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The Analytical Survey of Mercury Content in Whitening Cream Sold in Banjarnegara Regency's Traditional Market

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Abstract: Whitening cream is a mixture of active substances and the other additives that can be used to brighten the skin. Mercury is one of the whitening ingredients of the creams that can cause health problems when it is absorbed into the tissue. This study aims to identify whether there is a mercury content and how much mercury is contained in the whitening cream, which does not have a BPOM RI registration number (National Agency of Food and Drug Control, Republic of Indonesia) circulating in the market in Banjarnegara regency, Central Java, Indonesia. This research was conducted with purposive sampling, and 20 samples were obtained from the traditional market in Banjarnegara regency. The qualitative analysis method used the Potassium Iodide (KI) color reaction test and amalgam formation with copper rods, while the quantitative analysis used the Cold Vapor Mercury Analyzer at a wavelength of 253.7 nm. The results showed the presence of three positive samples containing mercury at average levels of 0.04217 mg / kg (ppm), 0.01814 mg / kg, and 0.91000 mg / kg. These findings suggest that mercury was added at the whitening cream and distributed in the market. Customers should be more careful when choosing a whitening cream to prevent health problems.

Keywords: Amalgam test and mercury analyzer-CV, KI test, mercury, whitening cream.

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1. INTRODUCTION

Cosmetics are pharmaceutical preparations widely used to support a person's daily appearance. Cosmetics can also help to improve the dry, dull, or blemished skin (1,2). They have both positive and negative impacts on public health (3). Whitening cream is one type of cosmetic that is most widely used to improve the facial skin, such as by making it brighter and whiter. One of the ingredients that can be used as lightning or bleaching is mercury (4).

Indonesia has issued regulations to control what ingredients are safe and can be used to a certain extent through the BPOM (Indonesian national agency of drug and food control (Badan Pengawas Obat dan Makanan) RI. It has been confirmed that many cosmetic products contain heavy metals such as mercury, arsenic, and hydroquinone that exceed the specified limits regulated by 21CFR700.13 FDA and BPOM RI (2,5). This issue, therefore, causes BPOM RI to issue a *public warning* number B-IN.05.03.1.43.12.17.5965 on December 11, 2017, concerning cosmetics that contain harmful ingredients so that they are withdrawn and are discovered to contain dangerous metals (6). During 2017, BPOM RI has also found 26 types of cosmetics containing hazardous materials. In the use of cosmetics, they must be adjusted to the rules of their use (7).

In 2018, there was a whitening cream that was positively found to contain mercury. It was found in the preliminary research of several researchers, such as Walangitan *et al.* (2018) and Mona et al. (2018). The mercury concentration of three samples from seven samples circulated in the Manado district, which were measured by using the Atomic

Absorption Spectrophotometry (AAS) method, was 0.1299 mg/kg, 0.1822 mg/kg, and 0.0566 mg/kg, respectively (8,9). Furthermore, mercury concentrations in two samples of six taken from the Manado region, measured by using the Mercury Analyzer method, were 229.38 ppm and 101.17 ppm (9).

According to BPOM RI in 2019, heavy metal mercury (Hg) should be not found inside cosmetics (2). Besides, the World Health Organization states that each country has its own policy regarding the amounts of mercury that can be used in cosmetic products (10).

According to Tranggono et al. (2014), mercury is proposed to be a skin whitening ingredient as it has the potential to be a reducing agent of skin color with a very strong whitening power (11). Mercury ions are considered to inhibit melanin synthesis of skin pigment in melanocyte cells (12,13). Melanin in people with dark skin is more abundant than in people with brownish yellow skin. It can be said that the function of melanin in the skin is to give the skin a brown color (14). When this process is inhibited, melanin will not be formed. This is the basis for the mechanism of various bleaching active ingredients to reduce the melanocyte cells that produce melanin. In the initial use of mercury, it can irritate and cause dependence. When the use is stopped, it will cause an itchy and uncomfortable feeling on the facial skin (15,16). Furthermore, mercury can also cause skin cancer with long-term use (17).

