Formulation And Evaluation Of Red Clay-Polyherbal Face Pack

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Abstract
Herbal face packs have been significantly popularized in the past few years as natural alternatives to conventional chemicals-made cosmetic products. With an increasing focus on sustainability and a demand of chemical-free skincare, individuals are turning to herbal face packs for their rejuvenating and nourishing properties. This abstract provides an overview of herbal red clay face packs, their ingredients, benefits, and application methods. Herbal face packs are composed of various botanical powders, and clays, carefully chosen for their unique properties and skincare benefits. Skin detoxification, unclogging pores, debris removal, dead cell exfoliation, promoting cell regeneration and reduction of blemishes, acne scars and pigmentation are numerous benefits of herbal face packs experienced by consumers. As excellent absorbent properties, skin tightening and mineral rich composition clays was the safe option and been used topically on skin across the globe since centuries because of their beneficial effects, this research hasusedFrench red clay for formulation and incorporated with various herbs having their own topical actions on skin. Evaluation of the formulation provided good results promising that the formulation may use by humans after further important tests.

Keywords: Herbs,polyherbal, red clay, Ginseng, face pack.

INTRODUCTION

As an important part of the human body, the facial skin became an indicator of an individual's overall health[1]. Nowadays, it becomes everyone's desire to get fair, radiant, and healthy facial skin. Mostly, teenagers and youngsters are experiencing skin problems such as pimples, blackheads, dark circles, and they highly concerned about it[2].Conventional cosmetics like face mask, creams, lotions, face washes and many more promises to get rid from these problems but they are also well known by individuals for its dark side that is side effects. The chemicals such as parabens, sulfates, phthalates, toluene, and synthetic fragrances and colors are used in these preparations and they are highly toxic and harms the skin on long time usage[3].

Since ancient times, humans already have knowledge that most of the plants nearby us have possess some beneficial properties to maintain healthy skin[4]. In Ayurveda, usage of various plants powders or extracts and clays are mentioned for getting healthy and glowing skin. The facial therapy involves the application of some herbal powders with water, milk or oils as a face pack are known as "Mukha-lepan" and those paste are known as "Mukha-lepa."The main intension to use such herbal mixture was toheal damaged skin as well as to prevent acne, rashes and get clear and bright complexion[5].

Significant progress has been achieved in the study of skin biology and herbal skin cosmetic formulations during the past few decades. Commercial skin care products are widely accessible and include ones made for skin whitening, photo protection, and antiaging[6]. The assumption that natural and home remedies are safer and have fewer adverse effects than synthetic ones have led to a widespread acceptance of their usage. Herbal face packs are used to preserve skin suppleness, boost muscular energy, enhance blood circulation, and clean pores of debris. Herbal cosmetics are nontoxic and less likely to cause allergic responses, among other advantages[7].

Natural face packs are enriched with essential vitamins that are crucial for maintaining the health and radiance of our skin. These ingredients offer multiple benefits for our skin. Using natural facial packs is uncomplicated and easy[8]. They aid in taking care of our skin and promote improved blood circulation in the facial veins, thus showcasing their effectiveness. Herbal clay face packs are a great way to cleanse, detoxify, and nourish your skin. It's important to note that the effects of these facial packs are typically short-term, requiring application 2-3 times a week for consistent radiance[9].

MATERIALS AND METHODS

Red clay, Ginseng, Orange Peel, Amla, Red sandalwood, Neem, Rose Petals.

A) Pre-formulation studies

1. Organoleptic characterization

Evaluation of all the powdered ingredients performed on various organoleptic parameters, including color, odor, appearance, texture, and consistency. These assessments involved visual examination, while texture was evaluated through touch and sensation.

