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(RESEARCH ARTICLE)

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Formulation and evaluation of sunscreen spray gel with lime peel extract (*Citrus aurantifolia*)

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Abstract

Background: Prolonged exposure to sunlight poses detrimental effects on the skin. Lime peel extract, containing over 60% flavonoids, demonstrates significant potential as a sunscreen agent due to the presence of conjugated aromatic benzene rings capable of absorbing UV-A and UV-B radiation. To mitigate sun-induced skin damage, lime peel extract has been formulated into a spray gel, offering rapid drying properties that enhance user convenience and comfort during application.

Objective: To evaluate the impact of varying concentrations of lime peel extract in sunscreen spray gel formulations on their physical properties and in vitro sun protection factor (SPF) values.

Methods: Lime peel crude extract was obtained using 70% ethanol as the extraction solvent. The extract was incorporated into sunscreen spray gel formulations at concentrations of 5% (F1), 10% (F2), and 15% (F3). These formulations were assessed for their physical properties, including pH, viscosity, spreadability, drying time, and adhesion, alongside their in vitro SPF values.

Results: The concentration of lime peel extract significantly influenced both the physical properties and SPF values of the spray gel formulations. All formulations (F1, F2, and F3) exhibited satisfactory physical stability and met the quality criteria for spray gel preparations. The SPF values for F1, F2, and F3 were determined to be 15, 20, and 30, respectively, indicating a progressive increase in photoprotective efficacy with higher extract concentrations.

Conclusion: The sunscreen spray gel formulations containing lime peel extract at concentrations of 5% (F1), 10% (F2), and 15% (F3) demonstrated physical stability and provided moderate to high SPF protection. Among the formulations, F3 (15%) exhibited the highest SPF value of 30, suggesting its superior efficacy as a sunscreen agent.

Keywords: *Citrus aurantifolia*; Spray Gel; Topical Sunscreen Formulation; Spray gel

1. Introduction

Ultraviolet (UV) radiation from sunlight, specifically UV-A and UV-B rays, exerts deleterious effects on the skin, contributing to premature aging, sunburn, and an increased risk of skin cancer.[1] The human integumentary system possesses innate defense mechanisms, including melanin synthesis, stratum corneum thickening, and perspiration, which provide partial protection against solar radiation.[2] However, these natural defenses are often insufficient, necessitating the use of exogenous photoprotective agents such as sunscreen formulations.[3] Lime peel (Citrus aurantifolia) exhibits potential as a natural sunscreen agent due to its high flavonoid content, exceeding 60%.[4] Flavonoids, a prominent class of secondary metabolites within the phenolic compound group, possess strong UV-

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absorbing properties, effectively mitigating UV-A and UV-B radiation.[5] The UV absorption potential of lime peel flavonoids has been corroborated by preliminary studies involving total phenolic content assays.[5] Research has demonstrated the efficacy of lime peel extract as a sunscreen across various dosage forms. Extract concentrations have yielded Sun Protection Factor (SPF) values ranging from 4.4 to 40.15,[6] while gel, cream, and lotion formulations incorporating the extract have achieved SPF values between 11.36–20.68, 12.01–18.57, and 11.27–19.44, respectively.[7],[8] These findings substantiate the potential of lime peel extract as an effective photoprotective agent.To further advance the utilization of lime peel extract in sunscreen formulations, this study focuses on the development of a spray gel, an innovative dosage form offering rapid drying, enhanced user convenience, and improved application consistency. The objective is to evaluate the extract's impact on the physical characteristics, stability, and SPF value of the spray gel formulation, thereby establishing its suitability as an efficient, user-friendly photoprotective product.

2. Materials and methods

2.1. Materials

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 obtained from Sigma-Aldrich.

2.2. Equipment

An oven (Memmert), grinder, dehydrator, hotplate (Philips), rotary evaporator (Heidolph), water bath (Memmert), furnace, calipers, analytical balance (Mettler Toledo), UV-Vis spectrophotometer (Shimadzu UV-1280), pH meter (Mettler Toledo), ultrasonic cleaner (Branson), viscometer (Brookfield LV), and magnetic stirrer (C-MAG HS 7, IKA) were utilized for the study.

