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Analysis of Heavy Metal Content (Cd, Hg, and Pb) in Rice Harvested by Farmers in Dengilo District, Pohuwato Regency Using Atomic Absorption Spectrophotometry (AAS)

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ABSTRACT

Rice is the staple food for a significant portion of the global population, particularly in Indonesia. The objective of this study was to determine whether rice cultivated in Dengilo District, Pohuwato Regency, is contaminated with heavy metals. The research employed qualitative and quantitative methods using various reagents, including HNO3, 55% HCl, HClO4, H2SO4, Potassium Iodide (KI), NaOH, Na2CO3, and an Atomic Absorption Spectrophotometer (AAS) instrument. The results revealed that the qualitative tests showed positive contamination in rice samples A, B, and D, indicated by color changes and the formation of precipitates, while sample C tested negative. Quantitative tests confirmed the presence of heavy metals (Cd, Hg, and Pb) in all samples, although within permissible limits. Among the three metals, cadmium (Cd) exhibited the highest average concentration at 0.0465 mg/kg. This elevated Cd level is attributed to the repetitive use of phosphate fertilizers and pesticides, industrial waste discharge, vehicle combustion emissions, heavy metal residues from industrial processes, and atmospheric deposition. The study concludes that all rice samples tested positive for heavy metals (Cd, Hg, and Pb); however, their concentrations remain within the safe thresholds established by regulatory standards.

Keywords: *Rice, Heavy Metals, Atomic Absorption Spectrophotometry (AAS)*

INTRODUCTION

Rice (Oryza sativa L.) serves as the primary source of nutrition for a significant portion of the global population, making it a vital component of daily diets. Globally, rice is one of the most important staple foods, particularly in Asia, where it is consumed by the majority of its inhabitants. Asia accounts for approximately 90% of the world's rice production, underscoring the pivotal role of Asian farmers in the agricultural sector.

In Indonesia, a developing country where approximately 90% of the population relies on rice as a staple food, there is an urgent demand for rice free from heavy metal contaminants, which pose serious health risks. The quality of rice is influenced not only by agricultural practices but also by environmental factors, including heavy metal contamination linked to the use of inorganic fertilizers.

Rice is a cereal grain belonging to the family *Poaceae*, specifically within the genus *Oryza sativa*. In Indonesia, rice is highly versatile and thrives in diverse climatic conditions throughout the year. The world's leading rice producers are China and India, contributing approximately 35% and 20%, respectively, of total global production (Shafwati, 2012).

Food is considered safe when it is free from biological, chemical, or other harmful contaminants that may threaten human health. According to the Indonesian Government Regulation No. 29 of 2005 concerning Nutritional Data, Safety, and Quality, food safety encompasses all measures and conditions aimed at preventing contamination that could harm, disrupt, or endanger human health.

MATERIALS & METHODS

This study is an experimental research aimed at determining the heavy metal content (Cd, Hg, and Pb) in rice harvested by farmers in Dengilo District, Pohuwato Regency, using the wet digestion method and Atomic Absorption Spectrophotometry (AAS).

Materials and Equipment Used

Equipment used in this study includes:
Aluminum foil, stirring rod (*Gloryamedika*), porcelain crucible (*SMLaborta*), glass funnel (*Pyrex*), Erlenmeyer flask (*Pyrex*), measuring cylinder (*Pyrex*) 25 mL, volumetric flask (*Pyrex*) 25 mL, analytical balance (*Osuka*), test tube (*Pyrex*), test tube rack (*Saptamedica*), dropper pipette (*Saptamedica*), volumetric pipette (*Iwaki*), spatula (*SamMedical*), and Atomic Absorption Spectrophotometer (*Perkin Elmer*).

Materials used in this study include:

Rice samples obtained from paddy fields in Dengilo District, aquadest, HNO3, 55% HCl, HClO4, H2SO4, potassium iodide, NaOH, Na2CO3, and filter paper. All chemicals were pro-analysis (p.a.) grade unless otherwise specified.

