A Novel Approach to the Formulation and Standardization of Vyanghar Ghrita

Dr. Sayongeeta Patil[1], Dr. Swati Gadgil[2*]

PG Scholar, Department of Rasashastra and Bhaishajya Kalpana, Bharati Vidyapeeth (Deemed to be University), College of Ayurved, Pune 411043, India patilsayongeeta76@gmail.com

Professor, Department of Rasashastra and Bhaishajya Kalpana, Bharati Vidyapeeth (Deemed to be University), College of Ayurved, Pune 411043, India
swati.gadgil@bharatividyapeeth.edu

Corresponding author*
Dr. Swati Gadgil
Professor, Department of Rasashastra and Bhaishajya Kalpana, Bharati Vidyapeeth (Deemed to be University), College of Ayurved, Pune 411043, India
swati.gadgil@bharatividyapeeth.edu

Abstract

Background: Vyanga is an Ayurvedic disorder clinically comparable to melasma and other forms of acquired facial hypermelanosis. Classical topical pastes may be effective in traditional practice but are limited by short shelf life, variable daily preparation and poor portability. The present pharmaceutical study developed Vyanghar Ghrita, a modified topical Siddha Ghrita derived from the Mukhakantikar Lepa concept, using Ghrita Manda as the lipid vehicle. Objective: To establish a reproducible manufacturing procedure and generate preliminary organoleptic, physicochemical, microbiological and phytochemical standards for Vyanghar Ghrita. Materials and methods: Haridra, Daruharidra, Manjishtha, Shuddha Gairik and Peetasarshap were used as equal parts of Kalka. Ghrita Manda, water and goat milk were incorporated using a standardized Sneha Paka procedure. Three pilot batches were prepared to assess batch consistency, followed by a scaled final batch. Raw materials were authenticated and evaluated by pharmacopoeial tests; Shuddha Gairik was examined by X-ray diffraction and Fourier-transform infrared spectroscopy. Finished-product testing included pH, specific gravity, acid value, peroxide value, saponification value, iodine value, refractive index, fatty-acid profile, microbial quality and LC-MS profiling. Results: Pilot batches demonstrated consistent specific gravity (0.95), peroxide value (2.5 meq/kg), acid value (0.897-1.00 mg KOH/g) and saponification value (212.62-213.07 mg KOH/g). The final batch showed an acid value of 2.94 mg KOH/g, peroxide value of 2.32 meq/kg, iodine value of 35.66, refractive index of 1.4599 and saponification value of 251.83 mg KOH/g. The fatty-acid profile comprised 67.43% saturated, 28.05% monounsaturated and 4.36% polyunsaturated fatty acids, with trans fatty acids below 0.1%. Specified pathogens were absent. LC-MS detected curcumin as the dominant constituent (52.959%), followed by beta-sitosterol (14.058%) and other antioxidant or anti-melanogenic compounds. Conclusion: The study defines a practical SOP and a preliminary analytical fingerprint for Vyanghar Ghrita. The observed inter-batch consistency supports process reproducibility; however, the slightly elevated pH of pilot batches and the higher saponification value of the final batch require confirmation in additional batches and formal stability studies before definitive product specifications or clinical efficacy claims are established.

Keywords: Ayurvedic pharmaceutics; Ghrita Manda; LC-MS; melasma; Sneha Kalpana; standardization; Vyanghar Ghrita; Vyanga

How to cite this article: Patil S, Gadgil S. A novel approach to the formulation and standardization of Vyanghar Ghrita. Int J Drug Deliv Technol. 2026;16(74s): 200-212. DOI: 10.25258/ijddt.16.74s.23

.

1. Introduction

Melasma is a chronic, acquired hypermelanosis that predominantly affects sun-exposed areas of the face and is associated with cosmetic concern, recurrence and impaired quality of life. Its pathogenesis is multifactorial and includes ultraviolet exposure, hormonal influences, genetic susceptibility, inflammation, oxidative stress and altered melanogenic signalling. Although topical depigmenting agents, photoprotection and procedural therapies are widely used, outcomes are variable and recurrence is common [6-9]. These characteristics create a continuing need for

standardized, acceptable topical preparations that can support long-term use.

Vyanga is described in Ayurvedic literature among Kshudra Roga and is characterized by painless, dark discoloration of the face. Classical management emphasizes Dosha pacification, Rakta-related measures and the external use of Varnya or complexion-promoting drugs [10-13]. A formulation conceptually related to Mukhakantikar Lepa employs Haridra, Daruharidra, Manjishtha, Gairik and Sarshapa with a lipid and milk medium. The ingredients are traditionally associated with Varnya, Twachya, Shothahara, Raktaprasadana or Lekhana actions, providing a rational basis for investigation in facial hyperpigmentation.

Fresh Lepa preparations, however, present pharmaceutical and practical limitations. Their consistency depends on daily preparation, liquid quantity, particle size and application technique. Inclusion of milk increases perishability and the possibility of microbial deterioration. Such preparations are also difficult to package, transport and dose uniformly. Conversion into a medicated lipid dosage form can improve convenience, protect lipid-soluble constituents and provide a more homogeneous topical vehicle.

Sneha Kalpana is a classical pharmaceutical process in which herbal paste and aqueous media are processed with Ghrita or Taila until the aqueous phase is removed and the active constituents are transferred into the lipid matrix [2-5]. Ghrita is valued as a carrier capable of dissolving or dispersing both lipid-soluble and selected polar constituents. Ghrita Manda, the clear upper fraction of settled Ghrita, was selected for the present formulation because it is comparatively light, smooth and readily spreadable. Its selection was intended to improve facial application without changing the fundamental Sneha Paka principle.

