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Analysis of Rhodamin B in Lipstick Preparations in Padangsidimpuan City by Thin Layer Chromatography (TLC) Method

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A B S T R A C T

Rhodamin B is a synthetic dye in the form of a crystalline powder, green or reddish-purple, odorless, and in solution will be bright red with fluorescence or fluorescence. Rhodamin B is commonly used as a dye in the textile and papermaking industries. Rhodamin B can cause irritation in short-term exposure and has carcinogenic effects. This study aims to find out whether there is Rhodamin B in lipsticks circulating in the Padangsidimpuan City market. Samples were taken by *purposive sampling* and 5 red samples were obtained. To identify the presence of Rhodamin B, a thin-layer chromatography method was used using eluene as the phase of motion, namely methanol, ethyl acetate, ammonia (50:20:25) and a 254 nm UV lamp The results of the study showed that out of 5 lipstick samples identified, there was one lipstick sample that was identified as containing Rhodamin B.

INTRODUCTION

Cosmetics are ingredients or preparations intended to be used on parts of the human body (epidermis, hair, nails, lips and external genital organs) or teeth and oral cavity mainly to clean, increase attractiveness, change appearance, keep in good condition, improve body odor but are not intended to treat or cure a disease. Cosmetics come from the Greek word "kosmetikos" which means the skill of decorating and arranging the appearance to be more beautiful than the original (Muliyawan dan Suriana, 2013).

Lipstick in this case is the one that is usually used to beautify and emphasize the color of the lips. Lipstick is the most common type of cosmetic used by women. In America, it is believed that almost all of its women have worn lipstick. Thus, an increase in cosmetic marketing is only possible if the population increases (Mamdo, L.D dan Citraningtyas, F.G, 2013)

The selection of lipstick as a cosmetic must of course be based on the BPOM investigation in 2014, there are 9817 cosmetic products that do not have or meet the requirements, namely products that are distributed without permits and products with harmful or prohibited ingredients. From the results of the examination of the hazardous substances found are mercury, hydroquinone and the dye Rhodamin B. Rhodamin B is a synthetic dye, in the form of a crystalline powder, odorless, purplish-red in color, in the solution will be bright red with fluorescence (fluorescent). This dye agent is commonly used for the paint, textile and paper industries (Jusnita et all, 2017)

Short-term use of Rhodamin B on the skin can cause irritation to the skin. This synthetic dye can bind to organic proteins and macromolecules so that the skin becomes a storage place for Rhodamin B because the amount of Rhodamin B increases in the skin, so synthetic absorption of this substance can occur. Rhodamin B also exerts a detrimental effect on the lips if used as a dye in lipstick (Jusnita et all, 2017).

Thin-layer chromatography (TLC) together with paper chromatography (KKr) with its wide variety is generally referred to as planar chromatography. Thin-layer chromatography (TLC) was developed by Izmailoff and Schraiber in 1938. In thin-layer chromatography, the stationary phase is in the form of a uniform layer on a flat surface used by glass plates, aluminum plates, or plastic plates, although this planar chromatography can be said to be an open form of column chromatography (Rohman, A 2009).

THEORETICAL OVERVIEW

Lipstick is one of the cosmetic preparations that is polished to the lips which has the function of giving color, softness and health to the lips. Lipstick is meant to make the appearance more attractive. According to Tranggono and Latifah (2007), the main ingredients in lipstick are wax, oil, fat, acetoglycerides, dyes, surfactants, antioxidants, preservatives and fragrance agents. There are 2 dyes in lipstick based on their source, namely, natural dyes are dyeing substances obtained from roots, leaves, flowers and fruits. Such as green dye from suji leaves and orange dye from carrots. While synthetic dyes come from the reaction between two or more chemical compounds, for example such as Rhodamin B.

Basically, dyes according to their origin consist of artificial (synthetic) dyes and dyes derived from nature (natural). The use of natural dyes in making lipstick formulations is one of the alternative ways for the benefit of the public to be able to use lipstick lipsticks that contain dyes derived from nature (natural). Natural dyes are usually dyes obtained or obtained from plants, animals, or mineral sources. This dye has long been used for food coloring and until now its use is generally considered safer than synthetic dyes (Afriyeni, 2016)

Synthetic dyes are basically made for industrial purposes which are in nature to facilitate human work with maximum results. Synthetic dyes are also claimed to be safe to use if they are used at maximum limits and on target. But along the way, synthetic dyes are often misused. One example is the use of Rhodamin B dye which is used for textiles but is abused in coloring lipsticks. The purpose of this use is intended to reduce the production cost of lipstick, because basically natural or synthetic dyes will be safer than synthetic dyes. Textile dyes such as Rhodamin B will also provide brighter colors than other dyes (Jusnita, 2019)

Rhodamin B is a dye in the form of a green or reddish-purple crystalline powder, odorless, and easily soluble, in a bright red fluorescent solution used as a dye for textiles, paints, paper or clothing.(chlorine) contained in Rhodamin B which is very reactive and harmful. The accumulation of Rhodamin B in the liver causes liver function disorders in the form of liver cancer and tumors (Sylvia, D, dkk. 2013).

