

Determination of Sun Protection Factor (SPF) for Various Sunscreens by UV Spectrophotometry

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ABSTRACT

The Sun Protection Factor (SPF) research has been very beneficial in recent times. Various Pharmaceutical ingredients formulated as Sunscreen creams and lotions must show an effective potency against UV Radiation. In this study, we tried to find the correlation between SPF and absorbance by evaluating various pharmaceutical excipients approved by Indian Pharmacopeia. Through this study, we could prove the correlation between absorbance and transmittance used for the evaluation of sunscreen SPF and the blockage of UV radiation. Mansur equation was used for the investigation and calculation of the samples, and the label claimed variation came due to the different amount of components present in the samples. Sunscreen with different SPF values (15, 20, 24, 30, 50, and 60) study results reveal a direct relationship between sunscreen absorption and SPF. This spectrophotometric method is simple, rapid, robust, sensitive, selective, and appropriate for the in vitro determination of SPF values of various sunscreens.

KEYWORDS

SPF; UV-Spectroscopy; Ethylhexyl Palmitate; Isopropyl Alcohol

INTRODUCTION

The most relevant indicator for the protection of skin from sunscreen is about to know as the sun protection factor (SPF). SPF values are directly proportional to UV radiation, meaning both SPF values and protection against sunburn are equally increased [1]. The properties of the substrate depend on its roughness, filter elution over the surface, pressure applied when spreading the product on the substrate, and the substantivity of the formula. The general idea is that sunscreen products should be able to protect the skin from ultraviolet (UV) radiation (290-400 nm) [2]. Long-term exposure to UVB (290-320 nm) and UVA (320-400 nm) can cause sunburn and skin cancer because UV is known to be the fastest cancer developer in humans. UV has many disadvantages, like decreased tissue repair functions and severe damage to DNA. People generally have 40% of skin cancer and 80% of basal skin cancers, 16% of squamous cell carcinomas, and 4% of melanomas. The skin's DNA can become damaged over time by UV radiation, solar radiation, or tanning, leading to mutations that result in 3.5 million skin cancer and around 11,500 fatalities in the US annually [3-6]. There are two skin cancers; one develops melanocytes and is known as melanoma, and the other develops in skin cells and is known as non-melanoma; it is the least common skin cancer [7-8]. Melanoma causes a high range of death in the US due to skin cancers. There are three types of UV: UVC, UVB, and UVA. Among these three, the UVC has the shortest wavelength, is the most dangerous, and is absorbed by the ozone layer [9-10]. The wavelength of UVC is about (200-280nm), UVB (280-320nm), and UVA (320-400nm). UVB radiation is not entirely absorbed by the ozone layer and is one reason for sunburn, and is responsible for chronic changes like immune suppression and photocarcinogenesis. The pre-maturation of skin is triggered by UVA, which penetrates deeper layers of the epidermis and dermis and promotes photoaging and photocarcinogenesis [11]. Sunscreen can be used for protection from both UV-A and UV-B radiation. The methodology accepted in evaluating SPF employs *in vivo* measurements in humans, considering all biological responses by the food and drug administration. Different instruments and substrates have been used as low-cost alternatives to determine the SPF *in vitro*. *In vitro* measurements are fast, practical, and economical but lack inaccuracy in SPF determination [12-13]. *In vitro* methods are applied by two processes, a. Measurement of absorption/transmission of UV radiation through the sample. b. Determined the absorption properties of the sample based on the spectrophotometric analysis of dilute solutions [14]. Several factors affect the determination of SPF values, such as different types of solvents used, sunscreens viscosity, different types of emulsion, cuvettes types, the interactions and effects of vehicle and other components, and the addition of other active ingredients, which can increase or decrease UV absorption of each sunscreen [15]. The stability of emulsions was determined during this study by measuring the pH and conductivity, and viscosity of emulsions. The objective of the proposed work was to determine the sun protection factor values for different formulations through UV spectrophotometry [16]. Many attempts have been attempted to develop *in vitro* SPF testing methods. Everyone has exposed to sunlight by electromagnetic spectra from the sun, UV radiation. It had the highest energy among the ground-reaching radiation. Generally, many of them are exposed to sunlight and UV radiation during the summer season in tropical areas [17-20].

Various analytical techniques have been reported for the determination and evaluation of sunscreens as well as SPF values by UV-spectroscopic, HPLC, and other methods [1-3, 5-6, 8, 11, 13-14].

The work aims to develop a new, easy, accurate, and reproducible method for determining SPF for various sunscreens and evaluating the relationship between sunscreen absorption and SPF. UV spectrophotometry quickly measured the amount of absorbing particles present in the sample, which help to calculate the label claimed variation by the Mansur equation.

