

IDENTIFICATION OF CORTICOSTEROID HARD DRUG CONTENT IN WHITENING COSMETIC PREPARATIONS CIRCULATING IN GORONTALO CITY

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ABSTRACT

This study aims to identify the corticosteroids in whitening cosmetic preparations circulating in Gorontalo City. The method used in this study was thin layer chromatography (TLC), with a qualitative approach and a descriptive (observational) type of research. The results showed that identification in the UV laboratory at a wavelength of 254 nm showed that from the 5 cream samples and 5 lotion samples for whitening cosmetic preparations tested, four of them were positive for the strong drug class of corticosteroid betamethasone valerate, namely with an Rf value on (cream sample E 0, 91), (lotion sample A 0, 91), (lotion sample B 1.0), and (lotion sample C 1.0) and have Rf values identical or similar to standard Rf values under the same chromatographic conditions.

Keywords: Corticosteroid, Whitening Cosmetic, Chromatography, Thin Layer.

INTRODUCTION

Cosmetics at this time have become a primary need for society, especially for women who think that beauty is synonymous with bright skin, cosmetics are used to treat and add to attractiveness. Cosmetics are materials or preparations intended for use on the external parts of the human body such as the epidermis, hair, nails, lips and external genital organs, or teeth and oral mucous membranes especially for cleaning, perfuming, changing appearance and/or improving body odor or protecting or maintaining the body in good condition, one way that is often used to care for the body is by using whitening cosmetics, but currently many illegal whitening cosmetic products circulating in the market contain dangerous ingredients [14].

According to the records of the World Health Organization WHO presentation on the use of chemicals in 2019 there were 40 types

of skincare consisting of 26 local product brands and 16 imported product brands that contain dangerous chemical ingredients that can cause skin diseases caused by cosmetic products that contain chemicals in them. dangerous [4].

In BPOM RI found a number of whitening cosmetic products that contained dangerous chemicals, namely mercury, hydroquinone, retinoic acid, and even corticosteroids. Cosmetics have become one of the important needs used by people, especially women who want to look attractive. The growing stigma that beautiful is synonymous with white skin has resulted in cosmetics that are in great demand by the public, namely whitening cosmetics. The high market demand for whitening cosmetics is certainly a profitable opportunity for businesses in the cosmetic sector. However, with this large opportunity, it becomes a loophole that can be exploited by

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business actors to produce illegal whitening cosmetics that use hazardous ingredients. In 2018, illegal cosmetics were found with a total of 126 billion and almost half of that amount was a type of whitening cosmetic. This illegal whitening cosmetic is sold in units and in packages consisting of day cream, night cream, liquid soap and toner [12].

There are quite a number of cosmetic dosage forms in circulation [7]. stated that most cosmetic preparations are in the form of liquids, creams, suspensions and powders. Of the several dosage forms, cream is the most widely chosen as a cosmetic dosage form, especially for skin care products. In accordance with the intended use, whitening cosmetics on the market can be in the form of skin lightening to brighten the skin and skin bleaching to fade black spots. The active ingredients used are of course different. Skin lightening usually contains acidic ingredients that function to brighten the skin, while skin bleaching usually contains active ingredients such as hydroquinone, mercury, corticosteroids or other ingredients that can whiten or fade black spots on the skin. The attractiveness of this product is quite high, especially for women who want to have white skin, so that many ordinary people make illegal cosmetic products that contain dangerous ingredients that have instant effects.

Referring to research data carried out by, [12]. BPOM RI found an increase in the number of findings of illegal whitening cosmetics in Gorontalo City. From the results of the testing by the BPOM, it was reported that in the Gorontalo area alone there were 2617 samples or 1.8% of illegal cosmetic products containing hazardous ingredients, one of which was corticosteroids which are closely related to whitening cosmetics [4].

