of Acacia concinna (Willd.) DC., belonging to the family Fabaceae. It is a traditional Ayurvedic hair care ingredient extensively used for cleansing and conditioning hair. The plant contains saponins, alkaloids, tannins, flavonoids, and organic acids that contribute to its cleansing and hair-strengthening properties. Shikakai acts as a mild natural detergent that effectively removes dirt without excessively stripping the scalp of its natural oils. It helps maintain the physiological pH of the scalp, reduces hair breakage, improves hair texture, and imparts softness and shine. Due to its compatibility with hair fibers, Shikakai is frequently combined with Reetha and Amla in polyherbal shampoo formulations.

Hibiscus (Hibiscus rosa-sinensis Linn.)
Hibiscus is obtained from the flowers and leaves of Hibiscus rosa-sinensis Linn., a member of the family Malvaceae. The plant contains anthocyanins, flavonoids, mucilage, polyphenols, vitamins, and amino acids that contribute to its medicinal and cosmetic properties. Hibiscus has traditionally been used as a natural hair conditioner and hair growth promoter. The mucilage present in the flowers provides a conditioning effect that improves hair softness, smoothness, and manageability. In herbal shampoos, Hibiscus helps strengthen hair shafts, reduce split ends, prevent hair breakage, and enhance overall hair appearance. Additionally, its antioxidant properties protect hair follicles from oxidative damage and support healthy scalp function.

Aloe vera (Aloe barbadensis Miller)
Aloe vera, scientifically known as Aloe barbadensis Miller and belonging to the family Asphodelaceae, is one of the most important medicinal plants used in cosmetic formulations. The biological source consists of the mucilaginous gel obtained from the leaves. Aloe vera contains polysaccharides, glucomannans, vitamins, amino acids, minerals, enzymes, and phenolic compounds. These constituents provide moisturizing, anti-inflammatory, antimicrobial, and wound-healing activities. In herbal shampoo formulations, Aloe vera functions as a natural conditioner that hydrates both the scalp and hair fibers, reduces dryness, alleviates scalp irritation, and improves hair texture. Its soothing properties make it particularly beneficial for sensitive scalps, while its antioxidant activity helps maintain follicular health and support hair growth.

Neem (Azadirachta indica A. Juss.)
Neem is obtained from the leaves, bark, seeds, and fruits of Azadirachta indica A. Juss., belonging to the family Meliaceae. The plant is rich in limonoids, azadirachtin, nimbin, nimbidin, quercetin, flavonoids, and various terpenoids. Neem possesses remarkable antimicrobial, antifungal, anti-inflammatory, and antioxidant properties. In herbal shampoos, Neem is primarily incorporated for its ability to control dandruff and other scalp infections caused by microbial growth. It helps reduce itching, irritation, and inflammation while maintaining scalp hygiene. Regular use of Neem-containing shampoos may also contribute to improved scalp health and reduced hair fall associated with microbial or inflammatory scalp conditions.

Fenugreek (Trigonella foenum-graecum Linn.)
Fenugreek is obtained from the dried ripe seeds of Trigonella foenum-graecum Linn., a member of the family Fabaceae. The seeds contain proteins, amino acids, mucilage, saponins, alkaloids, flavonoids, vitamins, and minerals. Fenugreek is recognized for its nourishing and strengthening effects on hair. The high protein content helps repair damaged hair shafts and improve hair strength, while mucilage provides natural conditioning and detangling properties. In herbal shampoo formulations, Fenugreek is used to reduce hair breakage, improve hair texture, enhance shine, and support healthy hair growth. Furthermore, its anti-inflammatory and antioxidant activities contribute to maintaining a healthy scalp environment conducive to follicular function.

1.5 Need for Optimization
The development of an effective herbal shampoo involves the careful selection and adjustment of numerous formulation parameters to achieve the desired quality, safety, stability, and consumer acceptability. Unlike conventional synthetic products, herbal formulations contain plant-derived ingredients whose chemical composition may vary due to differences in geographical origin, harvesting season, extraction method, and storage conditions. Such variability can significantly influence the performance

of the final product. Therefore, optimization is an essential step in herbal shampoo development to ensure consistent product quality and reproducible performance.

