J. Pharm. Res. 2026;25(3):191-196

Print ISSN: 0973-7200
Online ISSN: 2454-8405

Journal of Pharmaceutical Research

ORIGINAL ARTICLE

Development and Evaluation of Herbal Sunscreen Using *Rhododendron barbatum* Flower Extract

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ARTICLE INFO

Article history:
Received 07-06-2026
Accepted 06-08-2026
Published 22-08-2026

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https://doi.org/10.18579/jopcr/v25.i3.139

ABSTRACT

The damaging effects of ultraviolet (UV) radiation on the skin include skin burns, photoaging, hyperpigmentation, and skin cancer. Sunscreens are extensively utilized for UV protection. Herbal sunscreens have recently been considered because of their safety, antioxidant potential, and fewer side effects. The flowers of *Rhododendron barbatum* contain flavonoids, phenolics, tannins, and saponins that impart photoprotective properties to the flowers. Hydroalcoholic extraction was carried out using a maceration process, where ethanol and water were used in 70:30 ratio. The phytochemical analysis was undertaken on *R. barbatum* flowers. Three different formulations were used (F1, F2, and F3) containing 1ml, 2ml and 3ml hydroalcoholic extract. These were screened against various parameters, including colour, pH, spreadability, washability, stability, and patch tests, and SPF determination using the Mansur equation. The phytochemical analysis indicated the presence of flavonoids, phenols, tannins, and saponins. All experiments were done in triplicates (n = 3), and all results were calculated as mean ± SD. All formulations displayed desirable physicochemical characteristics. Among all formulations, F3 displayed the best characteristics in terms of homogeneity, spreadability, and stability. The pH of F3 was found to be 5.60 ± 0.17 and its spreadability was 5.50 ± 0.00 cm (mean ± SD, n = 3), which is favorable for topical application. The SPF value of formulation F3 was 8.36. The SPF activity is due to the presence of flavonoids and phenols that exhibit good antioxidant properties and UV absorptive properties. This helps prevent oxidative stress and UV-induced damage. The enhanced activity exhibited by formulation F3 can be attributed to the higher concentration of extracts present in this formulation. The herbal sunscreen cream was chemically and physically stable, safe, and showed moderate photoprotective efficacy. Therefore, it can be considered an alternative to artificial sunscreen creams. The developed formulation may serve as a promising natural sunscreen. However, further in vivo and clinical evaluation are required.

Keywords: Herbal sunscreen, *Rhododendron barbatum*, Ultraviolet radiation, SPF, Flavonoids, Photoprotection

INTRODUCTION

The skin is the biggest organ in the body. The skin is comprised of two major layers called the epidermis and the dermis. The epidermis, which is derived from the ectoderm, is the outer layer of the skin and acts as the body's interface with the external environment. Biological and physical features of the epidermis significantly contribute towards the skin's resistance to various environmental stresses, such as infectious organisms, chemicals, and ultraviolet rays. Keratinocytes are the most prevalent cells found in the epidermis, while the dermis comprises numerous immune cells and fibroblasts that are actively involved in several physiological reactions within the skin. The dermis forms the key protective barrier in the epidermis. Melanin content in the epidermis accounts for the most important factor for determining skin complexion and ultraviolet rays' susceptibility. Melanin is a large biopolymer molecule consisting of smaller units of several pigment types, synthesized from oxidation and cyclization of tyrosine[1].

Sunscreens were initially formulated with the primary purpose of preventing erythema. The end result of the primary index of the efficacy of a sunscreen is erythema, although SPF primarily indicates protection against UVB radiation. However, several studies on animals as well as humans have proven that there are several harmful effects which may follow sub erythema exposure to sunlight with UVR. Therefore, it became apparent that any sunscreen to effectively protect against harm other than sunburn must offer protection against the entire spectrum of the solar UVR spectrum[2].