Standardization is especially important for an innovative Ayurvedic formulation that does not have a dedicated pharmacopoeial monograph. Botanical identity, mineral purity, particle size, proportions, heating pattern, endpoint tests, filtration and storage can all influence product quality. A scientifically useful standard must therefore combine classical process controls with measurable analytical parameters and must clearly distinguish observed data from provisional acceptance limits [1,2,5].

The present work was designed as a pharmaceutical and analytical study, not as a clinical efficacy trial. It reports development of the manufacturing procedure, observations during scale-up, quality evaluation of raw materials, batch-to-batch comparison of pilot preparations and characterization of the final product using physicochemical, microbiological and LC-MS methods. The resulting data are intended to serve as a preliminary reference for future stability, safety and clinical studies.

2. Aim and Objectives

Aim: To develop and analytically standardize Vyanghar Ghrita as a reproducible topical Siddha Ghrita intended for further evaluation in Vyanga (melasma).

1. To authenticate and standardize the herbal and mineral raw materials used in the formulation.
2. To develop a pilot-batch procedure and finalize a scalable Standard Operating Procedure (SOP) based on classical Sneha Paka principles.
3. To document critical process parameters, Paka Siddhi Lakshanas, yield and in-process quality controls.
4. To evaluate batch-to-batch consistency using selected physicochemical parameters.
5. To characterize the final product by physicochemical testing, fatty-acid profiling, microbiological analysis and LC-MS fingerprinting.
6. To identify limitations and propose provisional quality attributes for future validation.

3. Materials and Methods

3.1 Study design and place of work

This was a pharmaceutical development and analytical standardization study. Raw-material authentication and selected in-house tests were performed at Bharati Vidyapeeth (Deemed to be University), College of Ayurved, Pune. Taxonomic authentication of the herbal drugs was obtained through the Interactive Research School for Health Affairs (IRSHA), Pune. Specialized physicochemical and microbiological testing was performed through Jeevanrekha Analytical Services, Chhatrapati Sambhajinagar, and LC-MS testing was undertaken with support from Poona College of Pharmacy, Pune, as stated in the supplied study record.

3.2 Ingredients and procurement

Haridra, Daruharidra, Manjishtha, raw Gairik and Peetasarshap were procured from a GMP-certified Ayurvedic pharmacy. Cow Ghrita was obtained from Katraj Dairy, Pune, and goat milk was obtained from an FSSAI-approved supplier. Materials were inspected for identity, colour, odour, taste, texture, foreign matter, insect infestation, fungal growth and signs of rancidity before use.

Table 1. Ingredients of Vyanghar Ghrita

No.	Ingredient	Botanical name / chemical identity	Part used	Proportion
1	Haridra Churna	Curcuma longa Linn.	Rhizome	1 part
2	Daruharidra Churna	Berberis aristata Linn.	Root bark	1 part
3	Manjishtha Churna	Rubia cordifolia Linn.	Stem and root	1 part
4	Shuddha Gairik	Red oxide of iron (Fe_2O_3-containing mineral)	Mineral	1 part
5	Peetasarshap Churna	Brassica alba Linn.	Seed	1 part

Figure 1. Ghrita Manda, herbal and mineral ingredients, and goat milk used in the formulation.

3.3 Authentication and pretreatment of raw materials

The four herbal ingredients were identified by macroscopic, organoleptic and taxonomic criteria. Powdered materials were cleaned, shade-dried where required, pulverized separately and passed through a fine sieve (approximately No. 80-100) to minimize coarse particles and improve uniform dispersion in the Kalka.

Raw Gairik was purified by Bharjana. The material was heated with Ghrita Manda under continuous mixing until the colour changed from bright red to a more uniform brick-red shade. The purified material was cooled, powdered and stored in a dry container. XRD and FTIR were used as complementary methods to document its mineral and functional-group profile.

Ghrita Manda was collected from cow Ghrita by allowing the product to remain undisturbed and separating the clear upper fraction. From 10 kg of Ghrita, approximately 3.5 kg of Ghrita Manda was obtained, giving a collection yield of 35%. This fraction was used as the Sneha Dravya in all pilot and final preparations.

3.4 Pilot batches and formulation ratio

Three pilot batches (A, B and C) were prepared under the same controlled conditions to evaluate reproducibility. Each pilot batch used 12.5 g total Kalka (2.5 g of each ingredient), 50 g Ghrita Manda, 200 g water and 50 g goat milk. The core Kalka:Sneha:water proportion was therefore 1:4:16, with goat milk additionally included in a quantity equal to the Sneha. Observations from these batches were used to define the final SOP and the analytical comparison range.

Table 2. Composition of the scaled final batch

No.	Material	Role	Quantity
1	Haridra Churna	Kalka Dravya	100 g
2	Daruharidra Churna	Kalka Dravya	100 g
3	Manjishtha Churna	Kalka Dravya	100 g
4	Shuddha Gairik	Kalka Dravya	100 g
5	Peetasarshap Churna	Kalka Dravya	100 g
6	Total Kalka	Paste phase	500 g
7	Ghrita Manda	Sneha Dravya	2,000 g
8	Water	Principal Drava Dravya	8,000 mL
9	Goat milk	Accessory Drava Dravya	2,000 mL

3.5 Standard operating procedure for Vyanghar Ghrita

1. Accurately weigh 100 g each of Haridra, Daruharidra, Manjishtha, Shuddha Gairik and Peetasarshap Churna. Combine and triturate to form a uniform Kalka.
2. Weigh 2,000 g Ghrita Manda. Heat gently until the raw odour subsides, avoiding smoking or overheating.
3. Add the 500 g Kalka to the warm Ghrita Manda and mix until uniformly dispersed.
4. Add 8,000 mL water gradually with continuous stirring, followed by 2,000 mL fresh goat milk.
5. Heat the mixture over Manda-Madhyama Agni. Maintain continuous or frequent unidirectional stirring to avoid sticking and local charring. Record temperature periodically.
6. As evaporation proceeds, monitor reduction in volume, change in colour and odour, cessation of frothing and separation of the Ghrita phase.
7. Assess Madhyama Paka by Varti formation, absence of crackling on ignition, Phena-shanti and development of characteristic Gandha and Varna.
8. When all endpoint criteria are simultaneously present, discontinue heating and filter the product while warm through double-layered muslin cloth.
9. Allow the filtrate to cool, record final weight and percentage yield, then transfer into labelled amber-coloured airtight containers.
10. Store below approximately 25 °C in a dry, light-protected place until testing or use.

