

FORMULATION AND EVALUATION OF HERBAL NIOSOMAL TABLET FOR HAIR CARE

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ABSTRACT

Nutricosmetics, the integration of nutrition and cosmetics, are becoming increasingly popular as advanced solutions for promoting skin, hair, and

beauty from the inside out. Among them, niosomal capsules are a potential delivery system that provides enhanced stability, bioavailability, and controlled release of the active components. The parameters of evaluation for such formulations are particle size, entrapment efficiency, zeta potential, in-vitro release, stability studies, and ability to penetrate the skin, to ensure efficacy and safety. The significance of nutricosmetics is their long-term potential to benefit beauty problems at a cellular level as opposed to only offering surface-level benefits. As awareness and demands among consumers rise regarding natural and non-invasive approaches to beauty, the worldwide market for nutricosmetics is expected to surge in magnitude and reach multi-billion-dollar market values, powered by innovations in nanotechnology and customization of skincare regimens. The growing consumer preference for sustainable, non-toxic, and efficient delivery systems has further fueled market growth. With increasing investments in nanotechnology and biotechnology, the niosome market is expected to witness significant expansion, reaching multi-billion-dollar valuations in the coming years. This growth is driven by rising demand for personalized medicine, innovative skincare solutions, and effective therapeutic delivery systems across the globe. This research article focuses on formulation evaluation and characterisation of niosomes made from the extracts of calyx of Hibiscus sabdariffa and leaves of Murraya koenigii. Incorporation of niosome in hard gelatine capsule and its evaluation.

INTRODUCTION

Nutricosmetics is a new discipline that combines nutrition and cosmetics, with the aim of beautifying from the inside out in the form of dietary supplements. They are rich in bioactive molecules, vitamins, minerals, and antioxidants that act from the inside out to nourish the skin, strengthen hair, and enhance general appearance. Unlike topical surface treatments, nutricosmetics act at the cellular level to yield lasting beauty benefits. (Lentjes, 2019)

Nutricosmetics promote beauty from the inside out by providing essential nutrients that support: (A.K Johnson, 2021)

Collagen synthesis: Collagen is an important protein that keeps skin elastic and firm. Ingredients such as hydrolyzed collagen, vitamin C, and peptides trigger collagen synthesis.

Skin hydration and protection: Hyaluronic acid, ceramides, and omega fatty acids retain moisture and fortify the skin barrier.

Antioxidant defence: Free radicals are responsible for aging and skin damage. Nutricosmetics that are high in vitamins A, C, E, and polyphenols fight antioxidant stress.

Hair and nail care: Biotin, keratin, and zinc fortify hair and nails, making them less brittle and encouraging growth.

1. Key Nutricosmetic Ingredients(Chopra et al., 2022)(Gokce et al., 2022)

Collagen Peptides: Enhances skin elasticity and minimizes wrinkles.Present in marine or bovine collagen supplements.

Hyaluronic Acid: Holds moisture, keeping the skin hydrated and plumper.Present in anti-aging supplements.

Vitamin C: Boosts collagen production and whitens the skin.Acts as a potent antioxidant.

Biotin (Vitamin B7): Strengthen nails and hair.Reduces hair loss and promotes scalp health.

Zinc: Controls oil production, minimizing acne.Maintains immune response and wound healing.

Omega-3 Fatty Acids: Preserves skin barrier function and minimizes inflammation.Present in fish oil and flaxseed supplements.

Ceramides: Enhances skin barrier and minimizes moisture loss.Present in wheat extracts. Polyphenols (e.g., Resveratrol, Green Tea Extract) It Protects skin from UV damage and aging and Possesses anti-inflammatory and antioxidant effects.

2. Benefits of Nutricosmetics(Orasan et al., 2016)

Skin Health: It reduces wrinkles and fine lines, improves skin elasticity and hydration and used in acne prevention and skin repair.

Encourages Hair and Nail Growth: Strengthens hair follicles, avoiding breakage, encourages shinier and thicker hair and prevents brittle nails and encourages nail growth.

Anti-Aging Benefits: Repels oxidative stress that causes premature aging, Boosts collagen and elastin and used in reducing pigmentation and age spots.

UV Protection: Certain nutricosmetics have carotenoids such as taxanthin and lycopene, which protect skin from sun damage.

