Formulation and Evaluation of Herbal Shampoo Containing Microencapsulated Neem, Onion, and Flaxseed Oils

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Abstract:

India has a long tradition of using herbal medicine, and natural formulations are increasingly preferred for hair care due to concerns about synthetic products and their side effects. This study focuses on the formulation and evaluation of an herbal hair oil and shampoo using onion, neem, and flaxseed extracts to promote hair health and address common hair issues such as hair fall, dandruff, and scalp infections. A preliminary survey was conducted to understand public preferences and challenges related to hair care products. Onion extract, rich in sulfur and antioxidants, stimulates hair growth and improves scalp circulation. Neem oil provides antibacterial and antifungal benefits, while flaxseed oil supplies omega-3 fatty acids that strengthen and nourish hair. The oils were extracted and microencapsulated using polyvinyl alcohol to enhance stability, and 1% of the encapsulated oil was incorporated into a shampoo formulation. The shampoo was evaluated for physicochemical parameters including pH, foam, stability, surface tension, and sensory attributes, all of which met standard quality criteria. Results indicate that the formulated microencapsulated herbal hair oil shampoo exhibit effective anti-hair fall, antifungal, and conditioning properties**,** offering a natural, stable, and eco-friendly alternative to synthetic hair care products.

Keywords: Herbal shampoo, Microencapsulation

Introduction

A shampoo is a cosmetic formulation designed for the cleansing of the hair and scalp, typically presented in a user-friendly form for convenient application. Its primary function is to remove accumulated sebum, scalp debris, and residues of hair-grooming preparations. In addition to its cleansing role, a complete shampoo formulation may exhibit secondary functions such as lubrication, conditioning, volumizing, prevention of electrostatic charge accumulation, and therapeutic benefits. Importantly, the formulation must ensure dermatological safety for long-term and repeated use [1].

In recent years, there has been a growing scientific and consumer interest in herbal and natural hair care formulations, motivated by increasing awareness of the potential adverse effects associated with synthetic cosmetic ingredients and a rising demand for eco-friendly and sustainable alternatives [2]. The present study aims to formulate and evaluate a herbal hair oil-based shampoo incorporating the therapeutic benefits of *Allium cepa* (onion), *Azadirachta indica* (neem), and *Linum usitatissimum* (flaxseed) oils, to address common hair-related concerns and enhance overall scalp and hair health.

Onion extract is a rich source of sulfur compounds, which play a vital role in stimulating hair follicle activity and improving scalp blood circulation. Additionally, its antioxidant constituents help mitigate oxidative stress by neutralizing free radicals, thereby preventing hair follicle damage. Neem extract, derived from *Azadirachta indica*, possesses potent antibacterial and antifungal properties, making it effective in the management of dandruff and various scalp infections. Furthermore, neem contributes to maintaining the scalp's physiological pH, thereby fostering an optimal environment for hair growth. Flaxseed oil is abundant in omega-3 fatty acids, which provide essential nourishment to the hair follicles, improving hair strength, elasticity, and overall texture while minimizing breakage and split ends [3].

Microencapsulation Technology

Microencapsulation refers to a physicochemical process wherein microscopic droplets or particles of a core material—either liquid or solid—are enveloped by a continuous coating or film of a polymeric substance. This process results in the formation of microcapsules with

diameters ranging from less than one micron to several hundred microns [4]. The encapsulating layer acts as a chemical, physical, and physicochemical barrier, protecting the encapsulated core from environmental factors such as oxidation, evaporation, and unwanted interactions with other formulation components. Among the various encapsulation techniques, coacervation is widely utilized due to its high efficiency and versatility. Coacervation involves the phase separation of a polymeric solution into a polymer-rich (coacervate) phase and a polymer-poor phase. The polymer-rich phase subsequently forms a coating around the core material, resulting in microcapsule formation.

The present study focuses on the microencapsulation of neem oil, onion oil, and flaxseed oil using a coacervation-based approach, employing suitable polymers as wall materials. The resulting microcapsules are intended for incorporation into a herbal shampoo formulation**,** aimed at improving the product's stability, efficacy, and controlled release properties.