2.3. Method

2.3.1. Preparation of simplisia

The lime samples were initially authenticated at the Botanical Department of Science College, Durg, to confirm their identity as Citrus aurantifolia. Mature lime fruits, characterized by their dark green color, were selected while still wet and thoroughly washed under running water to eliminate any dirt or impurities. The peels were carefully separated from the fruits using a lime peeler. The collected peels were dried in a dehydrator at 50 °C for one week to ensure complete drying. Once dried, the herbal material was ground into a fine powder using a grinder.

2.3.2. Extraction

The extraction process employed maceration with 70% ethanol. One kilogram of powdered herbal material was soaked in 10 liters of 70% ethanol in a 1:10 (w/w) ratio inside a covered container for 24 hours, with stirring for 10 minutes each day. This maceration process was repeated three times using 5 liters of 70% ethanol in a 1:5 (w/w) ratio. The resulting macerate was filtered through filter paper and evaporated using a rotary evaporator at 75 mbar pressure and a temperature of 50 °C. The liquid extract was further concentrated in a water bath at 50 °C until it reached a thick, paste-like consistency[9].

2.4. Determination of total phenolic content

2.4.1. Preparation of gallic acid stock solution

To prepare a 1000 ppm gallic acid stock solution, 10 mg of gallic acid was dissolved in 10 mL of analytical-grade ethanol. Serial dilutions of this stock solution were then performed to achieve final concentrations of 5, 10, 20, 30, and 40 ppm.

2.4.2. Preparation of 7.5% Na2CO3 solution

A total of 7,5 grams of Na2CO3 were weighed and dissolved in 100 mL of distilled water.[10]

2.4.3. Determination of operating time (OT)

A 300 µL aliquot of a 30 ppm gallic acid solution was combined with 1.5 mL of Folin-Ciocalteu reagent, stirred briefly, and allowed to stand undisturbed for 3 minutes. Subsequently, 1.2 mL of a 7.5% Na_2CO_3 solution was added and mixed thoroughly. The mixture was then left at room temperature for the duration of the operating time. The absorbance of

the solution was measured at 765 nm at intervals over a 0–60 minute period, and the time at which the absorbance stabilized was recorded as the operating time[11].

2.4.4. Determination of maximum wavelength

A 300 µL aliquot from each gallic acid solution (5, 10, 20, 30, and 40 ppm) was mixed with 1.5 mL of Folin-Ciocalteu reagent. The mixture was stirred and allowed to stand for 3 minutes. Next, 1.2 mL of a 7.5% Na_2CO_3 solution was added, and the mixture was thoroughly stirred until homogeneous. The solution was left at room temperature for the operating time (30 minutes). The absorbance was then measured at the maximum wavelength for gallic acid, and a calibration curve was generated based on the absorbance data[12].

2.4.5. Determination of total phenolic content

The lime peel extract was prepared by dissolving 10 mg of the extract in 10 mL of analytical-grade ethanol to achieve a concentration of 1000 ppm. The extract solution was then diluted with analytical-grade ethanol to a concentration of 100 ppm. Next, 300 µL of the diluted extract solution was mixed with 1.5 mL of Folin-Ciocalteu reagent, shaken, and allowed to stand for 3 minutes. Afterward, 1.2 mL of a 7.5% Na_2CO_3 solution was added, and the mixture was thoroughly mixed and incubated for 30 minutes at room temperature. The absorbance was measured at the maximum wavelength of 745.8 nm. The phenolic content was recorded as the mg equivalent of gallic acid per gram of sample. The measurement was repeated three times, and the total phenolic content was calculated using the following formula[12]:

$$\text{Total Phenolic Content} = C \times V \times FP/g$$

Note: C = concentration (mg/mL; V = volume of extract (ml); FP = dilution factor; g = the weight of sample used (gram)

2.4.6. Formulation of spray gel preparation

The formulation for the lime peel extract spray gel is outlined in Table 1. Methyl paraben and propyl paraben were dissolved in propylene glycol. Carbopol was dispersed in hot distilled water until it became homogeneous, after which triethanolamine was added, and the mixture was homogenized with the methyl paraben solution. HPMC was gradually added to a beaker containing hot distilled water and stirred until fully dissolved. The carbopol mixture was then poured into the HPMC solution and sonicated until uniform. The lime peel extract was dispersed in distilled water and sonicated to achieve a homogeneous extract solution. This extract was added to the HPMC and carbopol mixture, and distilled water was added to bring the total volume to 100 mL. The final mixture was sonicated for 5 minutes before being filled into spray containers[13].