Since there are a lot of reports stating that there are harmful effects of mercury on the use of bleaching creams by women in Indonesia and the survey results in the Traditional Market of Banjarnegara showed there were a lot of circulating cream products that were sold without BPOM RI registration numbers. Therefore, 20 samples of whitening cream without BPOM RI registration number that are circulated in the traditional market of Banjarnegara district were collected and then analyzed. This paper deals with the explanation of qualitative and quantitative analysis of mercury in the samples.

2. EXPERIMENTAL SECTION

2.1. Research Tools

Copper rods, sandpaper, filter paper, tongs, dropper pipettes, burners, tube racks, electric stoves (Maspion ®), beaker glass (Pyrex ®), 100 mL Erlenmeyer (Pyrex ®), volumetric flask 25 mL; 100 mL (Pyrex ®), volumetric pipette 1mL; 5mL; 10 mL; 25 mL (Pyrex ®), measuring pipette 5mL; 50 mL; 100 mL (Pyrex ®), test tubes (Pyrex ®), digital scales (Mettler Toledo AL 204), Mercury analyzer (AULA 254 ®) were used in the present study.

2.2. Research Materials

The materials used in this study were 20 types of facial whitening cream samples collected from traditional market of Banjarnegera Regency. The concentrated HNO_3 solution, concentrated HCl solution, and distilled water were purchased form Bratachem. The 0.5 N KI solution, $KMnO_4$, hydroxylamine hydrochloride, and $SnCl_2.2H_2O$ were obtained from Merck. The standard mercury solution was purchased form Sigma-Aldrich.

2.3. Procedures

2.3.1. Qualitative sample test

Sample preparation: Two (2) grams of sample were weighed using a digital scale, added to 25 mL of distilled water, and then went through wet destruction with 10 mL of aqua regia solution (the combination of concentrated HCl and concentrated HNO_3 at a ratio of 1:3. The wet destruction was done for 15 minutes, until the sample almost dried. The heating was conducted during the destruction using a hot plate. The end of destruction was then added 10 mL of distilled water. Furthermore, it was boiled briefly for 5 minutes, cooled, and filtered (15).

2.3.2. Mercury identification test

Color reaction test by using KI: The color test was done in the test tube (Pyrex ®). Two (2) drops of 0.5 N KI was added to one (1) mL of filtered sample in the test tube. The addition was done slowly through the wall of the test tube. The orange solid will appear if it is positively identified to contain mercury (8,18).

2.3.3. Amalgam test

Three (3) mL of filtered sample solution was added into the test tube, and then the copper rod was sanded until it was glossy. It was then dipped for some time into the test tube containing the tested solution. The copper rod will be coated with glossy grey patches if it is positively identified to contain mercury. The grey color will disappear if it is heated on a flame (18–20).

2.4. Quantitative Analysis of Samples Using a Mercury Analyzer

2.4.1. Sample preparation

Samples were weighed and placed in a 100 mL Erlenmeyer flask, followed by 10 mL of HNO_3 and $HClO_4$ with a ratio of 1:1. The mixture was then heated on a hot plate until it turned clear and a white smoke appeared. Afterward, the samples were filtered, and 50 mL were taken by using a volumetric flask (21).

2.4.2. Measuring mercury concentration using a Mercury analyzer (AULA 254®)

A 10-mL volumetric pipette was used to collect samples, which were then placed in a test tube. 0.1 mL of $KMnO_4$ (0.1%), 0.1 mL of hydroxyamine hydrochloride, and 0.5 mL of $SnCl_2.2H_2O$ were added. It was also measured with a Mercury analyzer at a wavelength of 253.7 nm (1,21,22).

3. RESULTS AND DISCUSSION

The qualitative analysis of mercury in the whitening cream samples from the traditional market of Banjarnegra Regency was done by using the KI color

reaction and Amalgam formation (18–20). The wet destruction was conducted before the test. The heating of organic samples in the presence of strong oxidizing agents such as single or combinations of mineral acids is known as wet digestion or destruction. The sample is completely oxidized to form an inorganic compound suitable for analysis if it is heated at a suitable temperature continuously for a sufficiently long time. Generally, HNO_3 is used for wet destruction as it cannot react with a saline solution such as HCl or H_2SO_4 (23). The wet destruction of this research used aqua regia solution, which is a mixture of HCl and HNO_3 with a ratio of 1:3 (24). HCl and HNO_3 were used because they are strong acids and oxidizing agents to break the bond of organic metals (Hg-organic) so that inorganic metals (Hg^{2+}) were formed.

