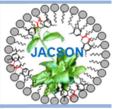


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Analysis of Mercury (Hg) in Whitening Cream Distributed in Palu City by Atomic Absorption Spectroscopy (AAS)

Endah Dwijayanti^{*1} and Susanti²

¹Departement of Chemistry, Faculty of Mathematical and Sciences, Islam University of Makasar, Departement Pharmacy, ²Faculty of Pharmacy Airlangga University of Surabaya, INDONESIA

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ABSTRACTS

This study aimed to obtain chemical data of mercury from some whitening cream distributed in Palu City. The whitening cream samples consisted of 3 groups (A, B and C), each sample of the group was extracted through wet destructions. The qualitative analysis was with HCl 2.0 M, KI 0.50 N, NaOH 2.0 N reagents, while the quantitative analysis was performed with Atomic Absorption Spectroscopy (AAS). The results of the qualitative analysis indicated that the A sample reacted with NaOH 2.0 N reagent delivered yellow precipitates and C sample with KI 0.50 N reagent delivered red precipitates. Both data indicated that both samples showed positive reaction of Hg (II). However, the B samples did not occur the positive reaction indicating Hg. Based on Indonesian National Agency of Drug and Food Control (BPOM) regulation, (No.HK.03.1.23.07.11.6662 of 2011) related to the requirements of microbe and heavy metals contamination in cosmetics, the Hg (II) concentration in cream should be no more than 1,000.0 μ g g⁻¹. AAS results found in present study demonstrated that A sample holded mercury content over the level required by the BPOM, which it was 4,554.00 μ g g⁻¹. The B and C samples were 47.18 and 780.32 μ g g⁻¹ respectively, both samples did not exceed the limits set by the regulation but continuous application might be toxic for body.

Keywords: whitening cream, mercury (Hg), wet destruction, atomic absorption spectroscopy * *Corresponding author:* endahdwijayanti.dty@uim-makasar.ac.id

1. Introduction

A cosmetic is material commonly applied on the outer part of human body. Today, it becomes important need since it is used routinely and continuously. In modern society, cosmetics is mainly purposed for self cleanliness, increasing physical attraction by make up, adding the self confidence, preventing premature aging and protecting the skin from the damage caused by ultraviolet light, pollution and another environment factor (Tranggono and Latifah, 2007).

These cosmetic products include, care creams, talcum and face powders, lipsticks, kajal, sindoor, eye makeup, mouth washes, etc. (Chauhan et al., 2010). One of the most widely used cosmetics for women is whitening cream. It is a mixture of chemical material and others claiming the benefit to whiten the skin and fade the black blemish as well. It is really useful for face which has a lot of flaws as it can return the skin brightness and decrease the black blemish (Peregrino et al., 2011; Parengkuan et al., 2013).

One of the active materials commonly added in whitening cream is mercury or so called *Hydrargyrum*, a chemical element symbolized as Hg. Mercury (Hg) is categorized in group of three heavy metals beside of lead (Pb) and Cadmium (Cd) with high toxicities which might be absorbed into human body via cosmetics, food or air (Dwijayanti et al., 2015). Mercury added to cream can create problems. The skin color might change, black blemishes, allergic, skin irritation. High dosage application might cause permanent damage of brain and kidney, impaired fetal growth. Furthermore, short term exposure in high dosage might experience vomiting, diarrhea, and damaged kidney, moreover, it is carcinogenic (can cause cancer) in human (BPOM, 2009).

Whitening cream without registration number from Indonesian National Agency of Drug and Food Control (BPOM) regulation remains in the market; one of them is in Palu City. The cream promises to have white skin within 5-7 days, thus, many women are interested to buy. When they were applying that cream, they complained about pain on the skin face, even it was peeled off. There are a lot of creams like this sold in traditional market in cheap price.