Table 1 Formula of Sunscreen Spray Gel with Lime Peel Extract (*Citrus aurantifolia*)

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

*K- = Negative Control (without addition of lime peel extract)

2.4.7. Testing of physical properties of the preparation

The physical properties of lime peel (*Citrus aurantifolia*) spray gel formulation were tested using the following methods for each observed formula with 3 replicates[14].

pH Test

The pH of the spray gel formulation was measured using a pH meter. The pH was assessed to ensure it falls within the required range for topical applications (4.5–6.5), which helps prevent irritation[15].

Viscosity Test

A 100 mL sample of the formulation was placed in a Brookfield viscometer with spindle number 61, set at a speed of 12 rpm. The viscosity reading was recorded once the value on the viscometer stabilized[16].

Spreadability Test

The formulation was sprayed onto a plastic film from a distance of 5 cm, and its spreadability was assessed using a caliper. The diameter of the spread area was measured as the parameter for evaluation[16].

Drying Time Test

The formulation was sprayed onto the inner forearm of a volunteer from a distance of 5 cm. The time taken for the formulation to dry was recorded using a stopwatch[17].

Adhesion Test

For the adhesion test, the formulation was applied to the inner side of the lower arm of a volunteer by spraying it from a distance of 5 cm. If the spray gel droplets dripped within 10 seconds, it was rated as "dripping." If the droplets remained adhered for more than 10 seconds, it was rated as "adhering."[18]

2.4.8. Stability testing of the preparation

The preparation was subjected to a cycle of cold storage (4±2°C) for 24 hours, followed by exposure to a hot temperature (40±2°C) for 24 hours, constituting one cycle. This procedure was repeated for a total of 6 cycles. The physical changes in the spray gel were observed at the start and end of each cycle, including assessments of organoleptic properties, pH, viscosity, spreadability, drying time, and adhesive properties.

2.4.9. SPF value testing of the preparation

The determination of the SPF value of lime peel extract begins with weighing each formulation, including F1 (5%), F2 (10%), F3 (15%), positive controls (NIVEA® sunscreen spray SPF 30, Wardah® UV Shield Essential Sunscreen Gel SPF 30, and Emina® Sun Battle SPF 30), and negative control (formulation without extract), amounting to 1 gram. The correction factor (CF) is determined by measuring the absorbance of the positive controls, which have known SPF values. Each weighted formulation is combined with 50 mL of 70% ethanol and sonicated for 15 minutes. The sonicated formulation is transferred to a 100 mL volumetric flask and filled with 70% ethanol up to the mark. The formulation is then filtered using filter paper, and the first 10 mL of the filtrate is discarded. An aliquot (filtered formulation) of 100 µL is pipetted into a 25 mL volumetric flask and diluted with 70% ethanol up to the mark. Subsequently, the absorbance is measured using a UV-Vis spectrophotometer. The absorbance spectrum of the sample in solution form is obtained at wavelengths ranging from 290 to 320 nm with a 5 nm interval, using 70% ethanol as the blank. The absorbance values for each concentration are recorded and used to calculate the SPF value .[19] The SPF calculation according to the Mansur equation is as follows, with EE x I representing a constant factor.

$$\text{SPF} = \text{CF} \times \sum_{290}^{320} EE(\lambda) \times I(\lambda) \times abs(\lambda)$$

Note: CF= Correction Factor; EE= Erythema Effect; I= Intensity of sunlight; abs = sample absorbance

3. Results and discussion

3.1. Extraction

The extraction in this study was performed using the maceration method, which is simple, does not involve heating, and does not require special equipment. The material was soaked to break the cell walls and membranes through a pressure difference. Secondary metabolites in the cytoplasm are dissolved in the organic solvent (Ditjen POM, 2000). The extraction of phenolic compounds was conducted using a mixture of 70% ethanol and water. Ethanol 70% has the appropriate polarity for extracting flavonoids and tannins. Additionally, it has low toxicity and readily evaporates.[20]

Stirring was performed to achieve concentration equilibrium. The re-maceration process was employed to extract any remaining compounds in the residue after solvent saturation.[21] The filtrate was concentrated using a water bath at 50°C, resulting in a concentrated extract weighing 195.6 grams with a yield of 19.55%. The yield value is related to the content of bioactive compounds in the raw material. A higher yield corresponds to a higher desired substance content (Ditjen POM, 2000).

