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Evaluation of Sunscreen Protection Factor Values (SPF) for some Aromatic Acids and their Salts of Mono- and Bivalent Metals by UV Spectrophotometer

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A B S T R A C T

This study aimed to determine the ultraviolet absorption for some carboxylic acids and their salts of mono- and bivalent metals as organic UV filters by UV spectrophotometer, as well as, calculate the values of the sun protection factor (SPF) for these compounds. The solutions of organic UV filters are subjected to absorbance measurements in the range of 290 to 320 nm, with five nm intervals, using the ultraviolet spectrometer. The experiments have been carried out in three different solvents: H_2O, MeOH, and EtOH. These salts included sodium benzoate, sodium salicylate, calcium benzoate, and calcium salicylate. The calculated sun protection factor (SPF) of these solutions was evaluated using the Mansur equation. All organic filters showed some sunlight protection properties. The best-calculated SPF values were 47.0 for salicylic acid, followed by the sodium salicylate salt at 40.3 and then the calcium salicylate salt at 39.7. These salicylic acid salts showed a high ability of UV absorbance compared to benzoic acid salts which showed SPF values of 11.5. This study presented organic UV filters with high SPF values and high solubility in polar solvents such as water and ethanol. Sodium and calcium salicylates would be recommended for use in the manufacture of sunscreen formulations.

1 Introduction

UV filters are chemical compounds that absorb ultraviolet radiation and minimize skin damage. UV filters can be classified into two groups according to their nature: inorganic UV filters and organic UV filters. Inorganic UV filters, also known as physical UV filters, are designed to reflect and diffuse rays to prevent them from penetrating the skin, while organic UV filters prevent rays from reaching the skin, both groups are classified as chemical UV filters (Dias-Cruz *et al*,. 2008; Chisvert & Salvador, 2007).

Minerals like titanium dioxide (TiO_2) and zinc oxide (ZnO) are widely used in sunscreen formulations such as broadband sunscreens that block UVA (290-320 nm) and UVB rays (320-400 nm) (Serpone *et al*., 2007).

Organic UV filters are compounds that contain organic compounds that have high absorption values in the ultraviolet range. These compounds typically consist of either single or multiple aromatic combinations and may be accompanied by double bond carbon links (Klimova *et al*., 2013; Li *et al*,. 2020). Figure 1 shows examples of organic filters that are commonly used in skin care products, such as sunscreens, to protect the skin from sunlight. Health encourages people to use sunscreen due to the harmful effects on the skin from excessive UV radiation, which mainly leads to skin cancer. Additionally, it has been found that a combination of UV filters is more effective than an individual ultraviolet radiation filter (Klimova *et al*., 2013; Li *et al*,. 2020).

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Organic UV filters must have terms that are heat stable, waterproof, non-toxic, easy to formulate, and safe for skin use (Nohynek & Schaefer, 2001). Three steps are outlined to explain how the skin absorbs UV rays (Klimova *et al*., 2015):

The entry of a substance into a particular pore or structure, such as the entry of a compound into the upper layer of the skin, is known as penetration.

Penetration is a layer-to-layer penetration, which is both a functional and a structural step and is the absorption of a substance into the lymphatic system.

Absorption is the process of absorbing through the skin and destroying the collagen layer of the skin.

The Sun Protection Factor (SPF) is a scale that measures the effectiveness of the sun's rays, helping to activate the natural defenses of skin protection from ultraviolet radiation (Brusie, 2020). The sun's protection factor's values indicate that the efficacy is determined by the absorption properties of each UV filtering material; since the skin is able to benefit from sunscreens because of their ability to absorb, reverse, and disperse UV rays; excessive exposure to the sun is closely linked to different skin types. (Catalano *et al*., 2020; Stiefel & Schwack, 2015).

Sunscreens are emulsions of chemical filters that contain water and oil, which can protect the skin from the harmful effects of UV radiation; the development of sunscreens is essential to keep the skin protected against UV rays, and preventing feedstock from interacting; sunscreen effectiveness can be achieved through the integration of soluble UVs into cosmetic formulations (Campos, Gaspar, 2006; Schulz, *et al*., 2002; Ngoc, *et al*., 2019; Mohiuddin, 2019).

Table (1) shows examples of allowance levels for some organic candidates in Canada, Australia, the European Union, and the United States (this is just a sample of countries) (Yarussi-King, 2017). Also, any organic filter needs to be licensed to allow it for example from the EU or FDA (Yarussi-King, 2017).

