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Determination of UV Filters on Some Sunscreen Products using HPLC-DAD

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ABSTRACT: To evaluate twelve sunscreen products purchased from the Saudi market using HPLC-DAD method, four UV filters (benzophenone-3, octinoxate, octisalate and homosalate) was investigated. Sample was weight, diffuse, centrifuge then analysis. Calibration curves of standards show good linearity, R^2 ranged from 0.9998 to 0.9999. Results ranged from 1.32% to 7.88% (w/w) under the permissible limits of SASO, which confirmed the safety of creams. Every sample has (1 to 3) sunscreen agents, while 4 creams don't detect anyone. The total UV filters concentrations on the same sample ranged from 2.4% to 16.88% (w/w). HPLC-DAD method was effectively monitoring of the commercial cosmetics.

Keywords: *HPLC-DAD, analysis, cosmetic control, Sunscreen, UV filters*.

I. Introduction

Sunscreens are important part of the fight to keep the skin beautiful and healthy and support to save the skin from sunburn, aging, and cancer [1]. A sunscreen has many forms: lotion, spray or gel which applied on the skin exposed to sunlight to absorb or reflect some of the sun's UV radiation [2]. Visible light/heat will emission as a result of absorption of UV, this explain how sunscreens function to care for human skin from dangerous UV radiation [3]. To slow or avoid anabasis of wrinkles and sagging skin, people should use sunscreen every day. This was a result of a study that covers 900 white people in Australia for 4.5 years [4]. Therefore, sunscreen agents are added to some cosmetics to deliver sunscreen function. However, long-term contact with the cosmetics that have excessive addition of sunscreen agents may increase the risk of developing a skin allergy to light as well as birth defects. As a result, the types and amounts of sunscreen agents in sunscreen cosmetics have been strictly regulated.

Several studies have been conducted on cosmetics, particularly sunscreens to quantitative determination of UV filters. ICP-MS/OES/AES were used to assessment an inorganic sunscreen agent (titanium dioxide) on sunscreen cosmetics[5][6]. Organic sunscreen agents in various cosmetic products were estimated using GC-MS[7][8]. Simultaneous determination of organic UV filters was successively achieved by using HPLC-UV/DAD [9-22].

Sunscreens are regulated by The Saudi Standards, Metrology and Quality Organization (SASO)[23]. There are only 27 active ingredients that are approved by SASO. There are 26 organic filters and 1 inorganic filter, titanium dioxide[24]. All compounds allowed from 2 to 10 %, and 25% for the

inorganic compounds TiO_2 and ZnO. Table I shows the toxicities of the sunscreen agents (allowed by the Food and Drug Administration (FDA).

Determination of four UV active ingredients sunscreen (Oxybenzone, Octinoxate, Octisalate and Homosalate) in 12 sunscreen products that collected from Saudi markets by using high performance liquid chromatograph (HPLC) instrument is the aim of this study.

Table I The toxicity of the four active ingredients of sunscreen (under investigation)

Chemical	Skin Penetration	Hormone disruption	Skin Allergy	Detect in mother's milk	References
Oxybenzone 2-Hydroxy-4-methoxybenzophenone (Oxybenzone) Use in USA sunscreen widespread	Yes 1-9%	Equal to estrogen in effect; join with endometriosis in women	Almost high	Yes	[25-30]
Octinoxate 2-Ethylhexyl-4-methoxycinnamate (Octylmethoxycinnamate) Use in USA sunscreen widespread	Yes less than 1%	Hormone-same action; thyroid and reproductive system	Medium	Yes	[31][30][29][27]
Octisalate Use in USA sunscreen widespread; stabilizes avobenzone	Yes	-	Rarely	-	[34][33][32]
Homosalate 3,3,5-Trimethylcyclohexyl salicylate stabilizes avobenzone	Yes less than 1%	Confuse estrogen, androgen and progesterone	-	Yes	[35][30][27]

