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Determination of Octyl Methoxycinnamate Levels on The Face Sunscreen Gel Using UV-Vis Spectrophotometry Method

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ABSTRACT

Long-term exposure to sunlight can have an impact on skin damage, such as sunburn, photoaging (premature aging), and dull skin. Ultraviolet (UV) radiation is a major cause of skin cancer, so many sunscreen gels contain UV filters. Octyl methoxycinnamate is an example of an active UV filter agent. This study aims to determine the octyl levels of methoxycinnamate in sunscreen gel preparations circulating in Magelang Regency. The analysis used in this research is qualitative and quantitative analysis. Qualitative study using thin layer chromatography (TLC) with silica gel F254 as stationary phase and mobile phase n-hexane: ethyl acetate (7.5 : 2.5) and then detected on UV light at 254 nm. Quantitative analysis using UV-Vis spectrophotometer at a maximum wavelength of 310.5 nm. The results of the TLC showed that samples X, Y, and Z produced purple stains and had the same Rf values between the samples and the standards. Octyl methoxycinnamate levels in sample X of 0.155%; Y 0.824%; and Z 1.954%. The results obtained by the relative standard deviation (RSD) of sample X 0.015%; Y 0.048%; and Z 0.921%. It can be concluded that the three tested sunscreen gel samples are safe for use by the general public.
Keywords: Octyl Methoxycinnamate; Thin Layer Chromatography (TLC); UV-Vis Spectrophotometry, Sunscreen Gel

INTRODUCTION

Sun exposure cannot be separated from daily activity. In reality, the majority of activities take place outside and are frequently in the sun. Sunlight has positive effects on the body's ability to produce vitamin D, fight off microbes, and enhance the condition of the cardiovascular system (Weller, 2016). However, prolonged and excessive sun exposure might affect the health of your skin. Sun exposure can cause skin damage such as sunburn (burning skin), photoaging (premature aging), and dull skin. Skin cancer is one of the more dangerous potential dangers (Chakraborty et al., 2018).

Ultraviolet (UV) radiation from the sun damage the skin. The broad spectrum of UV radiation is divided into three ranges, namely UVA, UVB and UVC. UVA has the longest wavelength (320–400 nm) but the least photon energy, UVB wavelength is in the middle range (280-320 nm), and UVC has the shortest wavelength (100-280 nm) but has the highest energy. Although UVA and UVB make up the majority of sunlight, each UV component can act differently and have various effects on the skin (Chakraborty et al., 2018). UVA rays have a radiation effect in the form of pigmentation which causes brown and reddish skin. UVB rays have a radiation effect that causes erythema (redness) that can cause skin cancer if exposed to this radiation for too long. Meanwhile, UVC rays are stuck in the uppermost layer of the earth's atmosphere and do not have time to enter the earth due to the presence of the ozone layer (Tahar et al., 2019).

Photoprotectors especially sunscreen are crucial in lowering the frequency of skin health issues brought on by UV rays, such as pigmentation and skin aging symptoms. Sunscreen has been discovered to be able to prevent and decrease the harmful effects of UV rays, which have the ability to absorb, reflect, and scatter sunlight (Donglikar & Deore, 2016). Recently, it was discovered that sunscreen shields the skin from dangers besides just reducing the harmful effects of UV rays (e.g. IR, blue light, and pollution). Theoretically, the activity of sunscreen agents should be restricted to topically to prevent negative systemic consequences (stratum corneum) because sunscreen can infiltrate the tissue. Montenegro (2013) conducted research on the penetration of topical sunscreen compounds that can be absorbed from plasma to urine (Montenegro & Puglisi, 2013).

Currently, octyl methoxycinnamate (OMC) represents the most common sunscreen agent in commercially available cosmetics, therefore has a very good UV absorption curve and good oil solubility. It is used as a UVB (280 nm–320 nm) filter although the low-energy tail of its absorption spectrum extends into the UVA (320 nm–400 nm) (Hanson et al., 2015). Due to their high lipophilicity and robust permeability, when these organic UV filters are coated on the skin's superficial parts, it should be taken into account that a significant accumulation will occur in the skin's deeper layers. In addition, OMC dosage was occasionally raised to achieve a high sunscreen protection factor (SPF) objective. The possible risk was made worse by the rising use of OMC. In the United States, European Union, and Australia, it was stipulated that the maximum dosage of OMC was 7,5-10% (Zhao et al., 2016).