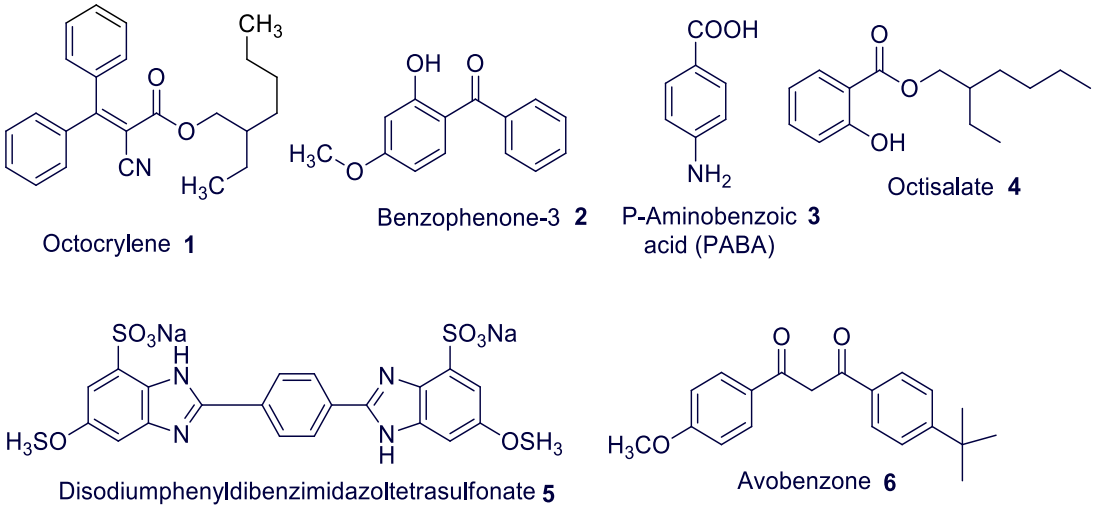

Figure (1): chemical structures of unknown organic UV filters (Klimova et al., 2013; Li et al,. 2020).

Table (1): The levels of organic candidates in four markets (Yarussi-King, 2017).

Sunscreen Active	European Union	United States	Australia	Canada
Octocrylene **1**	10% (as acid)	Up to 10%	10%	Up to 10%
Oxybenzone (Benzophenone-3) **2**	10%	Up to 6%	10%	Up to 6%
Aminobenzoic Acid (PABA) **3**	5%	Up to 15%	----------	Up to 15%
Octisalate (Etylhexyl Salicylate) **4**	5%	Up to 5%	5%	Up to 5%
Disodium phenyl dibenzimidazole tetrasulfonate **5**	10%	----------	10%	----------
Avobenzone (Butyl methoxydibenzoylmethane) **6**	5%	Up to 3%	5%	Up to 3%

2 Materials and Methods

2.1 Chemicals and Equipment

Sodium benzoate (98.00 - 100.50%) was bought from Riedel-de-Haen. Salicylic acid (99%) was purchased from ACORS. Ethanol (95%) and cyclohexane (99%) were obtained from BDH. Other chemicals were bought from Merk. Spectrophotometric determination of UV absorbance was carried out in a 1 cm path length cuvette (quartz) using a Jenway 6305 UV/visible spectrophotometer (single beam). Samples mixing were carried out using a vortex mixer (Bio Cote).

2.2 Calcium Benzoate Preparation

To a solution of 20 g (0.139 moL, 1.0 equiv) of sodium benzoate in distilled water (50 mL), the calcium chloride of 7.7 g (0.069 moL, 0.5 equiv) was slowly added at 25 ˚C with stirring. A white solid of insoluble calcium benzoate gradually precipitates is completed. The reaction mixture was stirred at 25 °C for an hour The mixture was then filtrated, washed with distilled water (25 mL), and dried to give calcium benzoate (8.8 mg, 23%) (Guang, 2021).

2.3 Calcium Salicylate Preparation

To a solution of 20 g (0.064 moL, 1.0 equiv) of sodium salicylate in distilled water (50 mL), the calcium chloride of 7.7 g (0.069 moL, 0.5 equiv) was slowly added slowly at25 ˚C with stirring. A white solid of insoluble calcium benzoate gradually precipitates is completed. The reaction mixture was stirred at 25 °C for an hour. The mixture was then filtrated, washed with distilled water (25 mL), and dried to give calcium salicylate (10.1 g, 26%) (Guang, 2021).

