ORIGINAL ARTICLE

Design, Optimization, and Characterization of Herbal Gel for Facial Application

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Abstract

The objective of this research is to create and test an herbal face gel for cosmetic uses that is comprised natural ingredients. A local market was the source of the amla (*Emblica officinalis*), masoor dal (*Lens culinaris*), and rose petals, which were then dried, powdered, geometrically mixed, and chemically evaluated as well as for organoleptic and physicochemical evaluation. Four separate mixtures, designated F1 to F4, were created using different ratios of components such *E. officinalis*, *L. culinaris*, rose petal powder, and polymers. All of the produced compositions were evaluated using a variety of metrics, including preformulation studies, pH, viscosity, spreadability, drying time, stability, statistical analysis, and an irritancy test. The viscosity of F1, F2, F3, and F4 was observed 15101.3 ± 0.55,149 44.9 ± 0.91, 16721.3 ± 0.90, and 16931.5 ± 0.96, respectively, and the pH was observed 5.4 ± 0.1, 5.6 ± 0.07, 5.5 ± 0.03, and 5.7 ± 0.1, respectively. The one-way analysis of variance approach demonstrated linearity for all answers at $P \leq 0.05$.

Key words: Carbopol, *Emblica officinalis*, facial gel, *Lens culinaris*, polyvinyl alcohol, polyvinylpyrrolidone, rose petals

INTRODUCTION

Cosmetics are over-the-counter items that are aimed to enhance the look of the skin by washing, beautifying, and enhancing attractiveness. Herbs have been utilized for cleansing, rejuvenating, and managing them since ancient times. The skin of the face is the largest portion of the body and reflects an individual's health.[1] It is made up of components such as amino acids, lipids, and carbs; thus, it requires a well-balanced diet to maintain it clean, shiny, and healthy.[2] The herbal paste is known as "mukhalepa" in Ayurveda which is used as a face therapeutic. This herbal paste is used to treat acne, pimples, scars, markings, and pigmentation on the face.[3] Cosmetic masks with clay scrubs' appeal can be ascribed to various psychophysiological effects. Several available commercially skin and hair care products contain the mystery and mythology of "moist soil" therapies. These products are commonly classified as therapy cosmetics and come as a result of jellies, viscous liquids, or pastes that are placed to the facial, body, hair, or scalp. The mask's aromas as well as drying time are intended to add towards the mask's or clay scrub's medicinal value. Several modern mask formulations mix pharmacological activities, physiological skin and hair enhancers, and cleaning ingredients with the curative feeling of the mask composition. Acne therapies, gritty body clay washes, alpha- or beta-hydroxy acid gel or clay masks, hair adjusters, and body stimulants or conditioners are among them.[4] They are normally kept on every day for 15–20 min to enable all of the moisture to evaporate, and the resultant film compresses and hardens, making it easy to remove. The exciting sensation of a regenerated face is achieved by the administration of a facial mask, whereas the colloidal and adsorption clays employed in these treatments eliminate oil and dirt out from skin of the face. Whenever the administered face pack is eliminated, the skin waste and grime placed on it are also excluded. Women can get eliminated of wrinkling, dark bags, blemishes, and acne using the face

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packs suggested in Ayurveda. Herbal facial packs improve skin's radiance and smoothness. We can get the most out of herbal face packs if we use them appropriately for our skin type. These face packs improve skin radiance and are the most effective ayurvedic therapy for increasing impartiality. Face packs are among the most ancient and attractive ways to cleanse the face. Ayurveda describes a variety of facial packs that include feeding, soothing, cleansing, astringent, and antibacterial effects. Face packs may be made at home using common household and kitchen ingredients. Herbal face packs are less expensive and have no adverse effects when it comes to organically achieving fair skin. Herbs have been used for cleansing, beautification, and management since antiquity. Cosmetics are described as items that are used for washing, grooming, enhancing attractiveness, or changing one's look.[5] Smooth, vibrant, and silky skin may be achieved with handcrafted natural face packs as well as mask. Mukhalepa is an Ayurveda herbal paste used to cure acne, pimples, scars, markings, and pigmentation on the face. The practice of putting an herbal mixture over one's face is known as "mukhalepana." This treatment is now known as face treatment.[3] Ayurvedic face pack gels can help minimize creases, blemishes, acne, and eye bags. They also improve the skin's attractiveness and suppleness.[6] Natural face pack gels do include certain essential vitamins that really are necessary for our skin's wellness and radiance.