Determination of octyl methoxycinnamate levels in cream dosage forms has been widely carried out by the High Performance Liquid Chromatography (HPLC) method. Among these studies was carried out by Purwaniati (2019) using the HPLC (High Performance Liquid Chromatography) method. Another method developed for the determination of octyl methoxycinnamate levels in gel preparations is UV-Vis Spectrophotometry (Purwaniati, pipit novita et al., 2019). The UV-Vis Spectrophotometry method was chosen for the quantitative analysis of octyl methoxycinnamate because it is practical and produces findings that are quick and accurate. The numbers that are read are promptly recorded by the detector and produced as digital numbers or regression graphs. In addition, the UV-Vis Spectrophotometry instrument has high accuracy with a relative error of 1%-3%, analysis can be carried out quickly and precisely, and can be used to define very small quantities of substances (Nabila et al., 2020).

In Magelang regency, there are still cosmetic sellers who sell cosmetic products without registration number. Therefore, it is important to determine whether the cosmetics for sale contain any dangerous chemicals. The purpose of this study was to examine whether there is an octyl methoxycinnamate content in samples of sunscreen gel products circulating in cosmetic sellers in Magelang Regency. In addition, there is a possibility that the use of octyl methoxycinnamate in sunscreen exceeds the specified usage level limit, it is necessary to carry out a feasibility test or analysis on sunscreen products to determine the amount of octyl methoxycinnamate content that has been used in the product. From the results of this analysis it can be interpreted whether sunscreen products are suitable or not suitable for use by the general public.

METHODS

Materials

The main material used in this study is 3 samples of sunscreen gel. This sample was taken from 3 cosmetic shops in Magelang Regency. Other materials used were octyl methoxycinnamate (Multi Jaya Kimia), isopropyl alcohol (General Labora), n-hexane p.a (Merck), ethyl acetate p.a (Merck), and distilled water.

Tools

The tools used are silica gel 60GF_{254}, capillary tube, chamber, Uv-Vis spectrophotometer, quartz cuvette, analytical balance, laboratory glassware (beaker glass, measuring cup, erlenmeyer, measuring flask, funnel), filter paper, volumetric pipette.

Methods

Sample Preparations

Sample preparations for analysis of sunscreen gel samples were weighed 1 gram then added 10 mL of isopropyl alcohol and stirred with a magnetic stirrer for 5 minutes. The solution was centrifuged for 10 minutes. Solution clear centrifuge results are taken and transferred into a 10 mL volumetric flask.

Thin Layer Chromatography (TLC) Analysis

The stationary phase used was Silica Gel 60 F_{254}. Silica gel 60 F_{254} was cut into 5 cm x 10 cm sizes, then 1 cm bordered on the top edge and bottom plate was marked with a pencil. The mobile phase used was pro-analysis of n-hexane: pro-analysis of ethyl acetate (7.5:2.5) (Pratiwi et al., 2018).

Standard octyl methoxycinnamate 1 mL is dissolved in a 10 mL measuring flask, add isopropyl alcohol up to the mark. The standard solution and the three samples were spotted on the TLC plate using a capillary tube at 1 cm from the bottom. The TLC plate that has been spotted is put into a chamber that has been saturated using filter paper. After the mobile phase reached the upper limit of the TLC plate, the spotting on the TLC plate was monitored under 254 nm UV light. The Rf value in the sample is calculated and compared with the standard Rf value for comparison.

Uv-Vis Spectrophotometric Analysis

Preparation of Octyl Methoxycinnamate Mains Solution 1000 ppm

Pipetted 1.0 mL of standard octyl methoxycinnamate, put into a 10 mL volumetric flask and isopropyl alcohol was added up to the mark. After that, 1.0 mL of the sample solution was pipetted into a 10 mL volumetric flask and isopropyl alcohol was added up to the mark. Pipette the sample solution again as much as 1.0 mL, put it into a 10 mL volumetric flask and add isopropyl alcohol up to the mark to obtain a level of 1000 ppm.