2. Rheological Evaluation

The process revolved around assessing the properties of all the ingredients. The samples were evaluated using a variety of physical criteria, including particle size, Hausner's ratio, bulk and tapped density, and angle of repose.

a) Angle of Repose- 10 gm samples were taken from individual ingredients for determination of the angle of repose (θ). A funnel was positioned at height of 2 cm above surface and sample was dropped into it. The height and radius of the resulting heap was carefully measured and calculated). Followingformula used to calculate the angle of repose (θ):

$$\text{Angle of repose}(\theta) = \tan^{-1} \times \frac{\text{Height (h)}}{\text{Radius (r)}}$$

b) Bulk Density (ρb)-50 gm samples from individual ingredients (M_s) weight on balance and transferred in the 100 ml measuring cylinder. The volume occupied by the samples (V) were observed. Bulk density of the samples was calculated using the formula:

$$\rho b = \frac{\text{Ms (in gm)}}{\text{V(in CC)}}$$

c) Tapped Density- The tapped density (ρt) of a powdered samples were measured by weighing 50 gm of the powdered sample and transferred into measuring cylinder with capacity of 100 ml. Initial volume (Ms) of the powder sample was recorded. The cylinder was placed on a tapped density apparatus at height of 3 mm and continuously tapped100 times for 1 minute to help the powder particles settle more efficiently.After the tapping completed, the final volume of the powder sample in the graduated cylinder was recorded and calculated using; the tapped volume (Vt) by subtracting the initial volume from the final volume.

$$\rho t = \frac{\text{Ms (in gm)}}{\text{Vt (in CC)}}$$

d) Hausner's Ratio- The Hausner's ratio is one of the quantification methods to check flowability of powders. Hausner's ratio is the ratio of tapped (ρt) density to bulk density (ρb) of the powder. Here is the formula to calculate Hausner's ratio:

$$\text{Hausner's Ratio} = \frac{\text{Tapped Density } (\rho t)}{\text{Bulk Density } (\rho b)}$$

e) Particle Size- Particle size analysis is a crucial step to determine the particle size distribution in a sample. The sieve shaker separates particles based on their size by passing them through a series of sieves with progressively smaller openings.The standard sieves were arranged with sieve numbers 10, 22, 36, 44, 65, 100, and 120, so, the coarsest sieve remained at the top, and the finest sieve was at the bottom.Accurately 50g of the sample was weighed and placed on the top sieve [10]. The sieve shaker was closed and shaken for 20 minutes.After shaking, the sample retained on each sieve was collected into separate papers and weight. The weights retained on each sieve were reported in the table against the corresponding sieve number.

B) Formulation of face pack

The powdered ingredients were sieved through a 40-mesh size individually. Optimization of formulations was done by preparingformulations; (F1,F2,F3 & F4), differentiation based on their ingredient's percentage weight. As per the formulation requirement the ingredients were weighed.Subsequently, all the ingredients were mixed and thoroughly triturated in a mortar-pestle. The process was not time bounded, so it was triturated till a uniform and even blend was achieved. Once prepared, the formulation was passed through a 120-mesh size sieve to get approximately 125 µm particle size.The face pack was assessed using various evaluation parameters.

The quantities of each ingredient used are detailed in **Table 1**.

Sr. No.	General Name	Scientific Name	Quantities for 100 gm			
			F1	F2	F3	F4
01	Red Clay	-	30	35	40	45
02	Ginseng powder	Panax ginseng	20	5	15	10
03	Orange peel powder	Citrus sinensis	10	10	10	05
04	Amla powder	Emblica officinalis	10	10	05	05
05	Red Sandalwood powder	Pterocarpus santalinus	15	20	10	05
06	Neem leaves powder	Azadirachta indica	10	10	05	10
07	Rose Petal Powder	Rosa indica	05	10	15	20

Table 1: Formulation composition for 100 gm of red clay-polyherbal face pack.

C) Evaluation of face pack

1. Morphological characterization

Organoleptic characterization of the formulations was done by assaying physical morphological characterization. Diverse organoleptic analysis was performed which encompasses color, aroma, visual appeal, texture, and consistency**(Table 2).**

2. Rheological Evaluation of formulation

The rheological characterization of the formulations; (F1, F2, F3 & F4) was done using various methods. Following are the standard tests suggested by IP to perform for rheological evaluation. It encompasses angle of repose. Particle size distribution, bulk density, tapped density and Hausner's ratio. The flow properties studies revealed that the formulation exhibited a free-flowing and non-sticky nature, meeting the desired criteria for a face pack powder **(Table 3 & 4)**.