It is widely accepted that ROS are the major cause of damage in the form of skin cancer, actinic keratosis, and photoaging caused by continuous exposure to sunlight. Therefore, the use of antioxidants, together with UV filters, in sunscreens can help increase the effectiveness of these products in preventing skin cancer and photoaging. The complexity of plant extracts and oils leads to different biological activities, including antioxidant activity, sun-blocking activity, anti-inflammatory, and immunomodulating activities. Moreover, the effectiveness of plant extracts in improving skin condition and curing different skin disorders has been thoroughly investigated. Plants possessing high antioxidant activity are considered to be highly attractive ingredients for inclusion into sunscreens for protection from sunlight-induced skin damages[3].

Rhododendron barbatum of Ericaceae family is an evergreen tree, widely distributed in the temperate regions of tropics[4].

can thrive under extreme climatic conditions at mountain peaks, arctic conditions, volcanos, swamps, rocks and are extensively distributed in temperate and cool subtropical regions. They are also referred to as Buransh in India. Local name – Lal Chimal[5].

The phytochemicals found in *R. barbatum* flowers include flavonoid, glycoside, phenolic, quercetin, oleanolic acid, β-sitosterol, which possess medicinal, antioxidant, anti-inflammatory, and photoprotective properties. This is because *R. barbatum* flowers possess numerous bioactive phytochemicals such as[4].

During the last few years, there has been much emphasis on the use of herbal sunscreens because of their natural source, antioxidative property, and safety aspect compared to chemical sunscreens. There are many plants like Aloe vera, *Camellia sinensis* (green tea), *Azadirachta indica* (neem), *Curcuma longa* (turmeric), *Phyllanthus emblica* (amla), and *Crocus sativus* (saffron), which have been studied for their photo-protective activity and use in herbal sunscreen preparations[6]. But very little scientific data is available about the formulation and physicochemical characterization of herbal sunscreen cream containing *Rhododendron barbatum* flower extract. Hence, this study has been done to formulate and characterize herbal sunscreen cream containing hydro-alcoholic extract of *Rhododendron barbatum* flower extract.

Table 1: Taxonomical classification

Taxonomic Rank	Classification
Kingdom	Plantae
Division	Magnoliophyte
Class	Equisetopside
Order	Ericales
Family	Ericaceae
Genus	Rhododendron
Species	*Rhododendron barbatum*

MATERIAL AND METHOD

Plant material:

Herbal sunscreen creams were prepared by using plant material such as *Rhododendron barbatum* (Family: Ericaceae). Fresh flower of *Rhododendron barbatum* were collected from Munsyari region of Uttarakhand, India. Plant identification was carried out based on morphological characteristics and available botanical literature ((Flora of British India, Hooker, 1875; Flora of Bhutan including a Record of Plants from Sikkim, Grierson and Long, 1983) before further processing.

Chemicals:

Stearic acid, Liquid paraffin, Methyl Paraben, Triethanolamine, Glycerin, Ethanol, Disodium EDTA, all chemicals and reagents used in the present study were of analytical grade and obtained from the laboratory of Amrapali university[7].

Extraction of plant material:

Fig. 1: Hydroalcoholic Extraction process of *Rhododendron barbatum* Flower

Maceration of *Rhododendron barbatum* Flowers: Fresh and healthy plant material of *Rhododendron barbatum* was procured and cleaned using water. It was shade-dried and converted to a fine powder using sieve number 80. A definite quantity of flower powder was taken (10g) after weighing and added with 70% ethanol followed by adding distilled water at 30%. This process was performed for 3 days continuously, with daily stirring after every 24 hours. Further, filtration and concentration were performed to get a

semi-solid extract called Hydroalcoholic Extract. The hydroalcoholic extraction of 10 g of dried *Rhododendron barbatum* flower powder yielded 1.5 g of crude extract, corresponding to a percentage yield of 15%. The extract was preserved in an airtight box Fig. 1[8].