Determination of Maximum Absorbance Wavelength of Octyl Methoxycinnamate

Pipetted 1.0 ml of 1000 ppm octyl methoxycinnamate mains solution into a 10 ml volumetric flask and then add octyl methoxycinnamate up to the boundary mark to obtain 10 ppm. Then the absorbance was measured at a wavelength of 400-800 nm using a visible spectrophotometer.

Determination of the Octyl Methoxycinnamate Standard Curve

Pipetted 500 µL of octyl methoxycinnamate mains solution with a micropipette, then put into a 10 mL volumetric flask and added isopropyl alcohol up to the mark. After that the solution was taken with a micropipette as much as 100; 200; 300; 400; 500; 600; and 700 µL, then put into a 10 mL volumetric flask and isopropyl alcohol was added to each solution up to the mark. This solution was homogenized, then the absorbance was measured at the maximum absorbance wavelength of octyl methoxycinnamate using a visible spectrophotometer. Then create a calibration curve so that the linear regression equation can be determined.

Determination of Total Octyl Methoxycinnamate Levels in Sunscreen Gel Samples

The sample is weighed as much as 1 gram and put into a beaker glass. Then 10 ml of isopropyl alcohol was added and stirred using a magnetic stirrer for 5 minutes. The sample was then centrifuged for 10 minutes at 1000 rpm. The solution was centrifuged for 10 minutes. Solution clear centrifuge results are taken and transferred into a 10 mL volumetric flask, then isopropyl alcohol is added up to the mark. The sample was then pipetted 1.0 mL, put into a 10 mL volumetric flask and added isopropyl alcohol up to the mark. After that, 1.0 mL of the sample solution was pipetted into a 10 mL volumetric flask and isopropyl alcohol was added up to the mark. Pipette the sample solution again as much as 1.0 mL, put it into a 10 mL volumetric flask and add isopropyl alcohol up to the mark to obtain a level of 1000 ppm. After obtaining the 1000 ppm level, the absorbance was measured at the maximum absorbance wavelength using a visible spectrophotometer and the treatment was repeated 3 times for each sample. The levels of total flavonoid compounds were determined using the linear regression equation of the calibration curve so that the total flavonoid levels in the sample were obtained.

RESULT AND DISCUSSION

Thin Layer Chromatography (TLC) Analysis

The stationary phase used in this solution is silica gel 60GF_{254}. Silica gel 60 F_{254} is silica gel with a pore size of 60 A. while F is a fluorescent/phosphorescent material with a wavelength of 254 nm (Simaremare, 2019). Spotting is carried out using a capillary tube and then put into a chamber that has been saturated using filter paper. If the elution process has reached the upper limit then the spots are observed under 254 nm UV light. The positive results contained octyl methoxycinnamate UV filter Rf stains on facial sunscreen gel samples that were the same as the standard standard stain length, namely octyl methoxycinnamate. Based on the detection results using 254 nm UV light for the three

Figure 1. TLC Results of Facial Sunscreen Gel Samples Under 254 nm UV Light (Standard Solution; Sample X; Sample Y; Sample Z)

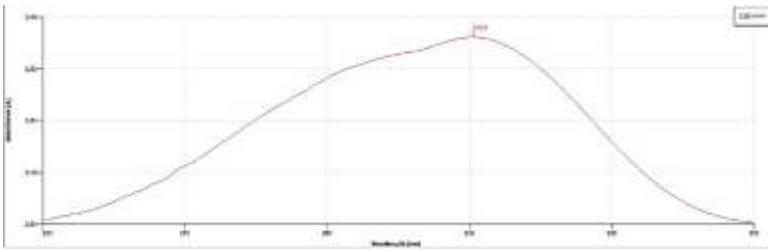

Figure 2. Maximum Wavelength of Octyl Methoxycinnamate

facial sunscreen gel samples consisting of samples X, Y, and Z, it can be interpreted that the facial sunscreen gel sample contains positive octyl methoxycinnamate.