3. Physico-chemical Characterization

The Physico-chemical characterization of formulations includes determination of various parameters such as loss on drying (LOD) at 105°C, determination of acid-insoluble ash, alcohol soluble extractive value, Total-ash value, water soluble value and pH. These parameters were followed as per the IP guidelines.

a) pH- The pH of the formulations; (F1,F2,F3 & F4) were determined by using digital pH meter at room temperature. The digital pH meter was calibrated using buffer solutions of pH-7 and pH-4. Calibration helps the device accurately measure pH values within the desired range. A 10% (w/v) dispersion of the formulations were prepared by mixing 10 gm of the formulations with 100 ml of distilled water. Once the calibration of pH meter was done, it underwent directly measurement of pH of 10% dispersion without any further dilutions. The numerical value of the preparation was provided by pH- meter.

b) Moisture Content-Loss on Drying (LOD) method was used for moisture content determination of(F1, F2, F3, F4). Precisely, 2gm of formulation was weighed using balance. A clean and dry glass bottle was weighed before adding the sample. The 2-gm sample was delicately placed inside the pre-weighed glass bottle. Subsequently, the sample along with glass bottle was placed into a hot air oven set at a temperature range100-105ºC. The temperature was set as per the composition of the preparation. Once the sample reached a constant weight, the moisture content was calculated using the following formula:

$$\text{Moisture Content (\%)} = \left[\frac{W2 - W3}{W2 - W1}\right] \times 100$$

Where:
W1 = Weight of the empty glass bottle
W2 = Weight of the empty glass bottle + sample before drying
W3 = Weight of the empty glass bottle + sample after drying

c) Ash Value- It is also known as total ash. It is a crucial parameter in the assessment of pharmaceuticals and herbal drugs. The known weight of sample was allowed to burn or incinerate completely in a standard muffle furnace. The inorganic residue left in the form of ash consists of inorganic mineral components such as salts, metals, and other non-combustible materials was assessed further.

Total Ash Value-

It is impossible to predict the drug identity or purity only from the ash content, but it can provide the insights into the quality of the sample. It was determined by accurately weighing 2 gm of sample into the crucible and weight was recorded. Muffle furnace was set at 550°C and place the crucible into it for 24 hours. Muffle furnace turned off and allowed to cool. The crucible was removed from the muffle furnace and ash sample was weighed and recorded as crucible plus ash sample (total weight).

$$\text{Weight of Ash (mg)} = (\text{weight of cruisible} + \text{Ash}) - (\text{weight of crucible})$$

$$\text{Ash(\%)} = \frac{\text{Weight of ash(mg)}}{\text{Initial sample weight}} \times 100$$

Acid Insoluble Ash Value-

25 ml of hydrochloric acid was added to the crucible containing total ash, and it was covered with a watch glass.The mixture was gently boiled for 5 minutes.The watch glass was then rinsed with 5 ml of hot water and added into the crucible.The insoluble matter was collected on an ash-less filter paper and washed with hot water until it became neutral.The filter paper containing the insoluble matter was transferred to the original crucible and dried on a hot plate.The crucible with the sample was ignited to a constant weight and left to cool in a desiccator for 30 minutes.After

cooling, the sample was weighed.The percentage of acid-insoluble ash was calculated in reference to the air-dried sample.

$$\text{Acid Insoluble Ash Value} = \frac{\text{Weight of Acid Insoluble Ash}}{\text{Weight of Air Dried Sample}} \text{x } 100$$

Water Soluble Ash Value-

25 ml of water was added to the crucible containing the total ash.The mixture was boiled for 5 minutes.The insoluble matter was collected on an ashless filter paper.The collected matter on the filter paper was washed with hot water.The filter paper with the insoluble matter was then subjected to ignition for 15 minutes at a temperature not exceeding 450ºC.After the sample was cooled, it was weighed.The percentage of water-soluble ash was calculated in reference to the airdried sample.