Table 2: Parameters used for extraction of *Rhododendron barbatum* Flowers

Parameters	Details
Plant used	*Rhododendron barbatum* Flowers
Extraction Method	Maceration
Solvent used	Hydroalcoholic solvent
Ethanol: water ratio	70:30(v\v)
Powder quantity	10g
Extraction Time	3days
Stirring interval	Every 24hr
Filtration method	Whatman filter paper
Final extract	Semisolid hydroalcoholic extract

Preliminary phytochemical test:

Table 3: Preliminary phytochemical screening of *Rhododendron barbatum* extract[9]

S. No.	Phytochemical test	Test	Procedure	Observation
1	Flavonoids	Shinoda test	2ml, extract + small piece of magnesium ribbon + 1ml concentrated HCL	Red color observed
2	Phenols	Ferric Chloride test	2ml, extract + 1ml ferric chloride solution	Black color observed
3	Tannins	Ferric Chloride test	2ml, extract + 1ml ferric chloride solution	Black color observed
4	Saponin	Foam test	2ml, extract + 5ml distilled water, shake well	persistent foam formation

Formulation of Herbal Sunscreen:

Preparation of Oil Phase: Stearic acid 4 g and liquid paraffin 2 ml were accurately weighed and transferred into a clean dry beaker. The ingredients were heated on a water bath at approximately 70°C until completely melted and add glycerin 2 ml into the molten mixture with continuous stirring to obtain a uniform oil phase.

Preparation of Aqueous Phase: Distilled water q.s. to 20 gm was taken in a separate beaker and heated to approximately 70°C.Methyl paraben 0.04 g and disodium EDTA (0.02 g) were dissolved completely in the heated aqueous phase and add triethanolamine 0.4 g slowly with continuous stirring to prepare a clear and uniform aqueous phase[10].

Emulsification and Incorporation of Hydroalcoholic Extract: The hot aqueous phase was added gradually into the oil phase with continuous stirring to form a smooth emulsion. The prepared hydroalcoholic extract of *Rhododendron barbatum* (3 ml) was incorporated slowly into the formulation with constant stirring to ensure uniform distribution. Continuous stirring was maintained until a homogeneous sunscreen cream was obtained, final formulation was allowed to cool to room temperature and transferred into a clean airtight container for further evaluation and storage Fig. 2[11, 12].

Fig. 2: Prepared formulations of F1, F2 and F3 herbal sunscreen

Table 4: Formulation and ingredient of Sunscreen

S. No.	Ingredient	F1	F2	F3
1	*Rhododendron barbatum* (Buransh) extract	1ml	2ml	3ml
2	Stearic acid	3g	3.5g	4g
3	Liquid paraffin	1.5ml	2ml	2ml
4	Methyl paraben	0.04g	0.04g	0.04g
5	Glycerin	2ml	2ml	2ml
6	Disodium EDTA	0.02g	0.02g	0.02g
7	Triethanolamine	0.4g	0.4g	0.4g
8	Purified water	11ml	9.5ml	8.54ml
	Total	**20g**	**20gm**	**20gm**

Evaluation of Herbal Sunscreen:

Physical Appearance: To observe the color, texture, consistency, and presence of any grittiness or lumps in the sunscreen cream. Visually inspect the prepared cream under proper lighting conditions. Observe White colored, smooth, uniform, soft, and non-gritty cream with pleasant appearance[13].

Homogeneity: To check the uniform distribution of ingredients in the formulation. A small quantity of cream was pressed between thumb and index finger to observe uniformity and presence of aggregates. Observed The formulation showed good homogeneity with no phase separation or coarse particles[13].

