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Spectroscopic Evaluation and Analytical Validation of Lead (Pb) Determination in River Water Using Atomic Absorption Spectrophotometry

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ABSTRACT

Purpose of the study: This study aims to determine the presence or absence of lead (Pb) in the Kelay River water and to quantify its concentration using atomic absorption spectrophotometry as a reliable analytical technique for environmental monitoring.

Methodology: This study used atomic absorption spectrophotometry (Varian Spectr AA) for analysis. Supporting tools included analytical balance (KERN ALJ 220-4 NM), volumetric glassware (Pyrex), and electric heater. Methods involved judgment sampling, acid digestion using HNO3, preparation of Pb(NO3)2 standard solutions, calibration curve construction, and linear regression analysis.

Main Findings: Lead (Pb) was detected in Kelay River water samples. The concentrations in samples D and E were 0.0773 mg/L and 0.0634 mg/L, respectively, exceeding acceptable limits. In contrast, samples A, B, and C showed concentrations below 0.01 mg/L. The calibration curve exhibited strong linearity with a high correlation coefficient.

Novelty/Originality of this study: This study applies an analytical spectroscopy-based approach to determine lead (Pb) levels in a specific river system with consideration of local environmental characteristics. It integrates calibration and detection limit evaluation, contributing to improved analytical reliability and providing new data for environmental assessment in underreported regions.

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1. INTRODUCTION

Heavy metal pollution in water is a significant environmental issue due to its toxicity, persistence, and ease of accumulation in the food chain [1], [2]. One heavy metal that has received significant attention is lead (Pb), which originates from industrial activities, mining, and domestic waste [3], [4]. The presence of Pb in water can have serious impacts on human health and aquatic ecosystems [5], [6]. Therefore, monitoring Pb concentrations in water is crucial to ensure environmental quality is maintained. Accurate and sensitive analysis is essential for detecting this metal at low concentrations.

In the context of chemical analysis, spectroscopy is a widely used approach to detect and quantify metal elements based on the interaction between electromagnetic radiation and atoms. Atomic absorption spectrophotometry works based on the principle of radiation absorption by free atoms at specific wavelengths specific to each element [7], [8]. For Pb, a commonly used wavelength is around 283.3 nm, which provides high sensitivity to changes in concentration. The relationship between absorbance and concentration follows the principle of linearity, which is the basis of analytical quantification [9], [10]. Thus, the spectroscopic approach not only provides quantitative results but also reflects the characteristics of the interaction of atoms with radiation [11], [12].

Although atomic absorption spectrophotometry has been widely used, the reliability of analytical results depends heavily on the validation of analytical performance and the interpretation of the resulting spectroscopic signals [13], [14]. Factors such as matrix interference, signal stability, and instrument operating conditions can affect the accuracy and precision of measurement results [15], [16]. Therefore, evaluating analytical parameters such as linearity, detection limit, sensitivity, and reproducibility is crucial for ensuring data quality [17], [18]. Furthermore, understanding the characteristics of spectroscopic signals allows for a more in-depth interpretation of the results. This approach aligns with developments in modern spectroscopic research, which emphasizes the importance of data reliability and interpretability.

The research conducted by Ihenetu et al., [19] focused on determining the level of heavy metal pollution in waters using a quantitative approach using spectrophotometry and atomic absorption spectrophotometry, followed by a health risk evaluation, thus focusing more on environmental aspects and toxicological impacts. Meanwhile, the research by Adinda et al., [20] also focused on determining Cd and Pb levels using atomic absorption spectrophotometry for health laboratory analysis purposes, with a primary focus on concentration results and their compliance with quality standards. Both studies share similarities in the use of spectroscopic techniques as a measurement tool, but still position spectroscopy only as an analytical instrument without a comprehensive in-depth exploration of signal characteristics, method validation, and analytical performance. In fact, in spectroscopic-based analysis, the quality of results is greatly influenced by the interaction of atoms with radiation as well as analytical parameters such as sensitivity, linearity, and detection limits that determine data reliability. Therefore, this study fills this gap by emphasizing comprehensive spectroscopic evaluation and analytical validation, thus not only producing Pb concentration data but also providing a deeper understanding of signal quality and method reliability in environmental analysis.

The novelty of this research lies in its approach, which focuses not only on determining Pb concentrations in river water but also on comprehensive evaluation of spectroscopic signal characteristics and validation of analytical performance. Unlike previous studies that generally position atomic absorption spectrophotometry as a quantification tool alone, this study emphasizes the relationship between absorbance response, the atomization process, and analytical parameters such as sensitivity, linearity, and detection limits [21], [22]. This approach provides a scientific contribution in the form of a deeper understanding of method reliability under complex environmental matrix conditions. The urgency of this research is driven by the need for analytical methods that are not only numerically accurate but also accountable in terms of signal quality and measurement stability. Thus, this research is important in supporting the development of more valid, sensitive, and interpretive spectroscopy-based environmental analysis.