Topical corticosteroids are one of the components of a formula that is widely used uncontrolled for facial whitening creams. Topical corticosteroids are often added as a

component to formulas in whitening and lightening products. The types of steroids that are often misused in cosmetics are dexamethasone, betamethasone, betamethasone 17-valerate, prednisone, prednisolone, methyl prednisolone, triamcinolone acetonide, fluocinolon acetonide, cortisone acetate. Steroids are added in whitening creams because they suppress melanin synthesis without destroying melanocytes. In addition, topical corticosteroids can reduce irritation or inflammation due to hydroquinone and tretinoin, the use of corticosteroids alone can have adverse side effects for skin health [9].

As mentioned by [1]. Topical corticosteroids have many benefits, but if used inappropriately they can cause adverse effects. The reasons underlying the misuse of topical corticosteroids on the face are skin lightening agents and overcoming acne. The most common side effect is acne and redness (exacerbation of lesions) that already existed, followed by facial dependence on corticosteroids, this occurs due to the use of corticosteroids that are not in accordance with their function.

Corticosteroids are actually drugs to treat inflammatory reactions in the body. Steroid drugs work by dilating capillaries and suppressing an overactive immune system. Steroids are only for treating skin problems, such as nummular dermatitis, atopic dermatitis, allergic and irritant contact dermatitis, psoriasis, bullous disease, and so on [9].

As reported by BPOM RI's official website, steroids are often used in cosmetic products, especially whitening preparations because steroids can be used as an anti-inflammatory which has a hypopigmentation or whitening effect. These effects are often abused by illegal cosmetic manufacturers. At the beginning of use, it will indeed give a stunning effect, where facial skin looks shiny,

glowing and blushing. However, long-term use will cause the skin to become inflamed. In lay people, the term "thin skin" is often known, blood vessels are visible, the skin becomes more sensitive, the face turns red, or the face blackens (spots) which cannot be completely lost (irreversible). If the use of the cream is stopped suddenly it usually causes side effects.

Side effects that can arise from using short-term topical corticosteroids such as reddish spots without itching, acne and excessive hair growth on the skin, while side effects that can arise from using long-term topical corticosteroids include seborrheid, perioral dermatitis, steroid rosacea, telangiectasia, cellulite, bacterial and fungal infections, acne, skin atrophy, pigmentation disorders, and rebound phenomena [3].

In identifying the content of topical corticosteroids in cosmetics, one of the methods recommended by BPOM is the Thin Layer Chromatography (TLC) technique. This is supported by research conducted by [8], who examined the Identification and Determination of Hydroquinone in Several Cosmetic Creams Using the Thin Layer Chromatography Method. The use of the TLC method offers a simpler, less expensive procedure, is able to analyze samples quickly and simultaneously, and allows multiple separation techniques and detection procedures to be applied. The TLC method can determine analyte levels, resolution values, coefficient of variance values, and recovery which describes the precision and accuracy of the method. Use of this method can eliminate errors caused by spot removal or extraction errors.

With the various factors and matters that have been stated above, the researcher is interested in further studying corticosteroids in whitening cosmetic preparations circulating in Gorontalo City.

RESEARCH METHODS

2.1 Tools and Materials

The tools used in this study were TLC plates, dropping pipettes, capillary pipettes, camber, stir bars, analytical balances, 25mL, 50mL measuring cups, 250mL glass beakers, centrifugation tubes, water baths, centrifuges, labels, ovens, freezers.

The materials used in this study were (N-hexane, ethyl acetate) silica gel GF245, and 10 samples of whitening cosmetics circulating in Gorontalo City.

2.2 How it Works

1. Collection of whitening cosmetic products circulating in the city of Gorontalo used as samples in this study.
2. Samples that have been collected are given labels that read Cream A to E as well as Lotion samples A to E.
3. Prepare tools and materials to be used in this study.
4. Prepare a TLC plate measuring 8x3 cm and give an upper limit of 1 cm and a lower limit of 1 cm then heat in the oven at 150°C for 10 minutes.
5. Saturate the standard solution into the chamber using the eluent ratio of N-hexane : Ethyl acetate (8:2).
6. Make a standard solution by weighing 2 grams of Betamethasone Valerate, put it in a beaker and dilute it with 96% ethanol and then stir it until it is homogeneous.
7. Preparation of the sample solution by weighing 700 mg of sample into each 5 ml centrifugation tube and adding 3 ml of 96% ethanol, then heating it in a water bath at 60°C for 15 minutes.
8. Then cool it to room temperature. Next, insert the centrifugation tube that already contains the sample into the freezer for 1 hour, then insert the centrifugation tube into the centrifuge for 20 minutes at 1500 rpm.
9. Spotting on the lower limit of the TLC plate, then inserting the plate into the chamber containing the solution then wait