2.4 Sample Preparation and Measurement of UV Absorption

0.5 g of each sample was weighed, transferred to a 100 ml volumetric flask, diluted to volume with the suitable solvent (Table 2), followed by stirring using a vortex, and then filtered through filter paper. 5 ml of the stock solution of the filtrate was transferred to a 100 ml volumetric flask, diluted to volume with the suitable solvent (H_2O or EtOH), so that the sample concentration is 250 ppm.

Table (2): The solubility of samples in water and ethanol

No	Sample name	Suitable solvent
A.	Benzoic acid	Ethyl alcohol
B.	Salicylic acid	Ethyl alcohol
C.	Sodium benzoate	H_2O
D.	Sodium salicylate	H_2O
E.	Calcium benzoate	Ethyl alcohol
F.	Calcium salicylate	Ethyl alcohol
G.	Acetophenone	Methyl alcohol
H.	Benzophenone	Methyl alcohol

The absorbance of the prepared solutions of all samples (A-H) was measured by the UV spectrophotometer in the range of 290-320 nm using a 1 cm quartz cell, and this was done with an increase of 5 nm in each measurement. The measurement process was repeated twice for one wavelength, then the average absorbance of each sample was calculated as shown in Tables (3 and 4).

Table (3): Absorbance average's values of organic acids, mono- and bivalent metals salts, and ketones of organic acids

Wave length (nm)	Benzoic acid	Salicylic acid	Sodium benzoate	Sodium Salicylate	Calcium benzoate	Calcium Salicylate	Acetophenone	Benzophenone
290	0.166	1.588	1.116	1.378	0.359	1.367	0.873	1.447
295	0.029	2.179	0.658	1.924	0.417	1.822	0.462	1.204
300	0.016	2.510	0.513	2.238	0.407	2.241	0.264	0.747
305	0.009	2.598	0.382	2.313	0.356	2.346	0.152	0.356
310	0.006	2.641	0.266	2.168	0.238	2.262	0.123	0.217
315	0.004	2.529	0.186	1.393	0.121	1.569	0.114	0.205
320	0.004	2.799	0.132	0.691	0.045	0.832	0.420	0.217

3 Results

3.1 SPF Chemical Samples Account

The Mansur mathematical equation (1) is used to calculate the SPF values of the samples (A-H) (Mansur, *et al*, 1986; Sayre, *et al*, 1979; Dutra, *et al*, 2004).

$$\mathbf{SPF= CF \times \sum_{290nm}^{320nm} EE\ (\lambda) \times I\ (\lambda) \times ABS(\lambda)} \quad \mathbf{(1)}$$

Where: CF is the correction factor (=10); "EE", the erythemal effect of radiation at wavelength λ; "I", the intensity of the solar spectrum; and "ABS", the absorbance at wavelengths 290-320 nm. "EE", "I", and "ABS" are values obtained or applied for every wavelength (λ). The values for each of the [EE(λ) x I(λ)] are constants have been reported by the authors as normalized on the basis of the work by Sayre et. al., and are shown in Table 4 (Dutra, et al, 2004).

Table (4): The values of EE x I as they were determined by Sayre et al (1979).

Wave length (nm)	EE X I
290	0.0150
295	0.0817
300	0.2874
305	0.3278
310	0.1864
315	0.0837
320	0.0180

4 Discussion

4.1. Research Strategy

The strategy of the study is focused on increasing the optical absorption of the organic filter by increasing the capacity of the exchange characteristic in π bonds that can increase the optical absorption of the organic filter because π bonds are responsible for the absorption of light in many organic molecules. When photons of light interact with a molecule, they can cause electrons in the π bonds to become excited and jump to higher energy levels. Then, the molecule is able to absorb light and generate an excited state.

The molecule's ability to absorb light can be enhanced by increasing the capacity of the exchange characteristic in π bonds, which increases its ability to accept and transfer electrons. This is because the exchange characteristic in π bonds is closely related to the conjugation of the molecule, which is the ability of electrons to delocalize across a series of adjacent atoms. When the exchange characteristic is elevated, conjugation is improved, resulting in a larger π electron cloud and, consequently, an enhanced absorption of light.

In addition, when using metal ions for salts, the solubility of this filter increases in very polar solvents such as water, methanol and ethanol. As shown in Figure (2), binary ions such as Ca^{+2} have the ability to double the size of the active organic filter, which allows it to have a good ability to absorb ultraviolet rays.

In general, the capacity of the exchange characteristic in π bonds can be enhanced to enhance its performance in applications such as solar cells or optical filters.