$$\text{Water soluble Ash Value} = \frac{\text{Weight of water soluble Ash}}{\text{Weight of Air Dried Sample}} \text{x } 100$$

4. Stability Studies

Stability testing of the formulations was performed to test the shelf life. The four formulations were packed in 12 glass vials (4×3), 1stset (F1, F2, F3 & F4) was stored for 30 days and another was kept for 60 days and third set for 90 days. Once the fixed duration completed, the formulations were evaluated for physical parameters such as color, odor, pH, consistency, and overall sensory feel.

RESULTS AND DISCUSSIONS

Evaluation tests

Morphological characterization

The formulations; (F1, F2, F3, F4) were subjected to organoleptic characterization through physical morphological analysis. The resulting face pack powders displayed a gentle light brown color. The formulations had pleasant-earthy aroma attributed to its blend of clay, citrus peels, and other thoughtfully chosen ingredients. The texture and particle size of the formulations were found to be well-suited for smooth and effective application on the skin, meeting the standards required for cosmetics formulations **(Table 2)**.

Table 2: Morphological characteristics of the formulation.

Sr. no	Parameter	Observation			
		F1	F2	F3	F4
01	Colour	Light Brown	Light Brown	Light Brown	Light Brown
02	Odour	Earthy-Pleasant	Earthy-Pleasant	Earthy-Pleasant	Earthy-Pleasant
03	Appearance	Fluffy	Fluffy	Fluffy	Fluffy
04	Texture	Free flowing	Free flowing	Free flowing	Free flowing

Rheological Evaluation of formulation

The rheological (Flow) properties of the formulations; (F1, F2, F3, F4), were assessed using several methods by following IP guidelines. The rheological studies revealed that the formulations exhibited a free-flowing and non-sticky nature, meeting the desired criteria for herbal face pack powder (see **Table 3 & 4**).

Table 3: Observation for the particle size determination Particle size observation table

Sieve No.	Aperture Size as Per I.P (µm)	Arithmetic Mean Size of opening (d) (µm)	Weight of Granules Retained on Sieve over Size(gm) (∑n)	% wt. of Granules Retained (Under Size) N	Cumulative% Granules etained (Under Size)	Weight Size (n×d) ∑(n×d)	% Under Size	% Over Size (100% Under Size)
10	1700	1205	2.2	2.20%	2.2	2651	2.2	97.8
22	710	922.5	2	2.00%	4.2	3874.5	4.2	95.8
36	425	602.5	2	2.00%	6.2	3735.5	6.2	93.8
44	355	302.5	3	3.00%	9.2	2783	9.2	90.8
65	250	215	3.3	3.30%	12.5	2687.5	12.5	87.5
100	150	137.5	4.69	4.69%	17.19	2363.63	17.19	82.81
120	125	62.5	10.2	10.20%	27.39	1711.88	27.39	72.61
Pan	0	0	71.5	71.50%	98.89	0	98.89	1.11
			98.89 gm			19807		

Table 4: Flow property evaluation results for herbal face pack.

Rheological evaluations:

Sr. no	Parameter	Observation		Inference
01	Angle of repose (θ)	F1	30°	Excellent flow
		F2	32°	Good flow
		F3	42°	Passable
		F4	38°	Fair flow
02	Bulk density (ρ_b)	F1	1.03	Good flowability
		F2	1.05	Good flowability
		F3	1.07	Passable
		F4	1.06	Fair flow
03	Tapped density (ρ_t)	F1	1.15	Good flowability
		F2	1.20	Good flowability
		F3	1.34	Passable
		F4	1.26	Fair flow
04	Hausner's ratio	F1	1.12	Good flow
		F2	1.15	Good flow
		F3	1.26	Passable
		F4	1.19	Fair flow
05	Particle size (µm)	F1	125	Very fine
		F2	125	Very fine
		F3	125	Very fine
		F4	125	Very fine

Physico-chemical Characterization

pH- Digital pH-meter was used to measurement the pH of the formulations; (F1, F2, F3, F4). At room temperature, the pH of the formulation was found to be as below, indicating its acidic nature, which is beneficial for skin health **(Table 5)**.