Mercurium (I) is produced by combining cold concentrated and moderately concentrated nitric acid (HNO3) with excessive mercurium, which is the reaction that occurs between a mercury metal and concentrated HNO3. The resulting reaction is depicted in Equation 1.

$$6Hg + 8HNO_3 \rightarrow 3Hg_2^{2+} + 2NO\ (evaporated) + 6NO_3^- + 4H_2O \quad (1)$$

By using excessively concentrated heated nitric acid (HNO_3), mercurium (II) ions are formed with the following Equation 2.

$$3Hg + 8HNO_3 \rightarrow 3Hg^{2+} + 2NO\ (evaporated) + 6NO_3^- + 4H_2O \quad (2)$$

Mercury (Hg) is very distinct from the reagents used in the systematic qualitative analysis, and it is therefore included in two different analytic classes. Mercury ions (I) belong to the first cation group, while mercury ions (II) belong to the second cation group (18).

The analysis was carried out using potassium iodide. The reaction that occurs between mercury and potassium iodide is given by Equation 3 (25).

$$Hg^{2+} + 2KI \rightarrow HgI_2 + 2K^+ \quad (3)$$

The reaction produced an orange-red solid of HgI_2 (19), as shown in Figure 1.

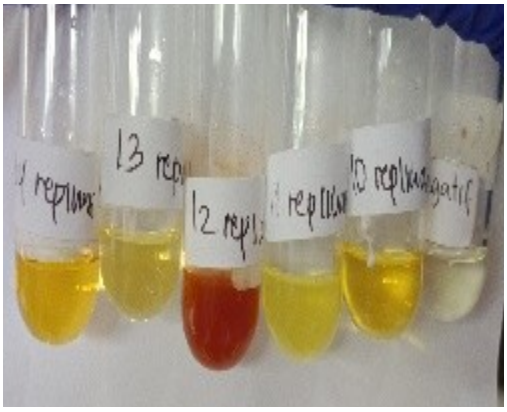

Figure 1: KI test of whitening cream.

Amalgam reaction formation is carried out by using Cu or a sanded copper rod. Amalgam is a metal solution from mercury produced by copper rods that can be dissolved by mercury. If it is positively identified to contain mercury, there will appear a layer containing grey glossy spots on the surface of the copper rod, which indicates a form of metal mercury (18,19,26,27). It happens from the results of the reaction (18,27), as in Equation 4.

$$Cu + 2Hg^{2+} \rightarrow Cu^{2+} + 2Hg \quad (4)$$

Based on that reaction, 2Hg solid produced the layer with glossy grey patches.

Figure 2: Amalgam test of whitening cream.

The result obtained was negative amalgam with no glossy grey patches on the surface of the copper rod. It is because the sample contained less mercury that made it hardly detected. The KI color test and amalgam qualitative analysis are summarized in Table 1.

Table 1: The Result of the Qualitative Analysis Using a Color Test (Potassium Iodide Test) and the Formation of Amalgam.

Sample Code	Replication	KI Color Test	Amalgam Formation Test	Information (+/-)
1	1	Clear	No glossy grey color change	-
	1.1	Clear	No glossy grey color change	-
2	2	Clear	No glossy grey color change	-
	2.1	Clear	No glossy grey color change	-
3	3	Clear	No glossy grey color change	-
	3.1	Clear	No glossy grey color change	-
4	4	Clear	No glossy grey color change	-
	4.1	Clear	No glossy grey color change	-
5	5	Clear	No glossy grey color change	-
	5.1	Clear	No glossy grey color change	-
6	6	Clear	No glossy grey color change	-
	6.1	Clear	No glossy grey color change	-
7	7	Clear	No glossy grey color change	-
	7.1	Clear	No glossy grey color change	-
8	8	Clear	No glossy grey color change	-
	8.1	Clear	No glossy grey color change	-
9	9	Clear	No glossy grey color change	-
	9.1	Clear	No glossy grey color change	-
10	10	Clear	No glossy grey color change	-
	10.1	Clear	No glossy grey color change	-
11	11	Clear	No glossy grey color change	-
	11.1	Clear	No glossy grey color change	-
12	12	Red and orange solid	No glossy grey color change	+
	12.1	Red and orange solid	No glossy grey color change	+
13	13	Clear	No glossy grey color change	-
	13.1	Clear	No glossy grey color change	-
14	14	Clear	No glossy grey color change	-
	14.1	Clear	No glossy grey color change	-
15	15	Colored and red solid	No glossy grey color	+