Preliminary Survey

Recognizing the importance of hair care and consumer perception in the development of effective formulations, a preliminary survey was conducted using a Google Form-based questionnaire. The objective of the survey was to gather insights into individuals' experiences, preferences, and challenges related to hair care and the use of hair products. The collected data were analyzed to understand current consumer trends, product satisfaction levels, and expectations, thereby providing valuable input for the development of improved, evidence-based herbal hair care formulations aimed at promoting optimal hair and scalp health.

2. Materials and Methods

2.1. Materials Used

The following materials were utilized in this study: neem oil, onion oil, flaxseed oil, methyl cellulose, ethyl cellulose, and polyvinyl alcohol (PVA). All reagents and chemicals used were of analytical grade.

2.2. Survey on the Use of Herbal Hair Oil

Survey Objective

A consumer survey was conducted to evaluate current customer preferences and perceptions regarding various brands of herbal hair oil and their usage patterns.

Survey Design

A structured questionnaire consisting of 11 questions was developed and distributed through Google Forms**.** The questionnaire included items related to:

- Preferred brand of herbal hair oil
- Price considerations
- Use of home remedies for hair care
- Key ingredients commonly used in herbal hair oils
- Awareness and usage of herbal hair oils formulated using novel formulation technologies

Study Population

The survey was conducted online, and responses were obtained from 143 participants aged 18 years and above. Participation was voluntary, and confidentiality of responses was maintained throughout the study. The results were summarized in Figure 1.

2.3. Preparation of Microspheres:

Microspheres were prepared using the solvent evaporation technique with neem oil as the model drug. Initially, 50 mL of acetone was added to a beaker and stirred for 15 minutes to ensure homogeneity. Subsequently, 1 mL of neem oil was added to the solvent system and stirred for 5 minutes to obtain a uniform dispersion.

To this organic phase, 1 g each of methyl cellulose and ethyl cellulose were added and the mixture was stirred for 30 minutes to achieve complete dissolution of the polymers. The resulting organic phase was then added dropwise to an aqueous phase containing 5 mL of 1% polyvinyl alcohol (PVA), which served as a stabilizing agent. The mixture was agitated at 500 rpm to form a fine emulsion.

A small portion of the emulsion was diluted and examined under a microscope to observe droplet formation. Microspheres containing other oils such as onion oil and flaxseed oil were prepared using the same procedure.

2.4. Formulation of Herbal Shampoo:

To formulate a base of shampoo specific amount of sodium lauryl sulphate, cetyl alcohol and distilled water solution containing methyl paraben and propyl paraben whose formula is shown in Table 1. Sodium lauryl sulphate, cetyl alcohol were added into demineralized water and were mixed gently to avoid making any foam. Then, methyl paraben and propyl were added and mixed gently. Previously prepared microsphere-based emulsion of the oils was added to the basic shampoo formulation (1%), after which it was mixed gently and then topped up with water upto 20 mL where needed. [5,6]

Table 1: Formulation Composition of 20 mL Herbal Shampoo

Ingredient	**Quantity given**	**Quantity taken**	**Use**
SLS (sodium lauryl sulphate	45%	9gm	Surfactant, cleansing agent, foaming agent
Cetyl alcohol	7%	1.4gm	Thickening agent,
Distilled water	100ml	20ml	As vehicle
Perfume	quantity sufficient	Quantity sufficient	Fragrance
Methyl paraben, propyl paraben	Quantity sufficient	Quantity sufficient	Preservative
Mixture of neem oil,onion oil, flaxseed oil containing microsphere	1gm	1gm	Active ingredient

2.5 Evaluation of Microspheres

1. Microscopy

The morphology, shape, and structural integrity of the prepared microspheres were evaluated

using optical microscopy. Samples were mounted on glass slides and examined under a light microscope equipped with a 10×/0.45 objective lens. Microscopic observation was utilized to assess the uniformity and surface characteristics of the microspheres, as well as to optimize the rate of addition of the phase-inducing agent during preparation. This technique provided detailed information on the wall morphology and overall structural features of the microspheres. The results were shown in figure 2.