This study aims to evaluate the spectroscopic characteristics and analytical performance in determining Pb levels in river water samples using atomic absorption spectrophotometry. In addition to determining Pb concentration, this study also emphasizes the analysis of the relationship between absorbance signals and concentration and the validation of relevant analytical parameters [23], [24]. This approach is expected to yield results not only quantitatively accurate but also provide a solid basis for spectroscopic interpretation [25], [26]. This study also contributes to understanding the influence of environmental matrices on the resulting spectroscopic response. Thus, this research is expected to strengthen the application of spectroscopic methods in environmental analysis in a more comprehensive manner.

2. RESEARCH METHOD

2.1. Tools and Materials

This study utilized various tools and materials to support the analysis of lead (Pb) levels in water samples. The equipment used included aluminum foil, plastic bottles, porcelain cups with lids, and heat-resistant glass funnels. Laboratory glassware such as Erlenmeyer flasks, graduated cylinders, and volumetric flasks, each with a high degree of precision, was also utilized [27], [28]. The weighing process was carried out using a high-precision analytical balance to ensure the accuracy of the mass of the materials used. For the analysis of lead (Pb) levels, an atomic absorption spectrophotometer equipped with an electric heating system was used, along with various tools, such as volumetric pipettes, to ensure accurate volume measurements.

The materials used in this study included distilled water as a solvent, concentrated nitric acid (HNO3) as a reagent for the sample digestion process, and lead nitrate (Pb(NO3)2) as a standard solution for creating a

calibration curve. The sample analyzed was water from the Kelay River, taken from the research site in Berau Regency. The use of high-purity chemicals minimized the possibility of contamination during the analysis process. Furthermore, all materials are prepared in accordance with established laboratory procedures to ensure consistent measurement results [29], [30]. This combination of tools and materials is expected to optimize the analysis process and produce accurate and scientifically reliable data.

2.2. Sample Collection

Water samples were collected using a judgment sampling technique, in which sampling locations were selected based on the researcher's consideration to ensure representativeness of environmental conditions [31], [32]. Samples from the Kelay River were obtained from five different locations determined by their distance from the mining conveyor area. At each location, samples were collected from three points across the river, from the left bank to the right bank, to ensure spatial representation. The collected subsamples at each location were combined into a composite sample and stored in sampling bottles. The sampling points consisted of location A at the mining conveyor area, location B at a distance of 100 meters, location C at 500 meters, location D at 1 kilometer, and location E at 1.5 kilometers from the conveyor.

2.3. Sample Preparation

Sample preparation was carried out by transferring 50 milliliters of water sample into a beaker. Concentrated nitric acid (HNO3) was then added until the pH of the solution was below two to preserve and dissolve the metal content. The solution was heated using an electric heater until the volume was nearly dry [33]. Subsequently, distilled water was added, and the solution was filtered through filter paper into a 50 milliliter volumetric flask. The volume was then adjusted to the mark using distilled water to obtain a solution ready for analysis.

2.4. Preparation of Blank Solution

The blank solution was prepared by transferring 2 milliliters of 0.5 molar nitric acid (HNO3) into a 100 milliliter measuring cylinder. The solution was then diluted to the mark with distilled water. This blank solution was used as a control to eliminate the influence of solvents and reagents during measurement. The use of a blank is essential to ensure that the measured absorbance originates solely from the analyte of interest [34], [35]. This procedure enhances the accuracy of the analytical results.

2.5. Preparation of Lead Standard Stock Solution

The lead stock solution was prepared by weighing 1.6 grams of lead nitrate (Pb(NO3)2) and transferring it into a 1000 milliliter volumetric flask [36]. Subsequently, 10 milliliters of concentrated nitric acid (HNO3) were added to facilitate dissolution. The solution was then diluted with distilled water to the calibration mark to obtain a stock solution with a concentration of 1000 parts per million. This stock solution served as the primary source for preparing standard solutions of lower concentrations. Careful preparation was conducted to ensure the accuracy of the concentration.

2.6. Preparation of Standard Solutions and Calibration Curve

The calibration curve was established by preparing a series of standard lead solutions through stepwise dilution. A 100 parts per million standard solution was prepared by diluting 10 milliliters of the 1000 parts per million stock solution to 100 milliliters. Subsequently, standard solutions with concentrations of 0.1, 0.5, 1.0, 1.5, and 2.0 parts per million were prepared by pipetting appropriate volumes into 100 milliliter volumetric flasks. Each solution was added with 3 milliliters of 0.5 molar nitric acid (HNO3) and diluted to the mark with distilled water. These standard solutions were used to establish the relationship between concentration and absorbance. The calibration curve was then constructed based on the measured absorbance values.

2.7. Calibration Measurement

Calibration measurements were performed by introducing each standard solution into the atomic absorption spectrophotometer [37], [38]. Measurements were conducted at a wavelength of 283.3 nanometers, which is specific for lead detection. The absorbance values obtained for each concentration were recorded. These data were used to construct the calibration curve and determine the linear regression equation. The resulting equation served as the basis for calculating lead concentrations in the samples.