2. Materials and methods

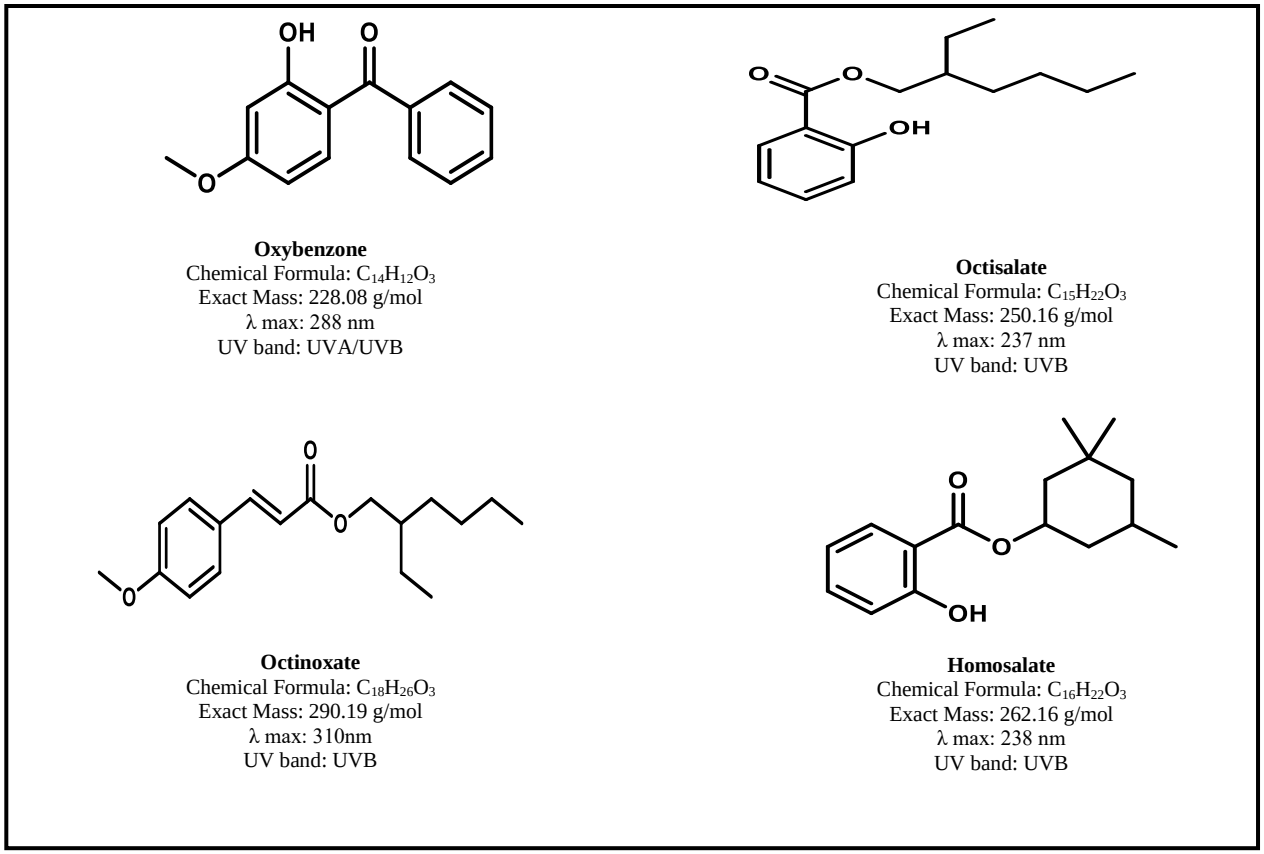
To estimate the quality of twelve sunscreen products, HPLC method is used for qualitative and quantitative analysis of four active ingredients. Figure 1 shows the chemical structures, formula, molar mass, **λ** max and UV band of four sunscreen agents that will be determined in this work.

2.1 Instrumentation

The HPLC analysis was carried out using Agilent 1260 system, equipped with solvent delivery module, quaternary pump, autosampler, Diode array detector (Agilent Technologies, Germany).Setting and data processing were achieved using ChemStation [Rev. B.01.03 SR2 (204)].

The separation was performed on an Agilent Eclipse Plus C18, x 100 x 4.6 mm I.D, 3.5 µm HPLC column (Agilent Technologies, USA)The temperature maintained at 25 ±2 ◦C. The LC system was operated isocratically using a mixture of methanol–water (80: 20, v/v) as mobile phase, at a flow rate of 1 mL/min. Injection volume was 5 µL.