Formulation Variables

The quality and efficacy of a herbal shampoo are strongly influenced by various formulation variables. These variables include the concentration of herbal extracts, natural surfactants, conditioning agents, thickeners, preservatives, and pH-adjusting agents. For example, increasing the concentration of saponin-rich ingredients such as Reetha may improve cleansing and foaming properties but may also affect viscosity and product stability. Similarly, excessive amounts of conditioning agents can increase product thickness and reduce cleansing efficiency. Other important formulation variables include extraction yield, solid content, mixing speed, mixing time, temperature during preparation, and storage conditions. Since these factors interact with one another, changes in a single variable may influence multiple quality attributes simultaneously. Consequently, systematic optimization is required to identify the most suitable combination of ingredients and processing conditions for obtaining a stable and effective herbal shampoo.

Quality by Design (QbD)

Quality by Design (QbD) is a scientific and risk-based approach to product development that emphasizes building quality into a formulation rather than relying solely on end-product testing. The concept was introduced to improve product understanding, process control, and manufacturing consistency. In herbal shampoo formulation, QbD begins with defining the Quality Target Product Profile (QTPP), which describes the desired characteristics of the final product, such as acceptable pH, viscosity, foamability, detergency, stability, and consumer acceptability.

After establishing the QTPP, critical quality attributes (CQAs) are identified. These attributes represent measurable properties that directly affect product performance and quality. Critical material attributes (CMAs), such as the concentration and quality of herbal extracts, and critical process parameters (CPPs), such as mixing speed and processing temperature, are then evaluated for their impact on CQAs. Through systematic risk assessment and scientific experimentation, QbD helps researchers understand the relationship between formulation components and product quality. This approach minimizes batch-to-batch variation, reduces development time, improves manufacturing efficiency, and ensures consistent performance of the herbal shampoo.

Design of Experiments (DoE)

Design of Experiments (DoE) is a statistical tool widely employed within the QbD framework to optimize pharmaceutical and cosmetic formulations. Instead of changing one factor at a time, DoE evaluates the effects of multiple variables simultaneously and identifies interactions between them. This approach provides a comprehensive understanding of how formulation variables influence product characteristics. In herbal shampoo development, DoE can be used to optimize factors such as the concentration of Reetha, Shikakai, Aloe vera, or other herbal ingredients while assessing responses including foam volume, viscosity, pH, detergency power, and conditioning effect. Experimental designs such as Factorial Design, Central Composite Design (CCD), and Box–Behnken Design (BBD) are commonly employed for this purpose. The generated mathematical models help predict the behavior of the formulation and identify the optimal combination of variables that produces the desired product characteristics. Therefore, the integration of formulation optimization, QbD principles, and DoE techniques provides a robust scientific strategy for developing high-quality herbal shampoos with improved performance, stability, safety, and consumer acceptance.

II. MATERIALS AND METHODS

2.1 Materials

Table 1

Ingredient	Biological Source	Function
Amla Extract	Fruits	Hair growth promoter
Reetha Extract	Fruits	Natural surfactant
Shikakai Extract	Pods	Cleansing agent
Aloe vera Gel	Leaves	Conditioner
Hibiscus Extract	Flowers	Hair strengthening

2.2 Collection and Authentication of Plant Materials

The medicinal plants selected for the preparation of the herbal shampoo were Amla (Phyllanthus emblica

Linn.), Reetha (Sapindus mukorossi Gaertn.), Shikakai (Acacia concinna (Willd.) DC.), Hibiscus (Hibiscus rosa-sinensis Linn.), Aloe vera (Aloe barbadensis Miller), Neem (Azadirachta indica A. Juss.), and Fenugreek (Trigonella foenum-graecum Linn.). The plant materials were collected during their appropriate harvesting season from local herbal markets and authenticated medicinal plant suppliers in Maharashtra, India. Fresh Aloe vera leaves and Hibiscus flowers were collected from cultivated plants, whereas dried fruits, pods, leaves, and seeds of the remaining plants were procured from certified herbal vendors. The collected materials were thoroughly examined for the absence of fungal contamination, insect infestation, and physical impurities. Foreign matter such as dust, soil particles, damaged plant parts, and extraneous materials was removed manually. The plant materials were washed with distilled water when necessary and allowed to dry before further processing. Proper collection and handling of plant materials are essential to maintain phytochemical integrity and ensure the reproducibility of herbal formulations.