2. Particle Size Analysis

The particle size distribution of the microspheres was determined using a particle size analyzer figure 3. Morphological characteristics, including shape and surface structure, were examined to evaluate uniformity and coating efficiency. [7]

2.6 Evaluation of Herbal Shampoo

1. Physical Appearance and Visual Inspection

The prepared shampoo formulations were evaluated for clarity, foaming characteristics, and fluidity through visual inspection. [8]

2. Determination of pH

The pH of a 10% (v/v) shampoo solution in distilled water was determined at 25 ± 2°C using a standard pH meter. Measurements were carried out in triplicate, and the mean ± SD values were reported. For comparative analysis, the pH of a 1% shampoo solution was also evaluated under identical conditions. [9]

3. Foaming Ability and Foam Stability

The cylinder shake method was employed to determine the foaming ability and stability of the formulations. A 1% (v/v) shampoo solution (50 mL) was transferred into a 250 mL graduated cylinder, which was sealed and shaken manually 10 times. The total foam volume was measured immediately after shaking and recorded as the foam volume after 1 minute**.** Subsequently, the foam height was measured at 1-minute intervals up to 4 minutes to evaluate foam stability.

4. Surface tension

Surface tension was determined using a stalagmometer to evaluate the shampoo's wetting and spreading ability, which are essential for efficient cleansing and foam formation. A reduction in surface tension compared to distilled water indicates improved surfactant activity contributed by the herbal ingredients and natural surfactants used in the formulation. [10]

5. Viscosity

Viscosity was measured using a Brookfield viscometer to ensure consistency, stability, and ease of application. Appropriate viscosity is critical to user acceptability, as it influences product flow, foam stability, and uniform distribution on the hair and scalp. [11]

6. Determination of % of solid contents

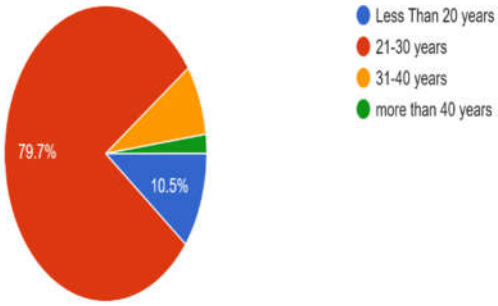
4 grams of shampoo were placed in a previously clean, dry and weighed evaporating dish. The dish and shampoo was weighed again to confirm the exact weight of the shampoo. The liquid portion of the shampoo was evaporated by placing the evaporating dish on the hot plate. The weight and thus % of the solid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