The observation result from 3 traditional markets in Palu demonstrated highest sale percentage of whitening cream owned by A (SP) equal to 77.77 % followed by B (UB Ginseng) 61.11 % and C (SJ) 50 %. They need to be analyzed through Atomic Absorption Spectroscopy (AAS) method. Previously research on mercury was conducted by Parengkuan et al. (2013) reported that 5 among 10 cream distributed in Manado, positively contained mercury with range 0.03 until 0.06 mg L⁻¹ after analyzed by qualitative and quantitative through Cold Vapour Atomic Absorption Spectroscopy while Armin et al. (2013) proved the three herbal cream distributed

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in Padang were mercury positive through Atomic Absorption Spectroscopy. It obtained range level of sample 0.28 % until 0.56 %. Similar research carried out by Trisnawati et al. (2016) reported the mercury content in some cream distributed in DTC Wonokromo market, Surabaya. It denoted but keep meeting BPOM standard. Based on that information above, it needs to conduct research about whitening cream spreaded in several traditional markets in Palu: Inpres market, masomba, and lasoani. From those markets there were 3 kinds of cosmetics mostly bought in various brands but BPOM unregistered.

Problem statement of this research covers; what are the three kinds of whitening cream containing mercury distributed in Palu and what mercury they have; how much the levels are, do they exceed the limit stated by BPOM. This research purposed to find out the presence of mercury, its types and its levels in three whitening cream distributed in Palu, through Atomic Absorption Spectroscopy analysis and to find that the mercury levels do not exceed the allowed limit set by BPOM as well.

The significances of this research are to comprehend the process of qualitative and quantitative analyses of mercury (Hg), finding a working principle of Atomic Absorption Spectroscopy *to* measure mercury levels, and provides information about mercury application effect in whitening cream.

2. Materials and Methods

2.1. Tools

Atomic Absorption Spectrometer, Stirring bar, funnel, beaker 250 ml, graduated cylinder 50 mL, hot plate, filter paper, laboratory flask, pipette in 10 ml volume, test tube rack, test tube and analytical balance.

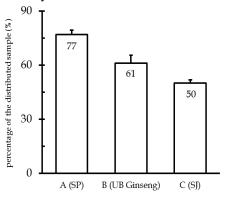


Fig. 1. Sample observation result in Palu City

2.2. Materials

The samples are whitening cream A (SP), B (UB Ginseng) and C (SJ) also some other chemical materials; alcohol, distilled water, HCl 2.0 M, HNO₃ 65.0 %, KI 0.50 N, NaOH 2.0 N, and $SnCl_2 2.0 \%$.

2.3. Working Principle

2.3.1. Sampling

The observed sample is the most well selling whitening cream in traditional markets in Palu City through observation method. It was obtained 3 kinds of BPOM unregistered whitening cream in different brand as the research samples (Fig. 1).

2.3.2. Sample solution preparation

The sample was prepared in wet destruction. Weighed 2 grams of whitening cream sample and added 10 ml concentrated HNO₃ 65.0 %. The mixture was heated on the hot plate at 80 $^{\circ}$ C until it got soluble. Added distillates 20 mL in the evaporation residue, reheat for 15 minutes. Let it cool and filtered. Bring the volume to reach 100 mL with distilled water.

2.3.3. Qualitative Analysis

2.3.3.1. Sample testing with diluted HCl 2 M

Pipetted 5 ml of sample solution and added with 5 drops of dilute HCl 2.0 M. it formed white precipitation if it was positive containing mercury Hg (I).

2.3.3.2. Sample Testing with KI 0.5 N

Pipetted 5 ml of Sample solution and added with 5 drops of KI solution 0.5 N. It formed green precipitation if it was positive containing Hg (I) and red indicating Hg (II).

2.3.3.3. Sample Testing with NaOH 2.0 N

Pipetted 5 ml of Sample solution and added with 5 drops of NaOH 2 N. It showed black precipitation if it was positive containing Hg (I) and yellow if it is Hg (II).

