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Photostability Study of Avobenzone in Commercial Sunscreen SPF 45 with Additional SolaStay® Quencher Upon Sun Exposure

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ABSTRACT

Avobenzone combined with various concentrations of Ethylhexyl Methoxycrylene (SolaStay® S1 quenchers) aims to see photostability studies. So that in this study tested the effect of SolaStay® S1 quenchers, SolaStay® quencher concentration, and the effect of irradiation time with sunlight on the photostability of avobenzone in commercial sunscreen formulations SPF 45. As well as determining the best SPF 45 commercial sunscreen formulations with the addition of Quenchers. The method used in this study is the spectroscopic method which uses two UV-VIS and HPLC spectrophotometer analyser. Based on the research data obtained in the presence of a quencher showed good photostability to avobenzone at certain quencher concentrations. Compared to F0, the effect of the addition of the quencher was very good, as seen from the relatively low decrease in the photodegradation rate compared to without the addition of the quencher. Viewed from the overall results, FSOL1 with the addition of 3% SolaStay® is the best SPF 45 sunscreen formulation of all formulations. This is seen from the small photodegradation results every time and the effect of skin effects was low.

Keywords: Avobenzone, Quencher, Photostability, Photodegradation, SolaStay®, Sunscreen

INTRODUCTION

The formulation of a sunscreen with high UV protection is an effective strategy to prevent the damaging effects of UV radiation to ensure optimal photoprotection. UV filters provide efficient protection against UVA and UVB radiation, are heat resistant, easy to photograph, user-friendly, cost-effective, waterproof, and non-toxic (1). The high capacity of the UV filter to absorb UV must remain stable for the entire period of sun exposure. The high capacity of the UV filter to absorb UV must remain stable for the entire period of sun exposure. Photostability is one of the important requirements of UV filters for sunscreens. Most commercially available sunscreen compounds exhibit photoreactions leading to the formation of hazardous products. Photostability refers to the ability of a molecule to remain intact under irradiation. Photostability is potentially an issue with all UV filters (2). Because if the UV filter is not stable, it will experience photodegradation over time (3). The occurrence of photodegradation reactions can cause harmful effects on skin cells.

One of the basic requirements regarding UV filters is photostability. In practice, when exposed to UV radiation, most UV filters undergo changes and photodegradation. One of the UV filters that are most susceptible to this kind of reaction is Butyl methoxydibenzoylmethane, better known as avobenzone, so it can be said that avobenzone is photo unstable. Other UV filters that are known to be not photostable are Octyl methoxycinnamate or octinoxate when irradiated with UV light they turn into cis-isomers which are significantly less active (4). Avobenzone is one of the best UVA UV filters (4), in the form of white to yellowish crystalline powder, which smells because it has an aromatic ring and is insoluble in water. The molecular weight of avobenzone is 310.4 g/mol with the molecular formula $C_{20}H_{22}O_3$ (5). The structure of the avobenzone is shown in figure 1.

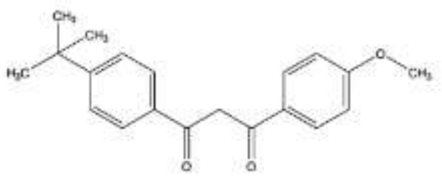


Fig. 1 The Structure of Avobenzone.

Avobenzone when irradiated will experience an increase in energy from the ground state to the excited state. It then disintegrates after reaching an excited state producing singlet oxygen. This singlet oxygen will react well with avobenzone. As a result, several products are formed that can cause irritation and allergic properties (6). The minimum avobenzone use concentration has been set at 2% and a maximum of 3% (7). avobenzone can be photostable if combined with other UV filters, modifying the structure of avobenzone and adding stabilizers or quenchers, and adding antioxidant compounds(8). So, with the study of the addition of quencher researchers want Avobenzone to be able to last longer and not be damaged by photons so that those who experience excitation are quenchant material itself.

SolaStay® S1 (Ethylhexyl methoxycrylene) has a molecular weight of 391.5 g/mol with the molecular formula $C_{25}H_{29}NO_3$, which is a viscous yellow liquid that is insoluble in water, designed for use in skincare and sun care products. SolaStay® S1 is included in the photostabilizer, which is a chemical that can take excited state energy from other molecules before these molecules change shape into new compounds (9). Methoxycrylene or SolaStay® was introduced by the Hallstar Company where this type of quencher can transfer the energy stored in a singlet excited state. Therefore a molecule that is in the singlet excited state does not even go to the triplet excited state but returns to the ground state and goes back to work (10).

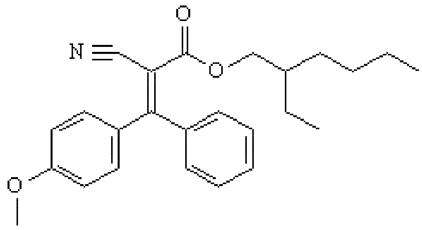


Fig. 2 The structure of Ethylhexyl Methoxycrylene (SolaStay® S1)

Before testing the photostability of each combination of sunscreens, it is necessary to review the effect of the physical and chemical properties of the test sample to see the quality of the formulation which includes homogeneity, pH testing, viscosity, and cream type testing (11). Homogeneity measurements were carried out based on visual observations using a quartz plate, pH and Viscosity testing were tested with a digital pH meter with electrodes that could be immersed directly into the emulsion and a tool for measuring viscosity used a Brookfield viscometer. After seeing a review of the physical and chemical properties, measurements were made using a UV-VIS spectrophotometer (12) and supported by other measurement variables such as HPLC..