3. RESULTS AND DISCUSSIONS:

3.1 Responses to *Use of herbal hair oil* survey conducted online using Google forms

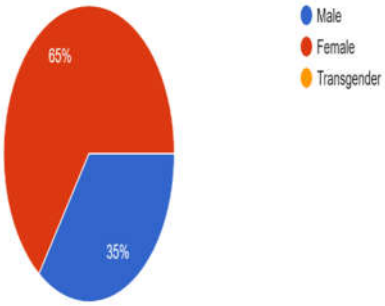
Your Age

143 responses



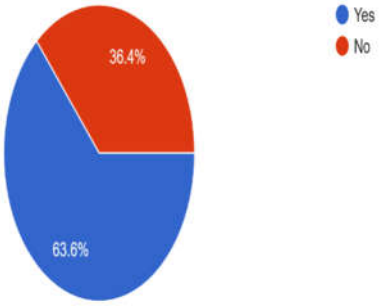
Gender

143 responses



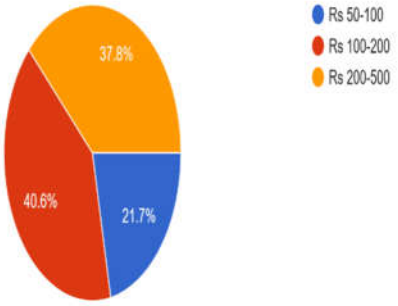
Are you using herbal Hair oil

143 responses



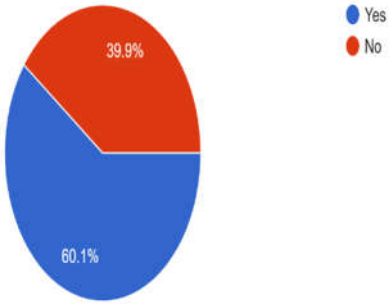
Cost of herbal hair oil

143 responses



Are you using any home remedies for hair

143 responses



if yes then which main ingredients you have chosen

143 responses

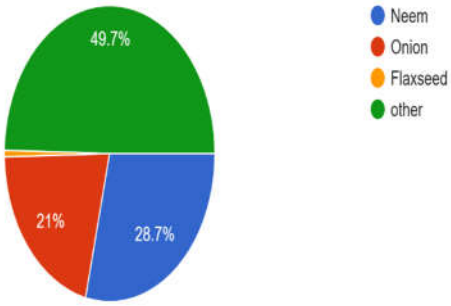


Figure 1: Survey Results on Herbal Hair Oil Usage Based on 143 Responses

1. Brand Preference

The responses indicated a diverse preference for various brands, with some respondents favoring well-established commercial brands, while others preferred homemade or locally available products.

2. Cost Considerations

A majority of participants considered affordability an important factor when purchasing herbal hair oil. Many respondents preferred mid-range priced products that offered a balance between quality and cost.

3. Use of Home Remedies

A significant portion of respondents mentioned using home remedies alongside commercial herbal hair oils. Popular home ingredients included coconut oil, amla, hibiscus, and fenugreek.

4. Key Ingredients

Participants identified several key ingredients in their preferred herbal hair oils, including:

- Amla (Indian gooseberry)
- Bhringraj
- Aloe vera
- Hibiscus
- Neem These ingredients were chosen for their perceived benefits in hair growth, scalp nourishment, and dandruff prevention.

5. Novel Formulation Technology

When asked about the use of herbal hair oil formulated using novel technologies, responses were mixed. While some participants showed interest in scientifically formulated products, others remained skeptical and preferred traditional formulations.