MATERIALS AND METHODS

Amla (*Emblica officinalis*) [Figure 1a], a member of the *Euphorbiaceae* family, is a popular herbaceous remedy used in a range of medicinal formulations for a variety of ailments. It is also known as Amla or Indian gooseberry. The berries are cooling, astringent, and refrigerant. Antibacterial, emmenagogue, anabolic, adaptogenic, antimicrobial, antiviral, antioxidant, anti-aging, cosmetics, and antiemetic properties that are all found in Amla fruits.[7,8]

MasoorDaal (*Lens culinaris*) [Figure 1b] belongs to the *Fabaceae*/*Leguminosae* family. Lentil had the greatest total antioxidant capability across pulses when assessed by ferric minimizing antioxidant potential and total radical-trapping antioxidant parameter, but it was second to wide beans when tested by trolox similar antioxidant capability.[9] Duenas *et al*. validated these observations, indicating that lentils had more antioxidant properties as beans.[10] Red lentils provide a variety of skin-beneficial properties. To begin with, it slows down the aging process.

Rose petals powder (*Rosa canina*) [Figure 1c] is high in antibacterial characteristics, as well as Vitamin K, C, and B, which have beneficial benefits. It also has a significant level of antioxidants.

Polyvinyl alcohol (PVA), polyvinylpyrrolidone (PVP), Carbopol 940, and ethanol were used for the preparation of face pack gel. PVA excels in film formation, emulsification, and adhesion. It has a great specific strength, elasticity, and an excellent shield to oxygen and odor. PVP has been utilized in the therapeutic and biomedical industries to create a variety of drug delivery methods, including orally, topical, percutaneous, and ocular delivery. Carbopol®, a brand of Lubrizol Company (Wickliffe, OH, USA), is a group of commercial polymeric material used as a thickener, suspending, scattering, and stabilizing ingredient in the cosmetology, pharmaceuticals, painting, and food industries.[11]

Preformulation studies

PVA, PVP, Carbopol 940, ethanol, and methanol were used for the preparation of the face pack gel.

The infrared spectral determination of PVA, PVP, and Carbopol 940

The infrared spectral analysis was analyzed using FT/IR-4600 type A model. The Nujol method was used for the preparation of the sample (PVA, PVP, and Carbopol 940). In this method, PVA, PVP, and Carbopol 940 were distributed (dispersed) in non-volatile Nujol (liquid paraffin) which has a similar refractive index to the drug, and then IR spectroscopy was conducted. For the preparation of the sample, 10 mg polymer was pulverized in a mortar and pestle. Then, two drops of Nujol were added and mixed to distribute the polymer in Nujol. Apply the paste to a KBr crystal plate (liquid cell) and sandwiched it with another plate.

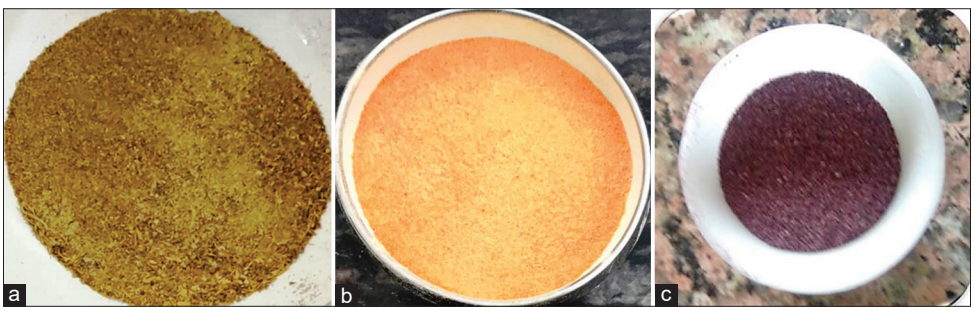

Figure 1: (a) Powder of Amla (*Emblica officinalis*), (b) Powder of Masoor Daal (*Lens culinaris*), (c) Rose petals powder (*Rosa canina*)

Differential scanning calorimetry (DSC) of PVA, PVP, and Carbopol 940

The melting temperature of PVA, PVP, and Carbopol 940 was measured by using DSC with a programmed heat at a rate of 10°C/min. PVA sample (~5 mg) was heated in crimped aluminum pans at a scanning rate of 10°C min^{-1} using dry nitrogen flow (30 mL min^{-1}) within a range of 21.83–203°C.

