

Article

The Effect of Antioxidants on the Photoinduced Degradation of Sunscreens

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Abstract

The stability of sunscreen formulations is a key factor influencing their protective efficacy and safety, particularly under UV exposure, which can induce photodegradation of UV filters. (1) Background: This study aimed to evaluate the effect of selected antioxidants (stabilized vitamin C, carrot macerate, and ferulic acid) on the sun protection factor (SPF) and photostability of sunscreen formulations. (2) Methods: Four oil-in-water creams with identical compositions, except for the antioxidant type, were prepared and characterized in terms of SPF, rheological behavior, pH, conductivity, stability, and sensory properties. SPF was determined using an in vitro spectrophotometric method before and after UV irradiation (30 and 120 min). (3) Results: Antioxidant incorporation increased initial SPF, with ferulic acid providing the most significant enhancement. After UV exposure, all formulations exhibited a decrease in SPF. However, the formulation containing ferulic acid maintained the highest absolute SPF value, indicating superior photostability. In contrast, vitamin C showed the greatest reduction in SPF, suggesting lower stability under irradiation. Physical characterization confirmed appropriate properties of the formulations, and sensory analysis identified the ferulic acid formulation as the most acceptable. (4) Conclusions: Ferulic acid demonstrated the most favorable impact on sunscreen performance, suggesting its potential as an effective multifunctional ingredient in sunscreens.

Keywords: sun protection factor; antioxidant; L-ascorbic acid; carrot macerate; ferulic acid; UV light

1. Introduction

Photoprotection can slow down or completely prevent skin damage caused by UV radiation. Sunscreens reduce skin damage through either chemical or physical mechanisms. There are also broad-spectrum filters that are effective against both UVA and UVB radiation, which can be combined with other UV filters [1].

The stability of photoprotective products is considered a fundamental prerequisite for ensuring proper photoprotection and safety. After absorbing radiation, UV filters transition into excited energy states. The absorbed energy is subsequently released primarily as heat, allowing the molecule to return to its original energy state. However, some UV filters may undergo photoisomerization, and in certain cases, irreversible bond cleavage can occur. These processes may result in the formation of photodegradation products, which can negatively affect the overall efficacy and safety of sunscreens. Some of these degradation products may react with cellular components, becoming toxic and potentially causing



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DNA damage. In addition, the stability of other ingredients present in photoprotective formulations may also be affected [2].

The photodegradation of UV filters plays a crucial role in the stability of sunscreen products. The outcomes of photodegradation may lead to reduced efficacy of the products, formation of by-products, and potential toxicity concerns. Several degradation pathways influence the stability of individual UV filters, with the most significant being photoisomerization, tautomerization, oxidative fragmentation, and triplet-state energy formation [3] (as shown schematically in Figure 1). Therefore, understanding the mechanisms of photodegradation is essential in the development of sunscreens that provide long-term UV protection without compromising consumer safety.

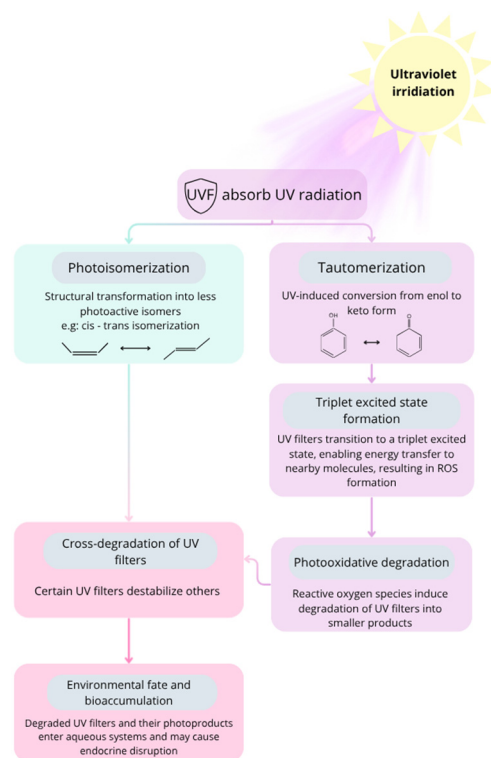


Figure 1. UV-induced transformation and degradation pathways of UV filters.

Antioxidants represent important substances that help protect the skin against the signs of aging. They act by reducing oxidative stress caused by unstable free radicals, which can damage cells, weaken the natural protective barrier of the skin, and accelerate the aging process. Skin aging is influenced by two main factors: intrinsic and extrinsic factors. Intrinsic factors include genetic predisposition and the natural chronological aging of the organism, while extrinsic factors mainly involve photoaging caused by exposure to UV radiation. Both types of aging are associated with changes in the physical, morphological, and physiological properties of the epidermis and dermis [4].

A wide range of antioxidants can be incorporated into sunscreen formulations, where they perform multiple functions. Their primary role is to mitigate oxidative stress in the skin, thereby reducing the visible signs of aging. In addition, they may also be useful in the treatment of certain UV-sensitive dermatoses. Numerous studies [5–10] highlight the importance of topically applied antioxidants in photoprotection. Antioxidants can contribute to the reduction in skin erythema and may also influence the immune response. Some organic UV filters become unstable after exposure to sunlight, which can lead to the formation of oxidized by-products. As a result, the phototoxicity of these products may increase while their protective efficacy decreases. For these reasons, compounds with

antioxidant activity are added to sunscreen formulations to enhance the stability of UV filters and help protect the skin from damage caused by free radicals [11].

Several approaches can be used to improve the photostability of photoprotective products, including the incorporation of antioxidants, encapsulation of active substances (UV filters), and the combination of multiple UV filters. These strategies contribute to increased stability of UV filters, preservation of their protective function, and overall improved efficacy of sunscreen products [2].

Among the antioxidants commonly used in photoprotection is vitamin C (L-ascorbic acid), which is well known for its ability to neutralize reactive oxygen species and regenerate oxidized vitamin E. Despite its strong antioxidant activity, ascorbic acid is chemically unstable, particularly in aqueous formulations, which has led to the development of more stable derivatives with improved skin permeation. In addition to its antioxidant effects, vitamin C has been shown to increase the skin deposition of mineral UV filters without increasing systemic absorption, thereby prolonging the photoprotective effect of sunscreen formulations [11]. Furthermore, vitamin C supports collagen synthesis and inhibits UV-induced alterations in elastin associated with skin photoaging [12]. Several studies also suggest that the combination of vitamins C and E may improve the stability of UV filters such as avobenzone [2,13].

Ferulic acid is another important antioxidant used in photoprotective formulations. Besides its antioxidant properties, it also exhibits anti-inflammatory activity and the ability to absorb UV radiation. Its derivatives have increased lipophilicity, which may enhance their suitability for topical formulations. Ferulic acid has been shown to reduce UVB-induced erythema and to exert synergistic effects with UV filters and other polyphenols, potentially increasing SPF values and improving UVA protection [11,14]. As Peres et al. [14] found, incorporation of ferulic acid into topical vitamin formulations improved the vitamins' chemical stability and strengthened their photoprotective properties, leading to decreased erythema and reduced corneocyte apoptosis.

