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Comparison of Lead (Pb) Content Analysis Using Atomic Absorption Spectrophotometry with Wet and Dry Destruction Methods in Body Lotion Preparations in Semarang City

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Abstract: Many body lotions are found in circulation without a BPOM RI registration number, which can raise concerns about lead metal contamination. The lead metal content in *body lotion* can be sourced from the raw material, namely water. Lead metal levels in water range from 0,011 mg/L to 0,076 mg/L. Determination of lead levels uses an Atomic Absorption Spectrophotometry instrument. The requirement for analysis using Atomic Absorption Spectrophotometry is that the sample must be in solution form, so a destruction process is necessary because the body lotion sample is in semi-solid form. This study aims to compare two commonly used destruction methods, namely wet and dry destruction. The methodology in this study consists of qualitative analysis, method validation, and quantitative analysis. The test results with qualitative analysis showed negative lead metal content. The results of the Atomic Absorption Spectrophotometry method validation were: linearity (R^2) of 0,9999; *LoD* of 0,024 mg/L; *LoQ* of 0,080 mg/L; precision (%RSD) of 0,61%; accuracy (*%Recovery*) of 100,09% with quantitative results of the percentage of wet digestion sample content, results were: 1,2264 ± 2,96%; 1,7687 ± 4,24%; 1,5036 ± 3,75%; 1,1807 ± 2,92%; and 1,5606 ± 3,88%. This can happen because the wet destruction method is better to use than the dry destruction method which can be seen from the *%Recovery*.

Keywords: Body Lotion, Lead, Wet Destruction, Dry Destruction, Atomic Absorption Spectrophotometry.

INTRODUCTION

Body lotion is a liquid emulsion preparation containing an oil phase and a water phase, with an emulsifier added to ensure stability and prevent separation between the two phases. Body lotion also contains one or more active ingredients in a semi-solid form to ensure quick and easy application, as well as rapid absorption into the skin. Some raw materials used in the production of body lotion may be contaminated with impurities, including heavy metals such as lead (Pb) (Megantara *et al*., 2017).

Qualitative analysis is a process for identifying compounds in a sample. This approach relies on the physical and chemical properties of a compound. In the qualitative analysis process, color tests and reaction tests are carried out to help reveal the characteristics of each compound in order to obtain interpretable information about a sample (Mustapa, 2024).

Atomic Absorption Spectrophotometry (AAS) is an instrument used in analytical methods to identify metallic and metalloid elements based on the absorption of radiation by free atoms. Atomic absorption spectrophotometry is a quantitative analytical technique used in various fields due to its selective and specific procedures, high sensitivity (ppm-ppb), and ability to create matrices that comply with standards (Dewi *et al*., 2021).

Wet destruction is a sample decomposition procedure involving single or mixed strong acids such as nitric acid (HNO_3), sulfuric acid (H_2SO_4), perchloric acid ($HClO_4$), and hydrochloric acid (HCl). Dry digestion is a sample digestion procedure involving heating at temperatures between 400-800°C until the sample is reduced to ash; however, the heating temperature used in this method depends on the type of sample being analyzed. Dry digestion is safer in terms of chemical reagents, but wet digestion is easier to control and condition. A clear solution in the digestion solution indicates that all the material has dissolved and the digestion process is complete (Aribowo *et al*., 2022).

This study aims to analyze the lead content in body lotions that do not have a BPOM RI registration number circulating in the city of Semarang using qualitative reaction testing methods and quantitative analysis methods, namely atomic absorption spectrophotometry.

METHODS

Instruments and Materials

The instruments to be used are atomic absorption spectrophotometry, analytical scales, glass beakers, ball pipettes, measuring pipettes, dropper pipettes, measuring flasks, test tubes, stirring rods, ovens, furnaces, hot plates,

porcelain dishes, funnels, aluminum foil, brown glass bottles, and Whatman No. 42 filter paper. The materials to be used in this study are 5 samples of body lotion available in Semarang City, Lead Nitrate or PbNO3 p.a (Merck), HNO_3 68% p.a., HCl 37% p.a., KI 0.5N p.a., NaOH 2M p.a., dilute HCl 2M p.a., HNO_3 0.05M p.a., distilled water, and deionized water (Onemed).

Sample Preparation

Sample preparation was carried out using two methods, namely wet destruction and dry destruction. Wet destruction was carried out by weighing 2 grams of sample and placing it in a beaker, then adding 5 ml of 68% HNO_3 and 15 ml of 37% HCl. The mixture is then heated on a hotplate at 100°C until the destruction process is complete, indicated by the disappearance of brown vapor and a clear solution. Wet destruction is performed by first subjecting the sample to incineration using a furnace at a heating temperature of 500°C until complete, indicated by the sample turning into white ash.