MATERIALS & METHODS

Reagents and samples

A commercially available sunscreen SPF 45 currently available in the Indonesian market was investigated but with a photoactive substance i.e., avobenzone only, as shown by the product material on the packaging. used quencher SolaStay® S1 as an additional ingredient (stabilizer) in an SPF 45 sunscreen formulation, ethanol (96%), Aquadest,

Instrumentation

Sunscreen photostability as measured by a Spectrophotometer (UV/Vis T70 UV/Spectrophotometer and High-Performance Liquid Chromatography (HPLC; CBM-20A SHIMADZU), quartz plate as reactor (25 mm in diameter and 5 mm in thickness).

Sample Preparation

The sample consisted of 4 pieces, namely commercial sunscreen SPF 45 without added SolaStay® S1 quencher, sunscreen SPF 45 added with 3 variations of SolaStay® quencher concentration, which can be seen in Table 1 below:

Table 1: Sample Name and Composition

No.	Code	Sunscreens SPF 45	Quencher (%)
			SolaStay® S1
1.	F0	✓	-
2.	FSOL1	✓	3
3.	FSOL2	✓	4
4.	FSOL3	✓	5

Emulsion Stability

Stability testing includes homogeneity, pH, viscosity, and cream-type testing of the SPF 45 sunscreen formulation. For homogeneity, each sample was weighed as much as 5 mg and spotted onto glass preparations. Then attached with a second quartz glass. After that, the top quartz glass is pulled horizontally slowly. For pH testing, SPF 45 sunscreen formulation using a digital pH meter 15 g of SPF 45 sunscreen formulation is weighed and put into a 50 mL beaker glass. Then the electrode is dipped into it, and wait until the number on the screen stabilizes and the pH value of each formulation is recorded. For viscosity, 25 g of SPF 45 sunscreen formulation was weighed and put into a 50 mL beaker. The appropriate spindle is attached to the tool and then dipped in each SPF 45 sunscreen formulation. The results of the viscosity of the cream can be seen from the number indicated by the stable needle (in cP units) and the viscosity value of each formulation is recorded. For cream type Testing the type of cream is done by staining method using methylene blue. 0.0050 mg of SPF 45 sunscreen formulation was weighed and then a drop of methylene blue was observed and the color distribution was observed.

Sunscreen Irradiation for Photostability

The SPF 45 sunscreen formulation sample (see Table 1) was weighed 0.005 g and applied to a quartz plate and then attached to a second quartz plate (quartz plate 25 mm in diameter and 5 mm thick) (13). Then place under the sun in the optimal intensity range from 11.00 to 14.00 (UTC+07:00) based on observations of the intensity of sunlight in Padang city. Samples of the SPF 45 sunscreen formulation were irradiated in sunny weather with light intensity measured in the range of 90,000–20,000 lux using a lux meter. Variations in the time the samples were irradiated by direct sunlight were 30, 60, 90, and 120 min.

Photostability Testing Using Spectrophotometer UV-VIS

Samples that had been irradiated by the sun for 0, 30, 60, 90, and 120 min were dissolved in a solution of ethanol and distilled water (40:60) in a 50 mL volumetric flask. Then stirred using a magnetic stirrer for 30 min, while stirring the volumetric flask containing the sample was wrapped with aluminium foil so as not to be exposed to sunlight. After that, it was measured using a UV-Vis spectrophotometer and looked for the percent photodegradation of avobenzone before and after being irradiated by sunlight at a wavelength of 359 nm with the formula:

$$\text{Photodegradation of Avobenzone (\%)} = \frac{\text{Abs before} - \text{Abs after}}{\text{Abs before}} \times 100\% \quad (1)$$

Photostability Testing Using *High-Performance Liquid Chromatography* (HPLC)

Samples that had been irradiated by sunlight for 0, 30, 60, 90, and 120 min were dissolved in a solution of methanol and distilled water (88:12) in a 50 mL volumetric flask. Then it was stirred using a magnetic stirrer for 30 min, during which the volumetric flask containing the sample was mixed was covered with aluminum foil so that it was not exposed to sunlight using the mobile phases of methanol and distilled water (88:12) and a flow rate of 1 mL/min. Then the standard solution and sample were injected sequentially, then the chromatogram results were obtained in the form of retention time peaks. The percentage of avobenzone present in the samples before and after irradiation is calculated using the formula:

$$\text{Photodegradation of Avobenzone}(\%) = \frac{\text{Sample area before} - \text{Sample area after}}{\text{Sample area before}} \times 100\% \quad (2)$$