Carrot macerate, with antioxidants as its main component in the form of carotenoids, including β -carotene, lutein, and zeaxanthin, has also demonstrated photoprotective potential. Studies indicate their ability to neutralize free radicals generated following UV exposure, thereby contributing to DNA protection and reduction of oxidative stress. Beneficial effects have been reported after both systemic and topical administration of β -carotene [15].

The present study investigated the effect of the antioxidants mentioned above (vitamin C, carrot macerate, ferulic acid) on the SPF value and photostability of sunscreen formulations.

The studied antioxidants were selected based on the review by Jesus et al. (2023) [11], which analyzed the most commonly used antioxidants in sunscreen formulations. Tocopherol and its derivatives ranked among the most frequently utilized antioxidants and were therefore incorporated into all formulations. As a lipophilic antioxidant, tocopherol primarily stabilizes the oil phase of the cream. However, its effect on SPF values immediately after preparation and following UV exposure was not investigated in this study. Other commonly used antioxidants included vitamin C and ferulic acid, which were also incorporated into the formulations. Carrot macerate was selected to evaluate its potential efficacy, as it is intended to be prepared for future use as a natural stabilizing agent in cosmetic products.

The aim of the study is to determine to what extent the type of antioxidant used in a sunscreen formulation may influence, on the one hand, the overall SPF value, and on the other hand, the degree to which the SPF is preserved after exposure to UV radiation. The experimentally investigated antioxidants were vitamin C, carrot macerate, and ferulic acid. Specifically, four sunscreens incorporating various antioxidants were prepared, and their

sun protection factor (SPF) was determined. The study further investigated the effect of UV radiation on the stability and SPF values of the formulations. Rheological properties and pH values were also assessed to characterize the formulations. The stability of each preparation was evaluated through a series of stability tests, and finally, sensory analysis was conducted on the two best-performing samples to assess their suitability for practical application.

The UV filter system was selected to provide broad-spectrum photoprotection and to achieve a high level of UV protection suitable for daily-use sunscreen formulations. The selection of individual UV filters was based on their complementary absorption profiles and compatibility within the formulation, allowing effective coverage of both UVB and UVA spectral regions. The final combination was optimized through preliminary formulation studies, which showed that the simultaneous incorporation of all selected UV filters resulted in the highest SPF values, suggesting a beneficial synergistic effect.

2. Materials and Methods

Beautyderm® K10 and Span® 80 were obtained from Merck KGaA (Darmstadt, Germany); cetyl alcohol and glycerol 85% were supplied by Galvex (Banská Bystrica, Slovakia); Cosgard® preservative, ferulic acid, carrot macerate, Olivem® 1000 MB, stabilized vitamin C (sodium ascorbyl phosphate), and xanthan gum E415 were purchased from Ekokoza (Fryčovice, Czech Republic); purified olive oil and tocopherol acetate were provided by Fagron (Olomouc, Czech Republic); ethanol 96% was obtained from CentralChem (Bratislava, Slovakia); Sunhancer™ ECO SPF Booster was obtained from Lubrizol (Wickliffe, OH, USA); Tinosorb® M, Tinosorb® S, and Uvinul® MC 80 were purchased from BASF (Mannheim, Germany); Xylance was obtained from Vegis.sk (Poruba, Slovakia); purified water was produced by reverse osmosis at the Department of Galenic Pharmacy (Bratislava, Slovakia).

2.1. Preparation of the Sunscreens

Four photoprotective creams were prepared with identical compositions of UV filters and excipients. The samples differed only in the type of antioxidant used. The percentage composition of the individual samples is presented in Table 1.

Table 1. Composition of the sunscreens.

Component	F1	F2	F3	F4
Olive oil	17.8	17.8	17.8	17.8
Beautyderm® K10	4	4	4	4
Olivem® 1000 MB	6	6	6	6
Cetyl alcohol	5	5	5	5
Xylance	4	4	4	4
Span® 80	4.8	4.8	4.8	4.8
Booster	3	3	3	3
Tinosorb® M	10	10	10	10
Tinosorb® S	10	10	10	10
Uvinul® MC 80	10	10	10	10
Xanthan gum	0.2	0.2	0.2	0.2
Purified water	18.2	17.2	17.2	17.2
Glycerol 85%	6.5	6.5	6.5	6.5
Tocopherol	0.2	0.2	0.2	0.2
Stabilized vitamin C	-	1.0	-	-
Carrot macerate	-	-	1.0	-
Ferulic acid	-	-	-	1.0
Cosgard®	0.3	0.3	0.3	0.3

The specified amounts of lipophilic components (olive oil, Beautyderm® K10, Olivem® 1000, cetyl alcohol, Xylance, tocopherol, Tinosorb® S, Uvinul® MC 80, Span® 80) were weighed into a stainless steel mortar. The mortar was subsequently placed under an infrared lamp and heated to approximately 60 °C.

A measured amount of purified water was transferred into a glass beaker, followed by the addition of xanthan gum, which was allowed to hydrate and swell for approximately 2 h. The dispersion was then subjected to continuous stirring using a magnetic stirrer at low rotations to minimize air incorporation and ensure uniform hydration. After complete swelling, the remaining aqueous-phase components (glycerol and Tinosorb® M) were added. The mixture was subsequently heated under constant stirring on a hot plate to a temperature approximately 5 °C higher than that of the lipophilic phase.

The emulsification process was carried out by gradual incorporation of the hydrophilic phase into the lipophilic phase under continuous stirring, ensuring controlled addition and formation of a stable emulsion. The resulting cream was stirred until it cooled to ambient temperature. Subsequently, a precisely weighed amount of the booster was incorporated, followed by homogenization of the formulation. Finally, four drops of Cosgard® were added as a preservative to enhance microbiological stability of the aqueous phase.

The remaining formulations (F2–F4) were prepared using an analogous procedure, with minor variations in the method of antioxidant incorporation. In formulation F2, stabilized vitamin C was added to the aqueous phase. In formulation F3, carrot macerate was incorporated into the lipophilic phase. In formulation F4, ferulic acid was introduced into the hydrophilic phase prior to emulsification.

2.2. SPF Determination in Sunscreens

The sun protection factor (SPF) values of the individual sunscreens were determined using an in vitro spectrophotometric method according to Mbanga et al. (2015) [16], based on the measurement of absorbance of ethanolic (96%, v/v) solutions over a defined wavelength range.

