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Research Article

Identification of Hydroquinone in Cosmetics Circulating in Kotamobagu City Using Chromatography Method

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ABSTRACT

The use of hazardous materials in facial whitening creams can endanger the health of users. Excessive use of hydroquinone can cause ochronosis, where the skin feels like it is burning and itching. Creams containing hydroquinone are widely circulated in the community and often cause undesirable effects. This study aims to analyze hydroquinone in cosmetics circulating in Kotamobagu City qualitatively using the Thin Layer Chromatography (TLC) method. This study was conducted using a qualitative method using TLC with silica gel GF254 as the stationary phase, methanol:chloroform (1:1) as the 8 mL mobile phase, and 5 samples of whitening cream. The samples that had been eluted on the TLC plate were analyzed using UV light to detect spots and calculate the Rf value. The results showed that the Rf value of the hydroquinone standard was 0.87 with a blackish-brown stain color. Meanwhile, the Rf value and stain color of samples A, B, C, D, and E were 0.37 brownish yellow, 0.62 purple, 0.87 blackish brown, 0.75 blackish brown, and 0.80 blackish brown, respectively. Based on the results of qualitative analysis using TLC, facial whitening cream cosmetics circulating in Kotamobagu City with samples C, D, and E were positive for containing hydroquinone.

Keywords: cosmetics; facial whitening cream; hydroquinone; kotamobagu; thin layer chromatography

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INTRODUCTION

The rapid development of the food and cosmetic industry requires strict supervision of the content of the ingredients used. Facial whitening cream is a cosmetic product containing a mixture of chemicals and other ingredients intended to brighten the skin (Warsi et al. 2022). Whitening creams contain active ingredients that can suppress or inhibit melanin formation, thus providing a whiter skin tone. One of the active ingredients in these whitening creams is hydroquinone (Kurniawan et al. 2022). Hydroquinone is one of the ingredients used as a mixture in whitening creams carried out by irresponsible individuals. Regulation issued by the Head of the Indonesian Food and Drug Authority Number 23 in 2019 concerning Technical Requirements for Cosmetic Ingredients has stipulated that the use of cosmetics containing hydroquinone compounds as whiteners is prohibited and is permitted only for the use of nail polish with the provision that the content is less than or equal to 0.02% (Megasari et al. 2022).

The use of hazardous materials in facial whitening creams can endanger the health of the user. The use of whitening creams containing hazardous materials such as hydroquinone does make the skin smooth and white, but then it will settle under the skin, and after years it will turn blackish blue and will trigger cancer. Creams containing hazardous materials are usually sold at affordable prices and promise satisfactory results. The cream usually does not list the chemical content, labeling, side effects and expiration date. The effects caused by the use of hydroquinone are characterized by skin irritation, vitiligo and even exogenous ochronosis (skin hyperpigmentation). Excessive use of hydroquinone can be risky to health because it can cause cancer (carcinogenic) and skin disorders, as well as ochronosis (blackening of the skin) (Nasiroh et al. 2024).

Research conducted by Tan et al. (2020), showed that from 2014 to 2019 there were 88 patients experiencing exogenous ochronosis due to hydroquinone contained in whitening cream, 92.04% of the patients were women. Research by Rubiyati and Setiawan (2016), reported that in the United States, the use of hydroquinone cream of more than 2% can cause skin irritation, itching, redness, and a burning sensation. Long-term use of 5-10 years will cause the skin to become bumpy like sand and bluish-black or ochronosis. Runtukahu et al. (2021), reported that a 39-year-old female patient experienced acquired exogenous ochronosis due to the use of 4% hydroquinone cream for skin whitening. The main symptoms of hydroquinone poisoning included diffuse hyperpigmentation of the face and neck, the appearance of blue-black macules, and the development of bluish-black hyperpigmentation in the areas of application. This condition occurred after three months of hydroquinone cream use. Exogenous ochronosis is a disorder that is often overlooked and requires attention for prevention.

Hydroquinone analysis in facial whitening cream preparations has been carried out widely, both qualitatively and quantitatively. Qualitative analysis can

be carried out using the color reaction method and thin layer chromatography (TLC), while quantitative analysis can be carried out using UV-VIS spectrophotometry and High Performance Liquid Chromatography (HPLC) (Pisacha et al. 2023). Chromatography is a simple, efficient and economical analytical technique for identifying components in a mixture, both qualitatively and semi-quantitatively. In cosmetic analysis, TLC is useful for analyzing the content of active ingredients such as hydroquinone, retinoic acid, and prohibited dyes and preservatives (Husna and Mita 2020).

There have been several cases of side effects from the use of whitening creams among the people of Kotamobagu, but no studies have tested the whitening cream preparations circulating in the community. Therefore, this study aims to qualitatively analyze hydroquinone in whitening cream cosmetic preparations A, B, C, D, and E circulating in Kotamobagu using Thin Layer Chromatography (TLC).