The effects of long-term use of rhodamin B color zar in food and cosmetics can cause cancer and impaired liver function. If exposed to a large amount of rhodamin B, in a short time there will be acute symptoms of rhodamin B poisoning. In addition to food or cosmetics, rhodamin B can also cause health problems, if inhaled there is irritation in the respiratory tract. Eyes affected by rhodamin B will also experience irritation which is characterized by reddened eyes and fluid deposits or udem in the eyes. If exposed to the lips, it can cause the lips to crack, dry, and itchy. In fact, the skin of the lips peels off (Muliyawan, 20130)

Thin-layer chromatography (TLC) is a simple chromatography technique that is usually used for the identification of organic compounds. This technique was developed in 1938 by Ismailoff and Schraiber. This method is quite sensitive with a few micrograms of snippets. In essence, TLC involves two changes, namely the stationary phase and the nature of motion. The quiescent phase can be a fine powder that serves as an absorbent surface (liquid-solid chromatography) or serves as a buffer for a layer of liquid substances (liquid-liquid chromatography). The working principle of TLC is that the mixture to be separated is dissolved in a suitable solvent. The commissioning was carried out using capillary pipes. The solvent is allowed to evaporate or removed with the help of a dry airflow. The layer is then inserted into a vessel containing a solvent about one cm deep that will act as a phase of motion. Then the vessel is tightly closed and the solvent is left for about 10-15 minutes. The point where the mixture is applied to the end of the plate or sheet is called the starting point and the way the snippet is placed is called the starting point. The solvent front line is

the upper part of the phase of motion or solvent as it moves through the layer and after development is complete, is the maximum height reached.

The hypothesis of this research is:

H1 : Lipsticks circulating in the city of Padangsidimpuan contain rhodamin B dye

H2 : Lipsticks circulating in the city of Padangsidimpuant do not contain rhodamin B dye

METHODOLOGY

Tool

Stirrer rod, chamber, Erlenmeyer (pyrex), filter paper, analytical balance, dropper, otol pipette, measuring cup, 254 nm uv lamp, oven, TLC plate.

Material

Amonia, asam chlorida pekat, aquadest, ethyl acetate, methanol plat TLC, Rhodamin B.

General procedure

Lipstick samples were taken from three market points in the city of Padangsidimpuan identified as many as 5 red lipstick samples, consisting of 2 samples from the Bonang Sagumpal Market, 1 sample from Saroha Market and 2 samples from the Sadabuan Impression Market. The samples marked A and B were taken from the Sangkumpal Bonang Market, samples C and D from the Sadabuan Presidential Market, Sample E from the Saroha Padang MarketMatinggi In identifying the Rhodamin B dye in lipstick samples was carried out by the thin-layer chromatography method. The testing process is carried out through several stages, including:

Lipstick Sample Test Solution Manufacturing

The lipstick sample was weighed ± 500 mg put into erlenmeyer, then 4 drops of 4 N hydrochloric acid were added, and 2 ml of methanol was added, and then homogenized was further sufficient with methanol up to 10 ml until dissolved, then stirred until well mixed and filtered using filter paper as solution A.

Raw Solution Manufacturing

An amount of approximately 50 mg of Rhodamin B is dissolved with methanol, put into erlenmeyer and then shaken until dissolved as solution B.

Mixed Solution Manufacturing

An equal amount of 1 ml A and 1 ml B solution is put into a small pot mixed, then homogenized as C solution.

Sample Identification

On a 20 X 20 cm TLC plate, it is activated by heating it in the oven at a temperature of 100 0C for 30 minutes. Solutions A, B, and C are applied to the KLT plate using a capillary pipe at a distance of 1.5 cm, from the bottom of the

plate 1.5 cm, at the top of the plate measuring 0.5 cm, then left for a while until it dries. The TLC plate that has contained a snippet is put into a chamber that has been saturated with eluene with a moving phase in the form of 50 ml methanol, 20 ml ethyl acetate, and 25 ml ammonia (50 : 20 : 25), allowed the eluent to move up until it is almost close to the upper limit of the plate. Then the TLC plate is lifted and dried in the air. The appearance is observed visually pink spots and under 254 nm UV light if the yellow fluoroscent stain with a 254 nm UV lamp indicates that the sample is positive for Rhodamin B. The result is positive if the color of the spot between the sample and the standard is the same and the Rf price between the sample and the standard is the same or close to each other with a price difference of ≤ 0.2 (Afriyeni and Tilap, 2016).