2.3.4. Quantitative Analysis

2.3.4.1. Preparation of Mercury solution

Pipetted 5 ml of mercury solution and poured into laboratory flask 50 ml, the volume set to the limit sign with distilled water. In sequence, another mercury solution 1000 μ g L⁻¹ was made by putting 5 ml mercury solution into laboratory flask 50 ml, set to the limit sign with distilled water. The concentrations of mercury solutions were 10,000.0 and 1,000.0 μ g L⁻¹, respectively.

2.3.4.2. Calibration curve in making

Pipetted 0.5, 1.0, 1.5, 2.0, and 2.5 mL of 1,000.0 μ g L⁻¹ Hg solutions and put respectively into each laboratory flask 50 ml and set the volume to the limit sign with distilled water. The concentrations of mercury solutions were 10.0, 20.0, 30.0, 40.0, and 50.0 μ g L⁻¹, respectively.

2.3.4.3. Determination of Mercury levels

Pipetted 1 mL of sample solution and put into laboratory flask 100 ml and added with distilled water until it reached the limit sign then added with 5 mL, 2.0 % SnCl₂. AAS was set up for the mercury wave length which is 253.75 nm and continued to be read (Agorku et al., 2016).

3. Results and Discussion

The samples here were whitening cream A (SP), B (UB Ginseng) and C (SP) distributed in Palu City through observation method in some markets based on the most bought whitening cream but without BPOM registration number. It carried out qualitative analysis by using test reactor HCl 2.0 M, KI 0.5 N and NaOH 2.0 N to find the occurrence of mercury and its types in the cream then analyzed through

quantitative method which is sensitive to mercury in low levels–AAS method. This method has been hugely applied by previous researcher, one of them was by Rohaya et al. (2017); she implemented AAS method to analyze mercury levels in whitening cream. It proved that AAS has sensitivity and high specificity to Hg metal even in small amount. High specificity means the elements are determined in the presence of each other (Cantle, 1986).

Selecting wet destruction referred to mercury property which is easy to evaporate. If it is with dried one, it is worrying that it can easily evaporate at high temperature. Wet destruction method used strong acid, concentrated HNO_3 65 %. This solution was chosen due to its acting as strong oxidizing agent at high temperature and has ability to dissolve mercury and prevents the element precipitation, thus Hg inside the cream can be used both for qualitative and quantitative analyses.

Table 1. Qualitative test result

Sample	Sample experi	Qualitative Test Result Replication			_ Explana-
Name	ment				tion
	-	1 2		3	-
Sample A	HCl 2.0	-	-	-	-
	М				
	KI	-	-	-	-
-	0.50				
	Ν				
	NaOH	\downarrow	↓	\downarrow	+
	2.0 N	yello	yello	yello	
		W	W	W	
	HC1	-	-	-	-
G 1 D	2.0 M				
Sample B	KI	-	-	-	-
	0.50				
	N				
	NaOH	-	-	-	-
	2.0 N				
Sample C	HC1	-	-	-	-
	2.0 M				
	KI	↓ red	↓ red	↓ red	+
	0.50				
	N				
	NaOH	-	-	-	-
	2.0 N				

Qualitative analysis can be seen by the occurrence of precipitation as shown in Table 1 data where the A sample added by NaOH 2.0 N left yellow and C sample added with KI 0.5 N left red precipitates showing the positive reaction of both reagents with mercury (II) presented in the cream, it corresponds to Vogel literature book page 224 (Vogel, 2000), based on the reaction:

 $Hg^{2+} + 2KI \longrightarrow HgI_2 + 2K$

Mercury (II) in the whitening cream, reported by Clarkson (2006) that mercury compound mixture in whitening cream is most inorganic, especially mercury (II) which is more reactive, thus it can be whiter the skin faster within 5-7 days. Sample B gave no positive reaction toward mercury presence in qualitative test, therefore it required to quantitatively analysis using a sensitive method that is AAS.