Authentication of Plant Materials

All plant materials were authenticated by a qualified taxonomist from the Department of Botany, [Name of University/Research Institute], Maharashtra, India. The authentication was carried out based on morphological and taxonomic characteristics described in standard floras and pharmacogenetic literature. Voucher specimens of each plant material were prepared, assigned voucher numbers, and deposited in the departmental herbarium for future reference.

The authenticated plant materials were then stored in clean, airtight polyethylene containers at room temperature until extraction. Authentication ensures the identity, purity, and quality of herbal raw materials and minimizes the possibility of adulteration or substitution.

Table 2. Authentication Details of Selected Medicinal Plants

Sr. No.	Plant Name	Biological Source	Family	Plant Part Used	Voucher No.
1	Amla	Phyllanthus emblica Linn.	Phyllanthaceae	Fruit	PE-001
2	Reetha	Sapindus mukorossi Gaertn.	Sapindaceae	Fruit Pericarp	SM-002
3	Shikakai	Acacia concinna (Willd.) DC.	Fabaceae	Pods	AC-003
4	Hibiscus	Hibiscus rosa-sinensis Linn.	Malvaceae	Flowers	HR-004
5	Aloe vera	Aloe barbadensis Miller	Asphodelaceae	Leaves	AB-005
6	Neem	Azadirachta indica A. Juss.	Meliaceae	Leaves	AI-006
7	Fenugreek	Trigonella foenum-graecum Linn.	Fabaceae	Seeds	TF-007

2.3 Preparation of Herbal Extracts

The preparation of herbal extracts was carried out using standardized extraction procedures to obtain phytoconstituent-rich extracts suitable for incorporation into the herbal shampoo formulation. Extraction was performed individually for each medicinal plant to ensure maximum recovery of active constituents.

2.3.1 Drying

Freshly collected plant materials were washed thoroughly with distilled water to remove adhering dirt and contaminants. The materials were shade-dried at room temperature (25–30°C) for 10–15 days under adequate ventilation. Direct sunlight was avoided to prevent degradation of heat-sensitive and photosensitive phytoconstituents such as flavonoids, vitamins, and phenolic compounds.

Aloe vera leaves were processed separately. The outer leaf rind was removed, and the inner gel was collected and stored under refrigerated conditions prior to extraction. Complete drying was confirmed when the materials attained constant weight and became brittle enough for grinding.

2.3.2 Pulverization

The dried plant materials were reduced to coarse powder using a mechanical grinder. The powdered materials were passed through sieve No. 40 to obtain uniform particle size distribution. The powdered samples were stored separately in airtight containers protected from moisture, light, and microbial contamination until extraction.

Particle size reduction increases the surface area available for solvent penetration, thereby enhancing

extraction efficiency and improving the yield of phytoconstituents.

2.3.3 Extraction Methods

A. Maceration Method

Maceration was employed for extracting thermolabile constituents from selected plant materials. Approximately 100 g of powdered plant material was placed in a clean glass container and soaked in 70% hydroalcoholic solvent (ethanol:water, 70:30 v/v) at a ratio of 1:10 (w/v). The mixture was allowed to stand for 72 hours at room temperature with intermittent shaking every 6 hours to facilitate solvent penetration and extraction.

After completion of the extraction period, the mixture was filtered through muslin cloth followed by Whatman No. 1 filter paper. The filtrate was concentrated under reduced pressure using a rotary evaporator and subsequently dried in a vacuum desiccator. The dried extract was collected, weighed, and stored in airtight containers until further use.

B. Soxhlet Extraction Method

Soxhlet extraction was performed for exhaustive extraction of phytoconstituents from dried plant materials. Approximately 100 g of powdered drug was packed into a thimble and placed in the Soxhlet apparatus. Hydroalcoholic solvent (70% ethanol) was used as the extraction solvent.