Control Solutions

In this study, positive control samples with added lead were prepared using wet destruction and dry destruction methods. Negative control samples were prepared without added lead.

Qualitative Analysis

A total 1 ml of the test solution is reacted with several drops of reagents such as 0.5 N potassium iodide, 2 M NaOH, and 2 M dilute HCl. If the addition of potassium iodide reagent causes a yellow color change and the presence of a yellow-black precipitate, it is considered positive for lead. If the addition of sodium hydroxide reagent causes a white precipitate, it is considered positive for lead. If the addition of dilute hydrochloric acid reagent causes a white precipitate resembling needle-like crystals, it is considered positive for lead.

Quantitative Analysis

Calibration Curve Preparation

The calibration curve used was prepared with standard solutions with concentrations of 0.5, 1, 1.5, and 2 ppm. Then, the absorbance was measured at a wavelength of 283.3 nm.

Method Validation

The method validation parameters used in this study included linearity (r), limit of detection (LoD), limit of quantification (LoQ), precision (%RSD), and accuracy (%Recovery).

Determination of Lead Content in Samples

The samples were destroyed wet and dry, then transferred to a measuring flask and added with distilled water up to the 50 ml mark. The prepared samples were measured for absorbance using atomic absorption spectrophotometry at a wavelength of 283.3 nm.

RESULT AND DISCUSSION

Qualitatitve Analysis

In this study, qualitative analysis was performed by reacting a 1 ml sample with several drops of reagents such as 0.5 N potassium iodide, 2M sodium hydroxide, and 2M dilute hydrochloric acid.

Table 1. Qualitative Analysis Result

Reagent	A		B		C		D		E	
	DB	DK	DB	DK	DB	DK	DB	DK	DB	DK
KI 0,5 N	-	-	+	-	-	-	-	-	-	-
NaOH 2M	-	-	-	-	-	-	-	-	-	-
HCl encer 2M	-	-	-	-	-	-	-	-	-	-

Description:
DB: Wet Destruction
DK: Dry Destruction

The results show that testing of sample B using the wet destruction method with the addition of 0.5 N potassium iodide reagent was positive for lead (Pb) content, but samples tested using other wet and dry destruction methods, such as samples A, C, D, and E with the addition of KI, were negative for lead (Pb) content. Sample B using the wet digestion method showed a positive result for lead (Pb) content because the addition of 0.5 N potassium iodide reagent to the sample produced a reaction characterized by a color change to yellow and the formation of a yellow-black precipitate ($PbI_{2\ (s)}$) in the solution. The reaction between lead (Pb) and KI is as follows:

$$Pb^{2+}_{(aq)} + 2I^{-}_{(aq)} \longrightarrow PbI_{2(s)}$$

Testing of samples with the addition of 2M NaOH reagent using wet destruction and dry destruction methods showed negative results for lead (Pb) content. The addition of NaOH reagent was positive for lead (Pb) content if there was a change such as a white precipitate ($Pb(OH)_{2(s)}\ 2Na^{+}_{(aq)}$) in the solution. However, no such changes, such as the formation of white precipitates, were observed in any of the samples, and they were all tested negative for lead (Pb) content. The reaction between lead (Pb) and NaOH is as follows:

$$Pb^{2+}_{(aq)} + 2NaOH_{(aq)} \longrightarrow Pb(OH)_{2(s)} + 2Na^{+}_{(aq)}$$

Samples with the addition of dilute HCl reagent are considered positive for lead (Pb) content if they show a white precipitate resembling needle-like crystals ($PbCl_{2(s)} + H_{2(g)}$), but none of the samples using the wet destruction and dry destruction methods showed such changes. Therefore, samples using the wet destruction and dry destruction methods with the addition of dilute HCl reagent in this study can be considered negative for lead (Pb) content. The reaction between lead and dilute HCl is as follows:

$$Pb^{2+}_{(aq)} + 2HCl \longrightarrow PbCl_{2(s)} + H_{2(g)}$$

The qualitative analysis of lead (Pb) in body lotion samples without BPOM registration numbers yielded negative results for lead (Pb) content. This may be due to the low concentration of lead contamination in the samples, resulting in minimal binding of the test reagent to the metal, thereby producing less noticeable results. Qualitative analysis cannot detect lead (Pb) at very low concentrations. In sample B, wet destruction with the addition of KI reagent showed a positive result, which may be a false positive or indicate the presence of other metals in the sample, as KI reagent can also be used to determine the presence of other metals.

Method Validation

Method validation is the process carried out to demonstrate that the analytical procedure is suitable for its intended purpose. In this study, the validation parameters used for atomic absorption spectrophotometry were linearity, limit of detection (LOD), limit of quantification (LOQ), precision, and accuracy.