3.6 In-process controls and Paka Siddhi

Critical in-process controls included fineness of Churna, smoothness of Kalka, uniform mixing, controlled flame intensity, prevention of charring, periodic temperature recording, disappearance of free aqueous phase and confirmation of all endpoint signs before filtration. Varti Pariksha was treated as a practical endpoint: the Kalka had to form a soft, non-sticky wick that burned with a steady flame and without crackling, indicating minimal residual moisture.

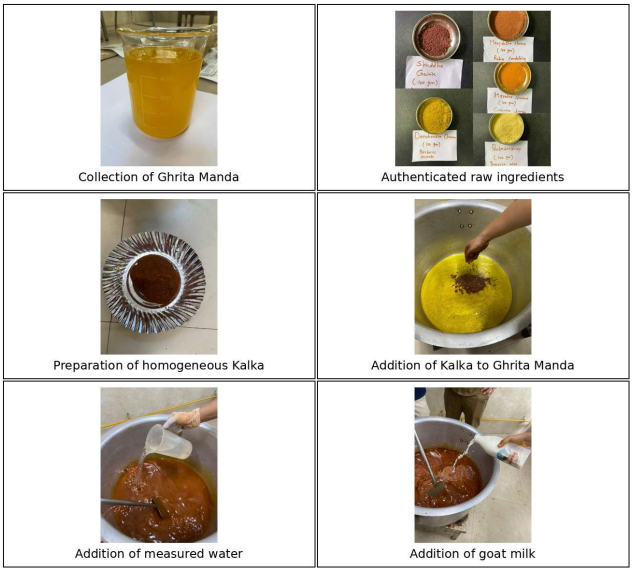

Figure 2A. Initial stages: raw materials, Kalka preparation and addition of liquid media.

Figure 2B. Controlled Paka, filtration and final packaging of Vyanghar Ghrita.

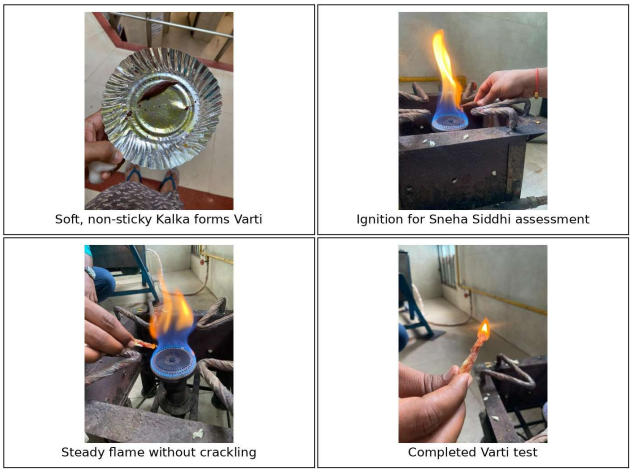

Figure 3. Sneha Siddhi assessment by Varti formation and ignition without crackling.

3.7 Analytical evaluation

Analytical procedures were selected from the Ayurvedic Pharmacopoeia of India, Indian Pharmacopoeia, WHO quality-control guidance, AOAC methods and relevant BIS procedures, as applicable to herbal powders, mineral materials and lipid-based topical preparations [1,2,5]. Because Vyanghar Ghrita has no dedicated monograph, available reference ranges were used as interpretive comparators rather than as definitive product specifications.

Table 3. Analytical tests performed

Material / stage	Tests
Herbal raw materials	Organoleptic examination, moisture content, total ash, water-soluble extractive, alcohol-soluble extractive and pH; TLC fingerprinting as available.
Shuddha Gairik	Organoleptic examination, X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR).
Pilot batches A-C	pH, specific gravity, acid value, peroxide value and saponification value.
Final Vyanghar Ghrita	Organoleptic profile, saponification value, iodine value, refractive index, peroxide value and acid value.
Lipid quality	Fatty-acid fractions by AOAC 996.06.
Microbiological quality	Total viable count, yeast and mould counts, and absence of E. coli, S. aureus, Salmonella spp. and P. aeruginosa.
Phytochemical fingerprint	Methanol-water extraction and qualitative LC-MS profiling of major detected constituents.

3.8 LC-MS sample preparation and interpretation

Approximately 0.5 g of Vyanghar Ghrita was extracted with methanol:water (80:20, v/v). The mixture was vortexed, sonicated for 30 minutes and centrifuged at 5,000 rpm for 10 minutes. The supernatant was passed through a 0.22 µm PTFE membrane filter and transferred to LC-MS vials. Relative abundance values were used to construct a qualitative chemical fingerprint. They should not be interpreted as validated absolute assay values unless confirmed with reference standards, calibration curves and a fully validated quantitative method.

3.9 Data handling

Pilot-batch results were compared descriptively to assess inter-batch variation. Numerical results are reported as supplied by the analytical laboratories. No inferential statistical analysis was undertaken because the study was intended to develop a process and preliminary product profile rather than to test a clinical hypothesis.

4. Results

4.1 Raw-material quality

The herbal powders were uniform in appearance and free from visible contamination. Moisture values ranged from 6.40% to 9.46%, while total ash ranged from 2.10% to 4.45%. Extractive values varied in accordance with the phytochemical composition of each drug. Daruharidra showed the most acidic pH (4.5), whereas Haridra and Manjishtha each showed pH 6.5. These values provide baseline identity and quality data for the raw materials used in the formulation.