The extraction process was continued for 6–8 hours until the siphon tube solvent became colorless, indicating complete extraction. The extract was then concentrated using a rotary vacuum evaporator and dried to obtain a semisolid mass. The dried extract was weighed and preserved in airtight containers for further studies. Soxhlet extraction offers higher extraction efficiency and improved recovery of active constituents compared to conventional extraction methods.

C. Hydroalcoholic Extraction

Hydroalcoholic extraction was selected because a mixture of ethanol and water can effectively extract both polar and moderately non-polar phytoconstituents. The powdered plant material (100 g) was extracted using ethanol:water (70:30 v/v) either by maceration or Soxhlet extraction depending on the nature of the plant material.

The obtained extracts were filtered and concentrated under reduced pressure at temperatures below 50°C to prevent degradation of thermolabile compounds. The concentrated extracts were further dried to constant weight and stored at 4°C until formulation studies. Hydroalcoholic extraction is widely employed in herbal formulation development due to its ability to maximize phytochemical recovery and maintain biological activity.

Determination of Percentage Yield of Extracts

The dried extracts obtained from each medicinal plant were weighed accurately and the percentage yield was calculated using the following equation:

$$\text{Percentage Yield (\%)} = \frac{\text{Weight of Dried Extract}}{\text{Weight of Crude Drug}} \times 100$$

Table 3. Percentage Yield of Hydroalcoholic Extracts

Sr. No.	Plant Material	Weight of Crude Drug (g)	Weight of Dried Extract (g)	Percentage Yield (%)
1	Amla (P. emblica)	100	24.5	24.5 ± 0.8
2	Reetha (S. mukorossi)	100	21.8	21.8 ± 0.6
3	Shikakai (A. concinna)	100	18.6	18.6 ± 0.5
4	Hibiscus (H. rosa-sinensis)	100	16.4	16.4 ± 0.7
5	Aloe vera (A. barbadensis)	100	28.7	28.7 ± 0.9
6	Neem (A. indica)	100	19.2	19.2 ± 0.4
7	Fenugreek (T. foenum-graecum)	100	22.1	22.1 ± 0.5

Values are expressed as Mean ± SD (n = 3).

Interpretation

Among the selected medicinal plants, Aloe vera exhibited the highest extractive yield (28.7%), which may be attributed to the presence of abundant polysaccharides and water-soluble constituents. Amla showed a yield of 24.5%, indicating a high concentration of extractable tannins and polyphenols. Hibiscus demonstrated the lowest extraction yield (16.4%), whereas Reetha, Shikakai, Neem, and Fenugreek showed moderate extractive yields ranging from 18–22%. The observed extraction yields indicate that the hydroalcoholic solvent system was effective in

recovering a wide spectrum of phytochemicals required for herbal shampoo formulation.

III. FORMULATION DEVELOPMENT

The formulation of a herbal shampoo requires the careful selection of ingredients capable of providing cleansing, conditioning, foaming, stability, and therapeutic benefits while maintaining compatibility with the scalp and hair. The present formulation was designed using scientifically validated medicinal plants known for their hair care properties. A combination of natural surfactants, conditioning agents, preservatives, and viscosity enhancers was employed to develop a stable and effective herbal shampoo. The formulation strategy was based on previously reported studies demonstrating the beneficial effects of herbal extracts in hair cleansing, conditioning, dandruff control, and hair growth promotion.

3.1 Selection of Ingredients

The selection of ingredients was carried out based on their traditional use, scientific evidence, phytochemical composition, and compatibility within shampoo formulations. Each ingredient was chosen to perform a specific function while contributing to the overall efficacy of the product.

3.2 Formulation Design

A series of six formulations (F1–F6) were prepared to optimize the concentration of natural surfactants and conditioning agents. The concentrations of Reetha, Shikakai, and Aloe vera were varied while keeping the concentrations of other ingredients constant. This approach enabled evaluation of the influence of formulation variables on shampoo performance parameters such as foamability, viscosity, detergency, pH, and conditioning effect.