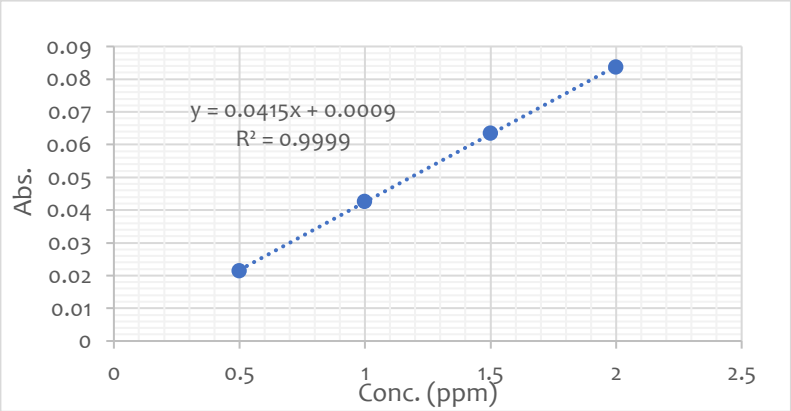

Figure 2. Curve Calibration of Atomic Absorption Spectrophotometry

The linearity results of this study showed a correlation coefficient (r) of 0.99995 and a coefficient of determination (R2) of 0.9999. This meets the requirement of r > 0.995. The LoD value obtained was 0.024 mg/L and the LoQ value obtained was 0.080 mg/L. Precision is expressed as %RSD with a percentage of 0.77% and is within the acceptance criteria of <2%. Accuracy is expressed as %recovery and ranges from 91% to 101%. For wet destruction method samples, the value obtained was 100.09%, and for dry destruction method samples, it was 91.21%; both meet the criteria of 80%-110%. All results meet the acceptance criteria, indicating that both methods are suitable for analyzing lead content in samples (Harmono, 2020; Sulistyani *et al*., 2021; Nurul & Sujana, 2020).

Determination of Lead Content in Samples

The concentration of lead in the samples was calculated using the linear regression equation obtained from the calibration curve by inserting the absorbance values obtained from each analytical method. Subsequently, calculations were performed to obtain the content of lead content present in the samples.

Table 2. Comparison of concentration determination results

Sample	Result (ppm)	
	Wet Destruction	Dry Destruction
A	1,2264	1,1133
B	1,7678	1,5542
C	1,5036	1,2682
D	1,1807	0,9814
E	1,5606	1,3927

The results obtained in determining the concentration in body lotion samples in Table 2 show that samples prepared by wet destruction and dry destruction have concentrations above the detection limit of 0.024 mg/L and the quantification limit of 0.080 mg/L on the Atomic Absorption Spectrophotometer instrument, so the results obtained from the body lotion samples are categorized as accurate. The percentage concentrations obtained from the body lotion samples using the wet destruction method were 2.96%; 4.24%; 3.75%; 2.92%; and 3.88%. The percentage concentrations obtained from the body lotion samples using the dry destruction method were 2.67%; 3.84%; 3.14%; 2.41%; and 3.41%.

The results of determining the concentration in body lotion samples between the wet destruction method and the dry destruction method show differences in the concentration and percentage obtained. This may occur because the recovery percentage or accuracy test on samples using the wet destruction method has a higher percentage compared to the recovery percentage of samples using the dry destruction method. The recovery percentage for wet digestion samples was 100.09% or equal to 100%, while for dry digestion samples, it was only 91.21%. In wet digestion samples, the analytes contained in the samples are not significantly lost because the digestion or decomposition process does not involve incineration, unlike dry digestion samples. The low concentration results in dry digestion samples are caused by the incineration process, which may result in excessive loss of analytes in the samples, and the primary purpose of this process is to convert organic compounds into inorganic compounds.

CONCLUSION

Samples prepared using wet destruction and dry destruction methods yielded the following lead (Pb) concentrations in wet destruction samples: 1.2265 ppm, 1.7687 ppm, 1.5036 ppm, 1.1807 ppm, and 1.5614 ppm. The concentrations obtained in the dry destruction samples were 1.1132 ppm, 1.5542 ppm, 1.2675 ppm, 0.9783 ppm, and 1.3927 ppm. The samples prepared using the wet destruction and dry destruction methods showed significant differences. This can be seen from the concentrations and percentage levels obtained. Samples prepared using the wet destruction method have higher concentrations and percentage levels compared to those prepared using the dry destruction method. This difference occurs because the dry destruction method involves a combustion process, which can result in lower concentration levels. In the study of five types of body lotion samples, all samples were found to contain lead (Pb), but the concentration levels in all body lotion samples are considered safe for use because the concentrations in all body lotion samples from Semarang City used in this study are below the limit set by the Indonesian Food and Drug Monitoring Agency (BPOM RI) Number HK.03.1.23.07.11.6622, which is 20 mg/L or 20 ppm.

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CONFLICT OF INTEREST

We declare that we don't have any conflict of interest.

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