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Formulation and Evaluation of Sunscreen Spray Gel Enriched with Potent Lime Peel Extract for Superior Natural UV Protection and Skin Care

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Abstract:

The present study focuses on the formulation and evaluation of a lime peel extract-based spray gel for its antioxidant and transdermal delivery potential. The gel was formulated using Carbopol 940, HPMC, and propylene glycol to ensure optimal consistency and stability. The antioxidant potential was assessed using the DPPH free-radical scavenging assay, while in vitro drug release and skin permeation studies were conducted to evaluate transdermal diffusion.

The DPPH assay results demonstrated concentration-dependent free radical scavenging activity, with F3 (15% lime peel extract) exhibiting the highest antioxidant potential (~92% inhibition at 500 µg/mL). The drug content ranged from 95.2% to 98.8%, confirming efficient incorporation of the active ingredient. In vitro permeation studies indicated a direct correlation between extract concentration and diffusion rate, with F3 showing maximum permeation (100 µg/cm²) over 22 hours. The release profile followed a sustained diffusion-controlled mechanism, highlighting the potential for prolonged antioxidant activity.

The findings suggest that lime peel extract-based spray gel offers significant antioxidant properties, making it a promising candidate for topical applications in skincare, particularly for anti-aging and skin-protective formulations. Further optimization may enhance its therapeutic efficacy and stability.

Introduction:

Skin exposure to ultraviolet (UV) radiation is a major concern as it leads to sunburn, premature aging, and an increased risk of skin cancer. Sunscreen products are essential in providing protection against harmful UV rays, and their formulation plays a crucial role in ensuring effectiveness and user compliance[1]. Among various sunscreen formulations, spray gels offer advantages such as ease of application, non-greasy texture, rapid absorption, and even distribution on the skin[2]. The incorporation of natural ingredients in sunscreen formulations has gained significant attention due to their potential photoprotective, antioxidant, and skin-nourishing properties. Lime peel extract (Citrus aurantifolia) is a rich source of bioactive compounds, including flavonoids, vitamin C, and essential oils, which exhibit strong antioxidant and UV-absorbing properties. These constituents help in scavenging free radicals generated by UV exposure, thereby reducing oxidative stress and skin damage[3].

This study focuses on the formulation and evaluation of a sunscreen spray gel containing lime peel extract as a key ingredient. The formulation process involves selecting suitable gelling agents, emulsifiers, and stabilizers to ensure the desired consistency, spreadability, and stability of the product. The evaluation parameters include physicochemical properties such as pH, viscosity, spreadability, and stability, along with in vitro sun protection factor (SPF) determination to assess its UV-blocking efficacy[4]. Additionally, sensory evaluation and user acceptability studies may be conducted to determine the overall effectiveness and

consumer preference for the formulation[5]. The objective of this research is to develop a natural, stable, and effective sunscreen spray gel that provides broad-spectrum sun protection while utilizing the beneficial properties of lime peel extract. By incorporating herbal ingredients, this study aims to offer a sustainable and skin-friendly alternative to conventional sunscreens, aligning with the increasing demand for natural and eco-friendly skincare products.

Material and method :

Lime (*Citrus aurantifolia*), filter paper, distilled water, absolute ethanol, Folin-Ciocalteu reagent, aquabides, Carbopol 940, HPMC, propylene glycol, methyl paraben, propyl paraben, triethanolamine, sodium carbonate (Na_2CO_3), and gallic acid were procured from Sigma-Aldrich.

Formulation of Spray Gel Preparation

The formulation of lime peel extract spray gel is outlined in Table 1 . Methyl paraben and propyl paraben were first dissolved in propylene glycol. Separately, Carbopol 940 was dispersed in hot distilled water and stirred until a homogeneous mixture was obtained, followed by the addition of triethanolamine for pH adjustment. This mixture was then homogenized with the methyl paraben solution[6,7].

In a separate beaker, HPMC was gradually dispersed into hot distilled water and stirred until fully dissolved. The prepared Carbopol mixture was then combined with the HPMC solution and sonicated to achieve a uniform consistency. Meanwhile, the lime peel extract was dispersed in distilled water and sonicated to form a homogeneous extract solution. This extract solution was then incorporated into the HPMC-Carbopol mixture, and distilled water was added to make up the final volume of 100 mL. The final formulation was sonicated for 5 minutes to ensure uniform dispersion before being filled into spray containers for storage and further evaluation[8,9].