The Rf value resulting from the standard standard of comparison and the three samples of facial sunscreen gel X, Y, and Z is 0.875. This can be seen from the results of observations made under 254 nm UV light, namely the appearance of purple stains on the reference standard spotting and the three samples X, Y, and Z and the same/parallel creepage distance (Figure 1). The purple stains result from the interaction between UV light and the chromophore groups which are bound by the auxochromes present in the stains (Oktaviantari et al., 2019). The results of the qualitative test it was continued for quantitative testing, which aims to measure the levels of octyl methoxycinnamate contained in each of these samples.

Determination of the Octyl Methoxycinnamate Maximum Absorbance Wavelength and Determination of the Curve Standard Octyl Methoxycinnamate

The working wavelength (λ_{max}) for OMC determination was found to be 310,5 nm (Figure 2), resulting in maximum absorbance values, with isopropyl alcohol being the best solvent for OMC. The maximum absorbance wavelength is used to determine the octyl methoxycinnamate standard curve as well determine octyl methoxycinnamate levels in the sample.

The octyl methoxycinnamate standard curve was determined using octyl methoxycinnamate standard solutions with a concentration of 0.05%; 0.1%; 0.15% 0.25%; 0.3%; and 0.35%. The series of levels were measured for absorbance at a wavelength of 310.5 nm. From the measurements, the results of the linear regression equation are y = 1136.4x + 0.3454 with value of relational coefficient (R^2) is 0.9955. Linearity is the ability of the analytical method to respond directly or with the help of

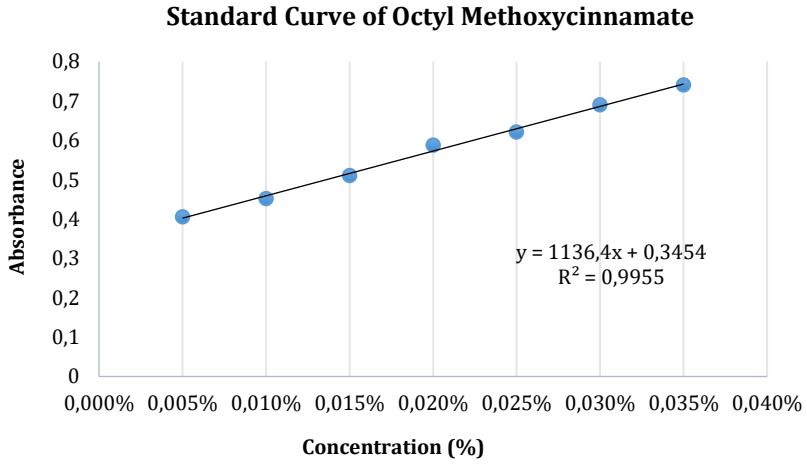

Figure 3. Standard Curve of Octyl Methoxycinnamate

a good mathematical transformation that is proportional to the analyte content in the sample (Haresmita & Pradani, 2022). A linear relationship is said to be ideal if it has values b=0 and r=+1 or -1 depending on the direction of the line (Harmita, 2004). The absorbance results from the seven concentration series solutions were then used as a standard curve (Figure 3).

The value of the relation coefficient is close to 1, indicating that there is a linear relationship between the concentration and absorbance of the reference standard solution, as seen in Figure 3. The linear standard curve indicates that when the absorbance increases, the concentration also increases proportionately. The relationship between concentration and absorbance is described in terms of r, and the sample solution's absorbance must also fall within the standard curve series' absorbance range (Suharyanto & Hayati, 2021).

Determination of Octyl Methoxycinnamate Levels in Sunscreen Gel Samples

One gram of sample was added with 10 mL of isopropyl alcohol, and stirred with a magnetic stirrer for 5 minutes. The sample solution was homogenized and then centrifuged at 1000 rpm for 10 minutes. Centrifugation was carried out so that the solution is easier to separate with the precipitating agent (Meixner et al., 2015). In this study, isopropyl alcohol was used as standard and sample solvent. Based on the chemical structure of the octyl methoxycinnamate compound, it is relatively polar, so a polar solvent is needed so that the sample can dissolve completely (Hanson et al., 2015). Isopropyl alcohol has a hydrophilic –OH group which will attract water molecules, so it can dissolve polar water molecules. In addition, isopropyl alcohol also has an alkyl group $–CH_2$. The alkyl group $–CH_2$ in isopropyl alcohol is an alkyl group that has a short carbon chain, so its polarity is very high. Therefore, octyl methoxycinnamate and isopropyl alcohol can mix and dissolve perfectly (Syahirah & Cahyati, 2021).