Herbal face pack gel preparation

The influence of two independent variables was investigated using a 2^2 factorial design focus on the model of experiments approach.[12,13] The values of the two components, the quantity of carbopol 940 (X1), and the quantity of PVP (X2) were appropriately entered. As output responses, pH (Y1) and viscosity (Y2) were used. Two criteria were assessed in this study, each at two levels. All feasible permutations (F1-F4) were tested in the experiments. Throughout the trial, all other composition and cognitive factors were held constant. Table 1 displays factorial combinations based on 2^2 different factorial designs.

Method of preparation

The herbal face mask gel was made by mixing several ingredients that have been prepared. Four variants of the formulation were made, with a comparison between different concentrations of carbopol 940 and PVP. For the optimization of herbal face pack gel, we used the 2^2 factorial designs. In this design, we used two-factor carbopol 940 (2 g and 3 g, low and high level, respectively) and PVP 1 g and 2 g, low level and high level, respectively, for the pH and viscosity. In this process, PVA was used as a film-forming agent, carbopol 940 and PVP as a gel base, and hydantoin as a preservative.[14]

Evaluation parameters of herbal face pack gel formulations

The pH determination

Since the preparation was used on the skin (topically), pH determination was essential to confirm its non-irritating effect. The digital pH meter (Rolex, India) was used for the determination of the pH of the prepared herbal face pack gel at ambient temperature. Face pack gel was weighed (2 g), and the dispersion medium was purified water (20 mL). The 4, 7, and 9 pH buffer solution was used for the calibration of the pH meter. The samples were repeated in a triplicate manner, and mean values were calculated.

Table 1: Optimization of formulation using 2^2 factorial design

Factor	Level used	
	Level -1 (low)	Level 1 (high)
Independent variables		
X_1 = Carbopol 940	2	3
X_2 = PVP	1	2
Dependent variables		
Y_1 = pH		
Y_2 = viscosity		

Spreadability determination

The formulated face pack gel spreadability was analyzed after 72 h of the formulation by determining the spreading diameter of preparation between the two glass plates after 1 min. A 400 mg face pack gel was weighed and placed on a 1 cm pre-marked circle on a glass plate and the second plate was placed on over it. When weight was increased on the upper plate and increased diameter of the gel was noted and calculated using equation.

$$S = \frac{m \bullet l}{t}$$

Where,

S=Spread ability of face pack gel, m = Weight place don upper glass plate l = Length upper of the glass plate, t = Time taken.

Viscosity measurements and rheological behavior

The viscosity of different face pack gels having various polymer quantities is shown in Table 2. As the viscosity of carbopol (3%) and PVP (1%) was optimum among other concentrations (2% and 3%) and (1% and 2%), respectively, so 3% of carbopol 940 and 1% PVP was found as a suitable one. The viscosity of different herbal face pack gels is shown in Figure 2. The herbal face pack gel preparation was analyzed for its rheological character by using (Brookfield Ametek, DV-E, USA) with a 40 mm cone with a 2.5° cone angle configuration of cone and plate. For the rheology status, the shear rate range was 50.67–487.08 s^{-1} at room temperature. The power law formula was used for the calculation of the flow index.

Drying time after application

The drying time of herbal face pack gel should be between 10 and 30 min. Drying gel for oily skin is often applied for a shorter period, whereas moisturizing and anti-aging pack gel are used for longer periods, sometimes overnight. Wash using cold/warm water, not hot.

Skin sensitivity test

A skin irritancy test was conducted on a 200–350 g weighted male healthy Wistar rat. Hairs of left dorsal skin and right dorsal skin of the rat were shaved and removed.