RESULT AND DISCUSSION

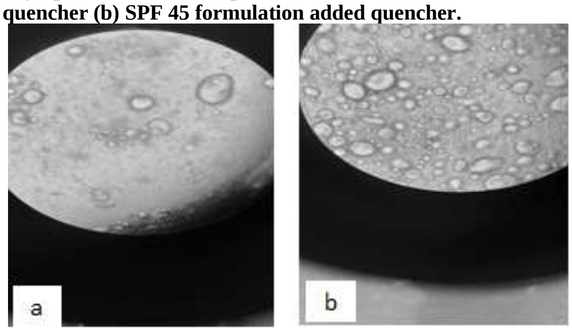
Emulsion Stability

Table 2: Sample Name and Emulsion Stability

No	Code Formulation	Homogeneity	pH	Viscosity (cps)	Cream Type
1.	F0	+ Homogeneity	5.10	10.760	O/W
2.	FSOL1	+ Homogeneity	5.09	14.580	O/W
3.	FSOL2	+ Homogeneity	5.13	14.900	O/W
4.	FSOL3	+ Homogeneity	5.10	26.721	O/W

The results showed that all SPF 45 sunscreen formulations showed good results, namely the cream was spread evenly and there were no lumps of particles that could be observed visually. This indicates that the SPF 45 sunscreen formulation is feasible to use and to test its photostability. Homogeneity in the emulsion system is influenced by the mixing technique or method used and the tools used in the emulsion manufacturing process. For further testing, observations were made using a microscope using a magnification of 40x in Figure 3 showing that the formulation looks homogeneous because the particle distribution is even between the oil phase and the water phase.

Fig 3: The homogeneity of the SPF 45 formulation using an Olympus CX31 microscope (a) SPF 45 formulation without quencher (b) SPF 45 formulation added quencher.



For pH and Viscosity result shows, the pH value of all SPF 45 sunscreen formulations is in the pH range that meets the pH criteria in cosmetics and has good emulsion stability when compared to F0. The pH value of cosmetics for the skin is in the range of 4–7 (14). FSOL2 can increase the pH value of F0, which is 5.13. The high concentration of SPF 45 sunscreen formulation did not have a significant effect on increasing the pH value and Viscosity relates to consistency. The viscosity must be able to make the preparation easy to spread and can stick to the skin. Preparations with a higher consistency will affect the application they are used in, where they will be difficult to flow or viscous so that the viscosity value is large (15). the viscosity standard for good sunscreen preparations is in the range of 2000–50,000 cps based on the result; it can be seen that the viscosity value of the formula (F0) before adding the quencher is 10,760 cp. The addition of a quencher can increase the viscosity value. It is said that the thicker an emulsion, the more stability will increase. Likewise, the results of this study show that with the addition of a quencher, the SPF 45 sunscreen formulation shows good formulation stability with an increase in the viscosity value.

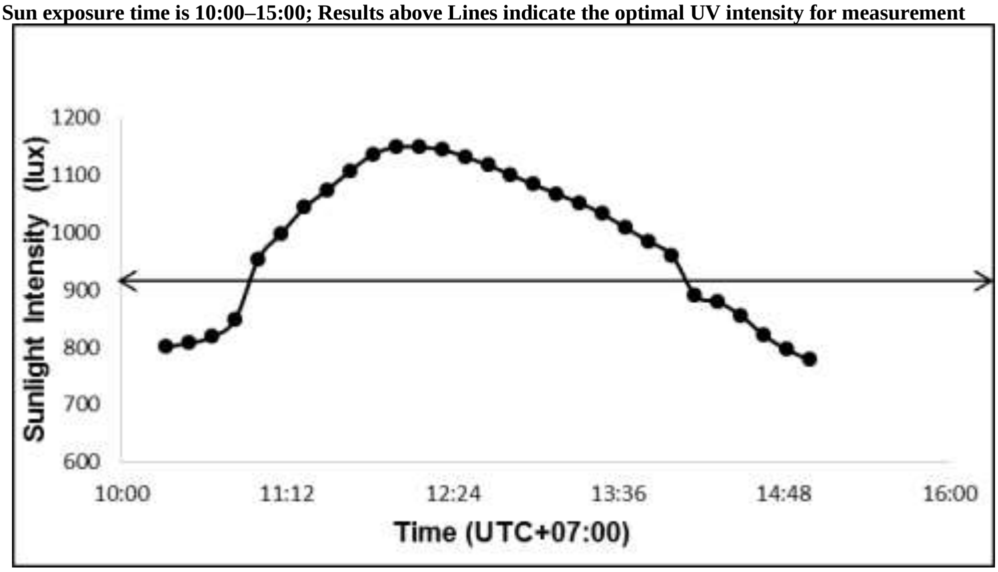
The cream type of SPF 45 sunscreen formulations shows the formulation has an O/W type where after adding methylene blue all samples form a soluble phase in the SPF 45 sunscreen formulation. Methylene blue (MB) functions to determine the water/oil soluble phase. Because as is known methylene blue (MB) dissolves in water after adding methylene blue (MB) it forms a soluble phase, meaning that the type is O/W where the dispersing phase is water. Water washable (O/W) cream, intended for cosmetic and aesthetic use. O/W type creams can be used on large areas of skin because the oil portion is smaller. When used on the skin, the oil phase will evaporate and can increase the concentration of water-soluble drugs in the attached/remaining film layers besides being easily washed off (16).