3.2. Determination of total phenolic content

The determination of total phenolic content was performed using the Folin-Ciocalteau reagent. Phenolic compounds can react with this reagent to form a solution with measurable absorbance. The Folin-Ciocalteau reagent oxidizes the hydroxyl groups of phenolic compounds, forming a blue-colored complex. This reaction proceeds slowly under acidic conditions, Na2CO3 was added during the test to create a basic environment and accelerate the reaction.[12]

The standard solution used was gallic acid, which is a simple, natural, and stable phenolic compound. During the reaction, the hydroxyl groups in the phenolic compounds react with the Folin-Ciocalteau reagent, forming a blue-colored molybdenum-tungsten complex. The intensity of the blue color increases with the concentration of phenolate ions formed. In other words, the higher the concentration of phenolic compounds, the more phenolate ions will reduce the heteropoly acid (phosphomolybdate-phosphotungstate) to form the molybdenum-tungsten complex, resulting in a darker color.[12]

The absorbance measurements of the gallic acid standard solution were used to construct a calibration curve. The curve, shown in Figure 1, follows a linear equation y = 0.0099x + 0.187 with a correlation coefficient (R) of 0.9989. This curve was used to determine the phenolic content of the sample. The average total phenolic content of lime peel extract was 42.66 ± 0.25 mg GAE/g, indicating that each gram of lime peel extract is equivalent to 42.66 mg of flavonoid.

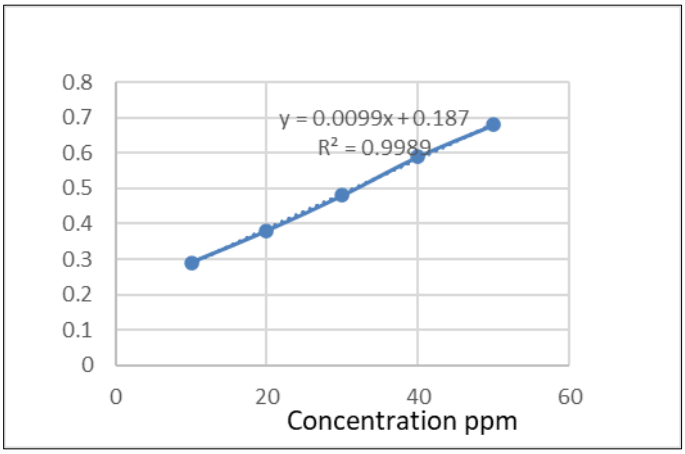

Figure 1 Gallic Acid Standard Curve

3.3. Testing of physical properties of the preparation

To achieve good and acceptable pharmaceutical formulations in society, the physical properties and stability of the preparations must be examined. Physical properties serve as determinants of the quality of pharmaceutical preparations. The physical characterization tests include organoleptic evaluation, pH, viscosity, spreadability, drying time, and adhesion. The results of physical property tests for the three formulas can be observed in Tables 2 and 3.

Tabel 2 Organoleptic test results of sunscreen spray gel formulation with lime peel extract

Formula	**Organolaptic**			
	Form	Odor	Color	Homogeneity
F1(5%)	Liquid	Lime smell	Yellow – brown	Homogeneous
F2(10%)	Liquid	Lime smell	Brown	Homogeneous
F3(15%)	Liquid	Lime smell	Dark brown	Homogeneous

Table 3 Results and significance test of sunscreen spray gel formulation with lime peel extract

Test	Formula	Results ± SD	Requirement
pH Test	F1 (5%)	5.7 ± 0.03	4.5 – 6.5
	F2 (10%)	5.5 ± 0.05	
	F3 (15%)	5.2 ± 0.05	
Viscosity	F1 (5%)	83.76 ± 0.97	< 150 cP
	F2 (10%)	115.07 ± 1.40	
	F3 (15%)	139.28 ± 1.78	
Spreadibility Test	F1 (5%)	6.2 ± 0.03	5 -7 cm
	F2 (10%)	5.6 ± 0.07	
	F3 (15%)	5.1 ± 0.07	
Drying Time Test	F1 (5%)	1.2 ± 0.08	< 5 minutes
	F2 (10%)	2.4 ± 0.06	
	F3 (15%)	3.3 ± 0.11	
Adhesion Test	F1 (5%)	37± 1	> 10 seconds
	F2 (10%)	62 ± 2	
	F3 (15%)	83 ± 2	