Research Procedure

Sample Collection

The samples used in this study were rice harvested by farmers from paddy fields in Dengilo District, Pohuwato Regency.

Qualitative Analysis

Qualitative analysis was performed using aqua regia, a solution containing a mixture of concentrated hydrochloric acid and concentrated nitric acid in a ratio of 3:1. Initially, 0.5 g of rice sample was weighed and treated with 10 mL of aqua regia solution. The mixture was left to stand for at least one hour, followed by heating for 5 minutes. Subsequently, potassium iodide, NaOH, and Na2CO3 solutions were added to each sample. The presence of heavy metals was indicated by observable color changes and the formation of precipitates.

Quantitative Analysis

For quantitative analysis, two variations of the wet digestion method were applied to the dry rice samples. In the first method, approximately 0.5 g of dried rice sample was weighed and treated with 2 mL of Aquamin and 9 mL of a concentrated HNO3 and HCl mixture (1:3). The mixture was heated at 105°C for 3 hours and 15 minutes. The resulting filtrate was then filtered and diluted with Aquamin in a 50 mL volumetric flask. In the second method, approximately 3 g of dried rice sample was weighed and treated with 25 mL of Aquamin and 8 mL of concentrated nitric acid. The mixture was heated on a hot plate at 105°C. After cooling, 5 mL of concentrated HNO3 and 3 mL of HClO4 were added, and the mixture was reheated for approximately 30 minutes. The filtrates obtained from both methods were filtered and diluted with Aquamin in a 100 mL beaker. These filtrates were directly analyzed using Atomic Absorption Spectrophotometry (AAS). This methodology allowed for precise determination of heavy metal contamination in the rice samples, providing insight into the safety and quality of rice produced in the study area.

RESULTS

Qualitative Test Results

The qualitative test aims to identify the presence of specific chemical compounds, whether organic (originating from living organisms) or inorganic (mineral-based). In this study, four reagents were used: KI, NaOH, Na2CO3, and HCl. The testing procedure involved preparing each sample as a reactant. Then, 1–3 drops of the sample solutions, labeled for identification, were added to each reagent. Changes, such as the formation of precipitates or color changes, were observed. A positive result for cadmium contamination was indicated by the formation of a white precipitate, mercury by a yellow color or yellow-orange precipitate, and lead by a white precipitate.

Tabel 1. Qualitative Test Results for Heavy Metals (Mercury, Cadmium, and Lead)

No.	Heavy Metal	Reagent	Sample A	Sample B	Sample C	Sample D
1	Mercury	NaOH	-	+	-	+
		KI	+	-	-	-
		Na_2CO_3	-	-	-	-
2	Cadmium	NaOH	-	-	-	-
		KI	+	-	-	-
		Na_2CO_3	-	-	-	-
3	Lead	NaOH	-	+	-	-
		KI	+	-	-	-
		Na_2CO_3	-	-	-	-

Legend:
(+) = Contains heavy metal
(-) = Does not contain heavy metal

Table 1 demonstrates that samples A, B, and D tested positive for heavy metals, as indicated by color changes or precipitate formation. In contrast, sample C tested negative, as no precipitate or color change was observed.

Quantitative Test Results

The quantitative test results in this study show the concentrations of heavy metals (Cd, Hg, and Pb) in rice samples from Dengilo District, Pohuwato Regency, as measured using Atomic Absorption Spectrophotometry (AAS). The data are presented in the table below.

Table 2. Heavy Metal Concentrations in Rice Samples Measured by AAS

No.	Sample	Mercury (mg/kg)	Cadmium (mg/kg)	Lead (mg/kg)	Description
1	A	≤0,01	0,0300	≤0,001	Below Limit
2	B	≤0,01	≤0,001	≤0,001	Below Limit
3	C	≤0,01	0,0225	≤0,001	Below Limit
4	D	≤0,01	0,1325	≤0,001	Below Limit
Average		**≤0,01**	**0,0465**	**≤0,001**	**Below Limit**

Grafik logam berat

Calibration Curve Graphs for Heavy Metals (Hg, Cd, and Pb) in Rice Samples from Dengilo District, Pohuwato Regency.