Sunscreen Irradiation for Photostability

Solar energy is the most promising choice because the source is not limited, so it is chosen as a source of UV light, so UV radiation observations need to be carried out which aim to find out the maximum intensity which is carried out in one place. The results of the intensity of sunlight show that the sample can be irradiated when the luxmeter shows the optimal intensity, namely from 90,000 lux-110,000 lux from 11.00 to 14.00 (UTC+07:00) (Fig. 4). In connection with research before which shows the relationship between light intensity measured from 10.00–14.00, showing the maximum value of light intensity resulting from that hour with light intensity in the range of 96,600-104,900 lux. The maximum light intensity shown on the clock shows the number of photons carried by sunlight.

Quartz plate used in research as a UV reactor. The UV reactor functions to transmit UV radiation to the sample. This is because the quartz plate has characteristics, one of which is high purity and can maximize heat absorption from UV rays due to high thermal resistance. The quartz plate is made with a diameter of 25 mm and a thickness of 5 mm.

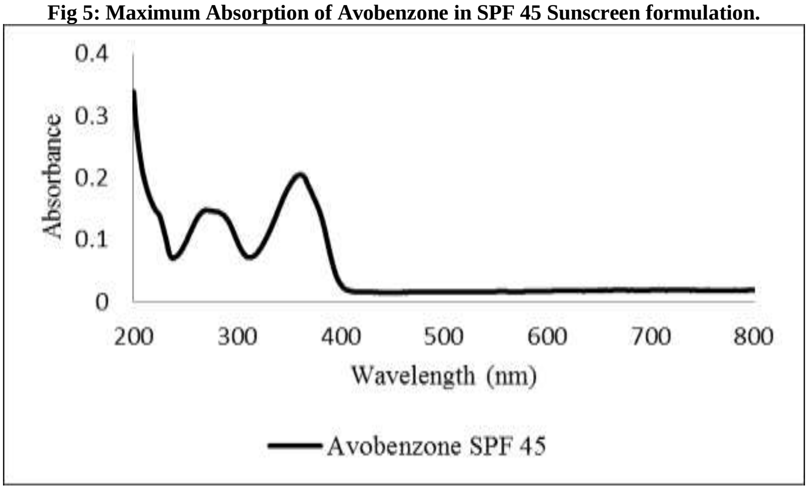
Fig 4: A histogram that displays the intensity of sunlight using a lux meter for trial days in August 2022 in Padang, West Sumatra, Indonesia. Sun exposure time is 10:00–15:00; Results above Lines indicate the optimal UV intensity for measurement



Photostability Testing Using Spectrophotometer UV-VIS

Variations in the time the samples were exposed to direct sunlight were 0, 30, 60, 90, and 120 min which were then measured with a UV-VIS spectrophotometer at a wavelength of 200 nm-800 nm. Determination of the percent degradation of avobenzone was calculated at a wavelength of 359 nm to see the photostability of avobenzone before and after being exposed to sunlight (17). This is because avobenzone has a maximum absorption at 359 nm (in Fig 5.)

Fig 5: Maximum Absorption of Avobenzone in SPF 45 Sunscreen formulation.



In the irradiated SPF 45 sunscreen formulation, the percent photodegradation on the base (without the addition of a quencher) was measured as a comparison to find out whether the SPF 45 sunscreen formulation with quencher could reduce the photodegradation of the formulation. The formulation is coded as F0 (without the addition of a quencher).