Commercially available face pack gel was applied as a control pack. The prepared face pack gel of 3 × 2 cm was applied as a test sample. The skin sensitivity test was performed on the unbraided skin of rats; the standard (control pack gel) pack gel was applied on the left dorsal surface of the rat, whereas herbs containing face pack gel were applied on a similar side of the right dorsal surface of the rat. The face pack gel was washed after 10, 25, 40, and 60 min by a using swab of fresh water, and the left dorsal surface and right dorsal surface of the rat's skin was examined for any edema and erythema.[15-17]

Thermodynamic stability of the prepared herbal gel

The herbal face pack gel must be thermodynamically stable without creaming, phase separation, and cracking. The thermodynamic stability of formulated face pack gel was assessed based on centrifugation and freeze-thaw cycles.[18,19] The strength of the interfacial film was reflected through centrifugation. The face pack gel preparations were centrifuged at 3500 rpm for 30 min. The freeze-thaw cycle was performed as face pack gel was filled in test tubes and then the tube was sealed hermetically and vertically kept for 16 h at −21°C in the freezer and that after the tubes were kept at room temperature (25°C) for 8 h. The freeze-thaw cycle was performed 3 times for getting reproducible results and the prepared herbal face pack was assessed for any changes.

Statistical analysis

One-way analysis of variance (ANOVA) was used for the statistical analysis of data collected from evaluated formulations.[20,21]

RESULTS AND DISCUSSION

Preformulation studies

Infrared spectral determination of PVA, PVP, and Carbopol

As shown in Figure 3, the identification score of PVA was 0.967. When PVA IR spectra matched to the existence of several groups was established. Figure 3 displays the different peaks found in the IR spectra, including OH vibrational stretching at around 3248.50 cm^{-1} and symmetric stretching of CH_2 at 2936.00 cm^{-1}.

The Figure 4 shows the identification score of PVP was 0.971. When PVP IR spectra matched to the existence of several groups was established. Figure 4 displays the different peaks found in the IR spectra, including NH vibrational stretching at around 3421 cm^{-1} and CH stretching vibration at 2922.00 cm^{-1}.

The Figure 5 depicts the identification score of Carbopol 940 was 0.967. When IR Carbopol 940 spectra matched to the existence of several groups was established. The different peaks found in the IR spectra, including OH vibrational stretching at around 3414.68 and symmetric stretching of CH_3 and C_3H_8OH at 2896.28 cm^{-1}.

Table 2: Composition of herbal face pack gel

Components (%w/w)	Formulation code			
	F1	F2	F3	F4
Emblica officinalis concentration	20	20	20	20
Lens culinaris concentration	15	15	15	15
Rose petals powder	15	15	15	15
PVA	24	24	24	24
Carbopol 940	2	3	2	3
PVP	1	1	2	2
Ethanol 95%	34	34	34	34
Tri-ethanolamine	15 drops	15 drops	15 drops	15 drops
DMDM hydantoin	1.2	1.2	1.2	1.2
H_2O	90	90	90	90

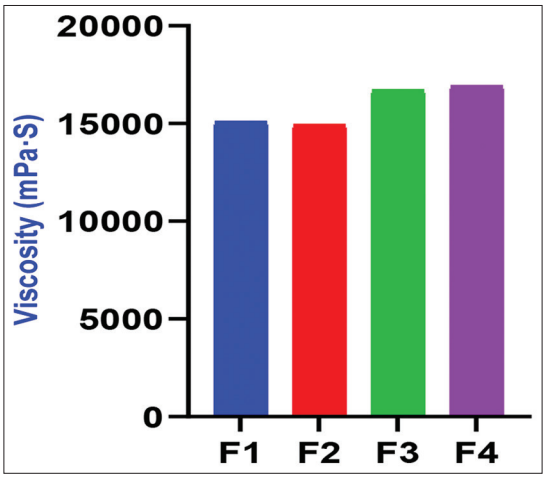

Figure 2: The viscosity of optimized herbal facial gel formulations (F1 to F4)