Gut-Skin Connection: Probiotics and prebiotics in nutricosmetics support a healthy gut microbiome, which indirectly improves skin clarity and reduces inflammation.

3. Future Trends in Nutricosmetics(Madhere & Simpson, 2010)

Personalized Nutricosmetics: Depending on DNA test or skin type.

Plant-Based Formulas: Vegan collagen and plant-derived antioxidants.

Microbiome-Targeted Supplements: Gut-targeted for healthier skin.

Sustainable Packaging and Sourcing: Green, clean-label.

4. Niosomes

Niosomes are vesicular non-ionic surfactant drug delivery systems and hence an efficient carrier of lipophilic and hydrophilic drugs. They are similar to liposomes but are more stable, cost-effective, and biocompatible. Due to their characteristic structure, niosomes have wide usage in the pharmaceutical, cosmetic, and biotechnology industries for controlled and targeted drug delivery.(Chauhan & Chauhan, 2021). Niosomes are made of **aqueousCore**– traps hydrophilic drugs, **bilayer Membrane** – Mixture of non-ionic surfactants and cholesterol that traps lipophilic drugs and **surfactants** – Gives structural stability and enhances drug permeability (Thabet et al., 2022). The drug is trapped inside the niosomal vesicle and is shielded from degradation (enzymatic, oxidative, or pH-dependent) before it reaches the target site.Due to their nano-size, niosomes can passively accumulate in target sites, especially in tumors and inflamed tissues, where permeability of blood vessels is enhanced(Moammeri et al., 2023).Sometimes cells recognize and engulf niosomes by phagocytosis or receptor-mediated endocytosis, leading to intracellular drug release (Witika et al., 2022) Niosomes are incompatible and biodegradable,

cost-effective stable, enhanced drug penetration, sustained release, Targeted Drug Release – Reduces systemic toxicity while enhancing therapeutic potency. Used in cancer therapy, gene delivery, anti-inflammatory drugs, and cosmetics. (Kaur et al., 2012, Yeo et al., 2017)

MATERIAL & METHODS

The calyx and leaves of *Hibiscus sabdariffa* and *Murraya koenigii* plant were collected from local area of Nagpur. The Mumbai-based Loba Chemicals Pvt. Ltd. Supplied us solvent methanol and ethanol. Quercetin, DPPH, ABTS, catechol, ferrozine, ferrous sulphate, guaiacol, gallic acid, sodium carbonate, ammonium molybdate, sulphuric acid, sodium phosphate, ascorbic acid, potassium ferricyanide, TCA, etc were purchased from Hi-Media, India. Hydrogen peroxide and Folin-Ciocalteu reagent were purchased from Central Drug House (P) Ltd. India. Every chemical was of the analytical grade.

1. Extraction

The coarse powdered of dried calyx of *Hibiscus sabdariffa* (100g) and leaves of *Murraya koenigii* (100g) was extracted with 650ml of ethanol by hot solvent extraction for 24hr using Soxhlet assembly. The extract was concentrated at 40°C to obtain dark reddish colour sticky mass. The extracts dried by evaporation weighed and subjected to phytochemical screening. (Luque de Castro & Priego-Capote, 2010)

2. Quantitative estimation

Phytochemical evaluation was done by performing tests for Carbohydrates, Proteins, Flavonoids, Alkaloids, Glycosides, polyphenols, Tannins, saponins etc. Quantitative estimation was done for estimating total flavonoid content in the extract.

Total flavonoid content: Stock solution of Rutin (1mg/ml) in methanol was prepared then Different aliquots of 100-1000µg/ml was prepared in a 10ml volumetric flask & 4 ml distilled water was added along with 0.3ml 5% NaNO₂. After 5 min add 0.3 ml of 10% AlCl₃ & further after 5-6 min add 2ml 1M NaOH. After the addition of NaOH, the solution turns red color. Incubate/kept at dark a place for 30 min. Absorbance was measured at 510 nm and Calibration curve was plotted as concentration v/s Absorbance. (Aryanti et al., 2021) (Salim et al., 2024)