for the solution to rise to the upper limit of the TLC plate, after that observe the TLC plate under 254 nm UV light.

RESEARCH RESULT

3.1 Research Result

Table 4.1 Product Description and Information on Cosmetic Products.

Cosmetic Preparations	Trademark	Utility	No.R BPO M	Expire d	Composition
Cream A	Yes	Yes	No	No	No
Cream B	Yes	Yes	No	No	No
Cream C	Yes	Yes	No	No	No
Cream D	Yes	Yes	No	No	No
Cream E	Yes	Yes	No	No	No
Lotion A	Yes	Yes	No	No	No
Lotion B	Yes	Yes	No	No	No
Lotion C	Yes	Yes	No	No	No
Lotion D	Yes	Yes	No	No	No
Lotion E	Yes	Yes	No	No	No

Source: Personal research data, 2023

Based on product descriptions and cosmetic product information from the table above is a description of the observed whitening cosmetics. The whitening cosmetic products observed were whitening cosmetic preparations such as creams and lotions. From the table above it can be seen that there are still many whitening cosmetic products that contain hazardous ingredients and do not have a BPOM registration number. Of the 10 samples of whitening cosmetic preparations in circulation, all of them had the name or trade

mark of the cosmetic product and their use on the cosmetic packaging, but the 10 samples of the cosmetic preparations did not have an expiration date and composition on the cosmetic preparation packaging.

3.2 Qualitative Test Results for Whitening Cosmetic Samples

The following are the results obtained from the identification of corticosteroid drug content in whitening cosmetic preparations circulating in Gorontalo City using the Thin Layer Chromatography test method while the TLC method was observed using UV light at a wavelength of 254 nm. The test results can be seen in table 4.2.

Table 4.2. Qualitative Test Results for Whitening Cosmetic Samples

Plate Code (KLT)	Test Variables (Cream)	Observation Results Under UV-254 nm Light	RF value
Plate A1	Standard solution	There are spot stains	1.0
	Sample Cream A	No stain spots	-
	Sample Cream B	No stain spots	-
Plate B2	Standard solution	There are spot stains	1.0
	Sample Cream C	No stain spots	-
	Sample Cream D	No stain spots	-
Plate C3	Standard solution	There are spot stains	0,91
	Sample Cream E	There are spot stains	0,91

Plate Code (KLT)	Test Variables (Lotions)	Observation Results Under UV-254 nm Light	RF value
Plate	Standard	There are	0,9

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C3	solution	spot stains	1
	Sample	There are	0,9
	lotion A	spot stains	1
Plate	Standard	There are	1.0
D4	solution	spot stains	
	Sample	There are	1.0
	lotion B	spot stains	
	Sample	There are	1.0
	lotion C	spot stains	
Plate	Standard	There are	1.0
E5	solution	spot stains	
	Sample	No stain	-
	lotion D	spots	
	Sample	No stain	-
	lotion E	spots	

Source: Personal research data, 2023

From the results of the identification of the strong drug content of corticosteroids in whitening cosmetic preparations carried out on 10 samples of whitening cosmetics circulating in Gorontalo City using the Thin Layer Chromatography method using a ratio of N-hexane and ethyl acetate eluents (8: 2) it was found that 4 whitening cosmetics positive for corticosteroids, the Rf values in standard solution and sample solution were the same, namely 0.91 and 1.0, it was concluded that the four whitening cosmetic products labeled cream E, lotion A, lotion B, and lotion C were positive for containing hard drugs. namely corticosteroids.