MATERIALS AND METHODS

Materials

70% of isopropyl alcohol and methanol were bought from the local drug store. Sunscreens from different manufacturers with different SPF (15, 20, 24,30, 50, and 60) were purchased from the local market, Visakhapatnam. All analytical grade solvents were used. Membrane filters (0.45 μm) purchased from Sigma-Aldrich (India) were used for the filtration of samples. Ethanol (Merck) of analytical grade was used for the preparation of working standard and dilution sample solutions

Instruments

The instrument used was a Shimadzu 1800 UV spectrophotometer with 1cm matched quartz cells for all measurements. The software used was UV probe 4.2 series. A digital analytical balance (Mettler Toledo, India) and ultrasonic sonicator (Spectra Lab, India) were used in the study. The validated pipette of 2, 10 mL; volumetric flask of 10, 100 mL; beakers of 50, 100 mL were made up of borosil glass.

Method

Sample Preparation and determination of SPF

1.0 g of sunscreen samples were weighed, transferred to a 100 mL volumetric flask, diluted to volume with 70 % isopropyl alcohol, followed by ultrasonication for 10 min, and then filtered through cotton. Three trials were performed for each solution by rejecting the first 5.0 ml. Further working standard and constant dilution solutions were prepared for each product with 50 % methanolic solution. The absorption spectra of samples in solution were obtained in the range of 290 to 450 nm using a 1 cm quartz cell, and 50 % methanol as a blank. The absorption data were obtained in the range of 290 to 320, every 5 nm, and 3 determinations were made at each point, followed by the application of the Mansur equation.

Mansur equation was used for the determination of SPF of different formulations.

The equation was developed by Sayre (1989). the equation follows:

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

- EE – Erythema effect spectrum
- I – Solar intensity spectrum
- Abs – Absorbance of sunscreen product
- CF – Correction Factor (10)
- λ – Wavelength (nm)

The maximum absorbance was recorded and then corrected to the same mass of 1.0 g. The corrected absorbance was calculated by the following formula:

$$A = \text{Measured } A \times 1.0 \frac{g}{\text{Mass of sample}}$$

The mean of corrected absorbance of the three measurements was calculated for accuracy and consistency.

The transmittance (T) of a sunscreen solution was calculated from the absorbance (A) based on the following equation:

$$A = -\log(T)$$

UV Visible Spectroscopy

Shimadzu 1800 Double beam UV spectrophotometer with 1cm quartz cuvette was used for the measurement of absorbance. Two cuvettes were taken and should be rinsed with distilled three times one cuvette was filled with a sunscreen sample and the cuvette should be cleaned with tissue paper to avoid fingerprints on cuvettes. Another cuvette filled with methanol as blank. The UV-Visible spectroscopy was set to scan from 220 to 400 and the wavelength produced should be noted after the scan.

RESULTS AND DISCUSSION

To examine a relationship between the absorbance and transmittance with the SPF value, sunscreens with various SPF values of 15, 20, 24, 30, 50, and 60 are performed by UV-Spectroscopy. According to Beer's rule ($A = \epsilon bc$), the absorbance of a material typically depends on the concentrations of the tested solution. Figure 1 displays the adjusted absorbance at the maximum of the sunscreens vs. SPF. The studies demonstrate a clear correlation between sunscreen SPF and rectified sunscreen absorption.

Wavelength(nm)	EE × I (normalized)
290	0.0150
295	0.0817
300	0.2874
305	0.3278
310	0.1864
315	0.0837
320	0.0180

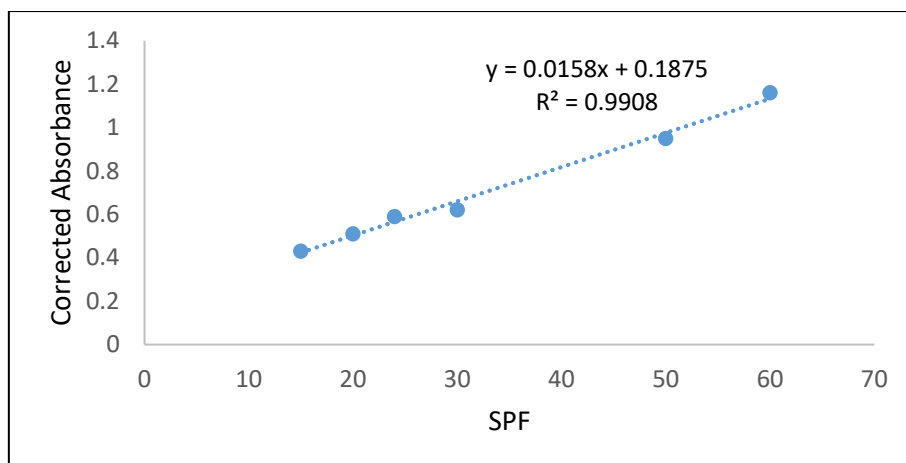


Figure 1: Absorbance of sunscreens at maximum absorbance (λ_{max}) versus SPF.