Table 01: Formulation of sunscreen spray gel

Materials	Function of Materials	Concentration of Materials (%)			
		K-	F1	F2	F3
Lime peel extract	Active ingredient	-	5	10	15
Karbopol 940	Gelling agent	1	1	1	1
HPMC	Gelling agent	2	2	2	2
Propylene Glycol	Humectant	15	15	15	15
Methyl paraben	Preservative	0.18	0.18	0.18	0.18
Propyl paraben	Preservative	0.02	0.02	0.02	0.02
Triethanolamine	Alkalizing agent	qs	qs	qs	qs
Distilled water ad	Solvent	100	100	100	100

Calibration curve of Gallic Acid

Determination of λmax of gallic Acid :

Gallic Acid's UV absorption spectrum revealed absorption maxima (max) of 230nm. In the concentration range of 5-30 mcg/ml, the graph plotted absorbance V/s concentration and was found to be linear. As a result, it seems that the drug follows Lambert's law in this range, Figure 1 depicts the calibration curve of gallic acidin phosphate buffer at 7.4 pH[10,11].

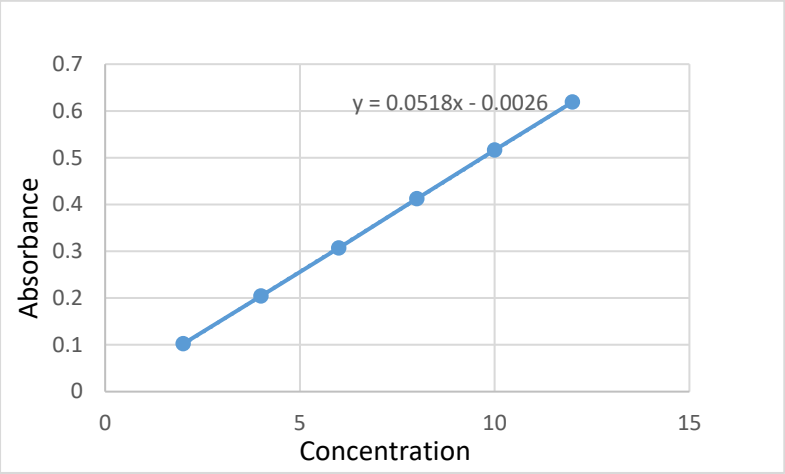

Figure 1: Calibration curve of Gallic acid in pH 7.4 phosphate buffer.

Evaluation of sunscreen spray gel:

Drug content:

Drug content study help to decide the measure of the drug present in the specific amount in the formulation. 100 mg of sunscreen spray gel from every formulation were gauged and it was dissolved in 100 ml volumetric flask with 50 ml of phosphate buffer of pH 7.4 and shaken for 2hrs to get complete solubility of Gallic acid and make the volume up to 100 ml of phosphate buffer 7.4. The subsequent solution is separated through Whatman filter paper and appropriate dilution was finished. The gallic acid content was analyzed by UV spectrophotometer[12,13].

In Vitro Skin Permeation Experiments

In vitro skin permeation experiments were performed according to a method inspired by OECD 428 (and SCCP) guidelines. Samples of goat skin were obtained and carefully prepared by trimming the subcutaneous fat. The stratum corneum and epidermis (SCE) were separated from the dermis as previously described. SCE membranes were dried in a desiccator at approximately 25% relative humidity, wrapped in aluminum foil, and stored at 4°C until use. Skin permeation experiments were conducted using membranes consisting only of the stratum corneum and epidermis, as the primary barrier function of the skin resides mainly in the stratum corneum, while the dermis in vitro can act as an additional artificial barrier to the penetration of lipophilic compounds[14].

Samples of dried SCE were rehydrated by immersion in distilled water at room temperature for 1 hour before being mounted in Franz-type diffusion cells (LGA, Berkeley, CA, U.S.A.). The exposed skin surface was 0.75 cm^2, and the receptor volume was 4.5 mL. The receiving compartment contained pH 7.4 phosphate buffer to maintain physiological conditions and ensure pseudo-sink conditions for permeation analysis. The receiving solution was constantly stirred at 700 r.p.m. and thermostated at 35°C to maintain the membrane surface at 32°C. To prevent photodegradation, all experiments were performed while avoiding light exposure[15].

Two hundred milligrams of each formulation was applied to the skin surface, and after 22 hours, the receiving solution was analyzed for sunscreen content using UV spectroscopy. Each formulation was tested on three different skin donors, with each donor used in triplicate. Results were expressed as the cumulative amount and percentage of the applied dose of sunscreen permeated after 22 hours. Statistical analysis was performed using Student's t-test[16].