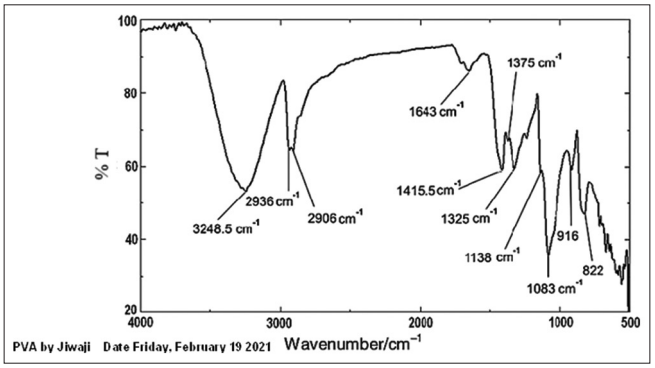

Figure 3: IR graph of polyvinyl alcohol

DSC of PVA, PVP, and carbopol 940

DSC thermograms of PVA revealed an exothermic peak at 197.86°C, which corresponds to the material's melting temperature and is shown in Figure 6 as a solid–liquid phase transition. DSC thermograms of PVP revealed an exothermic peak at 122.13°C, which corresponds to the material's melting temperature and is shown in Figure 7 as a solid–liquid phase transition. DSC thermograms of carbopol 940 revealed an exothermic peak at 262.61°C, which corresponds to the material's melting temperature and is seen in Figure 8.

Preparation and optimization of herbal facial gel

For topical delivery, herbs were loaded into a face pack gel system having carbopol 940 (2–3% w/w) and PVP (1–2% w/w) as a gel matrix. The optimized viscous system is suitable for topical delivery based on biophysical and sensorial evaluations. In the case of carbopol 940 (2% w/w) was added directly to prepare face pack gel, and it showed that it took more time for swelling than water. Even some tiny agglomerates of carbopol 940 (2% w/w) were observed. In carbopol 940 (3% w/w) cases, it was swollen in the water phase, and its pH and viscosity were optimum with good homogenization properties.

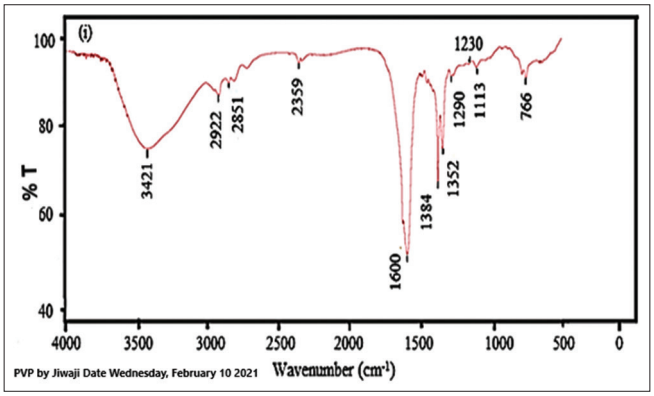

Figure 4: IR graph of polyvinylpyrrolidone

The pH and viscosity of optimized herbal facial gel (F1 to F4)

The pH of prepared herbal facial gel was observed 5.4 ± 0.1, 5.6 ± 0.07, 5.5 ± 0.03, and 5.7 ± 0.1 for all F1, F2, F3, and F4, respectively, which were closer to skin pH and shown in Figure 9. The viscosity of F1, F2, F3, and F4 was observed 15101.3 ± 0.55, 14944.9 ± 0.91, 16721.3 ± 0.90, and 16931.5 ± 0.96, respectively [Figure 2]. It was observed that F2 containing 3% carbopol 940 showed optimum viscosity in all three formulations.

One way ANOVA for pH

One way ANOVA method was used for statistical analysis of different formulations [Table 3].

Spread ability of herbal facial gel

Spread ability of face pack gel formulations was found to be 4.99 ± 0.07, 4.20 ± 0.04, 3.33 ± 0.03, and 3.25 ± 0.04 g.cm/s for F1, F2, F3, and F4, respectively [Figure 10].

Drying time after application

In a dish, 20 g of the prepared and optimized herbal facial gel composition was applied to the facial skin. Acne and blemishes