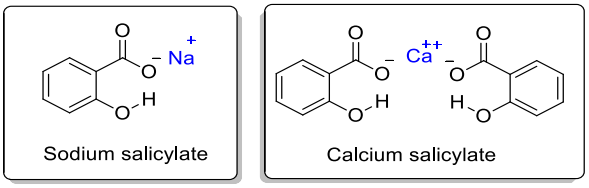

Figure (2): The structures of certain salts that are used.

4.2 Solubility Test of Samples of Organic Filters in Polar Solvents

Water was utilized as a solvent because it is the safest solvent to use on the skin. Table (5) shows that several attempts were made to obtain good solubility of the samples (A-H). The solvents are utilized because of their relative polarity, as revealed in Table 6.

Table (5): Solubility results for organic filters.

no	Organic UV filter	H_2O	MeOH	EtOH
A.	Benzoic acid	-	+	+
B.	Salicylic acid	-	+	+
C.	Sodium benzoate	+	+	+
D.	Sodium salicylate	+	+	+
E.	Calcium benzoate	-	+	+
F.	Calcium salicylate	-	+	+
G.	Acetophenone	-	+	+
H.	Benzophenone	-	+	+

Table (6): Used solvents arranged from the furthermost polar to the smallest polar.

Suitable solvent	Relative Polarity
H_2O	1.000
Methyl alcohol	0.762
Ethyl alcohol	0.654

4.3 SPF Values for Organic Filters

SPF values are numerical values that indicate the ability of a product to protect from the sun's rays. Thecalculated SPF values for organic filters are shown in Table (7).

Table (7): Calculated SPF values for organic filters.

no	Compound name	Calculated SPF values
A.	Benzoic acid	0.62
B.	Salicylic acid	47.04
C.	Sodium benzoate	11.5
D.	Calcium benzoate	11.5
E.	Sodium salicylate	40.3
F.	Calcium salicylate	39.7
G.	Acetophenone	7.5
H.	Benzophenone	7.9

In this study, the highest SPF value was found for salicylic acid, 47.04, followed by sodium salicylate (40.3), calcium salicylate (39.7), sodium benzoate (11.5), calcium benzoate (11.5), benzophenone (7.9), and acetophenone (7.8), as illustrated in Figure 1. These values show a high ability of salicylic acid and its salts to absorb UV rays compared to benzoic acid and its salts that showed lower SPF values. The reason of this low SPF values for benzoic acid salts may be attributed to the different structural construction between salicylic acid and benzoic acid. In the structure of salicylic acid, the presence of the electron-donating group (-OH) in the ortho position on aromatic ring increased the exchange capacity of π-bonds to give the compound a more reactive character than in benzoic acid, which lacks an electron-donating group on the aromatic ring (Figure 3).

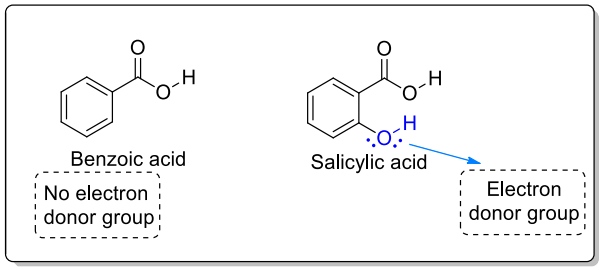

Figure (3): Comparison of the structural structures of the acids used.

The slight difference in the SPF values of the salicylic acid salts of sodium and calcium (E & F) may be attributed to the differences in the solvents (H_2O and EtOH) that effect on UV absorbency. On other hand, the SPF values did not change by changing the valence of the metal attached to the carboxyl group of the aromatic acid as observed from the obtained values. Thus, either mono- or bi-valent can be used in sunscreens formulations depending on which one is safer for the skin.

SPF values indicate the degree of protection a product offers against UVB rays, which are responsible for sunburn and skin cancer. The higher the SPF value, the greater the protection. It is recommended to use a sunscreen with an SPF value of at least 30. However, it is important to note that SPF values do not indicate protection against UVA rays, which can also cause skin damage. Therefore, it is important to use a broad-spectrum sunscreen that protects against both UVA and UVB rays.

Additionally, it is important to note that SPF values are not directly proportional to the amount of time aperson can spend in the sun without getting burned. Instead, the amount of time a person can spend in the sun without getting burned depends on a variety of factors, such as skin type, the intensity of the sun's rays, and the amount of sunscreen applied. Therefore, it is important to reapply sunscreen regularly and seek shade during peak sun hours to protect the skin from damage. Overall, this study suggests that salicylic acid and its salts may be more effective in providing protection against UV radiation compared to benzoic acid and its salts.