Approximately 1.0 g of each prepared sample was accurately weighed into a wide-neck flask and diluted with ethanol (96%; v/v) to a final mass of 100.0 g. The flask was sealed with cellophane film and subjected to ultrasonic treatment for 5 min to ensure complete dispersion. Then, the mixture was homogenized using a magnetic stirrer at 1000 rpm.

Subsequently, the sample solution was filtered through filter paper into a glass beaker. An aliquot of 5 mL of the filtrate was transferred into a 100 mL volumetric flask and diluted to the mark with ethanol (96%; v/v), followed by thorough mixing. A further 5 mL of this solution was then pipetted into a 25 mL volumetric flask, diluted to volume with ethanol (96%; v/v), and mixed again to obtain the final solution for analysis.

The prepared solution was analyzed by UV–VIS spectrophotometry in the wavelength range of 290–320 nm at 5 nm intervals, using ethanol (96%; v/v) as a blank reference. This procedure was applied to all four samples, with five parallel measurements performed for each formulation.

The SPF values were subsequently calculated using the following Equation (1):

$$SPF = CF \sum_{290}^{320} EE(\lambda) \cdot I(\lambda) \cdot Abs(\lambda) \quad (1)$$

where *Abs* represents the measured absorbance at a given wavelength, *CF* is the correction factor, *EE* denotes the erythemal effect spectrum, and *I* corresponds to the solar intensity spectrum [16]. The predetermined *EE* × *I* values corresponding to the individual wavelengths are listed in Table 2.

Table 2. Predefined $EE \times I$ values at specific wavelengths [16].

Wavelength (nm)	$EE \times I$
290	0.0150
295	0.0817
300	0.2874
305	0.3227
310	0.1864
315	0.0837
320	0.0180

The prepared sunscreens were subjected to controlled UV irradiation to evaluate the effect of UV exposure on SPF values. Approximately 1.1 g of each formulation was applied using a syringe onto Transpore™ 3M tape (3M Company, St. Paul, MN, USA; width 7.5 cm). The samples were evenly distributed over a pre-marked circular area (diameter 2.7 cm), ensuring consistent layer thickness across all tested samples. Three parallel measurements were carried out.

The edges of the tape were fixed onto a metal holder to allow uniform UV irradiation not only of the sample surface but also of its surrounding area. The samples were placed in a laminar flow cabinet equipped with a UV lamp and exposed for predetermined time intervals.

After 30 min of irradiation, the samples were removed. Then, 1.0 g of the formulation was carefully transferred from the Transpore™ tape into a wide-neck flask using a spatula, and its SPF value was determined according to the previously described method. The same procedure was followed for samples that were exposed to UV irradiation for 120 min. The values of absorbance and calculated factor (SPF) are recorded in Supplementary Materials, Tables S1–S4.

2.3. Physical Characterization of the Sunscreens

2.3.1. Electrical Conductivity

Electrical conductivity was determined using a microammeter (Metra, Blansko, Czech Republic). The electrode was immersed in the sample, and the measurement was recorded after stabilization of the reading. Subsequently, the electrode was rinsed with distilled water and carefully dried prior to the next measurement. Each determination was performed in triplicate for every sample. All measurements were conducted at a controlled temperature of 25 ± 0.5 °C.

2.3.2. pH Determination

The pH of individual samples was determined using a method adapted and slightly modified according to Omar et al. (2021) [17]. First, 1.0 g of each sample was accurately weighed into a vial and diluted with 9 mL of distilled water. The distilled water was preheated to approximately 80 °C to facilitate rapid dispersion of the cream. The mixture was thoroughly shaken until a homogeneous dispersion was achieved and the temperature decreased to approximately 25 ± 0.5 °C.

Subsequently, the electrode of a pH meter (SevenDirects SD20, Mettler Toledo, Greifensee, Switzerland) was immersed in the prepared dispersion, and the measurement was recorded after stabilization of the reading. The electrode was then rinsed with distilled water and carefully dried before further use. For each formulation, five parallel measurements ($n = 5$) were performed to ensure data reliability. All pH determinations were carried out at a controlled laboratory temperature of 25 ± 0.5 °C.

2.3.3. Rheological Measurement

The rheological properties of the prepared photoprotective creams were evaluated using a rotational viscometer (RheolabQC, Anton Paar, Graz, Austria). Approximately 2 mL of each sample was introduced into the gap of the stationary cylinder up to the marked level, after which it was connected to the rotating part of the measuring system CC27. Prior to measurement, the samples were equilibrated to a temperature of 25 ± 0.5 °C for 20 min directly in the viscometer.

The rheological measurements were performed in two consecutive cycles. During the first cycle, the shear rate was gradually increased from 0 to 650 s^{-1} . Apparent viscosity (mPa·s) and shear stress (Pa) were recorded automatically every 10 s at the following shear rates: 72.2, 144.0, 217.0, 289.0, 361.0, 433.0, 506.0, 578.0, and 650.0 s^{-1} . In the second cycle, the same parameters were recorded at identical shear rates. However, the shear rate was decreased stepwise from 650 to 0 s^{-1} . The resulting flow curves were used to evaluate the thixotropic behavior of the formulations. The obtained experimental data were analyzed and graphically processed using RheoCompass™ software (version 1.31.69). Between individual measurements, the measuring system was thoroughly cleaned, rinsed with distilled water, and dried to ensure the accuracy and reproducibility of the results [18].

The hysteresis loop area (HLA) was determined as the area enclosed between the upward and downward flow curves in the shear stress–shear rate diagram. The area was calculated by numerical integration of the difference between the shear stresses measured during the increasing and decreasing shear-rate ramps using the trapezoidal rule. The HLA value was used as an indicator of the degree of thixotropy, with larger values corresponding to a greater extent of structure breakdown and slower structural recovery.

2.4. Stability Assessment of the Sunscreens

2.4.1. Centrifugation Test for Stability Assessment

The stability of the prepared formulations F1–F4 was evaluated using a centrifugation test based on the method described by Navarro-Pérez et al. (2021) [19], with minor modifications to accommodate laboratory conditions. Approximately 10 g of each sample was weighed into glass test tubes, which were subsequently sealed. The samples were then subjected to centrifugation at 3000 rpm for 5 min. After centrifugation, the samples were removed and visually assessed for any signs of instability, such as phase separation or changes in texture, in order to evaluate the stability of the individual photoprotective creams.

2.4.2. Freeze–Thaw Cycling Test

The stability of the prepared formulations F1–F4 was further assessed using a freeze–thaw cycling method [20], with slight adaptations to suit the available experimental conditions. Approximately 10.0 g of each sample was transferred into sealable glass test tubes. The samples were stored at -18 ± 0.5 °C for 24 h, followed by storage at room temperature (25 ± 0.5 °C) for an additional 24 h. This sequence constituted one complete cycle. The procedure was repeated four times in total. After each cycle, the samples were visually examined for any changes, including phase separation, alterations in consistency, or other signs of destabilization.