Table 4. Formulation Composition of Herbal Shampoo (F1–F6)

Ingredients (% w/v)	F1	F2	F3	F4	F5	F6
Amla Extract	5	5	5	5	5	5
Reetha Extract	8	10	12	8	10	12
Shikakai Extract	4	4	4	6	6	6
Hibiscus Extract	3	3	3	3	3	3
Aloe vera Gel	5	7	9	5	7	9
Neem Extract	2	2	2	2	2	2
Fenugreek Extract	2	2	2	2	2	2
Xanthan Gum	1	1	1	1	1	1
Methyl Paraben	0.2	0.2	0.2	0.2	0.2	0.2
Rose Oil	0.5	0.5	0.5	0.5	0.5	0.5
Citric Acid	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.
Distilled Water	Up to 100	Up to 100	Up to 100	Up to 100	Up to 100	Up to 100

Method of Preparation

All herbal extracts were accurately weighed according to the formulation composition. Xanthan gum was dispersed in a small quantity of distilled water with continuous stirring until a uniform gel was formed. The herbal extracts of Amla, Reetha, Shikakai, Hibiscus, Neem, and Fenugreek were dissolved separately in distilled water and added gradually to the xanthan gum dispersion. Aloe vera gel was incorporated with continuous stirring to obtain a homogeneous mixture.

Methyl paraben was dissolved separately and added to the formulation as a preservative. Rose oil was then incorporated as a fragrance. The pH of the formulation was adjusted to approximately 5.5–6.0 using citric acid solution. Finally, the volume was adjusted with distilled water and the formulation was stirred continuously until a smooth, homogeneous shampoo was obtained. The prepared formulations were stored in airtight containers at room temperature for further evaluation.

3.3 Optimization by Design of Experiments (DoE)

To optimize the herbal shampoo formulation, a Design of Experiments (DoE) approach was employed within the Quality by Design (QbD) framework. Optimization was carried out using a simplified Box–Behnken Design (BBD) to evaluate the influence of critical formulation variables on the quality attributes of the shampoo. The experimental design facilitated the systematic investigation of formulation factors and their effects on product performance while reducing the number of experimental trials required.

Three independent variables were selected based on their significant contribution to cleansing, conditioning, and viscosity characteristics of the herbal shampoo. The concentrations of Reetha, Shikakai, and Aloe vera were varied at different levels, while other formulation components remained constant throughout the study.

Independent Variables (Factors)

- X_1 = Reetha Concentration (%)
- X_2 = Shikakai Concentration (%)
- X_3 = Aloe vera Concentration (%)

Dependent Variables (Responses)

- Y_1 = Foam Volume (mL)
- Y_2 = Viscosity (cP)
- Y_3 = Detergency Power (%)

The generated experimental data were analyzed to determine the relationship between formulation variables and response parameters. The optimized formulation was selected based on maximum foam volume, acceptable viscosity, and superior detergency power.

Table 5. Experimental Design Matrix for Herbal Shampoo Optimization (F1–F6)

Batch	X_1 Reetha (%)	X_2 Shikakai (%)	X_3 Aloe vera (%)	Y_1 Foam Volume (mL)	Y_2 Viscosity (cP)	Y_3 Detergency Power (%)
F1	8	4	5	132 ± 2.1	2850 ± 35	68.5 ± 1.2
F2	10	4	7	145 ± 2.5	3015 ± 42	73.4 ± 1.1
F3	12	4	9	158 ± 2.8	3240 ± 38	78.2 ± 1.4
F4	8	6	5	136 ± 2.3	3185 ± 40	70.8 ± 1.3
F5	10	6	7	149 ± 2.6	3425 ± 45	75.6 ± 1.5
F6	12	6	9	162 ± 3.0	3678 ± 48	81.3 ± 1.2

Values expressed as Mean ± SD (n = 3).

Interpretation: The results indicated that increasing the concentration of Reetha (X_1) significantly enhanced foam volume and detergency power due to its high saponin content. Increasing Shikakai concentration (X_2) improved conditioning characteristics and contributed moderately to viscosity enhancement. Aloe vera concentration (X_3) primarily influenced viscosity and conditioning properties by increasing the thickness and smoothness of the formulation. Among all batches, F6 exhibited the highest foam volume (162 mL), viscosity (3678 cP), and detergency power (81.3%), indicating superior overall performance and suitability as the optimized formulation.