3.2 Results of microsphere:

1. Microscopy:

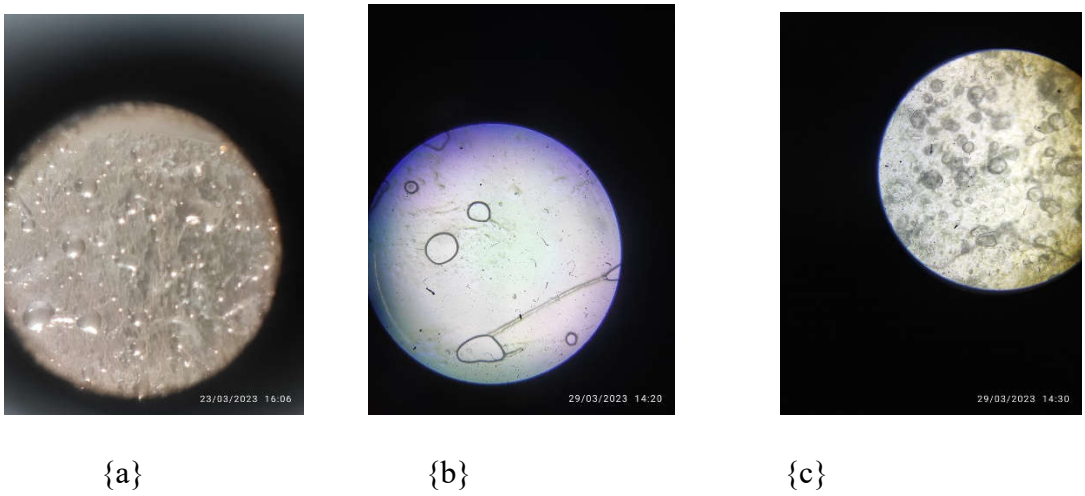
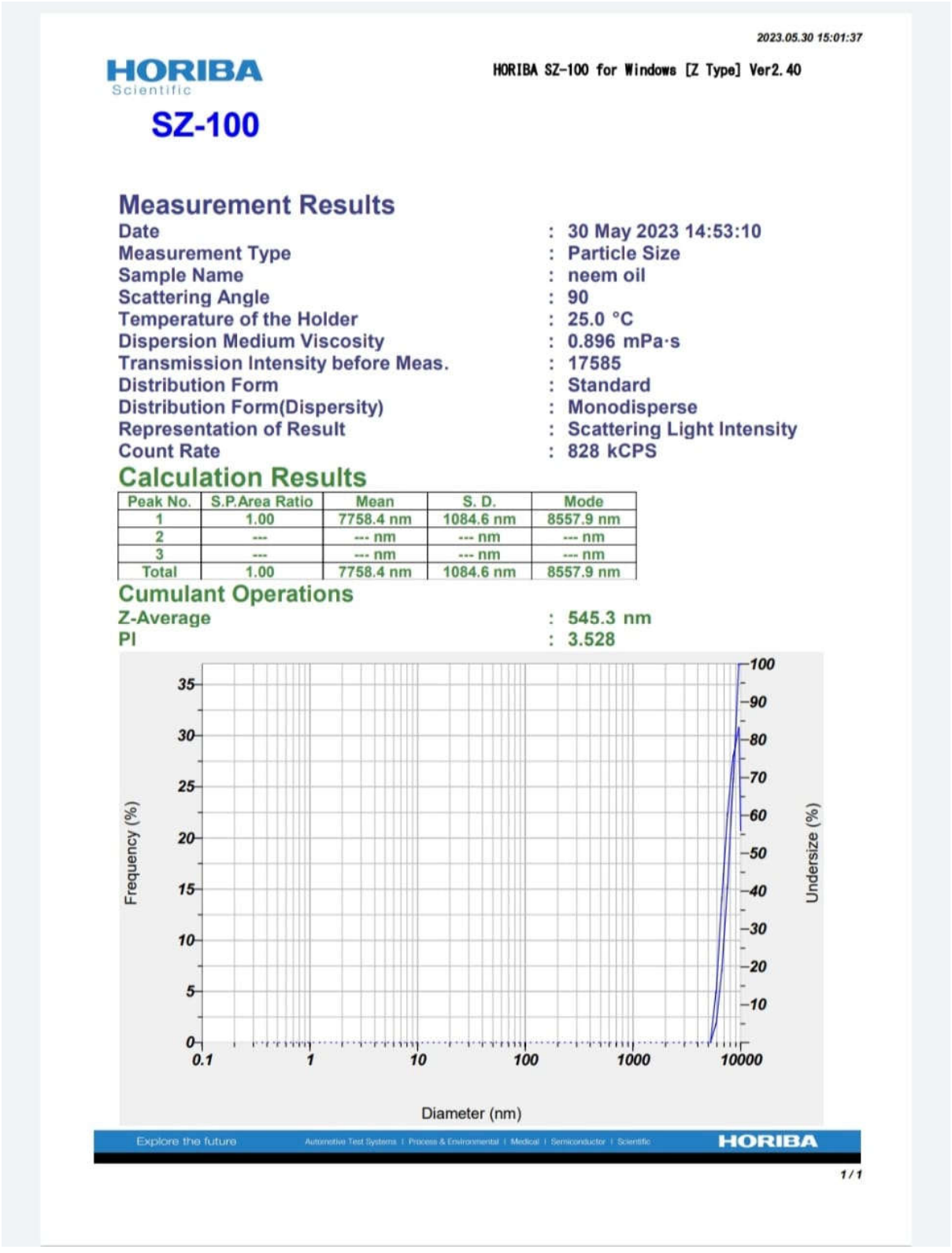


Figure 2: Images of the microsphere {a}. neem oil, {b}. onion oil, {c}. flaxseed oil.

2. Particle size:

The Particle size can be determined by the particle size analyzer. It was checked at Annasaheb Dange college of B pharmacy, Ashta.



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HORIBA SZ-100 for Windows [Z Type] Ver2.40

SZ-100

Measurement Results

Date	: 30 May 2023 14:53:10
Measurement Type	: Particle Size
Sample Name	: neem oil
Scattering Angle	: 90
Temperature of the Holder	: 25.0 °C
Dispersion Medium Viscosity	: 0.896 mPa·s
Transmission Intensity before Meas.	: 17585
Distribution Form	: Standard
Distribution Form(Dispersity)	: Monodisperse
Representation of Result	: Scattering Light Intensity
Count Rate	: 828 kCPS

Calculation Results

Peak No.	S.P.Area Ratio	Mean	S. D.	Mode
1	1.00	7758.4 nm	1084.6 nm	8557.9 nm
2	---	--- nm	--- nm	--- nm
3	---	--- nm	--- nm	--- nm
Total	1.00	7758.4 nm	1084.6 nm	8557.9 nm