2.4.3. Stability Monitoring over Time

The physical stability of the formulated sunscreens was evaluated following established cosmetic stability testing principles [21]. Samples were transferred into sealed containers and stored at controlled room temperature (25 ± 0.5 °C) for the duration of the study. Stability was monitored over a 12-week period, with evaluations conducted at 4, 8, and 12 weeks. At each time point, formulations were subjected to visual inspection to detect

signs of physical instability. Particular attention was given to organoleptic and structural parameters, including changes in color, phase separation phenomena (e.g., creaming or coalescence), and alterations in consistency such as thickening or loss of homogeneity. These characteristics are widely considered critical indicators of emulsion destabilization and product degradation.

2.5. Sensory Analysis

Sensory analysis of the selected formulations was carried out using 12 volunteer participants. The evaluation focused on key physicochemical and sensory attributes of the tested photoprotective creams, including appearance, spreadability, softness upon skin contact, stickiness, residual film after application, and absorption capacity.

Each participant compared two samples according to predefined evaluation criteria [18,22]. The evaluation questionnaire template is provided in Supplementary Materials as Table S5. Individual attributes were assessed using a five-point rating scale, where the lowest score indicated the least favorable perception and the highest score reflected a high level of volunteer satisfaction with the sensory properties of the sample. The mean values for each evaluated parameter were calculated and graphically illustrated for better visualization and comparison.

2.6. Statistical Analysis

The results are presented as the mean of three or five independent measurements (physical characterization: $n = 3$; SPF determination: $n = 5$) $\pm$ standard deviation (SD). Statistical differences between groups were evaluated using Student's *t*-test. A *p*-value ≤ 0.05 was considered statistically significant. Levels of significance are indicated in figures and tables.

3. Results

3.1. SPF Determination

The prepared photoprotective creams exhibited a pleasant texture and were odorless, indicating good initial sensory properties. Formulations F1, F2, and F4 displayed a white to slightly yellow coloration, whereas formulation F3 showed a subtle orange tint. This difference in appearance can likely be attributed to the presence of carrot macerate in formulation F3.

Following the preparation of the individual creams, the primary parameter evaluated was the SPF. Based on the measured SPF values of the tested sunscreen formulations, the final samples were further subjected to a series of additional analyses. These included the assessment of physical properties and stability, as well as consumer perception evaluated through sensory analysis.

Firstly, SPF was determined immediately after preparation while also assessing the effect of added antioxidants on this parameter. The absorbances and SPF values are recorded in Supplementary Materials, Tables S1–S4; these present the absorbance values of the ethanolic solutions of the individual sunscreen formulations F1 to F4, which differ in their antioxidant content (F1—reference formulation, F2—containing vitamin C, F3—containing carrot macerate, and F4—containing ferulic acid). Absorbance was measured at wavelengths ranging from 290 to 320 nm at 5 nm intervals, using ethanol as the blank, which was also employed for sample extraction. The resulting SPF values were calculated according to Equation (1). The same procedure was repeated after 30 min and 120 min of UV exposure of the samples.

The reference sample, F1, which did not contain any antioxidant, achieved an SPF value of 41.67. In the remaining samples containing selected antioxidants, an increase in

SPF was observed in the following ascending order: F3 < F2 < F4. The most significant increase in SPF compared to the reference sample F1 was recorded for the formulation F4, containing ferulic acid, with an enhancement of 5.93 SPF units. For sample F2, the increase was 1.53 SPF units, while for sample F3, it was 0.26 SPF units. In the case of ferulic acid, our findings are consistent with those reported by Jesus et al. (2023) [11], who observed an increase in SPF from 19.7 to 26 during in vivo testing.

In the next step, attention was focused on the stability of the formulations and the effect of UV radiation on their SPF values. All samples were exposed to UV radiation for 30 and 120 min. The SPF values of the sunscreen formulation without UV exposure and after 30 and 120 min of UV exposure are compared in Figure 2. Based on these results, the smallest decrease in SPF was observed for sample F1, with the decline gradually increasing in the order of F3 < F4 < F2; i.e., the most pronounced reduction in SPF was recorded for the formulation containing vitamin C.

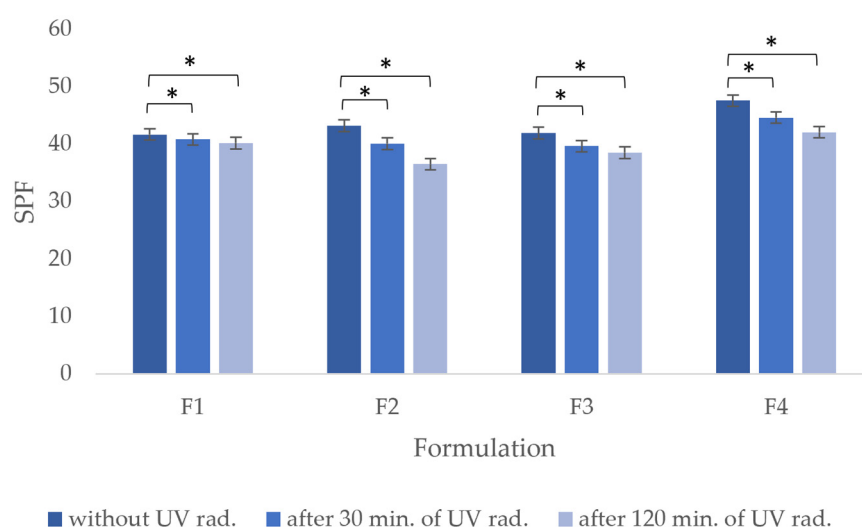


Figure 2. SPF values of sunscreen formulations (F1–F4) evaluated in vitro: (a) without UV exposure (dark blue column), (b) after 30 min of UV exposure (blue column), (c) after 120 min of UV exposure (light blue column). Columns marked with an asterisk (*) are statistically significantly different at the $p < 0.05$ significance level.

Based on our results, it can be concluded that the relative decrease in SPF between the initial measurement and after 120 min of UV exposure was lowest for sample F1, which contained no antioxidant. However, when evaluating absolute SPF values after exposure, formulation F4 containing ferulic acid achieved the best performance, maintaining the highest SPF value (42.07 ± 0.47) and thus providing the most effective UV protection, even after two hours of irradiation. The SPF values of the remaining formulations after exposure decreased in the following order: F1 > F3 > F2. The greatest reduction in SPF was observed for sample F2 containing vitamin C, with a decrease of up to 15.46%.

3.2. Conductivity Measurement

The measured conductivity values are presented in Table 3. Based on the obtained results, electrical conductivity increased in the following order: F3 < F1 < F2 < F4. The lowest conductivity was observed for the sample containing carrot macerate, which can be explained by its hydrophobic nature. In contrast, ferulic acid and vitamin C were incorporated into the hydrophilic phase, thereby increasing its relative proportion and, consequently, the overall conductivity of the formulations. Based on these findings, it can be concluded that all prepared creams are oil-in-water (*o/w*) emulsions, as all samples could conduct electric current.