Sample Code	Replication	KI Color Test	Amalgam Formation Test	Information (+/-)
			change	
	15.1	Colored and red solid	No glossy grey color change	+
16	16	Clear	No glossy grey color change	-
	16.1	Clear	No glossy grey color change	-
17	17	Red colored and orange red solid	No glossy grey color change	+
	R17	Red colored and orange solid	No glossy grey color change	+
18	18	Clear	No glossy grey color change	-
	18.1	Clear	No glossy grey color change	-
19	19	Clear	No glossy grey color change	-
	19.1	Clear	No glossy grey color change	-
20	20	Clear	No glossy grey color change	-
	20.1	Clear	No glossy grey color change	-

Based on the results, there are three positive samples containing mercury, namely sample numbers 12, 15, and 17, as there is a permanent change in the orange color and a red solid using KI color test. This result is in accordance with the previous identification of mercury in the whitening cream sold in Manado, Banjarmasin, and Palu, Indonesia (8,25,26), even in Shijiazhuang, China (13). To ascertain the result and find out how much mercury is contained in the sample, a quantitative analysis was carried out on the three samples by using a mercury analyzer.

Mercury analyzer includes in the spectrophotometric analysis technique of atomic absorption the administration of cold vapor samples. Mercury has the unique characteristic of having a high vapor pressure at room temperature (0.16 Pa at 293 K). The vapor produced is stable and monatomic, so that the atomic composition of the mercury vapor can be read, without the use of a flame or other atomizing techniques with a wavelength of 253.7 nm (28). The first thing to be prepared was the standard curve. A mercury concentration in the sample preparation was then measured by using a mercury analyzer at a wavelength of 253.7 nm. The wavelength was selected as it indicated the best sensitivity and did not indicate an interaction with other metals in the samples. In the atomization process, it used $SnCl_2$, which was an olive reducing agent. The reaction occurred as described in equation 5 and the concentrations of mercury in the three (3) samples of whitening cream are represented in Table 2.

$$Hg^{2+} + SnCl_2 \rightarrow Hg^0 + Sn^{4+} + 2Cl^- \quad (5)$$

Table 2: The result of the quantitative analysis using mercury analyzer.

Sample Code	Sample weight (g)	Results (ppm)
12	1.0346	0.04217
15	1.0086	0.01814
17	1.0872	0.91000

Based on Table 2, upon conducting a quantitative test by using Mercury Analyzer with a wavelength of 253.7 nm, three samples were found to contain mercury, namely samples 12, 15, and 17, with an average level of 0.04217 ppm, 0.01814 ppm, and 0.91000 ppm, respectively. The concentration of the mercury was similar to the previous research that reported on mercury analysis in the whitening cream sold in Palu, Indonesia (25). Moreover, based on WHO 2011, the policy to limit the use of mercury depends on each country (10). It is necessary to conduct a routine check on whitening cream products sold in the market to minimize the spread of harmful cosmetics. It is also expected that the public will be more careful in selecting whitening cosmetics to use.

4. CONCLUSION

Based on the analysis of whitening creams sold in Banjarnegra Regency using KI color test, it was found that three (3) whitening creams contained mercury out of the 20 collected samples. The concentrations of the mercury in the three whitening creams were between 0.01814 -0.91000 ppm based on mercury analyzer. According to the results, the consumer must be more careful when choosing a whitening cream to prevent the negative effects of its use.

5. CONFLICT OF INTEREST

There is no conflict of interest for this publication.

6. ACKNOWLEDGMENTS

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