Cumulant Operations

Z-Average	: 545.3 nm
PI	: 3.528

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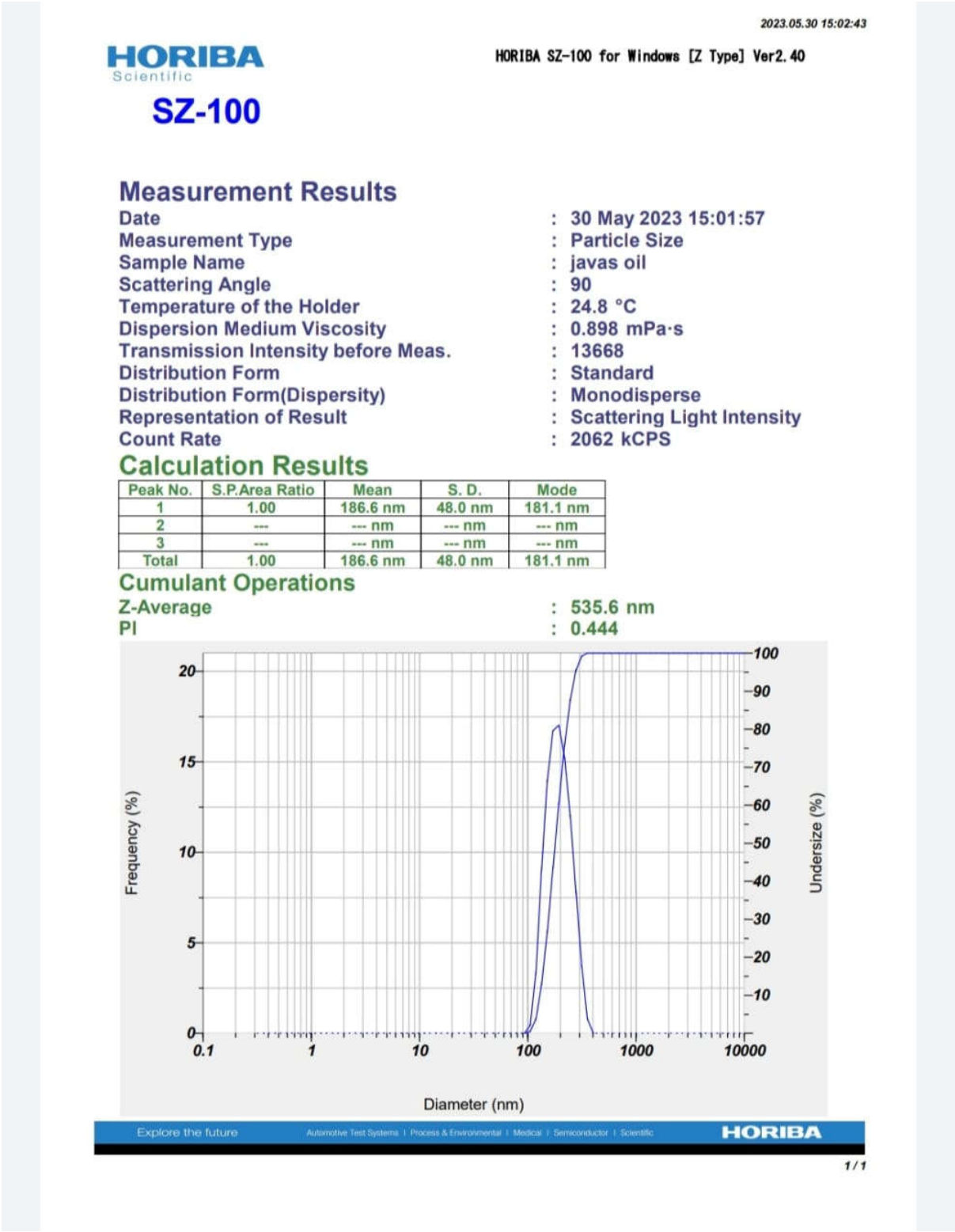
Figure 3: Particle size of neem oil