3. DPPH (2,2-Diphenyl-1-picrylhydrazyl) Radical Scavenging Activity

2,2-diphenyl-1-picrylhydrazyl (DPPH) assay is a general assay for determining the free radical scavenging capacity of compounds, extracts, or formulations. Dissolve 3.94 mg of DPPH in methanol (100 mL). Store the solution in a dark bottle at room temperature. Stabilize for 30 minutes prior to use. Prepare the test sample (pure compound or extract) of varying concentrations in methanol (Regupathi & Chitra, 2015). Prepare ascorbic acid as standard in identical solvent and concentrations. In a test tubes, add 1 mL of 0.1 mM DPPH solution. 1 mL of test sample of varying concentrations. As control, 1 mL of DPPH solution and 1 mL of methanol (no antioxidant). As blank, take 1 mL of methanol and 1 mL of sample (no DPPH), mixed well and incubate the reaction at room temperature in the dark for 30 minutes. Record absorbance at 517 nm using a UV-Vis spectrophotometer (Alam et al., 2013, Sundara rajan et al., 2017)

The percentage of **DPPH radical scavenging activity** is calculated using the formula:

$$\% \text{ Scavenging Activity} = \left(\frac{A_{\text{control}} - A_{\text{sample}}}{A_{\text{control}}} \right) \times 100$$

Where, A_{control} = Absorbance of DPPH solution (without sample).

A_{sample} = Absorbance of DPPH solution with test sample.

5. Formulation of niosomes by reverse phase evaporation techniques:

The procedure begins with the preparation of the niosomal dispersion by a suitable method such as thin-film hydration, ether injection, or reverse-phase evaporation. The niosomal suspension is mixed with a cryoprotectant such as sucrose, trehalose, or mannitol to prevent the aggregation of vesicles and damage to the membrane during freezing and drying. The formulation is pre-frozen at -40°C to -80°C for a few hours or quick freezing with liquid nitrogen to form uniform ice crystals. (Saafan et al., 2021) The frozen samples are placed in a lyophilizer where primary drying

(sublimation) is carried out under reduced pressure and controlled temperature (typically -40°C to -20°C), which allows the ice to convert directly into vapor without passing through the liquid phase. This is followed by secondary drying at a slightly higher temperature (0°C to 25°C) to remove residual moisture, ensuring final lyophilized niosomes are stable. The powder is recovered and stored in tight containers under controlled conditions to prevent moisture uptake and degradation. Lyophilized niosomes are reconstituted with a suitable buffer or solvent before application to restore their original characteristics. (Kawasaki et al., 2019)

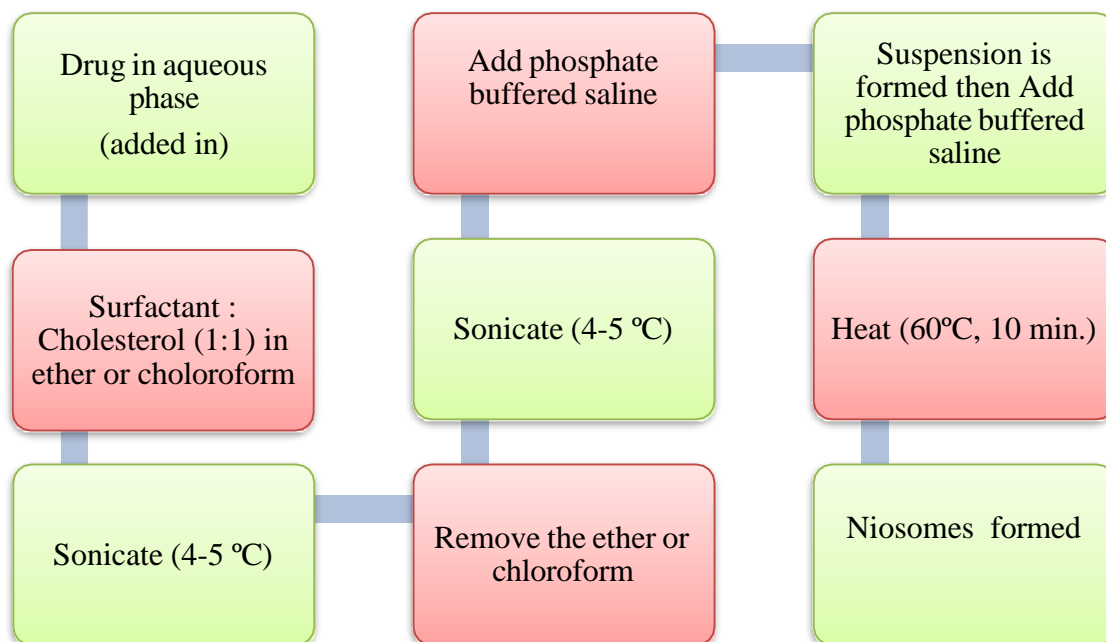


Figure 1: Flow chart of formulation of niosomes
Table 1. Formulation table of Niosomes