DISCUSSION

In the study of identifying the content of corticosteroids in whitening cosmetic preparations circulating in Gorontalo City using the Thin Layer Chromatography method to see whether or not there is corticosteroid content in whitening cosmetic preparations, this research was conducted in the Anaisis Chemistry Laboratory, Department of Pharmacy, Gorontalo State University for 3 days starting from 13-15 June 2023 and in the research process, the preparation process for testing was carried out for 1 day.

As mentioned by [14]. cosmetics at this time have become a primary need for society, especially for women who think that beauty is synonymous with bright skin, cosmetics are used to treat and add to attractiveness. This is what triggers many whitening cosmetic agents to misuse hazardous materials as additives in whitening cosmetics, one of the most frequently abused dangerous ingredients is corticosteroids. The reason for choosing corticosteroid drugs for observation is because corticosteroids themselves are a class of hard drugs that function to treat inflammation that are easily obtained freely and can suppress melanin synthesis in long-term use so that they can provide a fast and instant whitening effect.

The instant whitening effect provided by illegal whitening cosmetics has attracted many people to use these products. This makes the attractiveness of whitening cosmetics increase so that sometimes people pay less attention to safety aspects and the detrimental side effects caused by the use of illegal whitening cosmetics [10]. One of the cosmetic preparations that are often used are creams and lotions, so the samples used in this study were whitening creams and lotions.

At this stage of the research process, it begins with taking samples of whitening cosmetics circulating in Gorontalo City using the purposive sampling method, namely samples taken that meet predetermined criteria. 5 samples of whitening lotions by looking at different sample brands. The samples that have been collected are given labels that read cream A, cream B, cream C, cream D, and cream E as well as 5 lotion samples, namely lotion A, lotion B. lotion C, lotion D, and lotion E, and then a qualitative test is carried out. in whitening cosmetic preparations sold in Gorontalo City.

In the early stages, centrifugation of the sample aims to separate substances based on molecular weight by providing centrifugal force so that the heavier substances will be at

the bottom while the lighter substances will be above [3]. At this stage, 700 mg each of the whitening cosmetic samples was weighed into each 5 ml centrifugation tube and added 3 ml of 96% ethanol solvent, then heated in a water bath at 60°C for 15 minutes until the sample melted. then cool to room temperature. Next, insert the centrifugation tube which already contains the sample into the freezer for 1 hour, then insert the centrifugation tube into the centrifuge for 20 minutes at a speed of 1500 rpm. The next stage is the preparation of a standard solution in this study the standard solution used is Betamethasone Valerat, at this stage the standard solution is weighed as much as 2 grams put into a beaker and diluted with 96% ethanol then stirred until homogeneous, the selection of using 96% ethanol in this stage because this solvent is relatively non-toxic, easy to obtain and often used in cosmetic dilution.

The analysis with TLC begins with applying a small aliquot of the sample at one end of the stationary phase (TLC plate). The stationary phase used in this study was Silica gel GF245 with a size of 8x3 cm and then given an upper and lower limit of 1 cm each on the TLC plate. Then the lower boundary of the stationary phase containing the sample was dipped into the mobile phase (single solvent or a mixture of two pure solvents) in the mobile phase chamber used in this study using N-hexane and ethyl acetate solvents with an eluent ratio of 8:2 which has non-polar properties because it uses N-hexane (non-polar) solvents with a higher ratio much compared to ethyl acetate (semipolar) the selection of this eluent was based on the results of the orientation of the eluent that had been carried out which in this eluent produced the best separation with the highest number of stains after detecting spots on a 254 nm UV lamp, choosing to use UV 254 nm due to the fluorescence present on the plate, whereas at

366 nm UV fluorescence did not occur on the TLC plate [2].

The eluent consists of several types of solvents with a certain ratio that can separate a compound component [11]. Stated that the eluent is a solvent that is used as a mobile phase that carries and separates the components of the sample substance through the stationary phase in the elution process. The best eluent is the eluent that can separate these compounds properly. The polarity of the solvent in the eluent used reflects the polarity of the compound. Based on the standard requirements issued by BPOM, steroids are compounds that are (non-polar) in nature. So the use of N-hexane and ethyl acetate with an eluent ratio of 8:2 produces the best separation because the polarity of the solvent has the same properties as the compound to be identified [6].