Quantitation was performed at maximum wavelength of each compound. A Milli-Q system (Millipore, Bedford, MA, USA) was using to purified water. Balance (Ohaus Corp., Pine Brook, NJ, USA). Sonicator (NEXUL Ultrasonic Cleaner, NXP-1002,



Oxybenzone
Chemical Formula: $C_{14}H_{12}O_3$
Exact Mass: 228.08 g/mol
λ max: 288 nm
UV band: UVA/UVB

Octisalate
Chemical Formula: $C_{15}H_{22}O_3$
Exact Mass: 250.16 g/mol
λ max: 237 nm
UV band: UVB

Octinoxate
Chemical Formula: $C_{18}H_{26}O_3$
Exact Mass: 290.19 g/mol
λ max: 310nm
UV band: UVB

Homosalate
Chemical Formula: $C_{16}H_{22}O_3$
Exact Mass: 262.16 g/mol
λ max: 238 nm
UV band: UVB

Figure 1: Structures of sunscreen agents.

Kodo Technical Research Co. Ltd, Korea). Centrifuge instrument (Heraeus Labofuge 200, Kendro laboratory products, Germany).

2.2 Reagents

Homosalate (HOM) , Octisalate (OS) , Oxybenzone (OXB), methanol, HPLC grade (Sigma-Aldrich) (St. Louis, MO, USA), Octinoxate (OMC) (ACROS ORGANICS, USA).

2.3 Calibration of standards

Stock solutions involving 1.0 mg/mL of oxybenzone, octinoxate, octisalate and homosalate were processed by weighed accurate 5.0 mg of each substance and dissolved in 5 mL of methanol. Working solutions were arranged by further dilution with the mobile phase. All solutions were stored in tightly closed container away from light. Aliquots of the standard working solutions of oxybenzone, octinoxate, octisalate and homosalate were transferred into series of ten volumetric flasks with 10 mL capacity, then complete with mobile phase to the mark. The final concentrations were within the range of 7.5-100 µg/mL for all tested compound.

Ten planes of calibration solutions were injected 3 times to calculate the linearity of UV response at λ_{max} of each compound. By plotting the respective concentration against the peak area of each compound, The analytical calibration curves were constructed.

2.4 Sample collection and preparation:

Many types and brands of sunscreen are available in the Saudi market. Twelve sunscreen products were chosen (they are listed in Table II, and there prices ranged from 8 to 221 SR ($2.14 -58.67). Sample no. 7 (50 SR-13.35$ - no source inform) and sample no. 12 (221 SR- 59$ - France made) were the same brand but one was imitation (no. 7) and another was original brand (no. 12).

One g of each product was weighted and shifted to a 100 mL volumetric flask, diffused with 10 mL methanol by ultrasonication for 15 min. Completed to a volume with methanol, then centrifuged (10 min, 3000 g), filtered through 0.2 µm nylon syringe filters (Millipore Millex-GN).

Transfer 1 mL of the filtrate to a 10 mL volumetric flask, completed to volume with methanol and then analyzed by HPLC[14].

Table II: Sample collection

Sample	SPF	Country	Price (SR)
1	ND	Indonesia	10
2	15	Philippines	10
3	25	Thailand	8
4	20	Philippines	10
5	45	USA	20
6	50+	Spain	40
7	ND	ND	50
8	30+	Lebanon	60
9	50+	Italy	55
10	30	ND	90
11	100	FRANCE	100
12	30	FRANCE	221

3. Result and dissection:

The provided reference method was slightly modified to obtain suitable and acceptable chromatographic performance parameters using the available HPLC columns. The ratio of the organic modifier (methanol) was reduced to 80 % to obtain acceptable peaks without any interference from other compounds present in the tested sunscreen products. The beast flow rate of mobile phase is 1mL/min. Table V lists the four sunscreen agents (under investigation) which permitted using in sunscreen cosmetics according to GSO 1943/2009 in the Saudi Arabia (KSA), European Union(EU), the United States(USA), China(CHN) and Japan(JP).

3.1 Method validation

The calibration curves for all UV filters showing excellent linearity on a concentration range of 7.5-100 µg/mL, also the R^2 value of them exceeded 0.9998–0.9999. The slope of the calibration chart represents the sensitivity. The accuracy was assessed by recovery tests, the recoveries varied between 95 and 102%. The limit of detection (LOD) involves measuring increasingly more dilute concentrations of analyte (LOD = 3 × SDyx / Slope). The limit of quantitation (LOQ), is a minimum concentration which gives quantitative results with high confidence **(LOQ = 10 × SDyx / Slope)** [36].