Batches	Amount of drug (mg)	Amount of cholesterol (mg)	Amount of span 60 (mg)	Method followed
Batch-1	100	200	400	Ether injection method
Batch-2	100	200	200	Ether injection method
Batch-3	100	100	400	Ether injection method
Batch-4	100	100	500	Reverse phase Evaporation technique
Batch-5	100	100	100	Reverse phase Evaporation technique
Batch-6	100	100	200	Reverse phase Evaporation technique
Batch-7	100	100	300	Reverse phase Evaporation technique
Batch-8	5g	5g	15g	Reverse phase Evaporation technique
Batch-9*	5g	5g	15g	Reverse phase Evaporation technique

6. Evaluation of Niosomes

Particle size and zeta potential

Particle size and zeta potential of niosomes are determined by dynamic light scattering (DLS) or electrophoretic light scattering (ELS) techniques. Light scattering data are measured at a suitable angle (typically 90° or 173° for backscattering), and hydrodynamic diameter is calculated using the Stokes-Einstein equation (O'Brien et al., 1995).

The zeta potential is calculated by software from the Smoluchowski equation, and it is a measure of surface charge and colloidal stability of the niosomes. A rough estimate of zeta potential value of $> \pm 30$ mV is a measure of good colloidal stability, and low values are measures of the potential for aggregation (Marsalek, 2014) (Shah et al., 2014).

SEM (Scanning Electron Microscopy)

The scanning electron microscopy (SEM) is done to observe the microscopy of particles in high magnification. In this the sample is fixed, dehydrated, and covered with a conductive coating such as gold or platinum if non-conductive. The sample is mounted on an aluminum stub and placed in the SEM vacuum chamber. The sample is scanned with an electron beam, and secondary and backscattered electrons are produced due to interactions, and high-resolution images are created. Images are analyzed to examine surface morphology, particle size, and structural features. (Datye & DeLaRiva, 2023) (Abdullah & Mohammed, 2019) (Alam et al., 2013)

7. Formulation of Hard Gelatin Capsule

Lyophilized niosomes were filled in hard gelatin capsule in specific quantity and processed it for further evaluation processes.

8. Evaluation parameter of Hard gelatine Capsule

Weight variation

The weight variation test for hard gelatin capsules is performed to ensure uniformity in the fill weight of the capsules. A total of 20 randomly selected capsules are individually weighed using an analytical balance, and the average weight is calculated. Each capsule is then carefully opened, and the contents are emptied completely without losing any material. The stripped capsule shells are weighed, and the net fill weight is calculated by subtracting the shell weight from the total weight of the filled capsule. The single fill weights are compared with the average fill weight, and deviations are tested against limits in the pharmacopoeia: $\pm 10\%$ for capsule weight < 300 mg and $\pm 7.5\%$ for capsule weight > 300 mg. If two or more capsules fall outside the range given or one capsule falls outside twice the limit, the batch fails to meet acceptance. (Archer et al., 2020) (Samuel et al., 2018)

Disintegration test

The hard gelatin capsule disintegration test was performed with a USP disintegration apparatus to find out the time taken for the capsules to disintegrate in a simulated physiological condition. Six capsules were separately put in the tubes of the disintegration basket, each having a wire mesh at the bottom. The basket is then submerged in 900 mL of water or 0.1N HCl (mimicking gastric juice) at $37 \pm 2^\circ\text{C}$. (Šedbarè et al., 2023) The machine is set so that the basket oscillates up and down with a fixed frequency of 29–32 cycles per minute. The test is finished when there are no capsule residues left on the mesh, other than insoluble pieces of the shell. The capsules should completely disintegrate in 30 minutes unless otherwise directed. In case of failure of one capsule, the test is repeated using another six capsules, and at least five out of

six capsules should pass for the batch to qualify according to pharmacopoeial standards.(Ozon et al., 2023).