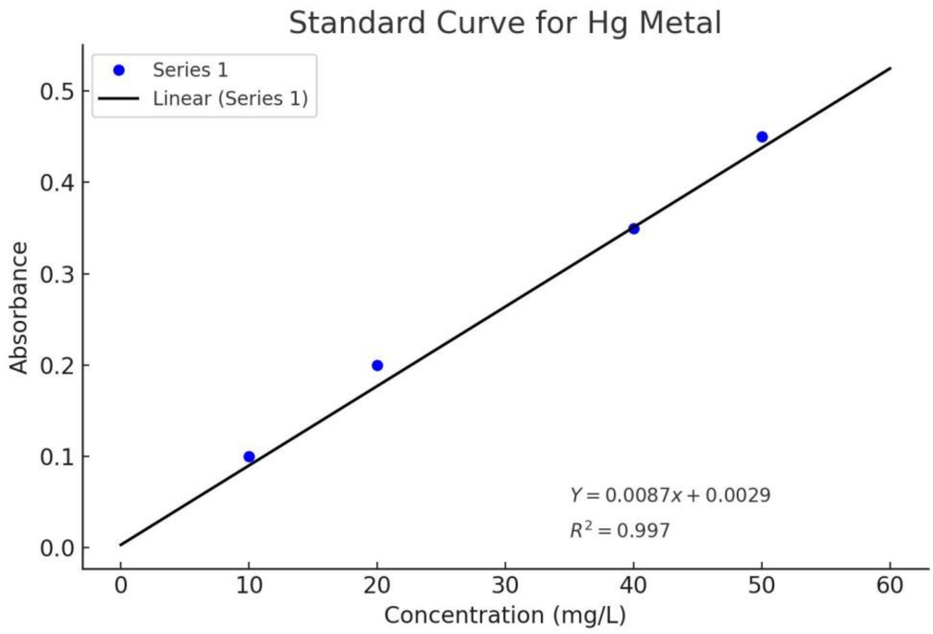

Figure 1. Mercury Graph

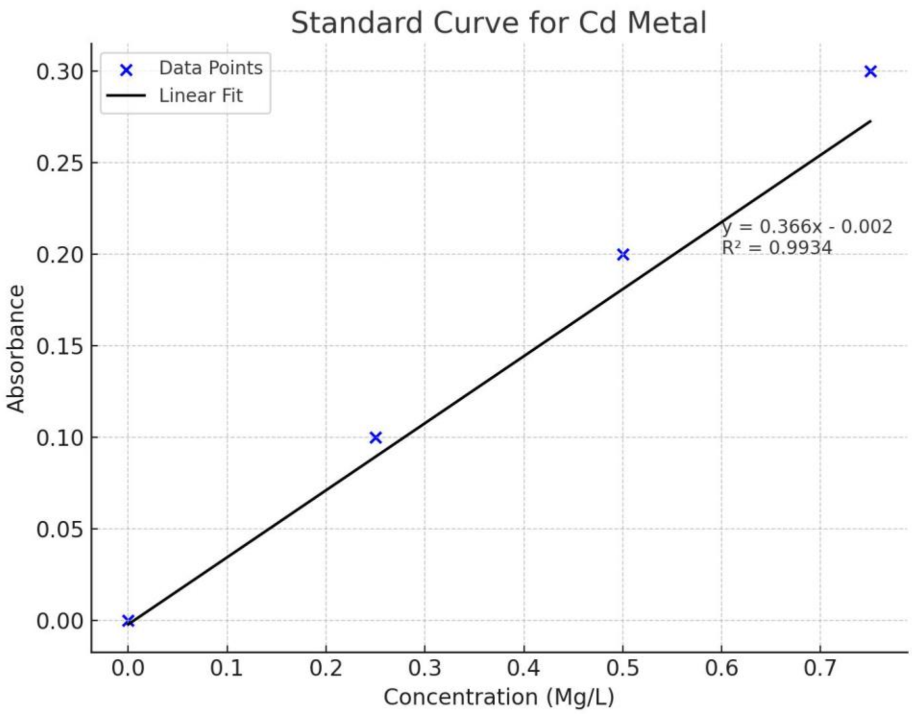

Figure 2. Cadmium Graph

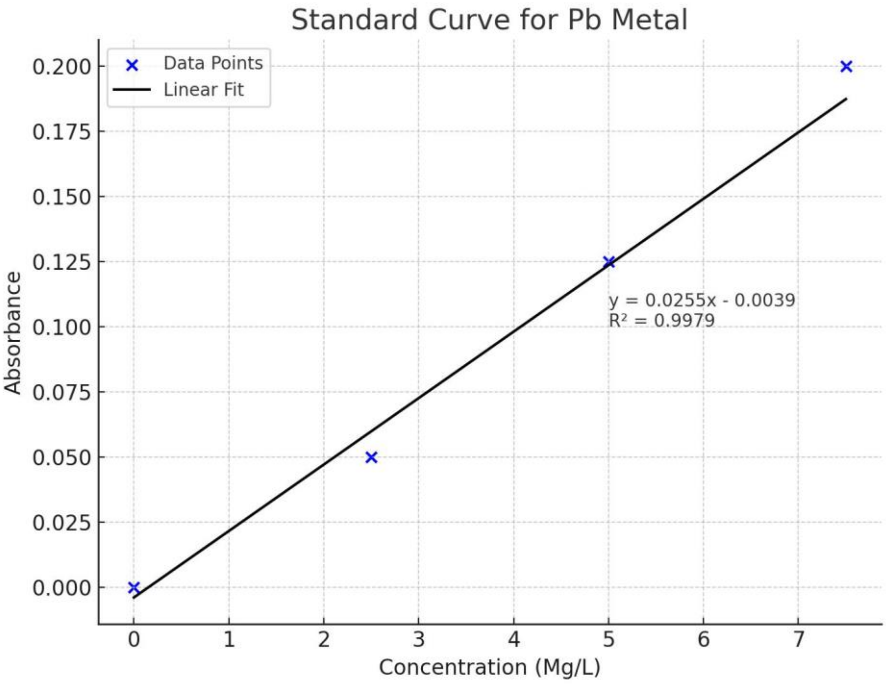

Figure 3. Lead Graph

DISCUSSIONS

The qualitative analysis of heavy metals (cadmium, mercury, and lead) revealed that two out of four rice samples tested positive for mercury contamination. Using reagents such as NaOH and KI, sample B and sample D were confirmed positive when tested with NaOH, as indicated by the formation of yellow/orange precipitates. Sample A tested positive with KI, evidenced by the formation of an orange precipitate. In contrast, sample C showed negative results as no yellow/orange precipitate was observed. Mercury testing is considered positive when a yellow/orange precipitate is formed, as supported by Hasma and Panaungi (2023).

Chemical Reactions Involved:
NaOH: $Hg^{2+} + 2OH^{-} \rightarrow HgO + H_2O$ (Yellow Precipitate)
KI: $Hg2^{+} + 2KI \rightarrow HgI2) + 2K^{+}$ (Yellow/Orange Precipitate
Na_2CO_3: $Hg_3^{2-} + CO_3^{2-} \rightarrow Hg_2CO_3$ (Yellowish Precipitate)
After conducting mercury testing, the samples were further analyzed for cadmium content. Based on qualitative tests for cadmium using reagents, only one sample (Sample A) tested positive with the KI reagent, indicated by the formation of a yellow precipitate.