The detection limit was determined to be within 0.77–1.2 µg/mL, and the quantization limit was determined to be 2.59–4 µg/mL. Table III shows all the last values.

3.2 Selectivity

Under optimized chromatographic conditions, no overlap was noticed and excellent separation of tested compounds was achieved, there were four acceptable peaks, for the four standards under investigation.

Oxybenzone, octinoxate and octisalate have pure chromatogram, while homosalate has unpure chromatogram.

The sharp peak of oxybenzone has appeared in 2.6 min at 288 nm, octinoxate 14.3 min at 310 nm, octisalate 17.8 min at 237 nm while homosalate has two peaks: the sharp one 19.7 at 238 nm and impurity peak 15.7 min as shown in Fig. 2.

By applying the spectral data developed by DAD, the purity of all separated compound was confirmed by the complete spectrum to ensure that no contamination of the matrix shared to peak response.

Table III: Linear range, linearity, accuracy, sensitivity, LOD and LOQ values

Analyte	Linear range (µg/mL)	R^2	Accuracy	Sensitivity (µg/mL)	LOD µg/mL	LOQ µg/mL
OXB	7.5 – 100	0.9999	100	13.21	0.7798	2.599
OMC	7.5 – 100	0.9999	95	19.413	1.117	3.724
OS	7.5 – 100	0.9999	101	10.426	0.9349	3.116
HOM	7.5 – 100	0.9998	102	9.2079	1.200	4.002

R^2: Linearity correlation coefficient

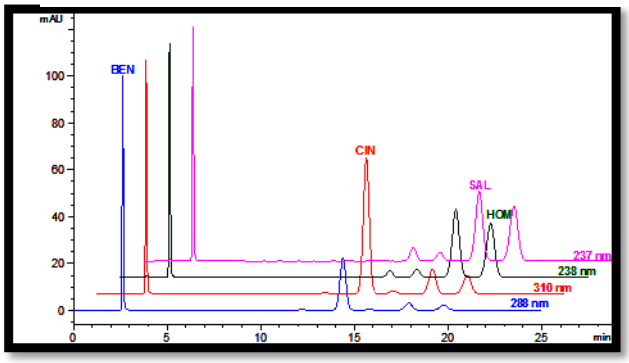


Figure 2: Chromatogram for a calibration curves of stock solution 100 µg/mL for all standards at different wavelengths.

3.3 Precision

The precision was resolved by testing the sample on two dissimilar days, applying the same instrument. The relative standard deviation (RSD) of peak area measurements (n = 3) were calculated. On the same day, the samples were tested in triplicate (intraday) and on two different days, they were tested in triplicate (interday). Thus, RSD of the 4 sunscreen agents in terms of concentration ranged within 0.12-1.90% (Table IV).

Table IV: Intra-day and inter-day precision (RSD, %) of the developed method

Analyte	RSD (%) intra-day (n=3)	RSD (%) inter-day (n=3)
OXB	1.87	1.90
OMC	0.19	0.12
OS	1.01	1.70
HOM	ND	ND

Table V: Permitted concentration of UV filters in USA, EU, CHN, JP and KSA [20] [24]

UV filters (active Ingredients)	Permitted Concentration				
	USA	EU	CHN	JP	KSA
OXB	6%	10%	10%	5%	10%
OMC	7.5%	10%	10%	20%	10%
OS	5%	5%	5%	10%	5%
HOM	15%	10%	10%	10%	10%

3.4 Sample analysis

Qualitative and quantitative results of the four UV filters on the investigated samples which analysed by HPLC analytical conditions were shown on table VI. As a result of analysis, that the percent of four organic UV filters in the twelve sunscreens were ranged for OXB (1.32-4.56%), OMC (2.40-7.88%), OS (3.28-4.44%) while HOM was present in one of the samples analysed (6.48%), all results within the designated limit according to SASO in KSA.