MATERIALS AND METHODS

Materials

The materials used were chloroform (PT. Kimia ARD), ethanol (PT. Kimia ARD), methanol (PT. Kimia ARD), N-hexane (PT. Kimia ARD), TLC plate Gf254 (Chromatography MERCK), and 5 samples of whitening cream A, B, C, D, and E.

Methods

Sample Preparation

The samples used were facial whitening creams circulating in the Kotamobagu city area and were taken randomly. Furthermore, they were prepared by weighing samples A, B, C, D, and E as much as 0.1 gram; then each sample was dissolved using 8 ml of 96% ethanol; then a hydroquinone comparison standard was made as much as 0.1 gram and dissolved with 8 ml of ethanol.

Organoleptic Testing

The prepared whitening cream samples were tested organoleptically to identify color, odor, and texture.

Qualitative Analysis

Analysis of hydroquinone content in samples was carried out qualitatively using the Thin Layer Chromatography (TLC) method. The first step was to prepare a TLC plate as a stationary phase and a mobile phase, namely methanol:chloroform (1:1), making as much as 8 ml for each sample. Then the hydroquinone standard and samples A, B, C, D, and E were spotted on different

TLC plates. The TLC plate containing the sample was inserted into a vessel containing a mobile phase that had been saturated in advance.

Identification using UV Light 254 nm and 366 nm

After being analyzed using the TLC method, the spots on the TLC plate were observed under UV light at 254 nm for the hydroquinone standard and UV light at 366 nm for 5 samples that were identified qualitatively to see the hydroquinone content in the 5 samples, which were marked by the color of the spots on the TLC plate.

Calculation of Rf Value

After qualitative analysis is carried out, the Rf value is calculated based on the stain results obtained on samples A, B, C, D, and E using the formula:

$$Rf = \frac{distance\ traveled\ by\ the\ sample}{distance\ traveled\ by\ the\ solvent}$$

RESULTS

The organoleptic test results can be seen in Table 1, where each sample had a different color, odor, and texture. Whitening cream samples C, D, and E were characterized as containing hazardous ingredients; namely, they were yellow in color, had a pungent odor, and had a soft to sticky texture.

Table 1. Organoleptic Test Results

Sample	Color	Odor	Texture
Whitening cream A	White	Pungent	Sticky
Whitening cream B	Yellowish white	Not too pungent	Soft
Whitening cream C	Yellow	Pungent	Soft
Whitening cream D	Yellow	Pungent	Sticky
Whitening cream E	Yellow	Distinctive and pungent	Sticky

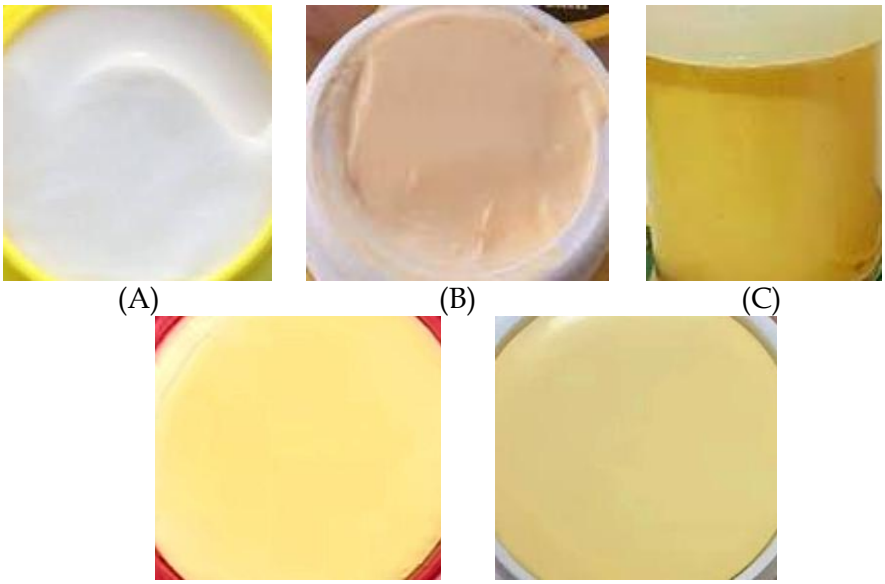

Figure 1. Whitening cream preparation

The results of qualitative analysis of hydroquinone using TLC can be seen in Table 2. The Rf value of the hydroquinone standard is 0.87 with a blackish-brown stain color. Meanwhile, the Rf value and stain color of samples A, B, C, D, and E are 0.37 brownish yellow, 0.62 purple, 0.87 blackish brown, 0.75 blackish brown, and 0.80 blackish brown, respectively. Whitening cream samples A and B were negative, while samples C, D, and E were positive for hydroquinone.