RESEARCH RESULTS

The lipstick sample used as a sample is a red lipstick sample which will test the results whether it is positive for containing textile ingredients, namely merrah lipstick. From the results of the research conducted from 5 lipstick samples circulating in several markets in Padangsidimpuan City, it can be seen in table 1 below

Table. 1 Results of Qualitative Test of Lipstick Samples

No	Sample	Value Rf Rhodamin B	Visual	UV 254 nm	Value Rf Sample	Test Results
1	Rhodamin B Rf	0,9	Pink	Yellow	0,9	Positives
2	Code A Rf 1	0,9	Purple	Non-Fluorinated	0,9	Negatives
	Rf 2	0,9	Orange	Non-Fluorinated	0,8	Negatives
3	Code B Rf 1	0,9	Purple	Non-Fluorinated	0,9	Negatives
	Rf 2	0,9	Pink	Fluorescence Yellow	0,9	Positives
4	Code C Rf 1	0,9	Orange	Non-Fluorinated	0,9	Negatives
	R 2	0,9	Orange	Non-Fluorinated	0,8	Negatives
5	Code D Rf 1	0,9	Orange	Non-Fluorinated	0,9	Negatives
6	Code E Rf 1	0,9	Purple		0,9	Negatives

	Rf 2	0,9	Orange	Non-Fluorinated Non-Fluorinated	0,9	Negatives

From the table, it can be seen that from as many as 6 samples with codes A, B, C, D, and E, there are no liptics that are proven to contain Rhodamin B circulating in the city of Padangsidmpuan.

DISCUSSION

Rhodamin B is a dye substance in the form of odorless crystals and is green or reddish-purple, in the form of a fluorescent bright red solution. To find out whether lipstick is safe to use or not, it is necessary to test qualitatively. One of the qualitative tests is using the TLC method. In addition to using the TLC method, you can also use the spectrophotometry method and use wool yarn which attracts rhodamin B on lipstick, but to get woll yarn is difficult to obtain.

In the qualitative analysis, first of all, sample solution is made, Rhodamin B raw solution is made, and mixed solution is made. For the manufacture of the sample solution first the lipstick sample is weighed ± 500 mg, Put into Erlenmeyer then added 4 drops of 4 N hydrochloric acid as a reagent to further clarify the red color of Rhodamin B contained in the sample, then dissolved with 2 ml of methanol then homogenized then sufficient with 10 ml of methanol, filtered, the filtrate obtained is then used for identification, the sample solution is considered as solution A.

Then for the manufacture of the raw solution of Rhodamin B by weighing 50 mg of raw Rhodamin B and dissolved with methanol up to 10 ml in Erlenmeyer, the solution is then shaken until dissolved, considered as solution B. Then for the manufacture of a mixed solution, the same amount of volume is taken from the sample solution and the raw Rhodamin B solution is 1 ml each, This mixed solution is considered as solution C. Then each solution A (Lipstick Sample), solution B (raw Rhodamin B) and Mixed solution (sample A and solution B) are identified by notifying each of these solutions on a 20 x 20 TLC plate with a fouling distance of 1.5 cm, from the bottom of the plate 1.5 cm and diluted using 50 ml methanol eluene (Motion phase), ethyl acetate, 20 ml and ammonia 25 ml (50 : 20 : 25).

After the TLC plate is removed, the stain from TLC is visually observed, and under 254 nm UV light if the yellow fluorosesy stain with a 254 nm UV lamp shows that the sample is positive for Rhodamin B dyes. The Rf value is calculated. Based on the identification results on 5 lipstick samples, it was found that there was Rhodamin B dye in the sample code B of Sagumpal Market, where in visual observation the stain that appeared on the KLT plate was easy red, and under 254 nm UV light if the yellow fluoroscent stain with a 254 nm UV lamp showed that the positive sample contained Rhodamin B dye close to a price difference of ≤ 0.2 (Afriyeni dan Nila, 2016).