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HORIBA Scientific

HORIBA SZ-100 for Windows [Z Type] Ver2.40

SZ-100

Measurement Results

Date	:	30 May 2023 15:01:57
Measurement Type	:	Particle Size
Sample Name	:	javas oil
Scattering Angle	:	90
Temperature of the Holder	:	24.8 °C
Dispersion Medium Viscosity	:	0.898 mPa·s
Transmission Intensity before Meas.	:	13668
Distribution Form	:	Standard
Distribution Form(Dispersity)	:	Monodisperse
Representation of Result	:	Scattering Light Intensity
Count Rate	:	2062 kCPS

Calculation Results

Peak No.	S.P.Area Ratio	Mean	S. D.	Mode
1	1.00	186.6 nm	48.0 nm	181.1 nm
2	---	--- nm	--- nm	--- nm
3	---	--- nm	--- nm	--- nm
Total	1.00	186.6 nm	48.0 nm	181.1 nm

Cumulant Operations

Z-Average	:	535.6 nm
PI	:	0.444

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Figure 4: Particle size of Flaxseed oil.

3.3 Results of Herbal Shampoo

1. Physical Appearance:

An ideal shampoo formulation should exhibit an aesthetically appealing physical appearance to ensure consumer acceptance. The prepared herbal shampoo was evaluated for its physical characteristics, including colour, odour, and transparency (Table 2). The formulation appeared pale yellow in colour with a pleasant odour**,** indicating satisfactory organoleptic properties.

Table 2: Physicochemical evaluation of formulated shampoo.

Sr. No.	Evaluation test	Results
1	Colour	Pale Yellow
2	Odour	Good
3	Transparency	Yellow Gel
4	pH (10% solution)	8.2 ± 0.08
5	% solid content	18.65
6	Foam type	Dense, Small
7	Viscosity	684.0cp
8	Surface tension	35.84 ±1.75 dyne/cm

Results are mean ± SD (n = 3)

2. Determination of pH:

The pH of a shampoo is a critical parameter that influences both hair and scalp health. A slightly alkaline or near-neutral pH helps to minimize irritation to the eyes and scalp while maintaining hair integrity. The pH of the formulated herbal shampoo was determined to be 8.2**,** which falls within the acceptable range for hair care products and ensures adequate cleansing efficacy without causing adverse effects.

3. Foaming Ability and Foam Stability:

Foaming properties are essential indicators of a shampoo's performance and consumer appeal. The formulated shampoo produced small, uniform, and stable foam, comparable to that of

commercial formulations. The observed foam stability and uniformity suggest the presence of an effective surfactant system, contributing to satisfactory cleansing and application characteristics.

4. Surface Tension:

Reduction in surface tension is directly correlated with the cleansing efficacy of a shampoo. The formulated herbal shampoo reduced the surface tension of water to 35.84 dyne/cm, demonstrating efficient surface-active properties and good cleaning potential Table 2.

5. Viscosity:

Viscosity determines the ease of application and handling characteristics of a shampoo. The viscosity of the formulated product was measured at 684.0 cP, indicating appropriate consistency for convenient dispensing and spreading during use.

6. Percentage of Solid Contents:

An optimal solid content of 20–30% is generally desirable for shampoo formulations to ensure easy application and rinsing. The solid content of the formulated herbal shampoo was found to be 18.65%**,** which falls slightly below the optimal range but remains acceptable, ensuring that the product can be easily applied and rinsed from the hair without excessive residue.

Conclusion

The survey highlights that consumers value affordability, natural ingredients, and effectiveness when choosing herbal hair oils. While traditional home remedies remain popular, there is also a growing interest in scientifically formulated herbal products. The insights from this survey can help manufacturers align their products with consumer expectations and preferences.

Good quality of essential oil is obtained under laboratory conditions. The main component of neem oil, onion oil and flaxseed oil are found to be very effective in formulated herbal shampoo. In the present study, microencapsulation of essential oils with poly vinyl alcohol (coating material) using microencapsulation technique was successfully carried out. The optimization was done by varying core to wall material ratio. During the characterization of microsphere, it was found that 1:1 ratio gives the highest yield.

The stability of microsphere in shampoo base is very significant. It was stable for 4 months at 45°C. Several tests were performed to evaluate and compare the physicochemical properties of the prepared shampoo. The prepared shampoo showed comparable result with that of marketed shampoo for quality control tests. Thus, a good quality of herbal shampoo is formulated which will be beneficial for hair growth and strengthening.

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