Chemical Reactions:
NaOH = $Pb^{2+} + 2OH^{-} \rightarrow Pb(OH)_2$ (White Precipitate)
KI = $Pb^{2+} + 2I^{-} \rightarrow PbI_2$ (Yellow Precipitate)
Na_2CO_3 = $Pb^{2+} + CO_3^{2-} \rightarrow PbCO_3$ (White Precipitate)

Positive results, indicated by changes in color, led to further testing of the samples for lead content. The lead analysis showed that 2 out of 4 samples tested positive using NaOH and KI reagents. With NaOH, only Sample B tested positive, marked by a white precipitate. For KI, only Sample A tested positive, indicated by a yellow precipitate. Samples C and D were negative as no color change or white precipitate was observed. Positive lead detection is confirmed by color changes or the presence of a white precipitate (Arifiyana, D., 2018).

Chemical reactions:
NaOH = $Pb^{2+} + 2OH^{-} \rightarrow Pb(OH)_2$ (White precipitate)
KI = $Pb^{2+} + 2I \rightarrow PbI_2$ (Yellow precipitate)
Na_2CO_3 = $Pb^{2+} + CO_3^{2-} \rightarrow PbCO3$ (White precipitate)

The absence of detectable heavy metals (Cd, Hg, Pb) in Sample C during qualitative testing may result from the low concentration of these metals in the sample. Advanced detection using Atomic Absorption Spectroscopy (AAS) is recommended, as AAS offers the advantage of measuring metal concentrations even at trace levels (Kristianingrum, 2012).

Based on quantitative testing using Atomic Absorption Spectroscopy (AAS), rice samples were found to contain heavy metals within safe limits. The average mercury (Hg) and lead (Pb) levels across all four samples were 0.001 µg/L, while cadmium (Cd) levels averaged 0.0465 µg/L. These results indicate higher cadmium levels compared to mercury and lead. Mariwy (2021) reported varying mercury levels in rice samples from different villages, ranging from 0.002 mg/kg to 0.027 mg/kg, with the highest levels linked to gold processing waste contaminating agricultural fields.

Although the heavy metal content is within permissible standards, caution is advised due to the potential for bioaccumulation. Heavy metals, once absorbed, cannot be excreted and may accumulate over time, causing severe health issues such as neurological disorders, kidney damage, organ dysfunction, reproductive problems, and cancer (Mulyadi, 2013). Heavy metals spread through air pollution, food, soil, or water, accumulating in the body with prolonged exposure.

Contamination in rice can originate from polluted water and soil, often due to industrial waste, mining, agricultural runoff, or pesticide use. Excessive use of chemical fertilizers can degrade soil quality, increase harmful mineral content, and elevate cadmium levels, disrupting photosynthesis by affecting chloroplasts and thylakoid membranes, thus stunting plant growth (Mahfodiyawati et al., 2016).

CONCLUSION

Based on the research findings regarding the analysis of heavy metal content (Cd, Hg, and Pb) in rice from Dengilo District, Pohuwato Regency, using Atomic Absorption Spectroscopy (AAS), it was observed that certain samples exhibited contamination. Qualitative testing revealed that samples A, B, and D were positive for heavy metals, as indicated by noticeable color changes during the tests, while sample C was negative, showing no color changes. Quantitative analysis showed that all samples contained cadmium (Cd), lead (Pb), and mercury (Hg) at levels below the standard limits for heavy metal contamination. The concentrations were as follows: cadmium (Cd) at 0.0465 µg/L, lead (Pb) at 0.001 µg/L, and mercury (Hg) at 0.01 µg/L. Among these, cadmium showed the highest concentration; however, it remains within acceptable safety limits. These results highlight the importance of ongoing monitoring to prevent potential health risks from heavy metal bioaccumulation, even when contamination levels are low.

Declaration by Authors

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