Then the Rf value (retention factor/creep time) of the Pasar Sagumpal B lipstick sample was pink and fluorescence yellow under a 254 nm UV lamp, the Rf value of 0.9 cm was in line with the Rf value of the raw Rhodamin B solution which was also 0.9 cm. The result was positive if the color of the spots between

the sample and the standard was the same and the Rf price between the sample and the standard was the same or close to each other with a price difference of ≤ 0.2 (Afriyeni and Nila, 2016). So it can be concluded that the sample with the code B is positive for Rhodamin B.

The result was positive if the color of the spots between the sample and the standard was the same and the Rf price between the sample and the standard was the same or close to each other with a price difference of ≤ 0.2 (Afriyeni and Nila, 2016)

1. Rf Value of Raw Rhodamin B Solution

$$\text{RF} = \frac{\text{Jarak noda}}{\text{Jarak Tempuh eluen}}$$

$$\text{RF} = \frac{16{,}3 \text{ cm}}{18 \text{ cm}}$$

$$= 0{,}9$$

It was seen that the stain was pink in nature. Spotted stain with yellow fluorinated 254 nm UV lamp

2. Sample A: Sagumpal Bonang Market

$$\text{Rf } 1 = \frac{16{,}8 \text{ cm}}{18 \text{ cm}}$$

$$= 0{,}9$$

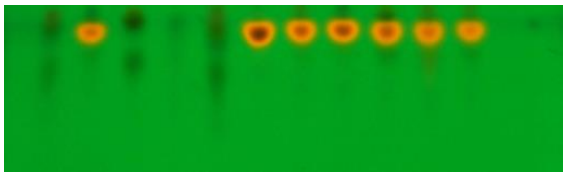

Fig.1 Uv 254 nm Sample Results

For lipstick samples with Codes A, C, D, E, did not contain Rhodamin B dye, judging from the test results Visually, the stain spots that appeared were not pink, and under 254 nm UV light the stain was not fluorescent.

From the table, it can be seen that there is one list with a positive code B that contains Rhodamin B textile dye. Regulation of the Minister of Health of the Republic of Indonesia Number: 239/MEN. KES/PER/V/85 concerning certain dyestuffs that are declared as hazardous substances. Rhodamin B is declared a dangerous dye and should not be used in cosmetic products. Negative impacts that can be caused by the use of Rhodamin B, namely irritation, liver damage and cancer (Praja, 2015)

According to previous research in the journal (Nina Jusnita, et al., 2017) out of 25 lipstick samples, there were four lipstick samples that showed Rhodamin B content, namely lipstick samples with codes D2 and D3 from the Yellow Bamboo Market and lipsticks with codes E5 and E6 obtained from Sunter Podomoro

Market. (Hurip Budi Riyanti, et al. 2018) Of the 11 lipstick samples circulating in four markets in the East Jakarta area, there was 1 sample (9.090% of 11 samples) that contained Rhodamin B. (Arifina, 2012) out of 7 blusher samples, there were 2 samples that were positive for Rhodamin B.

Different results were obtained by Mamdo, L.D and Citraningtyas, F.G, 2013 who studied the Analysis of Rhodamin B on Lipsticks Circulating in the Manado City Market From the results of the research carried out, it can be concluded that in 9 lipstick samples circulating in the Manado City market, the presence of Rhodamin B dye was not identified which was identified by Thin Layer Chromatography (TLC) and UV-Vis Spectrophotometry.

Although from the six samples studied from various markets in Padangsidimpuan City, the presence of rhodamin B dye was not identified, but caution is needed in the use of deep red lipsticks that are sold freely in the market. Synthetic dyes that are prohibited in Indonesia which are based on the Indonesian Minister of Health Regulation No.722 / Menkes / Per/ IX / 1988 concerning dye materials, are not allowed to use rhodamin B dye because this dye is only used for textile industry dyes such as fabrics, paper and paints. Rhodamin B contains the compound chlorine (Cl). Chlorine compounds are reactive and harmful inorganic compounds. This compound will try to achieve stability in the body by binding to other compounds in the body, this is what is toxic to the body. The use of rhodamin B is certainly harmful to health. The accumulation of rhodamin B in fat over a long period of time is continuously increasing in the body and can cause damage to the organs of the body until it results in death

CONCLUSIONS AND RECOMMENDATIONS

Based on the results of the research, it can be concluded that: Of the five lipstick samples taken from three market sources in Padangsidimpuan City, there is one sample that contains Rhodamin B, namely Pasar Sagumpal Bonang which shows the presence of pink visually, with a 254 nm UV lamp showing yellow fluoricity.

ADVANCED RESEARCH

For further research, you can do a Rhodamin B examination on other red cosmetics such as eyeshadow.

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