Moisture permeability

The USP requires determination of the moisture permeation characteristics of single-unit and unit-dose containers to ensure their suitability for packaging capsules. The degree and rate of moisture penetration are determined by packaging the dosage unit together with a color-revealing desiccant pellet, exposing the packaged unit to known relative humidity over a specified time, observing the desiccant pellet for color change (indicating the absorption of moisture), and comparing the pretest and posttest weight of the packaged unit. The difference in the weights gives the amount of moisture absorbed. An alternative method for the determination of moisture content of soft gelatin capsules is by toluene distillation method.(Masfria et al., 2023)

Stability of capsule

The stability test for hard gelatin capsules is performed to determine their physical, chemical, and microbiological stability under defined storage conditions. Capsules are stored in stability chambers at various conditions: long-term (25°C ± 2°C / 60% RH ± 5% RH), accelerated (40°C ± 2°C / 75% RH ± 5% RH), and intermediate (30°C ± 2°C / 65% RH ± 5% RH) for a given period of time. samples are removed and tested. Any detectable changes like capsule deformation, brittleness, color variation, or decomposition of the active ingredient are signposts of instability. (Osei-Asare et al., 2021)(Khan & Agrawal, 2018)

RESULT AND DISCUSSION

1. Phytochemical evaluation

During phytochemical evaluation *Hibiscussabdariffa* shows positive results for Carbohydrates, Proteins, Flavonoids, Phenols, alkaloids and Tannins while *Murrayakoenigi* shows positive Proteins, Flavonoids, Phenols, and Tannins.

Table 2: Phytochemical estimation of extract

List of Phytochemicals	Result of Ethanolic extract of <i>Hibiscussabdariffa</i>	Result of Ethanolic extract of <i>Murrayakoenigi</i>
Carbohydrates	+	-
Proteins	+	+
Flavonoids	+	+
Phenols	+	+
Alkaloids	+	-
Tannins	+	+
Glycosides	-	-

4. Total flavonoid content

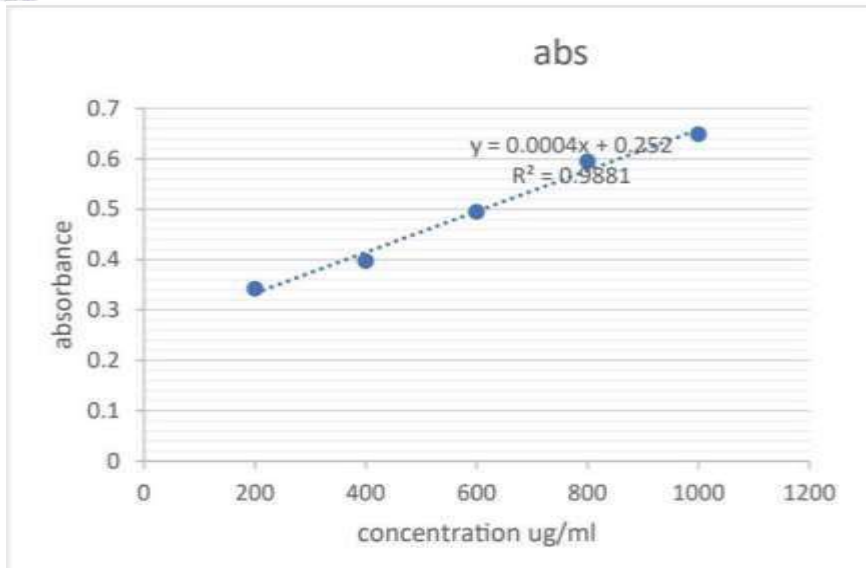


Figure 2: Calibration Curve of Rutin

$Y=0.0004x+0.252$ ($R^2=0.9881$)

Flavonoid content in Hibiscus extract was found to be 17.50 ± 0.52 mg / g in ethanolic extract and 20 ± 0.52 mg/g in methanolic extract.