Table 2 is different suncream samples those are containing several different types of active ingredients, which are evaluated by UV spectrophotometer and calculated by the Mansur mathematical equation, final amount of percentage present is calculated by comparison of the labeled claimed SPF and Calculated SPF.

Sample	Active Ingredients	Labeled SPF	Calculated SPF	(Found SPF/ Labeled SPF)*100
A	Butylmethylpropional, Alpha isomethyl ionone, geraniol, Benzyl alcohol, titanium dioxide.	15	13.03 \pm 0.05	86.86 %
B	Light liquid Paraffin, octyl methoxy cinnamate.	20	20.50 \pm 0.06	102.5 %
C	Dimethicone, Isononyl Isononanoate, Carbomer, Dimethiconol, Titanium dioxide	24	22.6 \pm 0.03	94.16 %
D	Ethylhexyl Palmitate, Niacinamide, Octododecenol, Titanium dioxide	24	23.76 \pm 0.04	99.00 %
E	Ethylhexyl salicylate, Niacinamide, Isopropyl isostearate, titanium dioxide	24	24.73 \pm 0.05	103.04 %
F	Citralol, Niacinamide, titanium dioxide	24	24.43 \pm 0.03	101.79 %
G	Glycerin, Stearic acid, Glycol Stearate, Titanium dioxide, Petrolatum	24	25.11 \pm 0.06	104.62 %
H	Alovera, Almond oil, Ross extract, Saffron extract.	24	11.26 \pm 0.05	46.91 %
I	Styrene/Acrylates Copolymer, Cetearyl Alcohol Titanium dioxide, Ceteareth-20	30	28.33 \pm 0.03	94.43 %
J	C12-15 Alkyl benzoate, TiO ₂ , Dimethicone, Glyceryl monostearate.	50	52.65 \pm 0.04	105.30 %
K	Glycerin, TiO ₂ , Copolymer, Dimethicone	60	54.05 \pm 0.06	90.08 %

The labeled SPF values are in the range of 15-60. Samples contain three, four, and five active ingredients. In samples, more than SPF-24, almost all samples' titanium dioxide is used. Two samples F and G almost have the same ingredients.

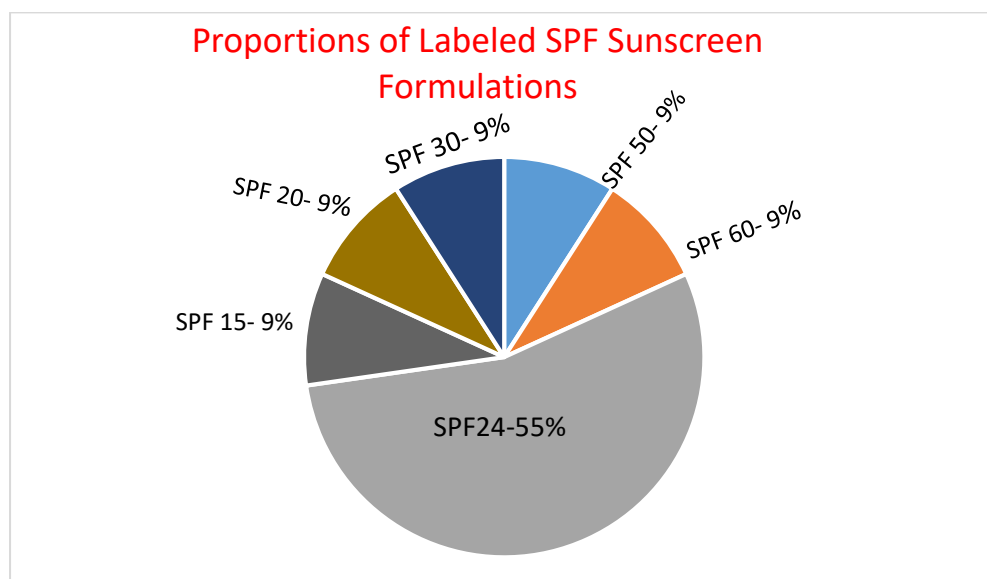


Fig.2: the pie diagram of sunscreen formulations of labeled SPF value SPF24 constitute 55% of all formulations.