Table 2. Results of qualitative analysis of hydroquinone using TLC

Sample	Result	Hydroquinone Standard	Rf Value	Color	Description
A			**Standard: 0,87** Sample: 0,37	**Standard: Blackish brown** Sampel: Blackish brown	- (negative for hydroquinone)
B			**Standard: 0,87** Sample: 0,62	**Standard: Blackish brown** Sampel: Purple	- (negative for hydroquinone)

C			**Standard: 0,87** Sample: 0,87	**Standard: Blackish brown** Sampel: Blackish brown	+ (positive for hydroquinone)
D			**Standard: 0,87** Sample: 0,75	**Standard: Blackish brown** Sampel: Blackish brown	+ (positive for hydroquinone)
E			**Standard: 0,87** Sample: 0,80	**Standard: Blackish brown** Sampel: Blackish brown	+ (positive for hydroquinone)

DISCUSSION

Facial whitening creams circulating in the Kotamobagu City market were taken randomly to be used as samples to be analyzed for the content of hazardous materials in cosmetics, namely hydroquinone, where the selected product brands are products that have been widely used by the Kotamobagu community and have been circulating in the market for a long time. Hydroquinone is an ingredient that is often added to whitening creams with the aim of whitening the skin. The use of whitening creams containing hydroquinone has very dangerous effects, namely ochronosis, epidemic atrophy, eczema, bacterial and fungal infections, dermatitis, warts and acne (Kurniawan et al. 2022).

Based on the organoleptic test, sample A is white, has a sticky texture, and a pungent odor. Sample B is yellowish-white, has a soft texture, and has a mild odor. Sample C is yellow, has a soft texture, and has a mild odor. Sample D is yellow with a sticky texture and a pungent odor. Meanwhile, sample E is yellow, has a sticky texture, and has a distinctive, pungent odor.

Whitening creams containing hydroquinone can generally be identified by several physical characteristics. These creams are often yellow in color, have a strong odor, and have a smooth but sticky texture. This strong odor often comes from perfumes intentionally added to mask the smell of harmful chemicals, such as metallic odors. Furthermore, the cream's sticky texture is also an indication of the presence of harmful ingredients. This is due to the strong binding properties

of metals, which can bind to surrounding metal ions and create that texture (Charismawati et al. 2021).

Hydroquinone analysis was carried out qualitatively using the TLC method, one of the simple analysis methods used to confirm the chemical compounds contained in the sample with its working mechanism based on the principle of absorption and partition determined by the stationary phase (absorbent) and mobile phase (eluent). The solvent ratio used was methanol:chloroform (1:1) as much as 8 ml with the aim of binding/attracting the compound suspected to be hydroquinone to the elution limit of 5 cm and causing spots. A mixture of methanol and chloroform can attract hydroquinone compounds maximally with a ratio of 1:1 (Odumosu and Ekwe 2010).

The next step is to saturate the eluent in the chamber with the aim that the eluent evaporates to fill the chamber so that the eluent as a mobile phase will elute properly so that accurate chromatography results are obtained (Novitasari et al. 2024). The next step is to spot the sample and hydroquinone standard on the TLC plate. The spotting distance is 1.5 cm from the end of the TLC plate. This sample spotting distance is so that there is no direct interaction between the mobile phase and the sample. The lower edge distance is too small or the amount of mobile phase is quite large, so that the spotting spot comes into direct contact with the mobile phase so that some of the sample molecules will dissolve in the mobile phase. This can cause the results obtained in the elution to be invalid. The spotting should also not be too large because it can reduce the resolution (Husna and Mita 2020). In this separation, the TLC plate used as the stationary phase is silica gel GF254 because it is able to fluoresce well under UV light with a wavelength of 254 nm and there is a chromophore group that will show colored spots (Agustin et al. 2021).

The elution results on the TLC plate show the Rf value and spot color of each facial whitening cream sample. The Rf value and spot color of samples A, B, C, D and E were 0.37 blackish brown, 0.62 purple, 0.87 blackish brown, 0.75 blackish brown and 0.80 blackish brown. These results were compared with the standard hydroquinone standard, which has a concentration of 0.2-0.3% with an Rf value of 0.87 and blackish-brown spots under 366 nm UV light. Whitening cream samples A and B were negative for hydroquinone, this was due to the Rf value and stain color being different from the standard standard. Meanwhile, whitening cream samples C, D and E were positive for hydroquinone, because they had Rf values and stain colors similar to the standard. This is supported by research by Charismawati et al. (2021), who tested the hydroquinone content in whitening cream under the same conditions, namely using the TLC method with a mobile phase of methanol:chloroform (50:50) and the sample Rf values of 0.73 and 0.85 were declared positive for hydroquinone. Meanwhile, based on the results of research by Sinurat et al. (2024), it was stated that blackish-sbrown stains were positive for hydroquinone. This is in line with research by (Meilyda et al. 2024)

which stated that brown color was positive for hydroquinone with an Rf value of 0.76. Other research also stated that positive hydroquinone was indicated by a blackish brown color (Werdiningsih 2024).

The results obtained in this study were qualitative in nature to determine whether there was hydroquinone content in the facial whitening cream samples. The next recommended research is to analyze the hydroquinone content quantitatively so that the levels in cosmetic preparations can be determined.

CONCLUSIONS

Based on the results of qualitative analysis using TLC, facial whitening cream cosmetics circulating in Kotamobagu City, samples A and B were negative, while samples C, D, and E were positive for containing hydroquinone.

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