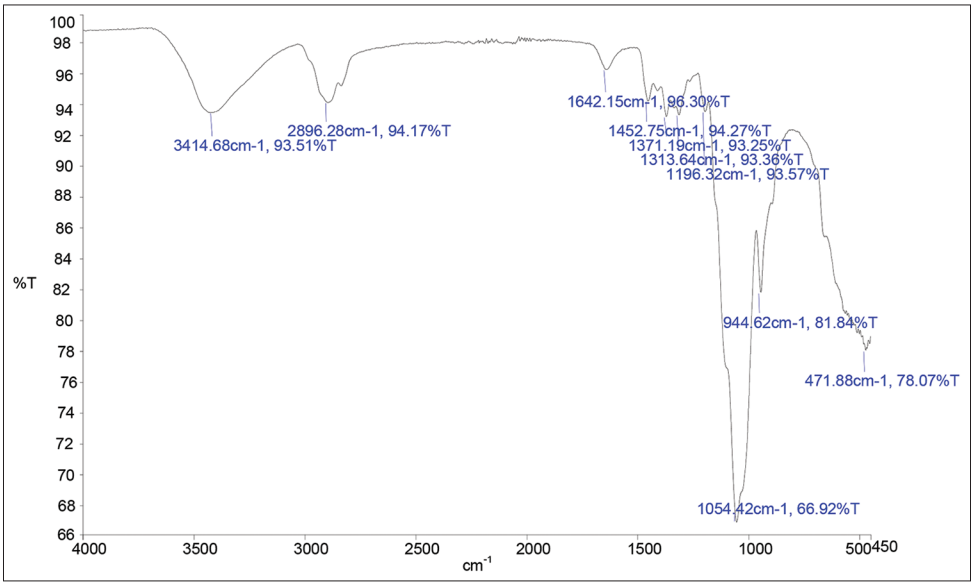

Figure 5: IR graph of Carbopol 940

should also be covered. Allow for thorough drying for 20–25 min before washing with cold water. The drying time of face gel depended on the environmental conditions, and drying time was observed between 18 and 30 min [Figure 11].