Table 4. Physicochemical evaluation of herbal raw materials

Parameter	Haridra	Manjishtha	Peetasarshap	Daruharidra
Moisture content (%)	9.46	7.02	9.00	6.40
Total ash (%)	4.45	4.26	2.10	3.10
Water-soluble extractive (%)	14.00	18.00	18.00	15.50
Alcohol-soluble extractive (%)	15.00	7.04	9.10	14.00
pH	6.5	6.5	5.5	4.5

4.2 XRD and FTIR profile of Shuddha Gairik

The XRD profile indicated a predominantly muscovite-containing mineral matrix with an iron-silicate hydroxide fraction and a smaller hematite fraction. The reported phase percentages were

normalized within the crystalline fraction; the separately reported global amorphous content was 7.5 ± 3.2%. The FTIR spectrum showed Fe-O, Si-O, Al-O-Si and hydroxyl-related bands, supporting the presence of iron oxide within a silicate-rich mineral matrix.

Table 5. XRD profile of Shuddha Gairik

Mineral phase	Quantitative fraction (%)
Muscovite	74.1 ± 0.5
Iron silicate hydroxide	23.9 ± 1.1
Hematite (Fe_2O_3)	2.1 ± 1.5
Global amorphous content (reported separately)	7.5 ± 3.2

Table 6. Major FTIR bands of Shuddha Gairik

Wavenumber (cm^{-1})	Assignment
692.50	Fe-O bending vibration
777.60	Fe-O lattice vibration
795.60	Fe-O stretching vibration
908.52	Si-O symmetric stretching
936.34	Si-OH deformation
998.53	Si-O stretching
1024.72	Al-O-Si / Si-O-Si stretching
1111.45	Si-O asymmetric stretching
3618.59	Structural O-H stretching
3649.69	Hydroxyl O-H stretching
3687.33	Surface O-H stretching

4.3 Pharmaceutical observations and yield

During initial mixing, the Kalka dispersed gradually in the molten Ghrita Manda. Addition of water and goat milk produced a yellow-orange emulsion. With controlled heating, the aqueous phase progressively evaporated, the preparation thickened and the colour deepened to reddish orange or golden-brown. Continuous stirring prevented adherence of Kalka to the vessel.

Temperature during the active evaporation stage was maintained approximately between 90 °C and 110 °C. The principal endpoint features were cessation of frothing, appearance of a clear lipid phase, formation of a soft non-sticky Varti, development of characteristic odour and absence of crackling during ignition. The total processing time was approximately four hours, of which 2.5-2.75 hours were required for active Paka.

The final yield from 2,000 g Ghrita Manda was approximately 1,700-1,750 g, corresponding to 85.0-87.5% of the starting Sneha. The main losses arose from Ghrita retained in the spent Kalka and from vessel, transfer and filtration losses. The product was a smooth, homogeneous, readily spreadable Ghrita with no visible phase separation or coarse particles.

Table 7. Approximate duration of the preparation stages

Stage	Activity	Duration
1	Gentle heating of Ghrita Manda until raw odour subsided	20-25 min
2	Kalka preparation, addition of Drava Dravya and initial mixing	15-20 min
3	Active Paka and evaporation with stirring	150-165 min
4	Endpoint assessment and confirmation of Madhyama Paka	15-20 min
5	Warm filtration, cooling and yield determination	20-25 min
Total	Complete manufacturing cycle	Approximately 4 h

4.4 Organoleptic profile of the final product

Table 8. Organoleptic characteristics of Vyanghar Ghrita

Parameter	Observed characteristic
Colour	Reddish orange to golden-brown
Odour	Pleasant, mildly sweet and characteristic herbal-ghee odour
Taste	Madhura with a mild herbal note
Dosage form	Medicated Ghrita; smooth lipid preparation
Consistency	Liquid to soft semi-solid depending on room temperature

Parameter	Observed characteristic
Appearance	Uniform and free from visible foreign particles

4.5 Pilot-batch physicochemical consistency

The three pilot batches showed close agreement for specific gravity, peroxide value and saponification value. Acid value increased slightly from 0.897 to 1.00 mg KOH/g but remained low. The pH values were narrowly distributed (6.87-7.03), demonstrating reproducibility; however, all were at or above the upper end of the cited comparator range. Therefore, pH should be treated as a formulation-specific characteristic requiring confirmation rather than as a confirmed pharmacopoeial compliance parameter.

Table 9. Physicochemical results of pilot batches A, B and C

Parameter	Batch A	Batch B	Batch C	Comparator used in source
pH	6.92	7.03	6.87	4.5-6.7
Specific gravity	0.95	0.95	0.95	0.91-1.03
Acid value (mg KOH/g)	0.897	0.942	1.00	≤3.0
Peroxide value (meq/kg)	2.5	2.5	2.5	≤5
Saponification value (mg KOH/g)	213.07	212.62	212.62	180-235

4.6 Final-batch physicochemical and lipid profile

The final sample retained a low peroxide value and an acid value just below the stated upper comparator. Its refractive index was compatible with a ghee-based lipid matrix. The saponification value (251.83 mg KOH/g) was higher than both the pilot values and the cited 180-235 range. This result should be rechecked in replicate final batches using the same method and laboratory before a product specification is assigned.

Table 10. Physicochemical parameters of the final Vyanghar Ghrita sample

Test parameter	Result	Method stated in source	Interpretation
Saponification value	251.83 mg KOH/g	API Part II, Vol. IV, Appendix 3.8	Higher than pilot batches and cited comparator; requires confirmation.
Iodine value	35.66	API Part II, Vol. IV, Appendix 3.9	Consistent with a relatively saturated lipid profile.
Refractive index	1.4599	API Part II, Vol. IV, Appendix 3.1	Compatible with a ghee-based lipid matrix.
Peroxide value	2.32 meq/kg	API Part II, Vol. IV, Appendix 3.11	Low primary oxidation at time of testing.
Acid value	2.94 mg KOH/g	API Part II, Vol. IV, Appendix 3.10	Near the stated upper comparator and should be monitored in stability studies.