Table 3. Basic physical and chemical properties of the prepared sunscreens.

Formulation	pH	HLA (kPa·s ^{−1})	Conductivity (μA)	Stability		
				Centrifuge	Freeze–Thaw	Over Time
F1	6.02 ± 0.05	73.02 ± 0.04	10.67 ± 1.53	no	no	yes
F2	7.85 ± 0.03 *	182.63 ± 0.05 *	24.67 ± 1.53 *	yes	yes	yes
F3	6.42 ± 0.35 *	71.74 ± 0.04 *	6.67 ± 1.15 *	no	no	yes
F4	4.19 ± 0.11 *	84.54 ± 0.05 *	20.67 ± 1.53 *	yes	yes	yes

* Statistically significant differences compared to the F1 reference sample ($p < 0.05$). HLA—hysteresis loop area.

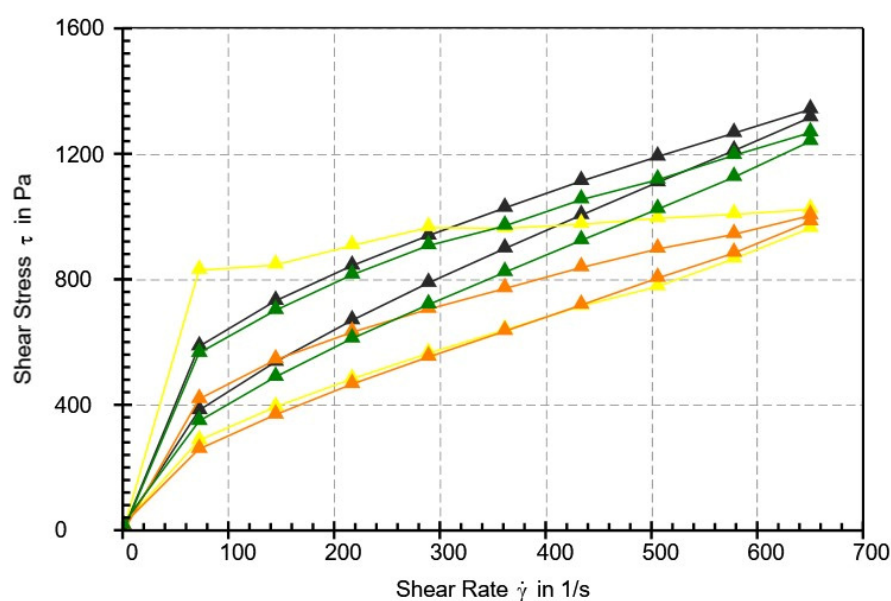
3.3. pH Determination

The optimal pH of the skin is in the range from 4 to 7 [23]. Although the natural pH of the skin is typically below 5.0, topical formulations with a pH within the range of 4 to 7 are generally considered physiologically compatible and non-irritating [24]. The measured pH values of the individual formulations are presented in Table 3. The pH values increased in the following order: F4 < F1 < F3 < F2. Three of the tested formulations fell within the physiologically acceptable pH range and can therefore be regarded as non-irritating. Only sample F2, containing vitamin C, exhibited a higher pH value of 7.85 ± 0.03 .

3.4. Rheological Behavior

Rheological properties provide a comprehensive understanding of the physical characteristics, structure, and stability of formulations [25]. In addition, they have a significant impact on the release of active pharmaceutical ingredients, such as vitamins and even the drugs themselves, which may be contained in dermal bases.

The rheograms illustrating the dependence of shear stress on shear rate are shown in Figure 3. The curve indicates that the photoprotective formulations exhibit time-dependent rheological behavior characteristic of thixotropic systems. As shear rate increases, the internal structure of the system is disrupted, leading to a decrease in apparent viscosity (see Figure 4). The apparent viscosity of the formulations increases in the following order: F3 < F4 < F1 < F2. The hysteresis loop area (HLA) increased following the incorporation of vitamin C or ferulic acid into the formulations (Table 3).

**Figure 3.** Rheograms of the formulations: F1 (black), F2 (yellow), F3 (orange), F4 (green).

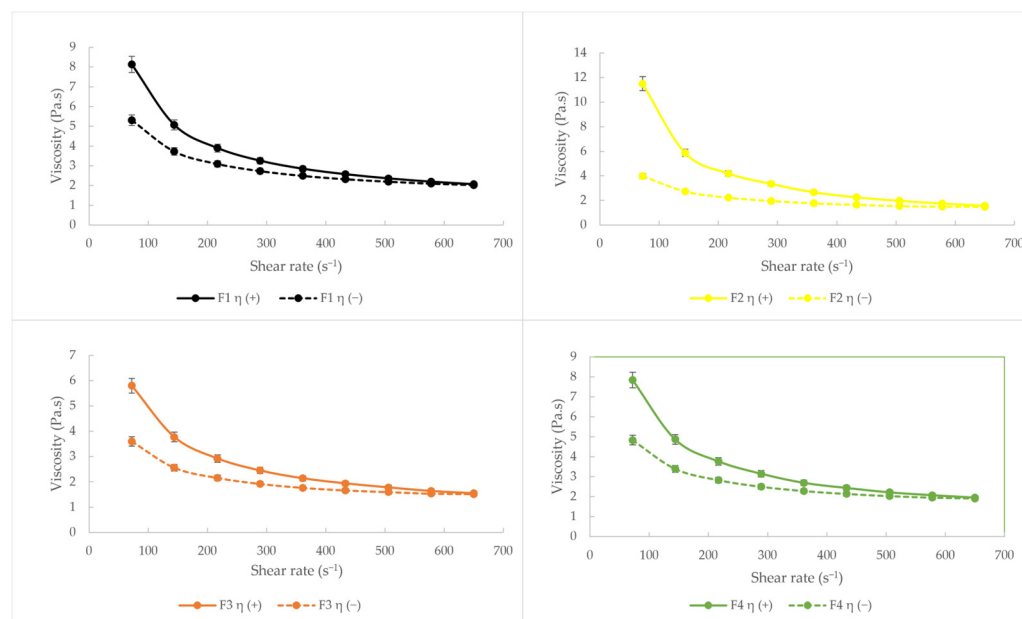


Figure 4. Dependence of apparent viscosity on shear rate for individual formulations. Solid lines indicate measurements obtained during increasing rotational speed (+), while dashed lines indicate measurements obtained during decreasing rotational speed (−).

3.5. Stability

Emulsions are thermodynamically unstable systems that naturally undergo changes over time due to various destabilization processes. These phenomena include gravitational separation of components (creaming or sedimentation), flocculation, droplet coalescence, Ostwald ripening, and phase inversion. When an emulsion is subjected to gravitational or centrifugal forces, oil droplets tend to move either upward (creaming) or downward (sedimentation). This process is driven by the density difference between the dispersed and continuous phases [26].