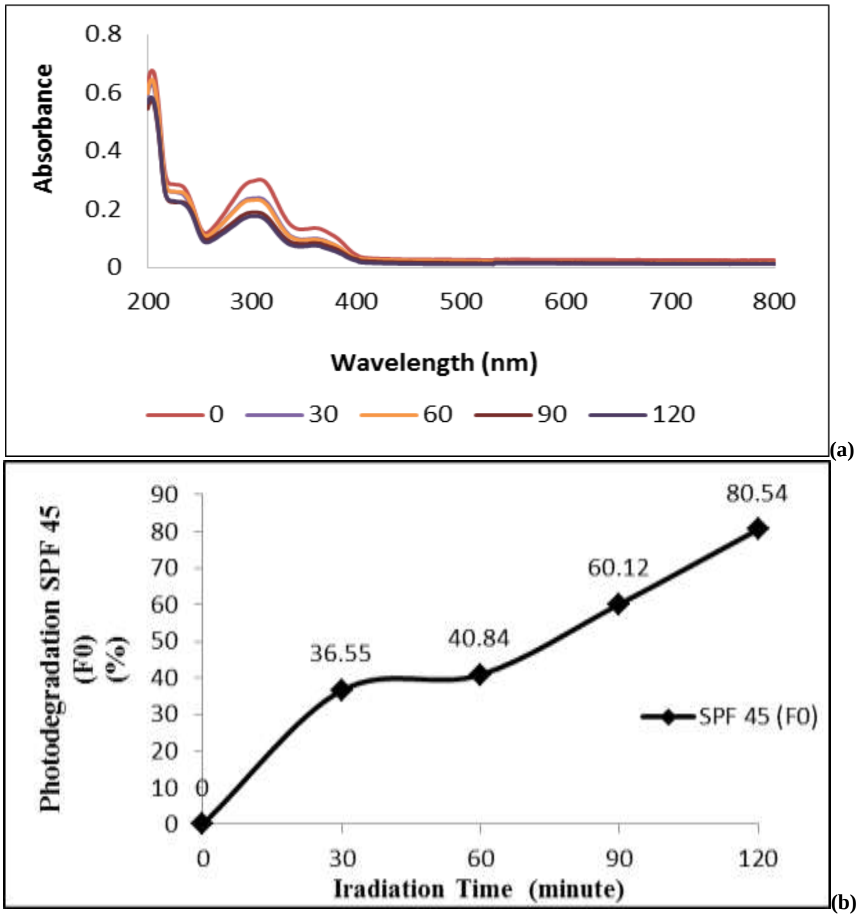
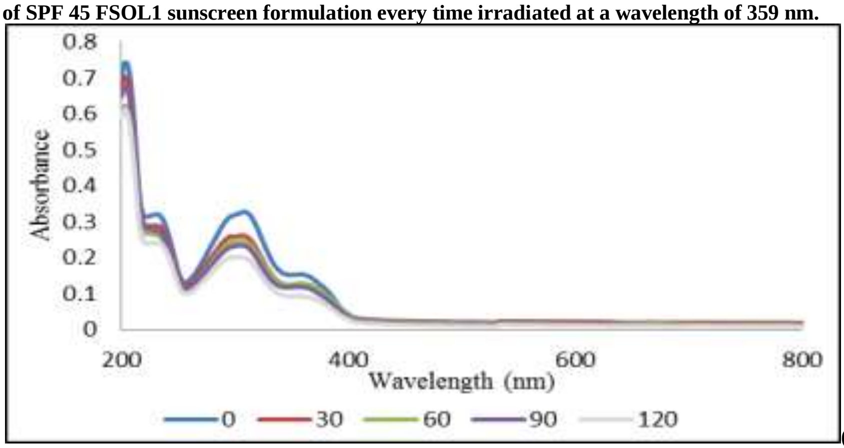


Fig 6: (a). Absorbance profile of SPF 45 F0 sunscreen formulation every time irradiated at a wavelength of 200-800 nm and (b). Photodegradation of SPF 45 F0 sunscreen formulation every time irradiated at a wavelength of 359 nm.

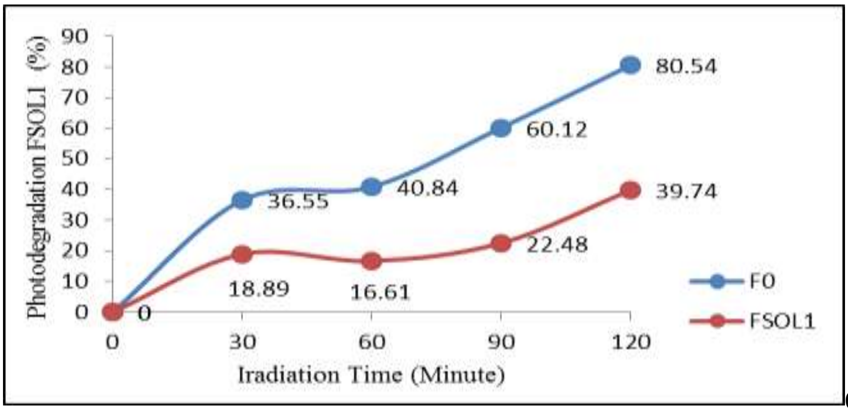
In Figure 6, it can be seen that the photodegradation of avobenzone in the SPF 45 (F0) sunscreen formulation is directly proportional to the irradiation time. The more the SPF 45 sunscreen formulation is irradiated, the greater the photodegradation, the highest percent of degradation is at 120 min reaching 80%. This is because avobenzone is unstable and easily damaged due to sunlight irradiation, so to reduce the damage to avobenzone, you can add a quencher, which is a photo stabilizer in sunscreen, which plays a role in absorbing excited energy back to its ground state. The quencher used in this study was SolaStay® S1. Where this combination is claimed to provide effective protection and can stabilize avobenzone by looking at its photodegradation and the quencher can not only absorb irradiation energy but also release energy as heat before it decays or reacts with the surrounding molecules (18).

Photodegradation of avobenzone with the addition of SolaStay® S1 with code FSOL1 can be seen in In Figure 7(b), is directly proportional to the irradiation time but at several points in time FSOL1 experiences a skin effect which is indicated by a decrease in the photodegradation value at 60 min caused by products that block light from entering the reactor wall which results in an inhomogeneous distribution of light in the reactor. FSOL1 has a high SPF 45 formulation photodegradation of 39.4% after irradiation in 120 min which when compared to F0 the high photodegradation after 120 min of irradiation is 80.54% so it can be said that the addition of quencher SolaStay® of 3% can stabilize avobenzone in the SPF formulation 45. The photodegradation value of the SPF 45 formulation in FSOL1 was generated successively for each time variation of 18.89%, 16.61%, 22.48%, and 39.4% which when compared with F0 photodegradation is produced successively each time variation is 36.55%; 40.84%; 60.12%; 80.54%.