Table 11. Fatty-acid fractions of Vyanghar Ghrita (AOAC 996.06)

Fatty-acid fraction	Result
Saturated fatty acids (SFA)	67.43%
Monounsaturated fatty acids (MUFA)	28.05%
Polyunsaturated fatty acids (PUFA)	4.36%
Trans unsaturated fatty acids	<0.1%

4.7 Microbiological quality

The measured total viable, yeast and mould counts were low. Escherichia coli, Staphylococcus aureus, Salmonella species and Pseudomonas aeruginosa were absent in the tested quantities. These findings support satisfactory hygienic preparation and storage of the tested sample, but they do not substitute for shelf-life microbiological monitoring.

Table 12. Microbiological results

Test	Result	Method
Total viable count	7 × 10^1 CFU/mL	IS 5402
Total yeast count	2 × 10^1 CFU/mL	IS 5403
Total mould count	2 × 10^1 CFU/mL	IS 5403
E. coli	Absent/mL	IS 5887 Part 1
S. aureus	Absent/mL	IS 5887 Part 2
Salmonella spp.	Absent/25 mL	IS 5887 Part 3
P. aeruginosa	Absent/mL	IS 13428 Annex D

4.8 LC-MS phytochemical fingerprint

Nine major constituents were reported in the LC-MS fingerprint. Curcumin was the dominant detected constituent, followed by beta-sitosterol and ferulic acid. Berberine, gallic acid, epicatechin and p-coumaric acid provided additional marker compounds associated with the constituent drugs. The relative-abundance profile is useful as a preliminary identity fingerprint, but quantitative marker limits should be set only after validation with authenticated standards.

Table 13. Major LC-MS-detected constituents and their relevance to hyperpigmentation research

Constituent	Relative abundance	Reported relevance
Curcumin	52.959%	Antioxidant, anti-inflammatory and melanogenesis-modulating activity.
Beta-sitosterol	14.058%	Anti-inflammatory and skin-protective activity.
Ferulic acid	5.034%	Antioxidant and photoprotective activity.
Berberine	2.413%	Antioxidant, anti-inflammatory and melanogenesis-related activity.
Gallic acid	2.375%	Free-radical scavenging and depigmenting potential.
Epicatechin	0.856%	Antioxidant and cytoprotective activity.
p-Coumaric acid	0.724%	Tyrosinase-inhibitory potential.
Piperine	0.322%	Bioavailability-enhancing and antioxidant activity.
L-Citrulline	0.313%	Supportive antioxidant and microcirculatory relevance.

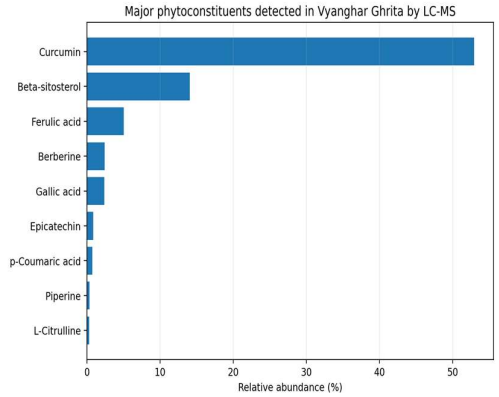

Figure 4. Relative abundance of major constituents detected by LC-MS.

5. Discussion

5.1 Pharmaceutical rationale

The principal pharmaceutical contribution of this study is the conversion of a perishable, freshly prepared Lepa concept into a standardized medicated lipid formulation. This modification directly addresses uniformity, portability and storage limitations that can interfere with routine topical use. It also creates a dosage form that can be manufactured under controlled conditions and subjected to conventional quality testing. The work should therefore be viewed primarily as dosage-form development rather than as evidence of clinical superiority.

Ghrita Manda was selected in preference to whole Ghrita because of its lighter consistency and ease of spreading. Classical descriptions attribute Sukshma and Varnya qualities to the upper clear fraction of Ghrita [14,15]. From a pharmaceutical perspective, a less viscous lipid phase may improve application over the facial surface and facilitate

distribution of lipophilic constituents. Nevertheless, comparative rheological or skin-permeation data for Ghrita and Ghrita Manda were not generated in the present study; the proposed advantage remains a rationale for future testing.

The formulation retained the classical Kalka:Sneha:water ratio of 1:4:16 and added goat milk in a quantity equal to the Sneha. This practical interpretation preserved adequate liquid for extraction while incorporating the milk component of the original Lepa concept. The consistency of pilot batches suggests that the ratio and operating conditions were reproducible at small scale. Scale-up to a 2,000 g Sneha batch was achieved without a major change in process sequence, although final-batch analytical differences indicate that scale can influence the product profile.

Classical Paka Siddhi tests remain valuable because they integrate several process variables: moisture removal, consistency of Kalka, separation of lipid phase and development of characteristic odour and colour. In this study, the simultaneous use of Phena-shanti, Varti formation and absence of crackling reduced dependence on a single subjective observation. These tests were also supported by controlled heating, temperature monitoring and immediate warm filtration, forming a practical hybrid of classical and modern process control.

5.2 Raw-material standardization

Quality of a polyherbal-mineral product is constrained by the quality of its ingredients. The observed moisture and ash values were useful for confirming that the herbal powders were dry and not grossly contaminated with inorganic matter. Water- and alcohol-soluble extractives generated ingredient-specific profiles that can be used in future procurement specifications. Because only single values were supplied, future work should report replicate determinations, mean, standard deviation and acceptance ranges.