3.4. Organoleptic test

The three formulas have the same color and homogeneity, but they differ in consistency and color. A good formulation is characterized by a pleasant odor, attractive color, good consistency, and homogeneity. The consistency of the spray gel preparation is in liquid form, in accordance with its definition, which is one form of gel formulation development, which is a water-based phase system comprising at least 10% to 90% of the formulation's weight. The term 'spray' is defined as a composition that can be dispensed from its applicator, such as an aerosol or spray pump. A homogeneous formulation refers to a preparation that does not contain coarse particles, has evenly dispersed particles, and has a uniform color.[22] Although the preparation is in liquid form, the consistency of each formula is different. Formula III (F3) has the thickest consistency because it has the highest concentration of lime peel extract, which is 15%. This result proves that the higher the concentration of the extract, the thicker the resulting formulation, with a more intense color pigmentation.

3.4.1. pH test

The obtained results indicate that an increase in the concentration of lime peel extract has an effect on the pH value of the formulation, causing it to decrease. This is due to the higher concentration of salicylic acid, amino acids, citric acid, and vitamin C in the lime peel extract. As a result, the pH value of the formulation decreases. All formulations have met the requirement for a good pH value, which is in line with the pH of the skin ranges from 4.5 to 6.5. If the spray gel formulation is too acidic, it may cause skin irritation. On the other hand, if the pH of the formulation is too alkaline, it may lead to dryness of the skin[23].

3.4.2. Viscosity test

The obtained results indicate that an increase in the concentration of lime peel extract affects the viscosity value of the formulation, leading to an increase in viscosity. This is because higher concentrations of lime peel extract result in a thicker formulation. Viscosity also influences the spreadibility, drying time, and adhesion of the resulting formulation (Lachman et al., 2008). All formulations have met the requirement for a good viscosity value for the spray gel formulation, which is below 150 cP.[24]

3.4.3. Spreadibility test

The results obtained indicate that as the concentration of lime peel extract increases, the spreadibility value of the formulation decreases. This is because higher concentrations of lime peel extract lead to a thicker formulation, reducing its ability to spread. Consequently, the opportunity for the active ingredients to come into contact with the skin diminishes, resulting in a decrease in the effectiveness of the formulation when applied topically.[25] All formulations demonstrated a spreading pattern when sprayed and met the requirement for an ideal spreadibility value for the spray gel formulation, which is 5-7cm.[26]

3.4.4. Drying time test

The results obtained indicate that as the concentration of lime peel extract increases, the drying time of the formulation also increases. This is because higher concentrations of lime peel extract result in a thicker formulation, which requires more time to dry. All formulations have met the requirement for a good drying time value for the spray gel formulation, which is less than 5 minutes to prevent stickiness on the skin and provide comfort for the consumer when applied.[10]

3.4.5. Adhesion test

The results obtained indicate that as the concentration of lime peel extract increases, the adhesion value of the formulation also increases. This is because higher concentrations of lime peel extract result in a thicker formulation, leading to a longer adhesion time and increased release of active ingredients. A sunscreen formulation is expected to adhere to the skin for a longer period of time to provide prolonged protection against ultraviolet radiation.[10] All formulations can be considered to adhere well to the skin as long as the formulation droplets do not drip from the skin within less than 10 seconds.[27]

3.5. Stability testing of the preparation

The entire sunscreen spray gel formula is stored at a cold temperature of 4°C ± 2°C for 24 hours and at a high temperature of 40°C ± 2°C for 24 hours (1 cycle). After that, a physical stability test is conducted for 6 cycles. The results of the physical stability test for the sunscreen spray gel formulation can be seen in Tables 4 and 5.