Eight samples 1, 2, 3, 4, 7, 8, 9, 12 were detect different concentrations of an active ingredients that ranged as total of (2.40%-16.88%). while four samples don't contain them. All concentrations of an active ingredient were under the permissible limits of SASO for the four active ingredients.

It was also observed that sample 12 contain oxybenzone (1.32%) and octinoxate (6.84%) while the sample 7 replicates in the brand contain octinoxate (6.84%) only. That indicate to the priority protect from sun radiation of sample 12 (an expensive one) than the replicate sample 7 (the cheaper one).

The largest total active ingredients components on samples was found on sample 9 which equal to 16.88%, its Italy made. Product label indicate the SPF was 50%, it represent the best of all samples and the best product on protect consumer skin. The lowest total active ingredients components on sample was found on the cheaper sample no.1, Indonesia made.

All compounds that identified on the analysis were agreeing with the compounds identified on the products label, except sample no. 12, it was also found to contain benzophenone-3, which is not included in the package label, their concentrations were in the range of 1.32–7.88% (w/w). Identified compounds and their quantities were in agreement with SASO.

3.5 A comparison of UV filters concentration (percent%) in this study with reported data in the literature.

The concentration (by percent%) of the four filters determined in this study are in comparison with those of UV filters concentration in some sunscreen products reported in the literature (Table VII). All UV filters (OXB, OMC, OS and HOM) under studied were drop in the ranges reported for sunscreen cosmetics in the literature. Addition to them submission within the designated limit of the SASO organization.

UV filters - Detection limits (%)					Total % of filters
Concentration in sunscreen product % w/w ± SD (RSD)					
Sample	OXB 10%	OMC 10%	OS 5%	HOM 10%	
1	ND	2.40±0.03 (1.25)	ND	ND	2.40
2	ND	5.82±0.09 (1.46)	ND	ND	5.82
3	ND	ND	ND	ND	ND
4	1.61±0.03 (1.57)	6.45±0.06 (0.93)	ND	ND	8.06
5	ND	ND	ND	ND	ND
6	ND	ND	3.28±0.06 (1.68)	6.48±0.06 (0.85)	9.76
7	1.32±0.01 (0.76)	6.84±0.05 (0.66)	ND	ND	8.16
8	ND	5.90±0.02 (0.34)	ND	ND	5.90
9	4.56±0.09 (1.87)	7.88±0.02 (0.19)	4.44±0.05 (1.01)	ND	16.88
10	ND	ND	ND	ND	ND
11	ND	ND	ND	ND	ND
12	ND	6.84±0.05 (0.66)	ND	ND	6.84

Table VI: Average results (%, w/w; n = 3) of 4 UV filters in commercial sunscreens.

Some centuries have different permissible limits of UV filters, such as Brazil and USA which allowed to HOM concentration to reach 15%, while SASO allowed it not excess than 10%. From table 3-14, Brazil study show the concentration of HOM reach to 11.2% and of OS reach to 5.1% which above the suggested safety limit of SASO organization (10% and 5% respectively). Korea study on 2011 shows that override concentration of OXB, OMC and OS of the permissible limits of UV filters on Korea, but no override concentration compared to SASO organization for OXB and OMC. Only OS (5.33%) override SASO permissible limit (5%).

USA research on three sunscreen shows allowed concentrations of all four UV filters except HOM filter which reach to 15.4%, that violate the permissible limits of it on USA (15%) and on Saudi Arabia (SASO 10%).

The rest studies on different countries had UV filters concentrations that fall within the permissible limits in them countries and on Saudi Arabia.

3. Conclusion:

The HPLC-DAD developed method is smooth, quick and convenient for the same time determination of: benzophenone-3, octinoxate, octisalate and Homosalate which commonly used in sunscreen formulations as sunscreen agents. Simple procedure to preparation the sample: dilution, centrifugation and filtration. From one to three organic sunscreen agents determined in a single sample, four samples had no detect any one of sunscreen agents. eight samples were detect different concentrations of an active ingredients that ranged as total of (2.40%-16.88%). All concentrations of an active ingredient were under the permissible limits of SASO for the four active ingredients.