The XRD and FTIR results provided complementary evidence for the identity of Shuddha Gairik. XRD indicated hematite within a predominantly silicate mineral matrix, while FTIR showed characteristic Fe-O and silicate-related bands. The relatively small hematite fraction and the high muscovite fraction illustrate why mineral raw materials should not be described solely by a nominal chemical formula. Future standards should include elemental analysis for iron and relevant toxic metals together with mineralogical profiling.

5.3 Batch reproducibility and physicochemical interpretation

The pilot batches demonstrated very close values for specific gravity, peroxide value and saponification value, indicating good short-term process reproducibility. The acid values were also low and narrowly distributed. These data support the use of the developed SOP for future pilot production.

The pilot-batch pH values were consistently close to neutral and slightly above the cited range of 4.5-6.7. Because pH measurement in a lipid product depends on sample preparation and aqueous dispersion conditions, the analytical procedure must be clearly fixed before an acceptance limit is defined. The consistent values may represent a genuine product characteristic related to goat milk, the mineral component or the measurement method rather than a processing fault.

The final batch showed a saponification value of 251.83 mg KOH/g compared with approximately 213 mg KOH/g in the pilot batches. A higher value generally corresponds to a greater proportion of lower-molecular-weight fatty acids, but the magnitude of the difference also raises questions about raw Ghrita source, sampling, batch scale, method transfer or laboratory variation. It would be scientifically inappropriate to declare full compliance without repeat testing. At least three independent final-scale batches should therefore be analysed in the same laboratory with method controls.

The final acid value of 2.94 mg KOH/g was below the stated limit of 3.0 but close to it, whereas pilot-batch acid values were below 1.0. This difference may reflect storage interval, scale, sample handling or hydrolytic change. The low peroxide value suggests limited primary oxidation at the time of testing, but acid and peroxide values should be monitored together during accelerated and real-time stability studies.

5.4 Fatty-acid profile and topical suitability

The lipid profile was dominated by saturated fatty acids, followed by monounsaturated and a small polyunsaturated fraction. This pattern is consistent with a ghee-rich matrix and explains the relative oxidative stability of the formulation. The MUFA fraction may contribute to emolliency and spreadability, while the low trans-fat level indicates that the controlled heating process did not produce a measurable increase in trans unsaturated fatty acids.

For a topical product, fatty-acid composition is relevant not only to stability but also to texture and interaction with the skin barrier. However, the present study did not measure viscosity, spreadability, occlusivity, skin irritation or in-vitro release under standardized conditions. These parameters should be incorporated into subsequent product-development work, especially if the formulation is intended for repeated facial use.

5.5 Microbiological quality

The low microbial counts and absence of specified pathogens indicate that the tested batch was prepared and stored hygienically. The removal of water during Sneha Paka and warm filtration likely reduced microbial risk. Nevertheless, goat milk is introduced at the beginning of the process and may increase microbial risk if Paka is incomplete. Routine manufacture should therefore include verified endpoint control, clean equipment, controlled filling and microbiological testing at release and during stability.

5.6 LC-MS profile and mechanistic relevance

The LC-MS fingerprint identified marker compounds that can plausibly originate from the herbal ingredients. Curcumin, the principal detected constituent, has reported antioxidant, anti-inflammatory and melanogenesis-modulating effects [17-19]. Berberine, ferulic acid, gallic acid and p-coumaric acid have also been investigated for antioxidant, photoprotective or tyrosinase-related activity [20-23]. Beta-sitosterol, epicatechin and piperine add anti-inflammatory, antioxidant or bioavailability-related properties [24-26].

The combined profile provides a rational chemical basis for further study in hyperpigmentation, but mechanistic plausibility is not equivalent to clinical efficacy. Relative abundance obtained from a qualitative LC-MS run can be influenced by ionization efficiency, extraction recovery and matrix effects. Therefore, the reported percentages should be used as a comparative fingerprint rather than as absolute composition. A validated assay for selected markers, particularly curcumin and berberine, would strengthen batch control.

5.7 Relevance to Vyanga and melasma research

Current melasma management generally requires sustained photoprotection and repeated topical treatment, yet irritation, incomplete response and recurrence remain common [6-9,29-32]. A stable, cosmetically acceptable Ayurvedic topical preparation may therefore merit evaluation, particularly where traditional practice is integrated with standardized manufacture. The present formulation combines a ghee-based vehicle with ingredients that are classically described as Varnya and that contain analytically detectable antioxidant or anti-melanogenic constituents.

The study does not contain patient outcomes, comparator data, lesion scores, colorimetry, dermoscopy or safety observations in human subjects. Accordingly, the results establish pharmaceutical feasibility only. A future clinical protocol should include standardized melasma severity scoring, colour measurements, validated quality-of-life measures, adverse-event monitoring, adherence assessment, sunscreen control and follow-up for recurrence [33-37].

5.8 Proposed preliminary quality attributes

On the basis of the present data, the following attributes may be used provisionally for future batch development: characteristic reddish-orange to golden-brown appearance; homogeneous, readily spreadable consistency; specific gravity near 0.95; low peroxide value; acid value below the applicable limit; absence of specified pathogens; and a reproducible LC-MS fingerprint with curcumin and berberine as key markers. Numerical release limits should not be finalized until data from multiple final-scale batches and stability studies are available.

6. Strengths and Limitations

Strengths of the study include documentation of raw-material authentication, use of three pilot batches, explicit recording of classical endpoint criteria, scale-up to a larger batch, and integration of physicochemical, microbiological, mineralogical and LC-MS methods. The study also provides practical manufacturing photographs and identifies process parameters that can be incorporated into a Batch Manufacturing Record.