5 .DPPH radical scavenging activity

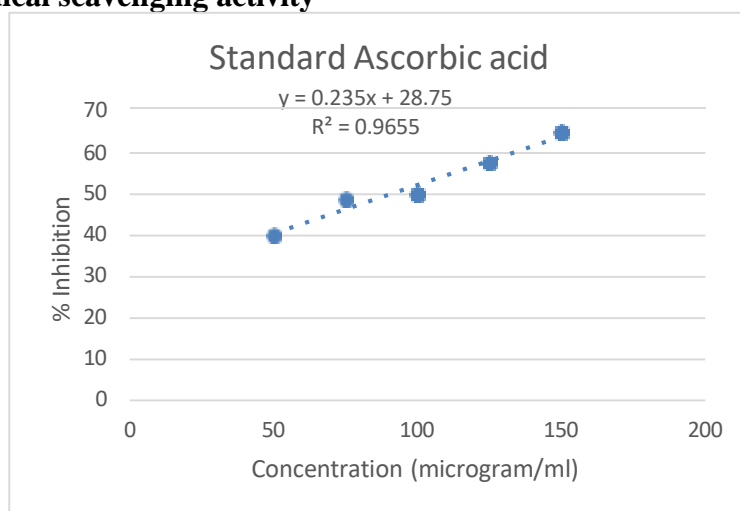


Figure 3: Calibration curve of standard ascorbic acid

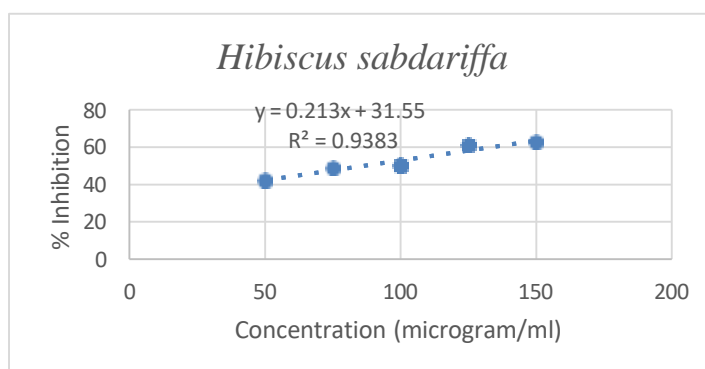


Figure 4: DPPH activity of *Hibiscus sabdariffa*

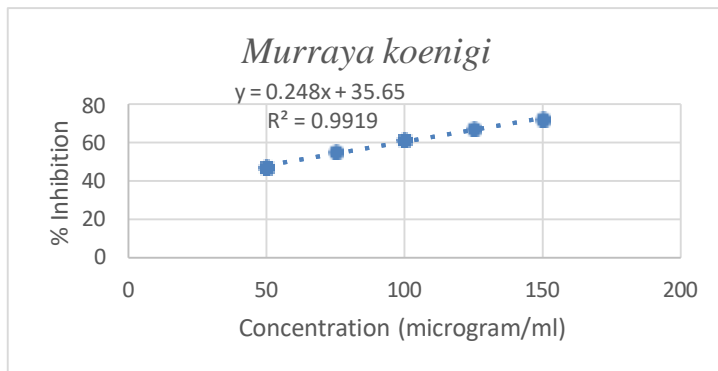


Figure 5: DPPH activity of *Murraya koenigi*

Table 3: IC₅₀ of standard Ascorbic acid, *Hibiscus sabdariffa* and *Murraya koenigi*

Particulars	IC ₅₀ Value
Ascorbic acid	90.42
<i>Hibiscus sabdariffa</i>	86.61
<i>Murraya koenigi</i>	57.86

2. Evaluation of Niosomes

Particle size and zeta potential

Table4: Particle size of formulation batches

Batches	Amount of drug (mg)	Amount of cholesterol (mg)	Amount of span 60 (mg)	Particle size
Batch-1	100	200	400	17523nm
Batch-2	100	200	200	5477nm
Batch-3	100	100	400	551.6nm
Batch-4	100	100	500	636.6nm
Batch-5	100	100	100	706.3nm
Batch-6	100	100	200	423nm
Batch-7	100	100	300	394nm
Batch-8	5g	5g	15g	320.1nm
Batch-9*	5g	5g	15g	264.1nm

The selected batch (Batch 9) of niosomes was further led for formulation and evaluation of capsule. Zeta potential of optimized batch was found to be -21.5mV