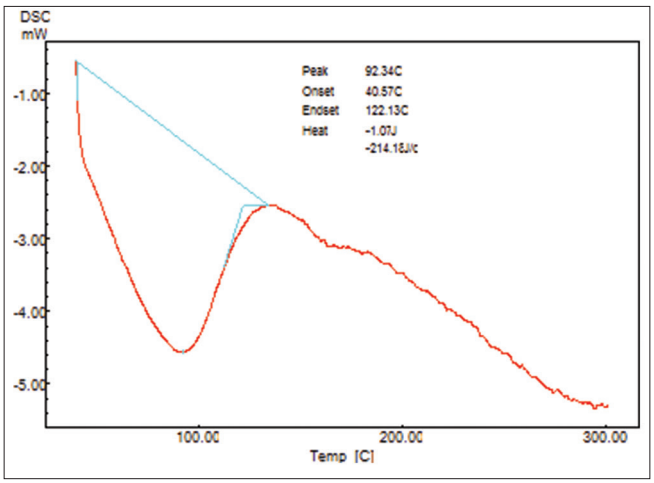

Figure 6: Differential scanning calorimetry graph of polyvinyl alcohol

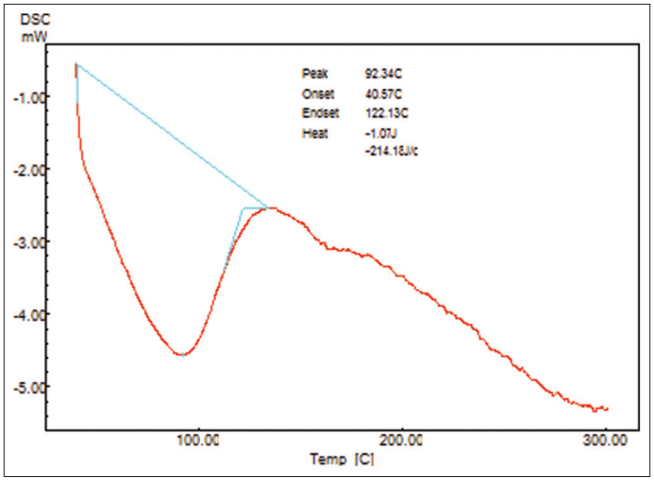

Figure 7: Differential scanning calorimetry graph of polyvinylpyrrolidone

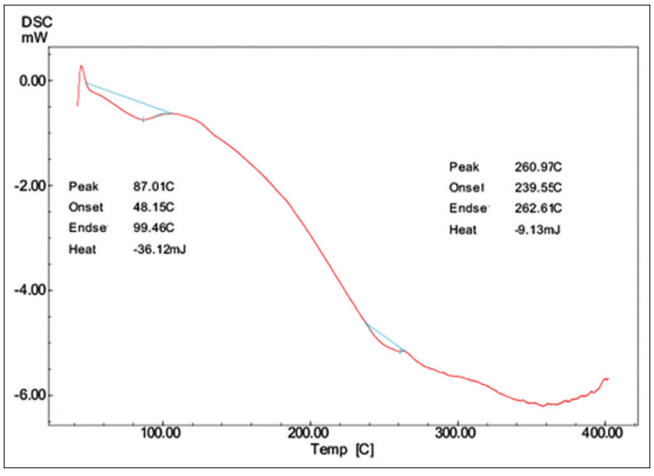

Figure 8: Differential scanning calorimetry graph of carbopol 940

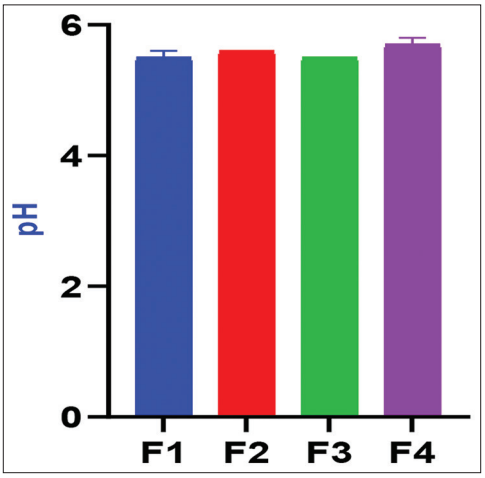

Figure 9: The pH of optimized herbal face gel formulations (F1 to F4)

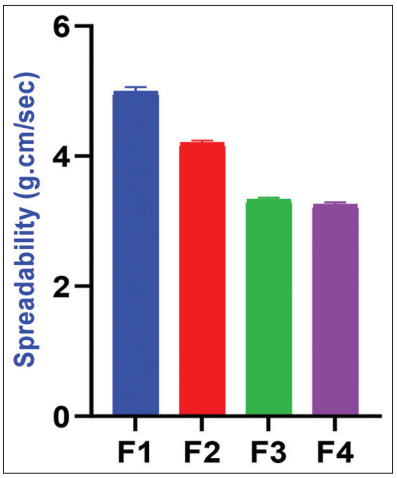

Figure 10: Graph for spread ability of formulated herbal facial gel (F1 to F4)

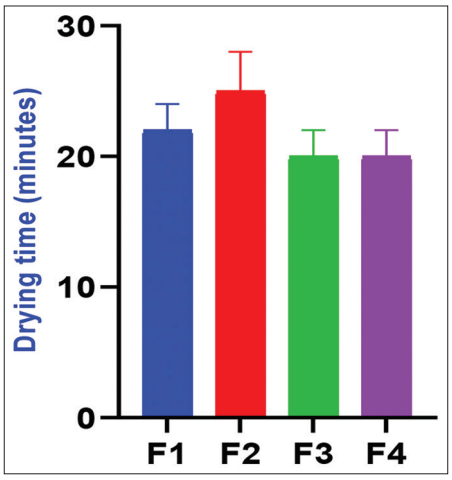

Figure 11: Drying time of formulated facial gel formulations (F1 to F4)

Scoring for skin sensitivity test: Skin sensitivity test

A skin irritancy test was conducted on a 200–350 g weighted male healthy Wistar rat. Hairs of left dorsal skin and right dorsal skin of the rat were shaved and removed. Commercially available face pack gel was applied as a control pack gel. The prepared face pack of 3 × 2 cm was applied as a test sample. The skin sensitivity test was performed on unbraid skin of rats; the standard (control pack gel) pack gel was applied on the left dorsal surface of the rat, whereas herbs containing face pack gel was applied on a similar side of the right dorsal surface of rat. The face pack gel was washed after 10, 25, 40, and 60 min by the using swab of fresh water and left dorsal surface and right dorsal surface of rat's skin was examined for any edema and erythema.

Edema and erythema were scored on a 0–4 scale, where 0 denotes no effect and 4 showing severe symptom. For every testing site, skin response cores at 10, 25, 40, and 60 min after the washing of applied face pack gel were summed and divided by three to observed mean sensitivity score/time point in Table 3. The results were compared to those sites where a control pack gel was applied. The average scores were summed and mean to obtain the initial irritation index.