The principal limitations are the absence of replicate statistics for most analytical tests, lack of real-time or accelerated stability data, incomplete method-validation information, no rheological or dermal-release testing, and no clinical efficacy or safety outcomes. The final-batch saponification value differed materially from the pilot results, and the pilot pH values exceeded the cited comparator range. Heavy-metal safety cannot be concluded because numerical elemental-analysis results were not supplied. These issues should be resolved before regulatory specification, shelf-life assignment or therapeutic claims are made.

7. Conclusion

Vyanghar Ghrita was successfully developed as a modified Siddha Ghrita using Haridra, Daruharidra, Manjishtha, Shuddha Gairik and Peetasarshap in a Ghrita Manda, water and goat-milk system. A reproducible stepwise SOP was established through three pilot batches and a scaled final preparation. Classical Sneha Siddhi criteria, practical yield, raw-material testing, low microbial counts and a multi-constituent LC-MS fingerprint support the pharmaceutical feasibility of the formulation.

The data should be regarded as a preliminary standardization profile. The consistency of pilot-batch parameters is encouraging, but the final-batch saponification value, near-limit acid value and formulation-specific pH require confirmation. Additional final-scale batches, validated marker assays, stability testing, dermal performance

studies and controlled clinical evaluation are required before Vyanghar Ghrita can be assigned definitive quality specifications or claimed to be effective for melasma.

8. Declarations

Acknowledgements: The authors acknowledge Poona College of Pharmacy, Jeevanrekha Analytical Services, Chhatrapati Sambhajinagar, the Pharmacy Section of Bharati Vidyapeeth (Deemed to be University), College of Ayurved, and IRSHA for their support in manufacture, authentication and analytical testing.

Conflict of interest: The authors declared no conflict of interest in the supplied manuscript.

Funding: No external funding information was provided in the supplied manuscript.

Ethics statement: This article reports pharmaceutical and analytical development and does not present human participant outcome data. Any subsequent clinical study should obtain institutional ethics approval and prospective registration as applicable.

Data availability: The numerical data presented in this manuscript were compiled from the supplied batch records and laboratory reports. Original analytical certificates should accompany journal submission where required.

9. Recommended Validation Framework

The present study provides a development-stage analytical profile. Conversion of these observations into formal release specifications requires a planned validation programme that separates critical process parameters from finished-product quality attributes. The programme should use at least three independent final-scale batches prepared from separately procured raw materials, with the same equipment, heating pattern and analytical laboratory wherever possible.

Process validation should document raw-material lot numbers, sieve size, Ghrita Manda yield, mixing time, temperature profile, evaporation time, endpoint observations, filtration conditions and final yield. Analytical validation should include replicate testing, method precision, sample-preparation controls and authenticated reference standards for selected LC-MS markers. The purpose is to determine whether the observed differences between pilot and final batches arise from true scale effects, raw-material variation or analytical variability.

Table 14. Proposed critical process parameter and risk-control matrix

Process element	Principal risk	Recommended control
Raw-drug identity	Substitution or adulteration	Taxonomic certificate, organoleptic profile, TLC/marker identity before release.
Moisture in powders	Microbial growth and variable extraction	Set ingredient-specific moisture limits and use moisture-protective storage.
Ghrita Manda quality	Rancidity or variable fatty-acid composition	Record acid value, peroxide value, refractive index and source batch before processing.
Particle size of Kalka	Non-uniform extraction and gritty product	Use a defined sieve and verify uniformity before charging.
Heating intensity	Under-processing or thermal degradation	Record temperature at fixed intervals; avoid smoking and charring.
Residual moisture endpoint	Microbial instability or spattering	Confirm Phena-shanti, Varti test and absence of crackling; consider moisture determination.
Warm filtration	Particulate contamination and product loss	Standardize cloth layers, filtration temperature and pressure.
Filling and storage	Oxidation, light exposure and contamination	Use clean amber airtight containers with defined fill volume and headspace.

A formal stability study is needed because the formulation contains a lipid phase, plant-derived constituents and a milk-derived processing medium. Acid value and peroxide value should be interpreted together, while appearance, odour and phase separation provide practical indicators of deterioration. Microbial monitoring remains essential even though the finished product contains little free water. Marker stability can be followed using curcumin and berberine or another validated pair representing different ingredients.

Table 15. Recommended stability-study design for future specification development

Study component	Suggested condition / test	Suggested interval

Study component	Suggested condition / test	Suggested interval
Long-term storage	25 ± 2 °C / 60 ± 5% RH	0, 1, 3, 6, 9 and 12 months
Accelerated storage	40 ± 2 °C / 75 ± 5% RH	0, 1, 2, 3 and 6 months
Photostability	Protected versus defined light exposure	Initial and end of exposure
Container compatibility	Proposed amber glass or final market pack	At each long-term time point
Physical quality	Colour, odour, homogeneity, phase separation, spreadability and viscosity	Every time point
Chemical quality	Acid value, peroxide value, saponification value, iodine value and refractive index	Every major time point
Marker fingerprint	LC-MS/HPLC identity and selected quantitative markers	0, 3, 6 and 12 months
Microbiological quality	Total counts and absence of specified pathogens	0, 3, 6 and 12 months

After analytical and stability validation, topical performance should be evaluated by standardized spreadability, rheology, in-vitro release, skin-irritation and, where ethically appropriate, dermal permeation methods. A subsequent controlled clinical study should use prespecified eligibility criteria, photoprotection, validated melasma severity measures, objective colour assessment, patient-reported outcomes and recurrence follow-up. These steps would allow pharmaceutical quality, safety and clinical performance to be evaluated as separate but connected evidence domains.