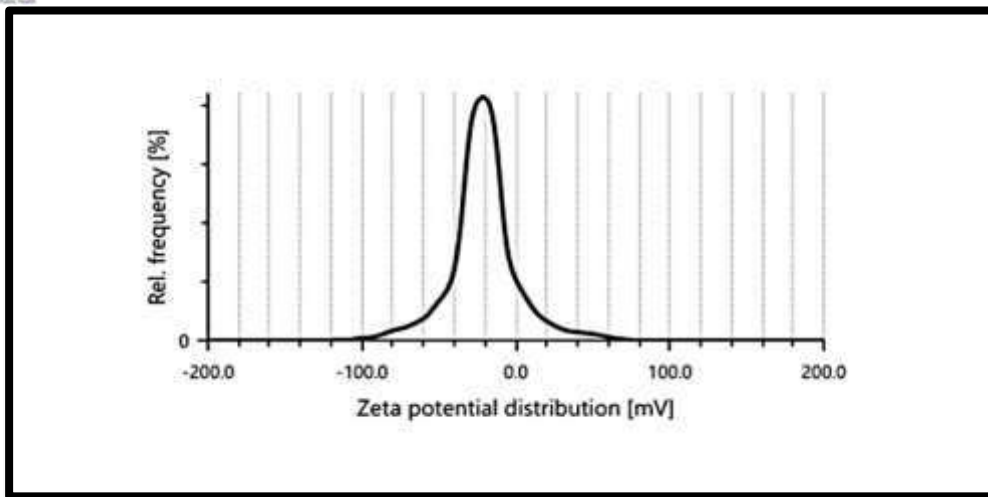


Figure 6: Zeta potential of niosomes

Scanning Electron Microscopy

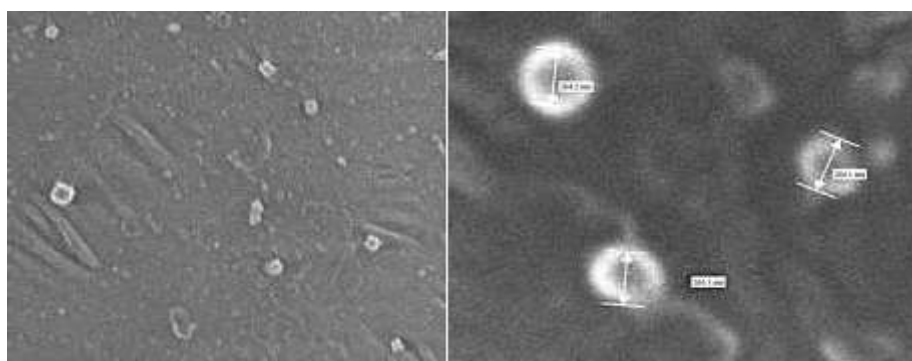


Figure 7: SEM analysis of niosome

3. Evaluation of Hard Gelatin Capsule

Weight variation of Capsules

Table 5: Weight variation of capsules

Sr.no.	Weight of individual capsule content (gm)	Average weight (gm)	% Weight variation (%)
1.	0.251	0.251	-0.74
2.	0.251	0.251	-0.74
3.	0.252	0.251	-0.74
4.	0.251	0.251	-0.74
5.	0.251	0.251	-0.74
6.	0.252	0.251	-0.74
7.	0.251	0.251	-0.74
8.	0.251	0.251	-0.74
9.	0.252	0.251	-0.74
10.	0.252	0.251	-0.74
11.	0.251	0.251	-0.74
12.	0.251	0.251	-0.74
13.	0.252	0.251	-0.74
14.	0.251	0.251	-0.74
15.	0.252	0.251	-0.74
16.	0.251	0.251	-0.74
17.	0.251	0.251	-0.74
18.	0.251	0.251	-0.74

19.	0.251	0.251	-0.74
20.	0.251	0.251	-0.74

The percent deviation for weight variation was found to be 5.5111 %

Disintegration time

The capsule was disintegrated in 16 mins at $37 \pm 2^\circ\text{C}$.

Moisture permeability test

The average weight difference in capsules after moisture permeability test was found to be 0.139 gm.

Stability study

Table 6: Stability study of capsules

Temperature	Humidity	Effectsoncapsulesshell
21-24 ⁰ C	60%	Capsulesbecomessofter
Greaterthan24 ⁰ C	Greaterthan45%	Morerapidandpronouncedeffects –unprotectedcapsulesmeltsand fuse together

CONCLUSION

Herbal niosomal capsules present a promising development in drug delivery, improving the bioavailability and therapeutic activity of herbal substances. Their capacity for enhanced solubility, stability, and targeted delivery makes them an attractive choice for contemporary herbal medicine. Through minimized side effects and controlled release, these formulations open the way for safer and more effective natural remedies. Ongoing research and development on this area will continue to enhance their potential, and they will become a valuable innovation in pharmaceutical and nutraceutical uses.

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