Table 5: Comparative study of prepared formulations

S. No	Test Name	Purpose	Method	F1	F2	F3
1	Physical appearance	To check the color, texture, and uniformity of cream	Visual observation under proper lightening	White, smooth cream	White, smooth cream	White, smooth cream
2	Homogeneity	To ensure uniform mixing of ingredients	Press small quantity between finger	Moderate homogeneity, tiny lumps	Very good homogeneity	Excellent homogeneity
3	Ph Determination	To assess skin Compatibility	1g cream dispersed in distilled water and measured using pH meter	5.56	5.51	5.58
4	spreadability	To assess skin application	Cream placed between glass slides and spread diameter measured	Moderate spreadability	Good spreadability	Excellent spreadability
5	Washability	To check removability from skin	Applied on skin and washed with water	Easily washable	Easily washable	Easily washable
6	Stability study	To evaluate formulation stability	Stored at room temperature and observed for change	Moderate Stable	Highly Stable, no phase separation	Highly Stable, no phase separation

pH Determination: To ensure the cream is skin-friendly and suitable for topical application. Weigh 1 g of cream and disperse it in 50 mL distilled water. Measure the pH using a calibrated digital pH meter, observed pH found to be 5.5–6 which is suitable for skin application[14].

The pH of formulation F3 was determined in triplicate and was found to be 5.60 ± 0.17 (n = 3), indicating that the formulation is within the acceptable pH range for topical skin application.

Spreadability: To determine how easily the cream spreads on the skin surface. Place 1 g of cream between two glass slides. Apply 500 g weight for 5 minutes and measure the spread diameter, Observed Cream showed good spreadability 5.5 cm spread diameter, indicating ease of application[15].

The spreadability of formulation F3 was determined in triplicate and was found to be 5.50 ± 0.00 cm (n = 3). This result indicates that the formulation possessed good spreadability, ensuring easy and uniform application over the skin surface.

Washability: To determine the ease of removal of cream from the skin using water. Apply a small amount of cream on skin and wash with tap water. Observe the ease of removal. observed, the cream was easily washable with water and did not leave excessive oily residue[16].

Stability Study: To evaluate the stability of the formulation under different storage conditions. Store the cream at room temperature and accelerated conditions (40°C ± 2°C) for 30 days. Observe changes in color, odor, consistency, and phase separation. observed No significant changes in appearance, color, pH, or consistency were observed during the study period, indicating good stability[13].

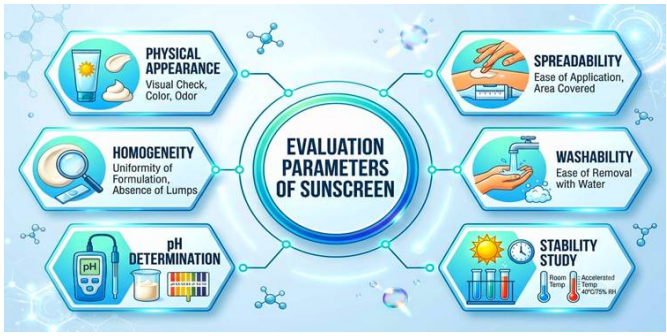

Fig. 3: Evaluation parameters for Sunscreen

SPF (Sun Protection Factor) Determination:

To evaluate the sun protection efficiency of the sunscreen cream. SPF value was determined using UV spectrophotometric method by measuring absorbance between 290–320 nm The formulation exhibited satisfactory SPF value, indicating effective sunscreen activity[17].

Absorbance of the sunscreen formulation was determined with the help of Ajanta UV-Visible Spectrophotometer (Model: UV-1900) within a wavelength range of 290–320 nm. All the measurements were done using quartz cuvettes of 1 cm path length, all measurements were performed in triplicate (n = 3), and the average values were used for analysis[18].