Thermodynamic stability of face pack gel

The data of stability studies are expressed in Table 4. Stability study of optimized herbal facial gel formulation (F2) was conducted for 3 months at different temperate conditions (25°C ± 2°C, 40°C ± 0.1°C, and 4°C ± 0.2°C). Optimized herbal facial pack gel preparation was stable and consistent for all evaluated parameters.

Statistical analysis

One-way ANOVA method was used for the statistical analysis of different formulations [Table 5 and 6]. When $P \leq 0.05$ than the values of all formulations were observed significant statistically. Linearity was observed for all responses at ≤0.05 *P*-value. Hence, it was obvious that obtained data were significant.

Table 3: Scoring for skin sensitivity test

Time of film detachment	Edema	Erythema
Acute singled skin irritation test		
10 min after washing face pack gel	0	0
25 min after washing face pack gel	0	0
40 min after washing face pack gel	0	0
60 min after washing face pack gel	0	0
Acute repeated skin irritation test		
10 min after washing face pack gel	0	0
25 min after washing face pack gel	0	0
40 min after washing face pack gel	0	0
60 min after washing face pack gel	0	0

Table 4: Stability study of optimized herbal facial gel preparation (F2)

Time	Temp.	Transparency	pH	Viscosity (mPa·S)	Drying time
1 day	4°C	Y	5.6±0.0	14945.9±0.91	26±3
	25°C	Y	5.5±0.06	14946.0±0.90	22±3
	40°C	Y	5.7±0.08	14945.8±0.92	24±3
1 month	4°C	Y	5.5±0.04	14944.3±0.90	25±3
	25°C	Y	5.6±0.05	14946.5±0.91	25±3
	40°C	Y	5.7±0.08	14943.2±0.94	22±3
2 months	4°C	Y	5.7±0.07	14944.0±0.90	26±3
	25°C	Y	5.6±0.03	14945.5±0.93	23±3
	40°C	Y	5.5±0.05	14945.8±0.92	22±3
3 months	4°C	Y	5.6±0.05	14945.9±0.91	25±3
	25°C	Y	5.6±0.07	14946.7±0.92	22±3
	40°C	Y	5.6±0.06	14945.5±0.90	24±3

*Values are shown as mean±SD (*n*=3), Y=Yes, N=No

Table 5: ANOVA table for pH of face pack gel

ANOVA table	SS	DF	MS	F (DFn, DFd)	*P*-value
Treatment (between columns)	0.08250	3	0.02750	F (3, 8)=5.500	0.0240
Residual (within columns)	0.04000	8	0.005000		
Total	0.1225	11			

ANOVA: Analysis of variance, SS: Sum of square, DF: Degree of freedom

Table 6: ANOVA table for viscosity of herbal facial gel

ANOVA table	SS	DF	MS	F (DFn, DFd)	*P*-value
Treatment (between columns)	9858640	3	3286213	F (3, 8)=4592570	0.0001
Residual (within columns)	5.724	8	0.7156		
Total	9858646	11			

ANOVA: Analysis of variance, SS: Sum of square, DF: Degree of freedom

CONCLUSION

Nowadays, people want a treatment option for many skin conditions that does not have any side effects. Cosmetics without adverse effects were made feasible thanks to herbal ingredients. According to reports, using herbal face packs is a reliable and efficient way to enhance the appearance of skin. As a consequence, the current study is a great attempt to create a herbal face pack utilizing readily available ingredients such as rose petal (*R. canina*) powder, masoordaal (*L. culinaris*), and amla (*E. officinalis*). It was claimed that the developed formulation had the qualities of a typical cosmeceutical skincare composition and was physicochemically stable.

ACKNOWLEDGMENTS

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ETHICS APPROVAL

Not applicable.

CONSENT FOR PUBLICATION

Not applicable.

AVAILABILITY OF DATA AND MATERIALS

The datasets of research were collected from experiments and analysis of variables during current study. These datasets are available from the corresponding author on reasonable request.

AUTHORS' CONTRIBUTIONS

PS designed and optimizes the study and developed the methodology. PS, SJ, and KD performed the experiments, collection of data and interpretation data. PS and SJ wrote the manuscript. VJ and PS contributed to manuscript revision and provided supervision. All authors read and approved the final manuscript.

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