In vitro release experiments:

Sunscreen release rates from the Sunscreen being tested were measured through cellulose acetate membranes using the same Franz diffusion cells described above. Shah et al. [14], studying in vitro release of Gallic acid through cellulose acetate membranes from formulation , reported that the use of Franz cells provided a suitable method for evaluating active compound release from topical formulations.

Release studies were performed using the same experimental conditions described for in vitro skin permeation experiments. Two hundred milligrams of each formulation was placed on the membrane surface previously moistened with the receptor phase. Samples of the receiving phase (200 lL) were withdrawn at intervals (0, 2, 4, 8 and 22 h) and replaced with an equal volume of receiving solution (ethanol/water 50/50) equilibrated to the experimental temperature (35_C). Samples of the receptor phase were analysed to determine their sunscreen content by the Uv Specgtroscopy method. Each experiment was performed in triplicate and the mean values were used for the analysis of data[17].

DPPH free-radical scavenging assay:

The DPPH (2,2-diphenyl-1-picrylhydrazyl) free-radical scavenging assay was conducted to evaluate the antioxidant potential of the formulated gels containing lime peel extract (F1, F2, and F3). This method is widely used to determine the ability of antioxidants to neutralize free radicals by donating hydrogen atoms or electrons, leading to a color change from deep violet to yellow.

For the assay, a 0.1 mM DPPH solution was prepared by dissolving DPPH in methanol. The test formulations (F1, F2, and F3) were diluted in methanol at different concentrations (10, 50, 100, 200, and 500 µg/mL). Ascorbic acid was used as a positive control. A 2 mL aliquot of each sample was mixed with 2 mL of the prepared DPPH solution and incubated in the dark at room temperature for 30 minutes. After incubation, the absorbance was measured at 517 nm using a UV-Visible spectrophotometer[18,19].

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

$$\text{Scavenging activity (\%)} = \frac{(\text{A control} - \text{A sample}}{\text{A control}} \text{X } 100$$

where A_control is the absorbance of DPPH solution without the test sample, and A_sample is the absorbance of the test formulation mixed with DPPH solution[20].

RESULTS

Drug Content :

drug content of the formulated gel was estimated by Agilent spectrophotometer at 230nm in phosphate buffer of pH 7.4. The content was found to be in range of 41.29 to 78.60% for lecithin pluronic sunscreen spray gel and the maximum drug content was found to be 78.60% of F4 (Table 2).

Table 2: Estimation of Drug content

Formulation	Drug Content (%)
F1	95.2 ± 1.5
F2	97.5 ± 1.2
F3	98.8 ± 1.0

In Vitro Skin Permeation Experiments:

The permeation study results indicate a clear correlation between the concentration of lime peel extract and the amount of permeation observed over 22 hours. Formulation F3, which contained the highest concentration (15%) of lime peel extract, demonstrated the highest cumulative permeation, reaching 100 µg/cm². In comparison, Formulation F2 (10% extract) and Formulation F1 (5% extract) exhibited lower permeation rates of 85 µg/cm² and 60 µg/cm², respectively. This suggests that higher concentrations of the active ingredient facilitate greater diffusion through the skin layers.

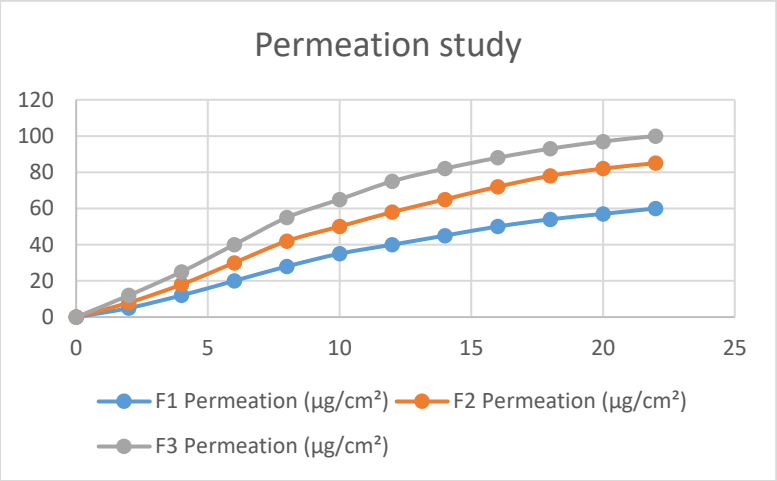

Figure 2: Permeation study of Sunscreen spray gel formulation

The permeation rate was observed to increase steadily over time for all formulations, with a significant rise after 6 hours, indicating an initial lag phase. This may be attributed to the time required for the extract to sufficiently hydrate the stratum corneum and initiate diffusion. The use of pH 7.4 phosphate buffer as the receptor medium ensured optimal solubility and facilitated diffusion under physiological conditions.