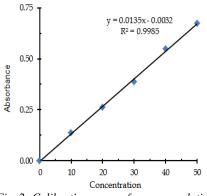


Fig. 2. Calibration curve of mercury solution

Quantitative test started by measuring absorption of standard solution of mercury using concentration series; 10.0, 20.0, 30.0, 40.0, and 50.0 μ g L⁻¹. Its calibration curve data are shown on Fig. 2 and linear regression equation y = 0.01351x - 0.00318 with its coefficient value of correlation (r), was 0.9985 indicating linear result as it fulfilled very strong correlation criteria (Jonathan, 2006).

Measured sample was the solution resulted from whitening cream destruction added with $SnCl_2$ 2.0 % as reductor. Quantitative analysis results are shown in Table 2.

Table 2. Absorption (y) result through AAS

1 001	Table 2. Absorption (y) result through AAS							
	mple ame*	Sample weight (g)	Adsor- ption (y)	Concen- tration (µg g ⁻¹)	Average concentration $(\mu g g^{-1})$			
А	A_1 A_2	2.0276 2.0354	0.6172 0.6293	4,529.5 4,600.2	4,554.00 ± 40.03			
	A ₃	2.0636	0.6286	4,532.3				
В	B_1	2.3166	0.1343	43.920	47.18 ± 2.83			
D	B ₂	2.0786	0.1344	48.990	47.18 ± 2.85			
	B ₃	2.0360	0.1306	48.630				
С	C_1 C_2	2.3696 2.1877	0.5843 0.5989	734.05 814.85	780.32 ± 41.66			
	C_2 C_3	2.1877	0.5989	792.06				
	5							

*Sample A (SP), Sample B (UB ginseng), Sample C (SJ)

Data result in Table 2 showed sample A had average levels over than required by the BPOM; $4,554.00 \pm 40.03$, while sample B equaled to 47.18 ± 2.83 and sample C equaled to $780.32 \pm 41.66 \ \mu g \ g^{-1}$. It informed that sample B and C remained under that of the BPOM guideline. Based on its regulation, No. HK.03.1.23.07.11.6662 of 2011 related to the requirements of mercury contamination in cosmetics, is supposed to be no more than 1,000.0 $\ \mu g \ g^{-1}$, but continuous application might be toxic for body as mercury does not only deposit under the skin layer but it can enter the skin pores and accumulate inside the blood, absorbed by some organs and at the end, it emerged problems, for instance, skin irritation, vomiting, permanent damage of brain and kidney; moreover it is carcinogenic for body (Dean et al., 1985; Agrawal and Mazhar, 2015).

It is similar to the rule of Ministry of Health, Permenkes RI No.445/Menkes/PER/V/1998 which ban the mercury usage in cosmetic; however it remains distributed in markets. It is proved by a research conducted by Rohaya et al. (2017) using unregistered whitening cream sample in Palu City. The result obtained indicated that mean value of those samples were 3.5– 5,000.0 µg g⁻¹, as well as research result by Carsten et al. (2013) reported that 45 % of whitening cream sold online in US and Taiwan; the US whitening cream contains mercury. It indicated that a lot of society still doesn't really understand about mercury danger on body.

Global health watch of WHO has delivered some researches on effects of mercury application in whitening cream. Those effects create skin color change, initially it might be white but slowly it turned to have black blemishes when the application was stopped; the skin got reddish, poignant and burnt. The Global health watch of WHO also stated the serious side effects of inorganic mercury common material found in soap and whitening cream, such as decreased immune system of skin to fight infection, trigger the kidney damage and might lead to skin cancer (Agorku et al., 2016).

4. Conclusion

Based on the research results, some points can be concluded result analysis of whitening cream spreaded in Palu is mercury positive and it is mercury Hg (II); quantitatively analyses showed sample A has average value of $4,554.03 \ \mu g \ g^{-1}$, sample B 47.18, and sample C 780.32. Among those samples, the sample A has higher levels than allowed by BPOM while sample B and C remain under the tolerated concentration.

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