References

1. World Health Organization. Quality control methods for herbal materials. Geneva: World Health Organization; 2011.
2. Ministry of AYUSH, Government of India. The Ayurvedic Pharmacopoeia of India. Part II. New Delhi: Ministry of AYUSH; 2008.
3. Sharangadhara. Sharangadhara Samhita. Vidyasagar PS, editor. Varanasi: Chaukhambha Orientalia; 2017.
4. Govind Das Sen. Bhaishajya Ratnavali. Shastri AD, editor. Varanasi: Chaukhambha Prakashan; 2019.
5. Central Council for Research in Ayurvedic Sciences. Protocol for testing of Ayurvedic, Siddha and Unani medicines. New Delhi: CCRAS; 2018.
6. Handel AC, Miot LDB, Miot HA. Melasma: a clinical and epidemiological review. An Bras Dermatol. 2014;89(5):771-782.
7. Ogbechie-Godec OA, Elbuluk N. Melasma: an up-to-date comprehensive review. Dermatol Ther (Heidelb). 2017;7:305-318.
8. Sheth VM, Pandya AG. Melasma: a comprehensive update. J Am Acad Dermatol. 2011;65(4):689-697.
9. Gupta AK, Gover MD, Nouri K, Taylor S. The treatment of melasma: a review of clinical trials. J Am Acad Dermatol. 2006;55(6):1048-1065.
10. Pallavi G, Virupaksha Gupta KL, Shreevathsa M, Chate VA, Balakrishna DL. Clinical evaluation of Varnya Gana Lepa in Vyanga (melasma). AYU. 2015;36(2):151-156.
11. Sushruta. Sushruta Samhita, Nidana Sthana: Vyanga Nidana Adhyaya. Varanasi: Chaukhambha publications.
12. Vagbhata. Ashtanga Hridaya, Uttara Tantra: Kshudra Roga Pratishedha. Varanasi: Chaukhambha publications.
13. Charaka. Charaka Samhita, Sutra Sthana: Sneha Varga. Varanasi: Chaukhambha publications.
14. Sushruta. Sushruta Samhita, Sutrasthana, Chapter 45: Dravadravyavidhi Adhyaya, Ghritamanda Gunah. Yadavji Trikamji Acharya, editor. Varanasi: Chaukhambha Surbharati Prakashan; reprint 2022.
15. Kaiyadeva. Kaiyadeva Nighantu, Drava Varga: Ghritamanda. Sharma PV, Sharma G, editors. Varanasi: Chaukhambha Orientalia; reprint 2018.
16. Government of India. Ayurvedic Formulary of India. Part I. New Delhi: Ministry of AYUSH.
17. Choi SY, Kim S, Kim H. Curcumin inhibits melanogenesis. Phytother Res. 2010.
18. Tu CX, Lin M, Lu SS, et al. Curcumin inhibits melanogenesis in human melanocytes. Int J Mol Med. 2012.
19. Priyadarsini KI. The chemistry of curcumin: from extraction to therapeutic agent. Molecules. 2014;19:20091-20112.
20. Wang YS, Tay YK, Kwong YL. Berberine and regulation of melanogenesis. Pigment Cell Melanoma Res. 2017.

21. Saija A, Tomaino A, Trombetta D, et al. Ferulic acid as a photoprotective antioxidant. Int J Pharm. 2000.
22. Choi JG, Kim N, Ju SA. Gallic acid suppresses melanogenesis. Int J Cosmet Sci. 2016.
23. An SM, Koh JS, Boo YC. p-Coumaric acid inhibits tyrosinase activity. Phytother Res. 2010.
24. Bouic PJD. The role of phytosterols and phytosterolins in immune modulation. Int J Immunopharmacol. 2001.
25. Srinivasan K. Black pepper and its pungent principle piperine: a review of diverse physiological effects. Crit Rev Food Sci Nutr. 2007;47:735-748.
26. Jug U, Naumoska K, Vovk I. (-)-Epicatechin as a contributor to antioxidant activity. Antioxidants (Basel). 2021;10(1):133.
27. Hassan-Danboyi E, Jimoh A, Alhassan A, et al. Antioxidant effects of L-citrulline supplementation in experimental metabolic stress. Niger J Exp Clin Biosci. 2021;9(2):95-102.
28. Mirenayat MS, Faramarzi M, Ghazvini MR, et al. Effects of short-term citrulline malate supplementation on oxidative stress and muscle damage. Hum Nutr Metab. 2024;36:200242.
29. Austin E, Nguyen JK, Jagdeo J. Topical treatments for melasma: a systematic review of randomized controlled trials. J Drugs Dermatol. 2019;18(11):1156-1171.
30. Desai SR, Alexis AF. Topical and systemic therapies in melasma: a systematic review. J Clin Aesthet Dermatol. 2023.
31. Sarkar R, Arora P, Garg KV. Cosmeceuticals for hyperpigmentation: current evidence and treatment strategies. J Cutan Aesthet Surg. 2022;15(1):1-12.
32. Pennitz A, Kinberger M, Valle GA, et al. Self-applied topical interventions for melasma. Br J Dermatol. 2022;187(3):309-323.
33. Balkrishnan R, McMichael AJ, Camacho FT, et al. Development and validation of a health-related quality-of-life instrument for women with melasma. Br J Dermatol. 2003;149(3):572-577.
34. Kwon IH, Choi JY. Circadian rhythm and skin-barrier repair. Ann Dermatol. 2019.
35. Marks R. Epidermal turnover time. Br J Dermatol. 1980.
36. Kittler H, Marghoob AA. Dermoscopy and disorders of skin pigmentation. Dermatol Clin. 2013.
37. Neema S, Chatterjee M. Dermoscopy of melasma. Indian Dermatol Online J. 2022.
38. Association of Official Analytical Chemists. AOAC Official Method 996.06: Fat (total, saturated and unsaturated) in foods. Gaithersburg, MD: AOAC International.
39. Bureau of Indian Standards. IS 5402, IS 5403, IS 5887 and IS 13428: microbiological examination methods. New Delhi: BIS.
40. Indian Pharmacopoeia Commission. Indian Pharmacopoeia. Ghaziabad: Government of India.