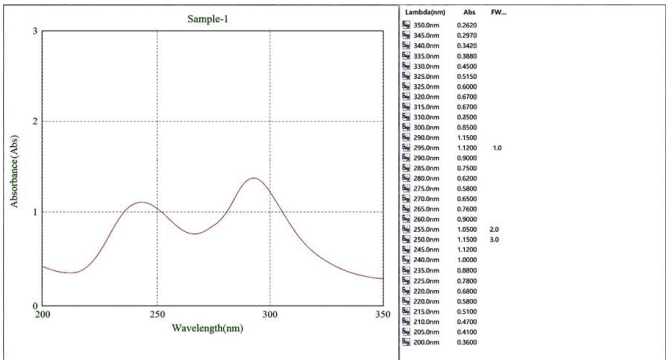

Fig. 4: UV absorption graph of F3 formulation

SPF Calculation by Mansur Method:

Using the absorbance values from your generated UV spectrum:

Wavelength (nm)	Absorbance
290	0.9000
295	1.1200
300	0.8500
305	0.8500
310	0.7500
315	0.6700
320	0.6700

Mansur Equation:

SPF = CF x ∑ EE (λ) x I(λ) x Abs (λ)

Where:

CF = 10

Standard EE × I Values

Wavelength (nm) EE × I

Standard EE x I Values:

Wavelength (nm)	EE × I
290	0.0150
295	0.0817
300	0.2874
305	0.3278
310	0.1864
315	0.0839
320	0.0180

Table 6: Detailed Calculation

Wavelength	Abs	EE x I	EE x I x Abs
290	0.9000	0.0150	0.01350
295	1.1200	0.0817	0.09150
300	0.8500	0.2874	0.24429
305	0.8500	0.3278	0.27863
310	0.7500	0.1864	0.13980
315	0.6700	0.0839	0.05621
320	0.6700	0.0180	0.01260

Sum of all values:

∑ (EE x I x Abs) = 0.8360

Final SPF

SPF = 10 x 0.8360

SPF = 8.36

RESULT AND DISCUSSION

All the herbal sunscreen formulations (F1, F2, and F3) prepared with the use of *Rhododendron barbatum* flower extract have exhibited satisfactory physicochemical characteristics. The phytochemical screening revealed that the formulations contained flavonoids, phenolics, tannins, and saponins which are known antioxidants and provide protection against UV radiation. F1, F2, and F3 formulations are homogeneous in texture, have a good appearance, appropriate pH, spreadability, and stability. F3 formulation has exhibited the best homogeneity, spreadability, stability, and performance. The SPF value of the optimized formulation (F3) has been 8.36 according to the results obtained by UV spectrophotometric method. It should be noted that SPF values of some other formulations developed using different plants have been in the same range (*Calendula officinalis, Camellia sinensis*). The better performance of F3 formulation compared with F1 and F2 formulations can be explained by the fact that it contains a larger amount of *Rhododendron barbatum* flower extract due to the larger number of flavonoids and phenolic compounds. Phytoconstituents have been characterized by UV absorption and antioxidant properties which increase photoprotective activity and stability of the formulation. Thus, the developed formulation can be used as a sunscreen preparation. However, the current study has some limitations since only SPF measurement.

CONCLUSION

In the current study, a sunscreen cream with the hydroalcoholic flower extract of *Rhododendron barbatum* was successfully designed and characterized. Out of all the formulations (F1, F2, and F3), the formulation F3 showed the best physicochemical properties such as good

homogeneity, pH (5.60 ± 0.17), spreadability (5.50 ± 0.00 cm), good washability, good physical stability, and absence of phase separation. Moreover, this formulation showed non-irritant nature in the patch test.

The optimized formulation showed an SPF value of 8.36, indicating its photoprotective capability. Flavonoids and phenolic compounds present in *Rhododendron barbatum* flower extract may provide their UV-protective and antioxidant activity. Hence, it can be said that the designed sunscreen cream showed good physical stability, safety and had photoprotective properties. Therefore, it could be concluded that *Rhododendron barbatum* flower extract can be successfully used in developing sunscreen formulations.

Even though the current study showed encouraging results, further in vivo photoprotective studies, stability studies, and clinical evaluations need to be conducted to prove the safety and effectiveness of the formulation. Further optimization of the formulation to improve the SPF value above 8.36 and feasibility of the formulation for large-scale manufacturing are some future prospects of the current study.

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