Fig 7: (a). Absorbance profile of SPF 45 FSOL1 sunscreen formulation every time irradiated at a wavelength of 200-800 nm and (b). Photodegradation of SPF 45 FSOL1 sunscreen formulation every time irradiated at a wavelength of 359 nm.



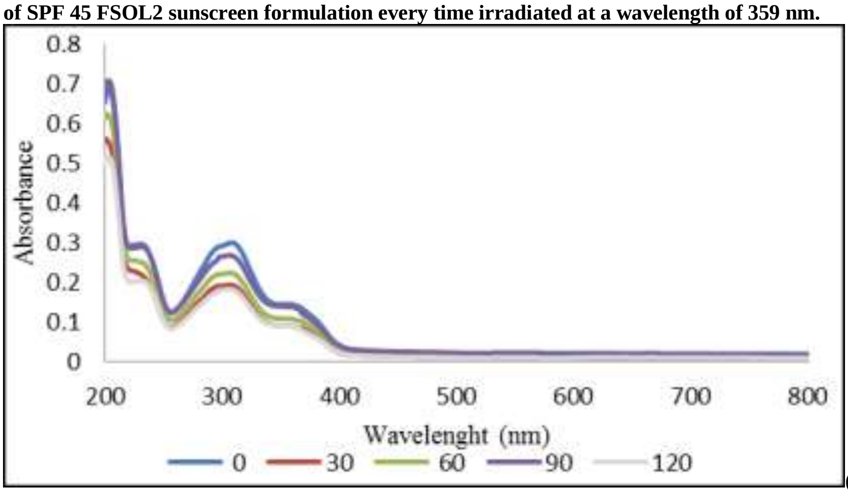
(a)



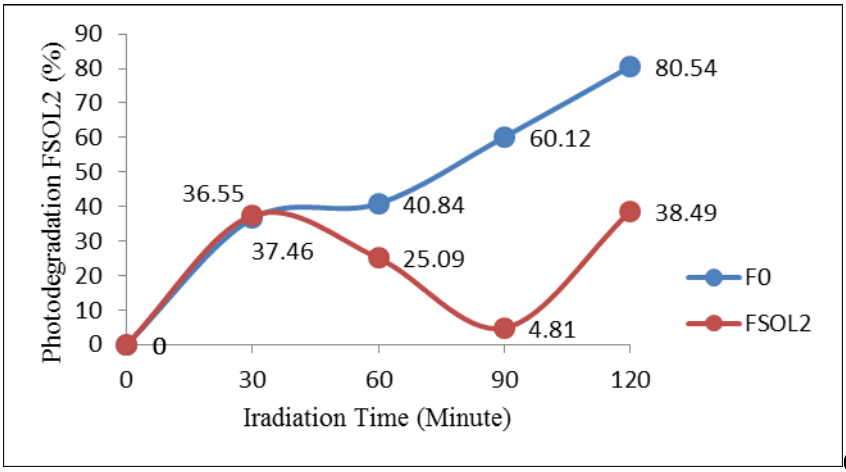
(b)

SolaStay® S1 is an avobenzone stabilizer that works in the singlet state because it has singlet energy below avobenzone which is 72.3 kcal/mol (18). Photodegradation of avobenzone with the addition of SolaStay® S1 with code FSOL2 can be seen in In Figure 8(b), FSOL2 has a high photodegradation of the SPF 45 formulation, namely 38.49% after irradiation in 120 min, which when compared to the high F0 photodegradation after 120 min of irradiation of 80.5%, so it can be said that the addition of quencher SolaStay® of 4% in the SPF 45 formulation is effective in stabilized avobenzone but seen in 30 min showed an increase in photodegradation, namely 37.46% from F0, which was 36.55% so that it can be said that SolaStay® of 4% was still not effective in reducing avobenzone photodegradation. Quencher SolaStay® is a recently discovered quencher and there are still very few commercial sunscreen products that use quencher SolaStay®. Several studies regarding the SolaStay® quencher only say that the combination of avobenzone and SolaStay® can act as a photostabilizer and make SolaStay® the main fotostabilizer in terms of energy levels (18).

Fig 8: (a). Absorbance profile of SPF 45 FSOL2 sunscreen formulation every time irradiated at a wavelength of 200-800 nm and (b). Photodegradation of SPF 45 FSOL2 sunscreen formulation every time irradiated at a wavelength of 359 nm.