Tabel 4 Organoleptic stability test results of sunscreen spray gel formulation with lime peel extract

Formula	**Cycle**	**Organoleptic**			
		Form	Odor	Color	Honogeneity
F1(5%)	0	Liquid	Lime smell	Yellow – brown	Homogeneous
	6	Liquid	Lime smell	Yellow – brown	Homogeneous
F2(10%)	0	Liquid	Lime smell	Brown	Homogeneous
	6	Liquid	Lime smell	Brown	Homogeneous
F3 (15%)	0	Liquid	Lime smell	Dark brown	Homogeneous
	6	Liquid	Lime smell	Dark brown	Homogeneous

Table 5 Results and significance of the physical stability test for the sunscreen spray gel formulation

Test	**Formula**	**Results ± SD**		**Requirement**
		Cycle 0	Cycle 6	
pH Test	F1 (5%)	5.7 ± 0.03	5.6 ± 0.05	4.5 – 6.5
	F2 (10%)	5.5 ± 0.05	5.4 ± 0.05	
	F3 (15%)	5.2 ± 0.05	4.9 ± 0.07	
Viscosity	F1 (5%)	83.76 ± 0.97	82.74 ± 1.51	< 150 cP
	F2 (10%)	115.07 ± 1.40	112.07 ± 1.56	

	F3 (15%)	139.28 ± 1.78	137.04 ± 1.71	
Spreadibility Test	F1 (5%)	6.2 ± 0.03	6.2 ± 0.03	5 -7 cm
	F2 (10%)	5.6 ± 0.07	5.7 ± 0.08	
	F3 (15%)	5.1 ± 0.07	5.3 ± 0.05	
Drying Time Test	F1 (5%)	1.2 ± 0.08	1.1 ± 0.05	< 5 minutes
	F2 (10%)	2.4 ± 0.06	2.2 ± 0.05	
	F3 (15%)	3.3 ± 0.11	3.2 ± 0.22	
Adhesion Test	F1 (5%)	37± 1	33 ± 2	> 10 seconds
	F2 (10%)	62 ± 2	55 ± 2	
	F3 (15%)	83 ± 2	71 ± 2	

3.5.1. Organoleptic test

The organoleptic properties observed in the formulation include the form and consistency, color, odor, and homogeneity of the spray gel. The results of the organoleptic testing of sunscreen spray gel formulations F1, F2, and F3 indicate that there were no changes in odor, color, and form of the formulation, and no visible phase separation throughout the 6 cycles of storage, both at cold and high temperatures. This indicates that the spray gel formulation exhibits good stability in organoleptic tests over the 6-cycle storage period.

3.5.2. pH test

The obtained results indicate that the formulation is stable, but there is a decrease in pH during the stability test. Changes in pH values during storage indicate reactions or damage to the components within the formulation, resulting in an increase or decrease in pH value.[28] This can occur due to oxidation reactions on the carboxylic acid groups of the acid compound in the extract, leading to the addition of hydrogen atoms and a decrease in pH value. Additionally, the use of transparent packaging is another factor contributing to the instability of the pH in the formulation as it allows light to interact and cause degradation reactions of secondary metabolites in the formulation.[29] This can be addressed by storing the formulation in a place that is not exposed to light and at an appropriate temperature. The choice of packaging should be tailored to the properties of the active substance and should protect the product from external influences. The use of buffers is also necessary in the formula to maintain the stability of the pH.

3.5.3. Viscosity test

The obtained results indicate that the formulation is physically unstable in terms of viscosity, but it still meets the viscosity acceptance criteria for a spray gel. The decrease in viscosity can be attributed to storing the formulation at high temperatures, which causes the active molecules in the formulation to move, weakening the intermolecular interactions and resulting in a decrease in viscosity .[8] Choosing an ideal storage temperature is important to maintain the viscosity stability of the formulation. Stability testing of the viscosity of the spray gel is crucial to ensuring that the formulation remains easy to spray through the applicator and adheres to the skin.

3.5.4. Spreadibility test

The obtained results indicate that the formulation is physically unstable in terms of spreading power, but it still meets the acceptance criteria for spreading power in a spray gel. This is due to a decrease in viscosity after storage, resulting in the weakening of the gel matrix's strength in the formulation, which leads to an increase in the spreading power of the formulation.[30]

3.5.5. [30]Drying time test

The obtained results indicate that the formulation is physically unstable in terms of drying time, but it still meets the acceptance criteria for drying time in a spray gel. This is due to a decrease in viscosity after storage, resulting in the formulation becoming more watery, which leads to a faster drying time.[14]

3.5.6. Adhesion test

The obtained results indicate that the formulation is physically unstable in terms of adhesion power, but it still meets the acceptance criteria for adhesion power in a spray gel. This is due to a decrease in viscosity after storage, resulting in the formulation becoming more watery, which leads to a decrease in the adhesion power of the formulation.[14]