Table VII: Levels of UV filters in sunscreen products in the literature, for different countries

Country	Cosmetics	Technique	OXB 10%	OMC 10%	OS 5%	HOM 10%	References
KSA	12 Sunscreens	HPLC	Found in 3 samples (1.32-4.56%)	Found in 7 samples (2.40-7.88%)	Found in 2 samples (3.28-4.44%)	Found in 1 sample (6.48%)	This study
Tokyo	2 Suntan lotion	GC-MS	(3.1-3.2%)	2.2%	ND	ND	[8]
	2 Foundation		ND	1.3%			
	2 Lipstick		(2.7%)	0.96%			
Italy	25 Sunscreen Products	HPLC	Found in 7 samples (0.5 -6%)	Found in 19 samples (2- 10%)	ND	ND	[37]
Brazil	10 Sunscreen Products	HPLC	Found in 6 samples (2- 4.9%)	Found in 5 samples (5.6-7.4%)	Found in 5 samples (3-5.1%)	Found in 5 samples (5.8%-11.2%)	[19]
USA	Cosmetic powder sample	HPLC	ND	3.28%	2.29%	ND	[38]
	Lotion sample		2.73%	2.63%	2.32%	2.01%	
	Lipstick sample		2.74%	2.41%	3.40%	3.04%	
Korea	5 Cosmetics	HPLC	Found in 1 samples (4.60%)	samples Found in 5 (.87-5.39%)	Found in 3 samples (.22-4.40%)	Found in 1 sample (9.31%)	[39]
Korea	101 commercial suncare products	HPLC	Found in 10 samples (3.04-5.37%)* $(2)^{+}$	Found in 96 samples (3.08-8.16%)* $(18)^{+}$	Found in 37 samples (1.87-5.33%)* $(2)^{+}$	ND	[16]
USA	3 Sunscreens	UHPLC	Found in 3 samples (2.32-5.39%)	Found in 3 samples (0.18-7.32%)	Found in 3 samples (2.91-4.15%)	Found in 2 samples (9.91-15.4) $(1)^{+}$	[40]
Syria	5 Sunscreens	RP-HPLC	Found in 2 samples (1.6-4.3%)	Found in 5 samples (4.24-7.5%)	Found in 1 samples (4.54%)	ND	[22]

*The range of concentration.
+The number of items which were over the authorized concentration.

Accurate, precise and a wide range of linearity obtained for the method, their sensitivities and LOD are convenient and successful for commercial sunscreen products analysis

. The regulatory authorities need to implement major quality controls and screening for products created to enter in direct and long-term touch the skin, particularly for products imported from different countries. Consumer should keep themselves updated with knowledge and an awareness of the ingredients present in the products they use.

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تقدير اربعة مرشحات للأشعة الفوق بنفسجية في منتجات الوقاية من اشعة الشمس باستخدام جهاز الكروماتوجرام السائل عالي الأداء – كاشف الصمام الثنائي

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مستخلص. لتقييم اثني عشر منتج من منتجات الحماية من الشمس المتواجدة في السوق السعودي باستخدام طريقة HPLC–DAD، تم تقدير أربعة مرشحات للأشعة فوق البنفسجية (Benzophenone–3, Homosalate, Octisalate and Octinoxate)وتم وزن العينة ثم تخفيفها وتشتيتها بالميثانول وأجهزة الطرد المركزي ثم تحليلها. تظهر منحنيات المعايرة للمعايير خطية جيدة، حيث R2 تراوحت (0.9998 إلى ٠,٩٩٩٩).

وجد ان تركيز المرشحات الاشعة الفوق بنفسجية قد تراوحت من (١,٣٢–٧,٨٨٪) وكانت ضمن الحدود المسموح بها في هيئة المواصفات والمقاييس السعودية ، والذي يؤكد على سلامة الكريمات، وتحتوي كل عينة على عوامل واقية من الشمس من (١ إلى ٣) ، في حين أن ٤ كريمات لم يكشف بها أيًا من هذه العوامل، وتراوحت تركيزات مرشحات الأشعة فوق البنفسجية الإجمالية على نفس العينة (٢,٤–١٦,٨٨٪). طريقة HPLC–DAD كانت فعالة لمراقبة جودة مستحضرات التجميل التجارية.

كلمات مفتاحيه: الكروماتوجرام السائل عالي الأداء – كاشف الصمام الثنائي، التحليل، التحكم في مستحضرات التجميل، واقي الشمس، مرشحات الأشعة فوق البنفسجية