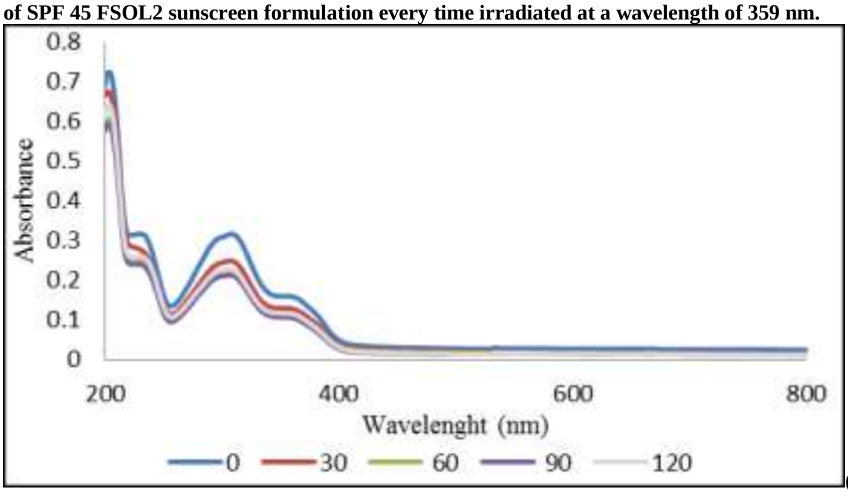
(a)



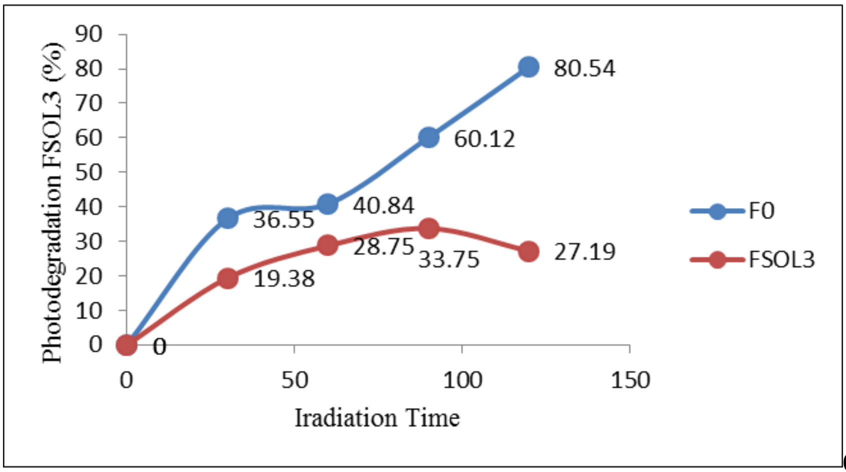
(b)

FSOL3 has a high SPF 45 formulation photodegradation of 33.75% after irradiation in 90 min which when compared to F0 the high photodegradation after 120 min of irradiation is 80.54% so it can be said that the addition of quencher SolaStay® of 5% can stabilize avobenzone in the SPF formulation 45. The photodegradation of avobenzone on FSOL3 is proportional to the length of time of irradiation except that at 120 min there is a slight decrease in the value of photodegradation due to the effect of skin effects which are the same as other SPF 45 FSOLs. FSOL1 and FSOL3 can be said to have the best performance in stabilizing avobenzone in the SPF 45 formulation. In the SPF 45 FSOL sunscreen formulation, the concentration series that experienced the smallest photodegradation was FSOL1 <FSOL3 < FSOL2. In research by (19) who examined the efficiency of quencher SolaStay® in stabilizing avobenzone, by measuring the fluorescence of avobenzone with the addition of SolaStay® with a concentration of 10 mmol it was proven that the role of a quencher could extinguish the excited state of avobenzone in $1.86/10^{-12}$ seconds thereby preventing unwanted chemical reactions and thereby contributing safely for photoprotection with stabilized Avobenzone. The effectiveness of adding 3% SolaStay® in the formulation was also carried out by (20), with the result that 3% SolaStay® can maintain 83.7% UVA absorbance meaning that as much as 83.7% is not degraded when compared to without the use of SolaStay® only 44.5% can maintain UVA absorbance.

Fig 9: (a). Absorbance profile of SPF 45 FSOL2 sunscreen formulation every time irradiated at a wavelength of 200-800 nm and (b). Photodegradation of SPF 45 FSOL2 sunscreen formulation every time irradiated at a wavelength of 359 nm.



(a)