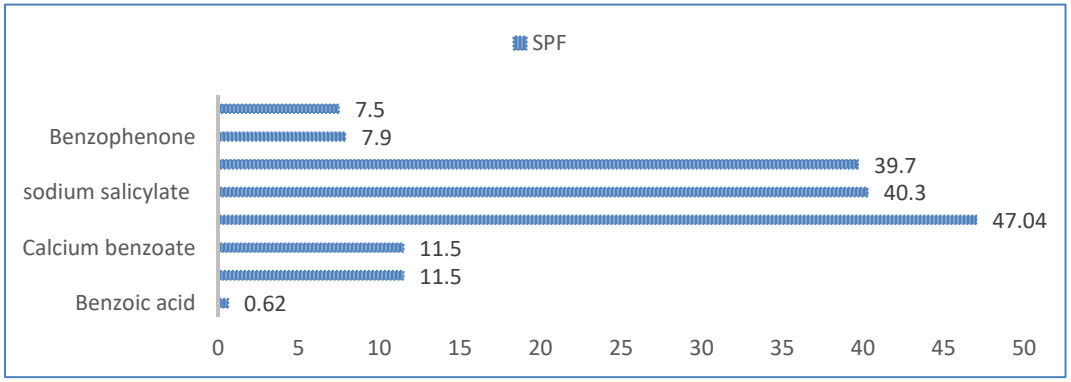

Figure (4): Comparison of the calculated SPF values for the samples used.

4.4 Comparison of the Obtained SPF Values with Previous Studies

Most of the previous studies that we conducted focused on natural extracts, whether from plant extracts or oils, instead of organic filters in our study, and did not exceed the SPF value 10 (Mosa, Makhlouf, 2019; Zayd, et al, 2019; Alfeetouri, Mosa, Jibreel, 2019; Mosa, Alsaady, Naser, 2022). Hence, we see that most of the organic filters give higher SPF values than filters extracted from plant samples, although salicylic acid was isolated from the bark of the willow tree and aspirin was prepared from it. In another study (Mbanga, et al, 2014), it was shown that the value of the SPF for some products containing organic filters (Table 7) written on the product and also calculated from Mansour's equation is much less than the values obtained from the filters under study.

Tables (7): calculated SPF values for organic filters in some formulations (Mbanga, *et al*, 2014).

no	Actives Ingredients	Labeled SPF	Calculated SPF
1	Avobenzene, Octylsalycilate, Titanium oxide, Terephthalylidène, Dicamphor sulfonic acid	15.00	15.24
2	benzophenone-3, octylmethoxycinnamate, octyldimethyl PABA	15.00	14.92

4.5 Uses of the Salts Under Study in Medicine and Food Industry

Sodium salicylate is used in medicine as an analgesic and antipyretic, also acts as a non-steroidal anti-inflammatory drug (NSAID) and induces apoptosis in cancer cells. However, calcium benzoate is used in the food industry as a preservative, its E number is E213; It is approved for use as a food additive in the European Union and other countries (Approved additives and E numbers, 2022). Sodium benzoate is widely used as a food preservative and pickling agent, and its E number is E211. Calcium salicylate is used as a preservative and also for the treatment of cancer (Cox, et al, 2021).

5 Conclusions

Measuring the SPF values of any organic filter is the best way to determine the effectiveness of sunscreen, so the higher the SPF value of the sunscreen, the greater the protection it provides against UV rays. In this study, the values of the SPF were measured for four aromatic salts added to their acids and samples of ketones, namely acetophenone and benzophenone, to compare them with the ability of the salts to absorb light.

This study presented organic filters that have a high ability to absorb in the UV region and gave high SPF values represented in salicylic acid, sodium salicylate salts, and calcium salicylate, and also distinguished from the rest of the filters by their ability to dissolve in highly polar solvents, which facilitates their use in skin care preparations to protect from Sun rays. Thus, this study contributed to the development of sunscreens to better protect against the sun's rays.

6 Recommendations

It is recommended to use salicylic acid salts in sunscreen products as organic filters which have a high ability to filter UV rays and prevent their penetration into the skin; as well as these salts dissolve in water which is the safest solvent to use on the skin. Further research is also recommended in the field of organic filters that are safe and easily soluble in polar solvents such as water.

Contrast of Interest: The authors declare that they have/ have no contrast of interest associated with this manuscript.

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