Based on a decision issued by BPOM in 2011, corticosteroids are included in one of the ingredients that are prohibited for use in cosmetics because they can cause various kinds of dangerous side effects. Even so, in the city of Gorontalo corticosteroids are often misused by some skin lightening agents in the form of creams and lotions. The most abused corticosteroids as an additional ingredient in whitening cosmetics are corticosteroids with super potent, potent, and moderate potential. This study aims to identify the content of corticosteroids in whitening cosmetic preparations circulating in Gorontalo City such as cream and lotion preparations using the Thin Layer Chromatography (TLC) method. After the elution process was carried out using two organic solvents, it was followed by the process of identifying the presence of corticosteroids in the sample. Identification was carried out using TLC where the R_f value was the parameter. The use of the TLC method offers a simpler, cheaper procedure, capable of analyzing samples quickly and simultaneously. The results of the identification test in the

laboratory after the TLC plate was observed under UV light at a wavelength of 254 nm, showed that of the 10 samples of creams and lotions for whitening cosmetic preparations tested, four of them were positive for the strong drug class of corticosteroid betamethasone valerate, namely with an Rf value on (cream E sample). 0.91), (lotion sample A 0.91), (lotion sample B 1.0) and (lotion sample C 1.0) and have Rf values identical or similar to standard Rf values under the same chromatographic conditions [5].

Based on the research data as contained in table 4.2, it can be seen that no spots were observed on the TLC plate code A1 (cream sample A, cream sample B), TLC plate code B2 (cream sample C, cream sample D), and TLC plate code E5 (lotion sample D, lotion sample E). The appearance of spot stains can be seen on the standard Betamethasone Valerate section only with an Rf value of 1.0, but it is different when observing the TLC plate code C3 (cream sample E, sample lotion A) and the TLC plate code D4 (lotion sample B, sample lotion C) it is found that , on the sample plate there were visible spots with Rf values of 0.91 and 1.0. With the similarity of the results of the Rf values of the standard solution and the sample, it was highly probable that the four whitening cosmetics contained corticosteroids. Retention/retardation factor (Rf) is a value or measure which is obtained based on the spot position of each solute on a thin layer chromatography plate. The Rf value is obtained by dividing the value between the distance from the start of the spotting of a compound until the compound spot stops when the elution process is complete (a) divided by the elution distance (b). The Rf value has a value range from 0.0 to 1.0, this value can vary due to various factors, such as sorbent quality, humidity, plate thickness, elution distance, and ambient temperature. the same as the standard [11].

Based on the 2011 BPOM regulations, the Rf value is a value that expresses the ratio between the distance traveled by the compound to the distance traveled by the mobile phase in the TLC plate, the addition of a slightly polar solvent such as ethyl acetate into a non-polar solvent such as N-hexane will significantly increase the Rf value. The Rf obtained by betamethasone valerate is 0.35-0.2 with blue-gray and purplish-blue spots. Supported by the results of research conducted by [3], states that face cream is tested positive for corticosteroids if it has an Rf value that is identical or similar to a standard Rf value under the same chromatographic conditions. The requirements for the Rf value of corticosteroids in cream preparations are not less than 0.35 and not more than 0.2 for betamethasone valerate [5].

CLOSING

Conclusion

This research is a descriptive qualitative study that aims to identify the content of corticosteroids in whitening cosmetic preparations circulating in Gorontalo City using the Thin Layer Chromatography method. Based on the results of a study conducted from 10 samples of whitening cosmetics, there were 4 cosmetics that positively contained corticosteroids. Corticosteroids were found in samples with Rf values (face cream sample E had an Rf value of 0.91 and lotion sample A had an Rf value of 0.91), (lotion sample B Rf value 1.0, and lotion C sample Rf value 1.0)

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