Centrifugation test: After centrifugation, the samples were visually inspected. In the formulations F1 and F3, small oil droplets appeared on the surface, indicating lower stability. In contrast, samples F2 and F4 can be considered stable based on this test.

Stability test using freeze–thaw cycles: Visual evaluation of the samples was performed after each cycle. In formulations F1 and F3, small oil droplets were observed during the final cycle, suggesting reduced stability. Based on these findings, samples F2 and F4 demonstrated higher stability.

Stability over time: The sunscreens were stored in sealed containers at room temperature (25 ± 0.5 °C). The stability of the formulations was visually evaluated at time intervals of 4, 8, and 12 weeks after preparation, focusing on changes in colour, phase separation, and the occurrence of creaming. All samples exhibited sufficient stability throughout the entire observation period. No significant changes were observed over the 12-week period. The samples showed no signs of destabilization, nor were any visible changes in color detected.

3.6. Sensory Analysis

Based on the results of the stability tests, formulations F2 and F4 were selected for final evaluation through sensory analysis. This analysis was conducted on a group of 13 female volunteers with an average age of 37.16 ± 9.52 years. The participants evaluated the sensory properties of the formulations, specifically softness, adhesiveness, spreadability, stickiness, residual feel, and absorption rate.

The evaluation was performed using a scale from 1 to 5, where 1 represented the lowest rating and 5 the highest. The mean value for each parameter is shown in Figure 5. Formulation F4 achieved the highest scores across all evaluated attributes, as clearly illustrated in Figure 5.

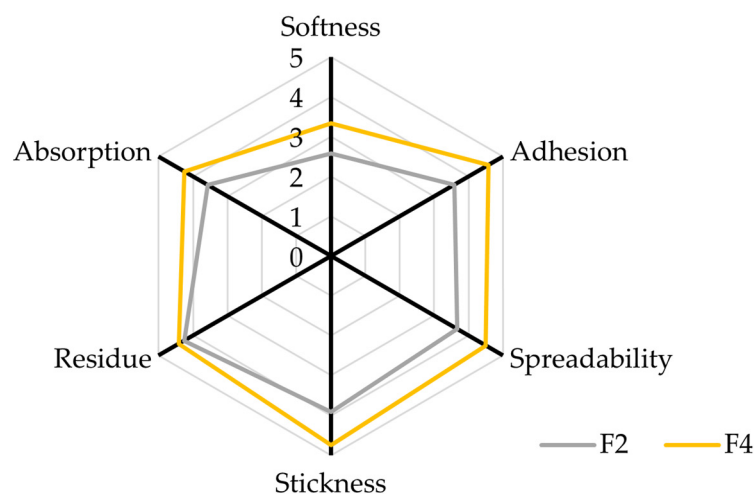


Figure 5. The main sensory attributes of the formulation F2 (with vitamin C) and the formulation F4 (with ferulic acid).

However, both samples received the lowest ratings in the softness parameter (F2: 2.58 ± 0.99 ; F4: 3.33 ± 1.23). Sample F2 achieved its highest score in the residual feel attribute (4.25 ± 0.75), while sample F4 performed best in terms of stickiness (4.75 ± 0.45). Overall, the results of the sensory analysis can be considered favorable, as all evaluated attributes—except for softness—achieved average scores above 3.5.

4. Discussion

Intrinsic skin aging is closely associated with the action of free radicals and oxidative stress, which lead to damage of cellular structures, degradation of collagen and elastin, and consequently to the formation of wrinkles and loss of skin elasticity [27]. Moreover, several forms of radiation can threaten skin health, including ionizing radiation, ultraviolet (UV) rays, infrared (IR) radiation, visible light, radiofrequency radiation, and microwaves [28].

Our goal was to develop a photoprotective sunscreen with an optimal formulation and to study the effect of various antioxidants on the product's photostability. The selection of UV filters used in the formulation was based on previous research [18], in which various combinations of chemical UV filters were evaluated. That study indicated that achieving high levels of photoprotection, with SPF values approaching 50, required the combination of all selected chemical UV filters at their maximum permitted concentrations. The primary objective was to develop a stable sunscreen formulation with the highest achievable SPF while maintaining acceptable physicochemical and sensory properties.

The chosen UV filter system was designed to provide broad-spectrum UV protection. Tinosorb® M (methylene bisbenzotriazolyl tetramethylbutylphenol, also known as bisoctrizole) is a broad-spectrum organic UV filter providing protection throughout the UVB and UVA regions, including substantial UVA-I coverage [29]. Tinosorb® S (bis-ethylhexyloxyphenol methoxyphenyl triazine, also known as bemotrizinol) is a highly photostable broad-spectrum UV filter covering both UVB and UVA wavelengths, with particularly strong absorption in the UVA-I region [30]. Uvinul® MC 80 (ethylhexyl methoxycinnamate, also known as octinoxate) is an efficient UVB filter with maximum absorption around 310–311 nm. It was included primarily to provide strong UVB protection [18] and to complement the broad-spectrum coverage provided by Tinosorb® M and Tinosorb® S.

Based on the results of a previous study [18], which confirmed the SPF booster effect, the same booster type, Sunhancer™ ECO SPF Booster, was selected in the formulations. It is a natural SPF-enhancing ingredient composed of micronized particles derived from sustainably sourced Carnauba wax and Rice bran wax. Carnauba wax contains cinnamic acid derivatives with UV-absorbing and antioxidant properties, which contribute to improved sunscreen performance. Rice bran wax is rich in bioactive compounds, particularly flavonoids, that provide protection against ultraviolet radiation [18,31].

For effective free-radical neutralization, topical antioxidants need to penetrate and remain within the skin after application. In contrast, physical UV filters should stay on the skin's surface rather than being absorbed, allowing them to block ultraviolet radiation while minimizing potential toxicity [32]. Additionally, antioxidants applied topically should accumulate locally in the skin reservoir instead of entering systemic circulation.

The role of antioxidants in dermal formulations is multifaceted. Antioxidants in skincare products perform several important functions, helping to stimulate regenerative processes and collagen synthesis, thereby promoting increased skin firmness and reducing wrinkles. At the same time, they improve the skin's overall appearance by enhancing its radiance [33]. On the other hand, they play a key role in stabilizing cosmetic formulations, as they inhibit oxidative and photodegradation processes not only during storage but also after application to the skin, thereby protecting active ingredients from degradation induced by external factors such as the effect of radiation.

Köckler et al. [34] investigated the photostability of UV filters using a method based on their incorporation into an oil-in-water emulsion applied to polymethyl methacrylate (PMMA) plates. The prepared samples were subsequently exposed to radiation from a solar simulator ($\lambda > 290$ nm) at a dose of 650 Wh/m² over defined time intervals. An irradiation period of 120 min was established as the threshold between acceptable and insufficient photostability, corresponding to the recommended reapplication interval of sunscreen products every two hours. In the present study, this methodology was adapted to the available laboratory conditions and instrumentation.