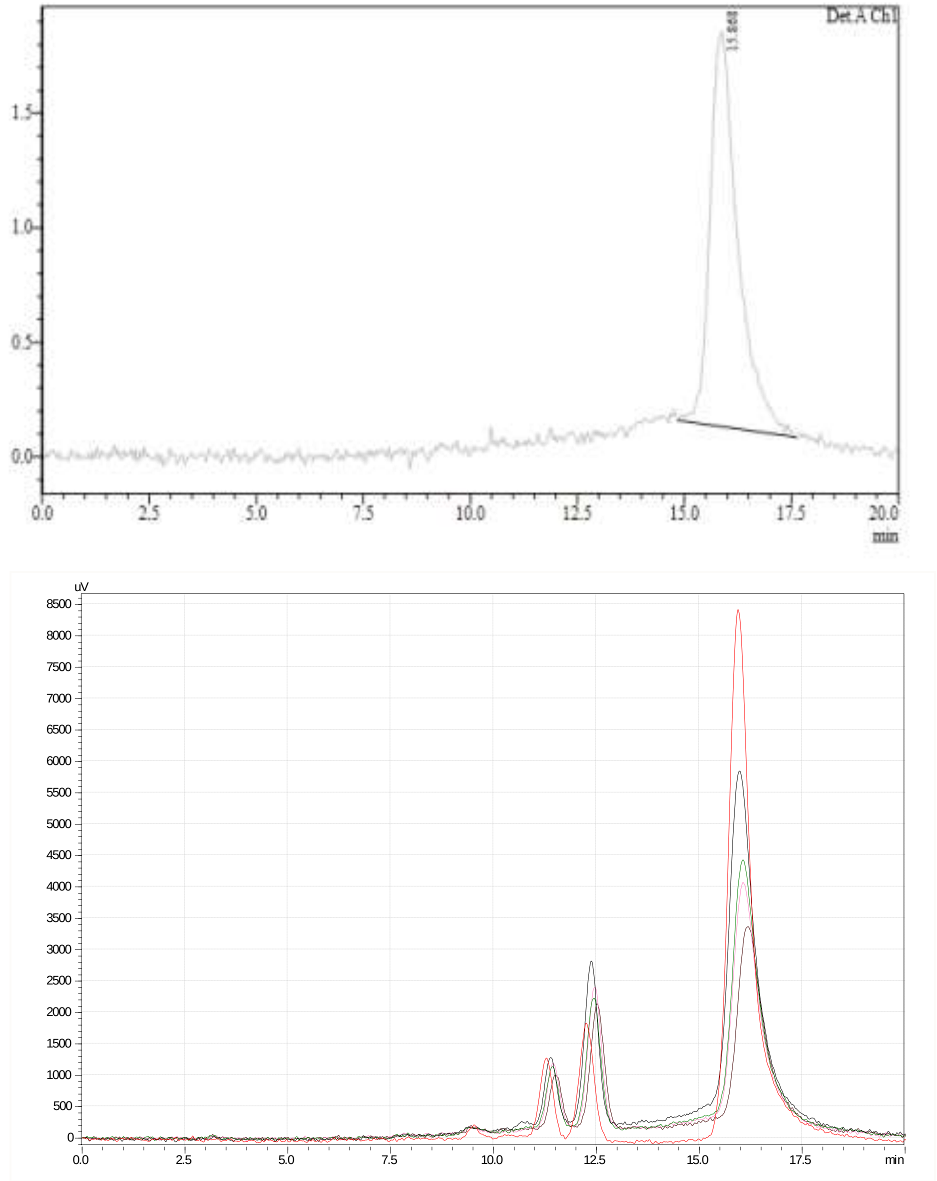


(b)

Photostability Testing Using *High-Performance Liquid Chromatography* (HPLC)

Determination of the wavelength according to UV-VIS spectrophotometry analysis and also from the validation of previous research methods obtained the maximum absorption of avobenzone at a wavelength of 359 nm. This is following the support of previous studies where avobenzone has maximum absorption in the wavelength range of 356 – 362 nm depending on the type of avobenzone used (17). The standard retention time of the avobenzone obtained is 15.868 min. Figure 10 shows peaks that are well separated from impurities and with a total retention time of more than 15 min which is following the standard. Figure 17 observes that avobenzone has an area that is directly proportional to the length of irradiation time, the longer the irradiation time, the more avobenzone will be degraded. Based on Fig. 10, in 120 min there was still 39% avobenzone left. This is different from the results using UV-VIS spectrophotometry analysis which still has approximately 60% avobenzone remaining. The difference in the remaining avobenzone produced is different because the HPLC method is the best in measuring photostability which indicates a compound specifically and with high accuracy using the UV-VIS spectrophotometry method alone is not specific enough in determining the photodegradation of a compound to be investigated.

Fig 10: (a). chromatogram profile of pure avobenzone in SPF 45 sunscreen and (b). chromatogram of the effect of irradiation time on HPLC chromatogram of sunscreen formulation SPF 45 (FSOL1) 0, 30, 60, 90, and 120 min.



Up to 120 min of irradiation, all SPF 45 formulations still showed absorption. Judging from the photodegradation value, there was still approximately 40-50% left that was not photodegraded. Photodegradation events will encourage changes in the molecular structure of the photosensitizer to its derivatives when irradiated continuously for a long time with high light intensity, causing changes in the chemical structure of the UV filter (21). In the sense that we cannot avoid photodegradation but we can slow down the rate of photodegradation by using a photo stabilizer in the SPF 45 formulation.

CONCLUSION

In the results of the emulsion stability review, all SPF 45 sunscreen formulations that had been added with SolaStay® S1 quencher had met the eligibility standards of formulations for marketing but could not prove an increase in photostability of avobenzone. After the addition of the quencher, it showed good photostability to avobenzone at certain concentrations of quencher. Viewed from the overall results, FSOL1 with the addition of 3% SolaStay® is the best SPF 45 sunscreen formulation of all formulations. Up to 120 min of irradiation, all SPF 45 formulations still showed absorption in the sense that we cannot avoid photodegradation but we can slow down the rate of photodegradation by using a photostabilizer in the SPF 45 formulation.

Declaration by Authors

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Conflict of Interest: The authors declare no conflict of interest.

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