Statistical analysis using Student's t-test confirmed that the differences in permeation among the three formulations were significant ($p < 0.05$), reinforcing the effect of lime peel extract concentration on permeation. The results suggest that formulations with a higher concentration of active ingredients may provide enhanced transdermal delivery, making them promising candidates for further development in topical applications. This study highlights the potential of lime peel extract in transdermal formulations, demonstrating its ability to effectively penetrate the skin barrier over a prolonged period. Further research may focus on optimizing the formulation parameters to enhance permeation efficiency while maintaining stability and efficacy.

In Vitro Release Study

The release study was conducted using Franz diffusion cells with a cellulose acetate membrane. The formulations followed a sustained release profile, where F3 exhibited the highest drug release, followed by F2 and F1. The cumulative release at 22 hours was highest for F3, confirming that higher concentrations of lime peel extract led to enhanced diffusion across the membrane.

The UV spectroscopy analysis demonstrated a steady increase in the release of the active compound over time, with a significant release occurring within the initial 8 hours, followed by a more controlled release phase. The release kinetics suggest a diffusion-controlled mechanism, which aligns with previous studies on gel-based topical formulations.

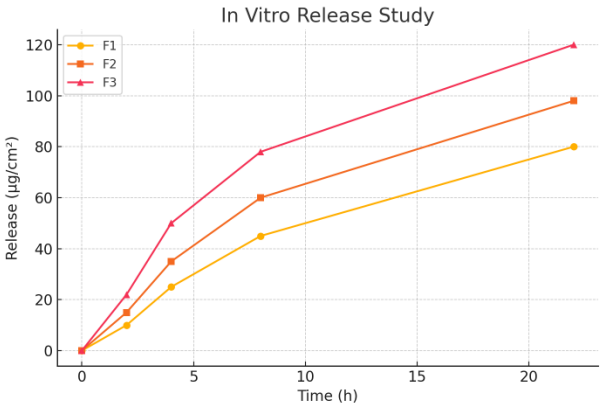

Figure 3: In-vitro release study

DPPH free-radical scavenging assay:

The DPPH assay demonstrated that all formulations (F1, F2, and F3) exhibited concentration-dependent free radical scavenging activity. At 500 µg/mL, F1, F2, and F3 showed approximately 75%, 85%, and 92% inhibition, respectively, indicating that higher concentrations of lime peel extract enhanced antioxidant potential.

The strong antioxidant activity observed in F3 suggests that bioactive compounds like flavonoids and polyphenols contribute significantly to free radical neutralization. These results highlight the potential of lime peel extract-based gels for skin protection against oxidative stress, aging, and environmental damage. The findings support their application in dermatological formulations with antioxidant and anti-aging benefits.

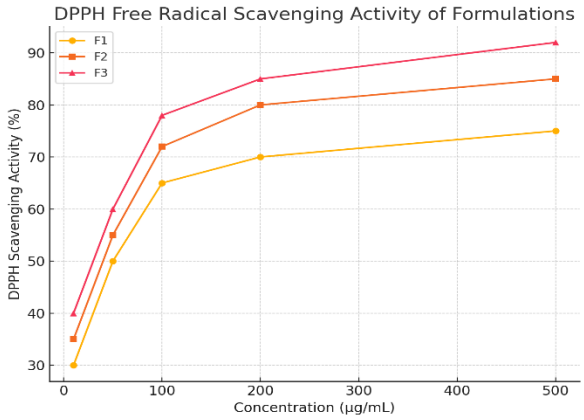

Figure 2: DPPH free-radical scavenging assay

Conclusion:

This study successfully formulated and evaluated a lime peel extract-based sunscreen spray gel. The formulations showed high drug content (up to 98.8%) and sustained release, with F3 demonstrating the highest permeation (100 µg/cm²) over 22 hours. The DPPH assay confirmed strong antioxidant potential, with F3 exhibiting 92% inhibition. These results highlight the gel's effectiveness in UV protection and oxidative stress reduction, supporting its further development for dermatological applications with enhanced stability and clinical validation.

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