Table 6. Evaluation Parameters of Herbal Shampoo Formulations

Parameters	F1	F2	F3	F4	F5	F6
Appearance	Light Brown	Light Brown	Brown	Light Brown	Brown	Dark Brown
Homogeneity	Good	Good	Good	Good	Good	Excellent
pH	5.48 ± 0.03	5.56 ± 0.04	5.61 ± 0.05	5.52 ± 0.03	5.67 ± 0.04	5.72 ± 0.02
Solid Content (%)	23.4 ± 0.5	24.8 ± 0.4	25.6 ± 0.6	24.1 ± 0.5	25.9 ± 0.4	27.2 ± 0.5
Surface Tension (dynes/cm)	34.8 ± 0.7	33.6 ± 0.8	32.5 ± 0.6	34.1 ± 0.7	33.0 ± 0.5	31.4 ± 0.6
Wetting Time (sec)	168 ± 3	154 ± 2	145 ± 3	160 ± 2	148 ± 3	136 ± 2
Dirt Dispersion Test	Moderate	Good	Good	Good	Very Good	Excellent
Foam Volume (mL)	132 ± 2.1	145 ± 2.5	158 ± 2.8	136 ± 2.3	149 ± 2.6	162 ± 3.0
Foam Stability (%)	84.5 ± 1.2	86.8 ± 1.4	89.2 ± 1.1	85.6 ± 1.3	88.4 ± 1.2	91.7 ± 1.0
Viscosity (cP)	2850 ± 35	3015 ± 42	3240 ± 38	3185 ± 40	3425 ± 45	3678 ± 48
Detergency Power (%)	68.5 ± 1.2	73.4 ± 1.1	78.2 ± 1.4	70.8 ± 1.3	75.6 ± 1.5	81.3 ± 1.2
Conditioning Performance	Good	Good	Very Good	Very Good	Very Good	Excellent
Skin Irritation Test	No Irritation	No Irritation	No Irritation	No Irritation	No Irritation	No Irritation
Stability (3 Months)	Stable	Stable	Stable	Stable	Stable	Stable

Values are expressed as Mean ± SD (n = 3).

IV. CONCLUSION

The present investigation successfully demonstrated the formulation, optimization, and evaluation of a polyherbal shampoo containing extracts of Amla, Reetha, Shikakai, Hibiscus, Aloe vera, Neem, and Fenugreek. The selected medicinal plants were chosen based on their traditional use and scientifically

reported benefits in hair care, scalp protection, cleansing, conditioning, and hair growth promotion. Hydroalcoholic extraction was employed to obtain phytoconstituent-rich extracts, ensuring maximum recovery of bioactive compounds suitable for incorporation into the shampoo formulation.

A systematic formulation approach was adopted using Quality by Design (QbD) principles and Design of Experiments (DoE) methodology to optimize the concentrations of critical ingredients. Six formulations were prepared and evaluated for various physicochemical and performance parameters, including pH, foamability, foam stability, viscosity, detergency power, wetting time, conditioning effect, skin compatibility, and stability. The evaluation results confirmed that all formulations possessed acceptable quality characteristics and complied with the desirable requirements of an ideal shampoo.

Among the developed formulations, F6 exhibited the most favorable performance profile, showing superior foam generation, detergency power, viscosity, conditioning effect, and overall stability. The enhanced performance of F6 may be attributed to the optimized combination of Reetha and Shikakai as natural surfactants along with Aloe vera as a conditioning and moisturizing agent. The formulation also demonstrated good scalp compatibility, as no signs of irritation or adverse effects were observed during evaluation.

The study highlights the effectiveness of herbal ingredients as multifunctional alternatives to synthetic chemicals commonly used in commercial shampoos. The optimized herbal shampoo offers natural cleansing, improved hair conditioning, antimicrobial protection, and enhanced consumer safety while maintaining environmental sustainability. Therefore, the developed formulation can be considered a promising herbal hair care product with potential for commercial development and future clinical evaluation.

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