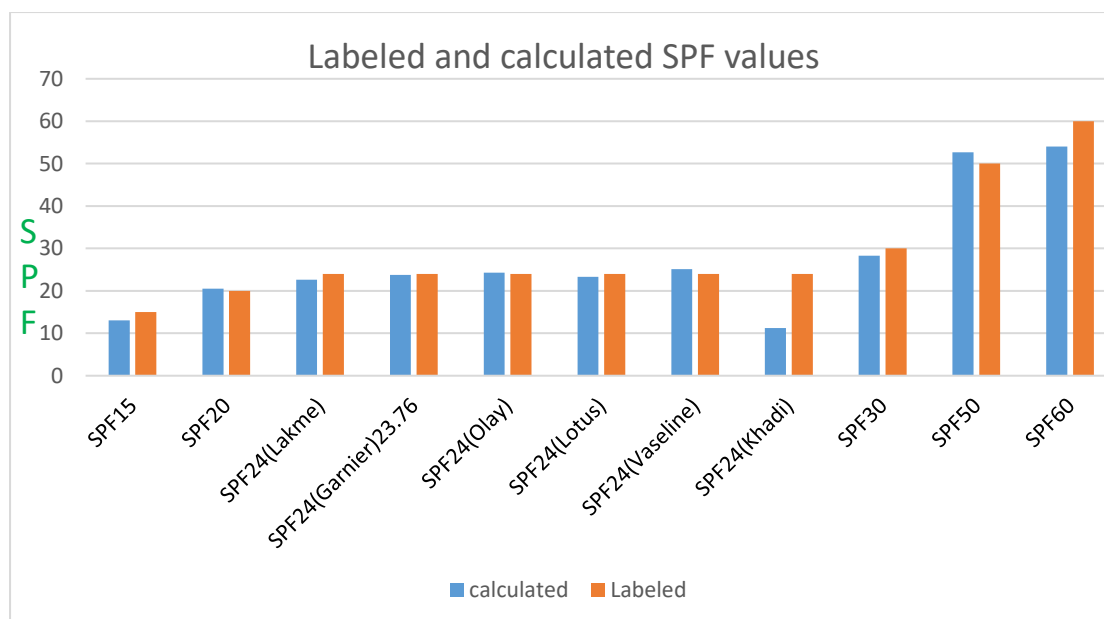


Fig.3 Labeled SPF and calculated SPF vs Sample

From Fig.3 slight variation was found in labeled and calculated SPF values for SPF values higher than 15.

DISCUSSION

The SPF sunscreens of different brands with different SPF values 15, 20, 24, 30, 50, and 60 were bought from the store, and observed the correlation between absorbance and transmittance was with help of SPF values. Generally, UV visible spectroscopy is used to measure absorbance but along with absorbance it measures transmittance. The absorbance found at

maximum wavelength is known as λ_{max} . The SPF value increases absorbance increases. There is an assumption that if SPF 24 blocks 80% of radiation, then SPF 30 will block 93% of UVB radiation which is false the difference between them is nearly 3%. Six different brands of SPF 24 were purchased from the store. They were compared with the standard sunscreen (Olay). For every sunscreen test, three trials were done, and normal absorbance was noted in the table. From the SPF brands two brands have the closest an incentive to standard sunscreen and one sunscreen is deviated from the brand S. The SPF tests D, E, F, G have closest values to the marked SPF. All other SPF esteems some are higher than named and some are lower than labeled. Test A, B, and C have a distinction of 1.97, 0.5, and 1.4 and for the examples H, I, and J have 12.74, 1.67, 7.35, 5.95, when compared with SPF 24. The distinction between named and marketed values is because of their composition difference. The sunscreens will assist with hindering the UVB rays to reach the skin. For the most part, absorbance is legitimately relative to SPF. This shows as the SPF is inversely proportional to absorbance.

APPLICATION OF DEVELOPED STUDY

The cosmetic industry could employ this newly developed approach for routine quality control examination of various sunscreen products with variable SPF values. The technique is simple, economical, and reproducible; it can be utilized rapidly in the industry and laboratories.

CONCLUSION

The study shows the correlation between absorbance and transmittance used for the evaluation of sunscreen SPF and for the blockage of UVB radiation. This method can be used in the cosmetics industry to evaluate the quality of finished and in-process sunscreen products. From the above results, we conclude that there is a variation in labeled and calculated values due to their composition.

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AUTHOR CONTRIBUTION

All authors Contributed Equally.

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CONFLICT OF INTEREST

The authors do not have any conflict of interest in this research work.

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