Octyl methoxycinnamate was chosen in this study because octyl methoxycinnamate (OMC) represents the most common sunscreen agent in commercially available cosmetics and has an excellent UV absorption curve and good oil solubility. Octyl methoxycinnamate is an ester derivative of cinnamic compounds which has a chromophore group in the form of an aromatic benzene ring connected by a conjugated double bond with an ester group and an ausochrome ether group which is directly connected to the chromophore system next to the benzene ring (Abdillah et al., 2017). the octyl structure of methoxycinnamate can be seen in Figure 4.

Determination of octyl methoxycinnamate levels is done by measuring the absorbance of the sample. Octyl methoxycinnamate concentration in the sample was calculated by subtituting the absorbance value of the sample solution into the linear regression equation of the solution y = 1136.4x + 0.3454. The results of measuring octyl methoxycinnamate levels in three samples of sunscreen gel are shown in Table I.

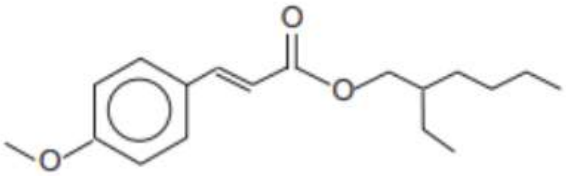

Figure 4. Structure of Octyl Methoxycinnamate (Narloch & Wejnerowska, 2021)

Table I. Octyl Methoxycinnamate Levels in Samples Gel Sunscreen

Sample Code	Replication	Octyl Methoxycinnamate Levels (%)	Average Octyl Methoxycinnamate Levels (%)	RSD (%)
X	1	0.1725	0.155	0,015
	2	0.1420		
	3	0.1522		
Y	1	0.8114	0.824	0,048
	2	0.7830		
	3	0.8782		
Z	1	2.1172	1.954	0,291
	2	2.1281		
	3	1.6172		

Based on the results above, it was stated that three samples contained Octyl Methoxycinnamate. The levels of each sample were X 0.155%; Y 0.824%; and Z 1.954%. As stated in the Regulation of the Head of Agency Drug and Cosmetic Supervision of the Republic of Indonesia No. HK.03.1.23.08.11.07517 Years 2011 concerning Material Technical Requirements Cosmetics, BPOM specifies that the maximum concentration of octyl methoxycinnamate that is allowed in the preparation sunscreen is equal to 10%. Based on the determination data contained in the sampel, the three samples contains octyl methoxycinnamate that were safe for usage (≤ 10%) (BPOM RI, 2011). The results of this research corroborate the previous research by Purwaniati, pipit novita et al., (2019) three samples tested, two samples containing octyl methoxycinnamate that were safe for usage.

Precision test shows repeatability the results of measurements carried out with 9 repetitions. Score precision expressed as % KV or relative standard deviation (RSD). Precision measurement results on sample got RSD value <2%. Result obtained the relative standard deviation of the sample sunscreen gel were X 0.015; Y 0.824; and Z 1.954. So that the measurement results on this research has complied requirements, namely <2% (Harmita, 2004).

CONCLUSION

The three samples of sunscreen gels X, Y, and Z circulating in Magelang Regency tested positive for octyl methoxycinnamate UV filters. The results of the TLC study showed that samples X, Y, and Z were purple in color and had same Rf value between sample and standard. The content of octyl methoxycinnamate in sample X was 0.155%; Y 0.824%; and Z 1.954%. The results obtained by the relative standard deviation (RSD) of the sunscreen gel sample were X 0.015%; Y 0.048%; and Z 0.921%. It can be concluded that the three sunscreen gel samples tested are safe for for usage by the general public.

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