3.5.7. SPF value testing of the preparation

The determination of the correction factor (CF) value in this study was done by measuring the absorbance of sunscreen products with known SPF values to ensure the calculation of SPF based on the formula. The absorbance values were then processed using the Mansur equation to determine the CF value used to account for the spectrophotometry and solvent usage (Allen & Ansel, 2014). The positive control sunscreen products used in this study included NIVEA® sunscreen spray SPF 30, Wardah® UV Shield Essential Sunscreen Gel SPF 30, and Emina® Sun Battle SPF 30. The results of the Correction Factor (CF) for the Positive Control of the Sunscreen Formulation can be seen in Table 6

Table 6 Results of correction factor (CF) for positive control of sunscreen formulation

Positive Control	SPF	Correction Factor (CF)	Results ± SD
NIVEA®	30	47.97	44.17 ± 0.05
Wardah®	30	40.29	
Emina®	30	44.25	

Table 7 Results and significance of spf testing for the spray gel

Formula	Results ± SD	SPF Categories
F1(5%)	15 ± 0.2	Medium
F2(10%)	20 ± 0.4	Medium
F3(15%)	30 ± 0.1	High

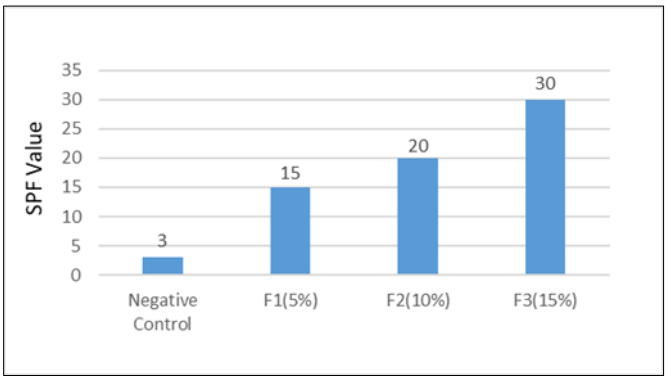

Figure 2 Graph of variation in extract concentration against SPF value

The selection of three different products with known SPF values as positive controls aimed to validate the chosen method for this study. The average CF value obtained from these three products was 44.17. This CF value would then be used to calculate the SPF value of the samples tested in this study. The SPF value testing on the negative control (formulation without extract) yielded an SPF value of 1,8872. The SPF value of the negative control indicated that the polymer in the gel spray without extract had no significant effect on the SPF value of the resulting gel spray formulation. The results of the SPF value testing for the gel spray formulation can be seen in Table 7, along with the graph showing the variation of extract concentration on the SPF value in Figure 2.

In accordance with the data presented in Table 7, the SPF values derived from the three formulations are classified within the medium to high range. This classification is established according to the protection range defined by the

Indonesian Food and Drug Monitoring Agency (BPOM). SPF values within the range of ≥6 - <15 are categorized as low, ≥15 - <30 as moderate, ≥ 30 - <50 as high, and ≥ 50 are classified as very high (BPOM RI, 2020). F1 and F2 belonged to the moderate protection category against UV rays, while F3 belonged to the high protection category. Increasing the extract concentration enhanced the SPF value in the sunscreen gel formulation due to the higher phenolic compound content in the formulation (Zuhroh, 2019). The sunscreen activity of the formulation was attributed to the presence of phenolic compounds in the lime peel extract, which had conjugated aromatic benzene groups capable of absorbing UV-B rays that can be harmful to the skin. Higher SPF values indicate longer protection against UV rays.[31]

4. Conclusion

The conclusion of this study is that the variation in the concentration of lime peel extract affects the physical properties and SPF value of the sunscreen gel spray formulation .Each formulation of the spray gel demonstrates good physical stability in viscosity, spreadibility, drying time, and adhesion tests. The SPF values derived from F1(5%), F2 (10%) dan F3 (15%) are classified within the medium to high range, with F3 (15%) having the highest SPF value of 30.15, classified as providing high protection against UV-rays.

Compliance with ethical standards

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Disclosure of conflict of interest

No conflict of interest to be disclosed.

Statement of informed consent

Informed consent was obtained from all individual participants included in the study.

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