PMMA plates are currently considered the preferred substrate for many in vitro sunscreen photostability studies because of their photostable, non-reactive nature and highly reproducible surface characteristics [34]. PMMA plates are recommended in both European and U.S. methodologies for the determination of UVA protection factor and critical wavelength and are therefore widely used for broad-spectrum sunscreen evaluation [35,36]. Furthermore, Ahn et al. [37] identified PMMA plates as the most suitable substrate among commercially available options, including Transpore™ tape and Vitro-Skin®, due to their mechanical strength, ease of handling, and reduced interference from substrate properties.

Nevertheless, the choice of substrate should depend on the specific objective of the study. Several investigations have demonstrated that although PMMA plates are advantageous for UVA and photostability testing, Transpore™ tape may provide better agreement between in vitro and in vivo SPF measurements. Garoli et al. [36] reported that Transpore™ tape showed stronger correlations with in vivo SPF values than PMMA plates. Similar observations were reported by Dimitrovska Cvetkovska et al. [38], who found that Transpore™-based methodologies produced a closer correlation between in vitro and in vivo SPF results than methods employing PMMA substrates.

For these reasons, Transpore™ 3M tape was selected in the present work for practical and methodological purposes. The porous structure of Transpore™ tape has been widely used in dermatological and sunscreen research and facilitates homogeneous spreading, retention, and subsequent recovery of semisolid formulations. In addition, its porous microstructure provides a textured surface that may better reproduce certain aspects of skin microrelief than the comparatively rigid PMMA substrate. Scalia et al. [39] demonstrated

that PMMA plates and Transpore™ tapes provide comparable results before irradiation, indicating that both substrates are suitable for non-irradiated *in vitro* sunscreen evaluation. However, after UV exposure, PMMA plates generally showed a greater reduction in SPF and a better correlation between spectrophotometric and HPLC measurements of UV-filter degradation, suggesting that PMMA plates may provide a more reliable assessment of sunscreen photostability.

In our experiment, we applied 1.1 g of sample on the Transpore™ tape because approximately 1 g is required for the subsequent spectrophotometric SPF analysis after extraction and homogenization of the sample in ethanol (96%, *v/v*). The additional 10% compensated for material losses during recovery from the tape. This was necessary to obtain a sufficient amount of irradiated sample for reliable SPF determination. A thinner layer could have been achieved by spreading the formulation over a larger area. However, this would have increased sample losses and reduced the accuracy and reproducibility of the measurements.

The results demonstrate that the type of investigated antioxidant significantly influences both the initial SPF and its preservation after UV exposure. The incorporation of antioxidants led to an increase in SPF compared to the reference formulation (F1), with the most pronounced effect observed for ferulic acid (F4). Peres et al. (2018) [14] demonstrate that ferulic acid not only improves formulation stability but also enhances the photoprotective efficacy of sunscreen systems. When combined with UV filters, ferulic acid exhibits a synergistic effect, significantly increasing SPF and UVA protection, thereby acting as a photoprotective booster and improving overall sunscreen performance.

A moderate increase was observed for vitamin C (F2) and carrot macerate (F3). These differences can be attributed to the distinct chemical structures and antioxidant mechanisms of the tested compounds.

The antioxidant activity of vitamin C is based on its chemical structure and its ability to act as an electron donor. It neutralizes free radicals and ROS (reactive oxygen species) through electron transfer, becoming oxidized to the relatively stable ascorbyl (semidehydroascorbate) radical. Further oxidation of this intermediate leads to the formation of dehydroascorbic acid [11,12]. One possible explanation for the decrease in SPF after 120 min of UV exposure is the gradual oxidation of vitamin C. As ascorbic acid undergoes oxidative transformations during its antioxidant action, its ability to neutralize free radicals may become limited under prolonged UV irradiation, which could be reflected in a reduction in SPF [40].

In the case of formulation F3 containing carrot macerate, the moderate increase in SPF may be associated with the presence of carotenoids. Their antioxidant activity is related to their ability to scavenge singlet oxygen and peroxy radicals generated during UV irradiation [41]. Through these mechanisms, carotenoids may contribute to the improvement of the photoprotective properties of the formulation, which may be reflected in a moderate increase in SPF.

However, when evaluating photostability, a different trend emerged. Although the reference sample without antioxidants (F1) showed the smallest relative decrease in SPF after UV irradiation, this formulation still exhibited lower absolute SPF values compared to F4. Importantly, the formulation containing ferulic acid maintained the highest SPF after 120 min of UV exposure, indicating superior long-term protective efficacy. In contrast, vitamin C showed the greatest reduction in SPF, which may be related to its susceptibility to photodegradation and limited stability under UV conditions.

The observed behavior highlights the dual role of antioxidants in sunscreen formulations. While some antioxidants can initially boost SPF, their effectiveness in stabilizing UV filters under prolonged irradiation varies significantly. Ferulic acid appears particularly ef-

fective due to its known ability to absorb UV radiation and stabilize reactive intermediates, thus preventing degradation of UV filters.

Antioxidants incorporated into sunscreens may contribute not only to SPF enhancement but also to biological photoprotection by reducing oxidative stress and inhibiting ROS-mediated cellular damage. Several antioxidants have been shown to suppress inflammatory pathways, decrease matrix metalloproteinase activation, and limit collagen degradation associated with photoaging [5].

Although vitamin C increased the initial SPF value, the formulation containing stabilized vitamin C showed the greatest decrease in SPF after UV irradiation. This finding agrees with previous reports describing the limited photostability and oxidative instability of vitamin C and some of its derivatives in topical formulations, particularly under prolonged UV exposure [42]. The improved SPF retention observed in formulation F4 may indicate a stabilizing effect of ferulic acid on the UV filters used in the formulation. Previous studies have demonstrated that ferulic acid can act as a UV absorber and radical scavenger [43], thereby limiting photooxidative degradation of sunscreen filters. Mancuso et al. (2021) [44] found that after absorbing UV light, ferulic acid forms a stable phenoxy radical, enabling it to terminate free radical chain reactions. Freire et al. (2023) [45] report that ferulic acid helped preserve avobenzone and octyl methoxycinnamate during thermal/photo stress. This preservation is directly linked to reduced degradation of filters under UV exposure. Lu et al. (2025) [46] describe enhanced photostability and UV resistance in a supramolecular system with avobenzone and ferulic acid.