Table 5: pH measurement of all formulations.

Sr. No.	Formulation	pH
01	F1	6.2
02	F2	6.2
03	F3	6.0
04	F4	6.1

Moisture content- The moisture content of the formulations; (F1, F2, F3, F4) were assessed by LOD using hot air oven at fixed temperature range and for fixed time duration. The observations are mentioned as below (**Table 6**):

Table 6: Observations of the formulation assessed for LOD.

Sr. No	Conditions	Observations (gm)			
		F1	**F2**	**F3**	**F4**
01	Weight of dry empty glass bottle (W1)	32.68	32.68	32.68	32.68
02	Weight of sample + glass bottle (W2)	34.68	34.67	34.67	34.69
03	Weight of W2 after drying (W3)	34.60	34.56	34.50	34.52
Final Moisture content		4.00%	5.52%	8.54%	8.52%

Ash value- Various techniques were used to determine the ash values ofF1,F2,F3 & F4. The solubility or insolubility of the ash in water and acid provided insights into the inorganic and earthy material present in the sample. The table below shows the different ash values obtained**(Table 7).**

Table 7: Various ash values of the sample.

Sr. no	Ash value	Observation (%)			
		F1	**F2**	**F3**	**F4**
01	Water-Soluble Ash	64.87	63.75	68.35	65.37
02	Acid-Insoluble Ash	54.65	59.25	60.12	55.46
03	Total Ash-value	17.00	15.00	17.00	18.00

Stability studies

The stability investigation reveals a minor pH variation in the formulations when stored up to 90 days that is acceptable and cannot harm skin, while no alterations were observed in the day-30 and day-60 formulations. However, it is worth

noting that there were no changes in color, odor, and other parameter under the rest stability conditions mentioned in the table (**Table 8**).

Table 8: Stability studies of the formulation at different temperatures for given parameters. Changes are mentioned in the observation table. (Abbreviations: N C- No change; N P- Not present).

Sr. No.	Parameter	Observations											
		Day-30				Day-60				Day-90			
		F1	F2	F3	F4	F1	F2	F3	F4	F1	F2	F3	F4
01	Odour	N C	N C	N C	N C	N C	N C	N C	N C	N C	N C	N C	N C
02	Colour	N C	N C	N C	N C	N C	N C	N C	N C	N C	N C	N C	N C
03	pH	6.2	6.2	6.0	6.1	6.2	6.2	6.0	6.1	6.0	6.0	5.9	5.9
04	Texture	N C	N C	N C	N C	N C	N C	N C	N C	N C	N C	N C	N C
05	Grittiness	N P	N P	N P	N P	N P	N P	N P	N P	N P	N P	N P	N P

CONCLUSION

The development of the herbal face pack formulation has yielded valuable insights into its therapeutic properties and aesthetic advantages. This formulation was meticulously crafted through the blending and trituration of various beneficial ingredients.After optimization, four distinct formulations (F1, F2, F3 & F4) were produced, each aimed at achieving the highest quality product for skincare. These formulations underwentassessments across multiple parameters, including morphological characterization, rheological evaluation, Physico-chemical characterization, stability studies. During this rigorous evaluation process, several shortcomings and drawbacks were identified in formulations F1, F3, and F4.Formulation F1 raised concerns as it can cause skin irritancy upon application as it contains high ginseng concentration. Additionally, formulations F3 and F4 exhibited slight pH changes after a 90-day stability assessment. However, it is worth noting that no significant flaws were detected during the evaluation of formulation F2.The thorough evaluation of formulation F2 demonstrated the most promising effects on the skin. Consequently, it can be considered a safe product with some recommended precautions for usage.

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