Adding ferulic acid to a topical formulation containing 15% L-ascorbic acid and 1% α -tocopherol increased the stability of both vitamins and enhanced photoprotection against simulated sunlight, raising protection from about fourfold to roughly eightfold, as shown by reductions in erythema and sunburn cell formation [47]. Queiroz et al. (2026) [48] confirmed the sun protection effect of ferulic acid alone, demonstrating that nanoemulsion-based formulations with low concentrations of encapsulated ferulic acid (0.2%, 0.3%, and 0.5%) exhibited progressively increasing SPF values (2.28, 2.70, and 2.96, respectively) in a concentration-dependent manner.

Carrot macerate also contains phenolic acids, including ferulic acid, as minor constituents [49], but its predominant antioxidants are carotenoids, especially β -carotene, and lipophilic antioxidants such as tocopherols [50]. Regarding its SPF-boosting effect, its performance is comparable to that of vitamin C in formulation F2. However, a key advantage is its superior stability, as it is less prone to degradation than vitamin C. On the downside, it imparts a slightly orange tint to the cream, which may not always be acceptable to consumers. The relatively small SPF enhancement observed for carrot macerate may be related to the lower concentration or reduced availability of active antioxidant constituents compared to purified antioxidant compounds such as ferulic acid.

The use of vitamin C as a stabilizer and antioxidant in the formulation is further limited by its pronounced instability, an increase in the formulation's pH, and a significant reduction in SPF following 120 min of UV exposure. However, if vitamin C is intended to be used as an active ingredient, its stability within the product could be improved through encapsulation in an appropriate carrier system. In general, nanoparticle-based formulations can be engineered to enhance targeted antioxidant delivery and improve retention within the skin [28].

The physical characterization further supports the suitability of the developed formulations. All samples were identified as oil-in-water emulsions, which are generally preferred for dermal application due to their favorable sensory and skin compatibility properties.

The pH values of most formulations were within the physiologically acceptable range. Formulations containing vitamin C slightly deviate from the recommended pH range

for dermal products. Although ascorbic acid is inherently acidic and would typically be expected to lower the pH, this effect can be influenced by the overall composition of the formulation and interactions of vitamin C with other ingredients. Ascorbic acid tends to degrade through oxidation, which can alter pH behavior over time. Despite exhibiting a somewhat higher pH compared to the other samples, no signs of irritation were reported by volunteers evaluating the sensory attributes of formulation F2 containing vitamin C.

Stability testing revealed that formulations containing ferulic acid and vitamin C demonstrated improved resistance to phase separation under stress conditions (centrifugation and freeze–thaw cycles), whereas formulations F1 and F3 were less stable. This suggests that certain antioxidants may also contribute to emulsion stability, likely by influencing the interfacial properties of the system.

Rheological analysis confirmed thixotropic behavior of all formulations, which is desirable for topical products, as it ensures good spreadability during application and structural recovery afterward. Thixotropy is a reversible rheological phenomenon typical of non-Newtonian fluids, characterized by a decrease in apparent viscosity under increasing shear stress, which disrupts the internal structure of the system. Once the shear stress is reduced, the material gradually returns to its original viscosity as its internal structure is rebuilt. The hysteresis loop offers qualitative insight into the extent of structural breakdown and subsequent recovery during the cycle of applied stress and relaxation [51]. As evident in Figures 3 and 4, the addition of the antioxidant was reflected in a change not only in apparent viscosity but also in HLA. This differed only slightly between formulations F1 and F3 ($73.02 \pm 0.04 \text{ kPa}\cdot\text{s}^{-1}$ and $71.74 \pm 0.04 \text{ kPa}\cdot\text{s}^{-1}$). However, statistical analysis confirmed that the difference was significant. In contrast, stabilized vitamin C and ferulic acid markedly increased HLA, with vitamin C causing an approximately 2.5-fold increase. From a technological perspective, this behavior may influence the product's spreadability and sensory properties during application. Generally, the observed thixotropic behavior is advantageous for topical sunscreen formulations, as apparent viscosity decreases during spreading, facilitating application onto the skin, while structural recovery after application helps maintain a uniform protective film.

Sensory evaluation further indicated that, among the two tested formulations (F2 and F4), the formulation containing ferulic acid (F4) was preferred by the volunteers, achieving the highest scores across most evaluated parameters.

Current cosmetic research increasingly focuses on multifunctional ingredients capable of simultaneously enhancing SPF, improving photostability, reducing oxidative stress, and providing favorable sensory properties [11]. Ferulic acid appears to fulfill several of these requirements. Despite these promising findings, several limitations of the present study should be mentioned. First, SPF values were determined using an *in vitro* spectrophotometric method rather than an *in vivo* approach. While *in vitro* methods are widely accepted for preliminary formulation screening and comparative studies, they cannot fully reproduce the complexity of sunscreen performance under real-life conditions on human skin. Second, photostability testing was conducted using Transpore™ tape instead of the PMMA plates more commonly employed in standardized photostability studies, which may limit direct comparison with data from other investigations. Furthermore, the study evaluated only a relatively small number of antioxidants and employed comparatively short irradiation times. These limitations highlight the need for future research.

5. Conclusions

In conclusion, the present study provides preliminary evidence that the incorporation of antioxidants may influence the SPF and photostability of sunscreen formulations. Within the limitations of the applied methodology, formulations containing ferulic acid exhibited

the highest SPF values and maintained a greater degree of photoprotective performance following UV exposure compared with the other tested antioxidants. In contrast, vitamin C and carrot macerate showed limited moderate effects, while vitamin C also appeared to exhibit lower photostability. These findings suggest that antioxidants may differ in their contribution to sunscreen performance and should be selected carefully during formulation development. The tested formulations displayed acceptable physicochemical characteristics, including suitable pH, rheological properties, and short-term stability, while sensory evaluation indicated generally good user acceptability, particularly for the formulation containing ferulic acid. Overall, the results suggest that ferulic acid may be a promising multifunctional ingredient for sunscreen formulations due to its antioxidant properties and its potential to enhance and maintain UV protection. However, further studies employing additional experimental approaches and broader evaluation methods are needed to confirm these observations and establish their practical significance.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/cosmetics13040186/s1>, Table S1: Measured Absorbances and Corresponding SPF Value for Formulation F1; Table S2: Measured Absorbances and Corresponding SPF Value for Formulation F2; Table S3: Measured Absorbances and Corresponding SPF Value for Formulation F3; Table S4: Measured Absorbances and Corresponding SPF Value for Formulation F4; Table S5: Sensory Evaluation Questionnaire; provided to 12 volunteers to facilitate consistent and accurate sensory assessment of the tested attributes of the prepared sunscreen formulations F2 and F4.

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Informed Consent Statement: Informed consent was obtained from all subjects involved in the study.

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Abbreviations

Abs	Absorbance
CF	Correction Factor
EE	Erythema Effect
HLA	Hysteresis Loop Area

I	Solar Intensity
<i>o/w</i>	Oil-in-Water
PMMA	Polymethyl methacrylate
SD	Standard Deviation
SPF	Sun Protection Factor
UV	Ultraviolet

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