2.8. Determination of Lead Concentration in Samples

The prepared sample solutions were introduced into the atomic absorption spectrophotometer for analysis. Measurements were carried out at the same wavelength used for the standard solutions, namely 283.3 nanometers. The absorbance values obtained were recorded and used to calculate the lead concentration based on the regression equation derived from the calibration curve [39], [40]. The analysis was conducted carefully to ensure data

consistency and reliability. The results were then interpreted to assess the level of lead contamination in the river water.

2.9. Data Analysis

Data analysis was performed using simple linear regression to establish the relationship between concentration and absorbance. The regression equation was expressed as a linear relationship between the independent and dependent variables [41], [42]. The slope and intercept values were used to calculate the concentration of lead in the samples. In addition, the correlation coefficient was determined to evaluate the strength of the relationship between the variables. The limit of detection was also calculated statistically based on the calibration data to assess the sensitivity of the analytical method.

3. RESULTS AND DISCUSSION

3.1. Determination of Absorbance of Standard Lead Solution

The absorbance data of the lead standard solution was used to create a linear regression equation. Several series of lead (Pb) standard solutions were prepared with various concentrations, namely 0; 0.1; 0.5; 1.0; 1.5; and 2.0 ppm, then operated on an Atomic Absorption Spectrophotometer. The data obtained are presented in Table 1.

Table 1. Results of standard lead (Pb) measurements

No.	Level (ppm)	Absorption (A)
1.	0.0000	-0.0005
2.	0.1000	0.0048
3.	0.5000	0.0135
4.	1.0000	0.0270
5.	1.5000	0.0402
6.	2.0000	0.0534

The absorbance data of the Pb standard solution shows an increase in absorbance values along with increasing concentration within the analyzed range. The absorbance value at a concentration close to zero indicates that the background signal contribution is relatively small and does not have a significant effect on the measurement. The consistent increase in absorbance from a concentration of 0.1 to 2.0 ppm reflects a linear relationship between the number of Pb atoms and the absorbed radiation energy. This indicates that the atomization process takes place effectively so that the number of Pb atoms in the gas phase is proportional to the solution concentration. Thus, these data indicate that the atomic absorption spectrophotometry method used has a stable signal response and is suitable for quantitative Pb analysis.

3.2. Determining the Standard Linear Equation

To determine the standard linear regression line equation, you can use the formula Y = ax + b. The complete calculation can be seen in Appendix 2. Based on the calculation results, the standard solution linear regression line equation is obtained:

$$Y = 0.02636X + 0.00066$$

With the correlation coefficient of X against Y is:

$$R = 0.99831$$

The obtained linear regression equation indicates a very strong relationship between Pb concentration and the resulting absorbance value. The slope value of 0.02636 reflects the sensitivity of the method, where each increase in Pb concentration results in a proportional increase in absorbance. The very small intercept indicates that the contribution of the background signal is relatively insignificant to the measurement results. The correlation coefficient value close to one indicates that the data has an excellent fit to the linear model. Thus, the obtained regression equation can be used reliably to determine the Pb concentration in samples based on the resulting spectroscopic response.

3.3. Determination of the Calibration Curve for Lead Standard Solution

Based on the absorbance data of a series of standard solutions, a standard/calibration curve can be created to measure the absorbance. The lead calibration curve is created based on the absorbance value of a standard lead solution, where Y is the absorbance of the standard lead solution and X is the lead solution concentration.

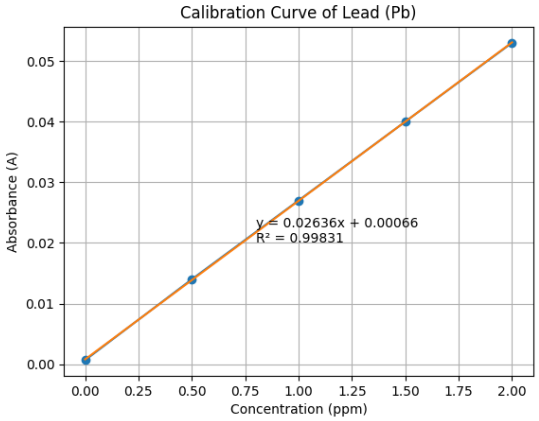

Figure 1. Lead (Pb) standard curve

The calibration curve shows a linear relationship between the absorbance value and the Pb concentration in the standard solution used. The slope of the curve reflects the sensitivity of the method, where small changes in concentration result in significant measurable changes in absorbance. An intercept value close to zero indicates that the influence of the background signal is relatively small and does not contribute significantly to the measurement results. A high coefficient of determination indicates that the linear model is able to accurately represent the relationship between signal and concentration. Thus, the obtained calibration curve can be used as a reliable quantification basis for Pb analysis in water samples.

3.4. Determination of Sample Solution Absorbance

The absorbance of the sample solution was obtained by injecting the Kelay River water sample solution into an atomic absorption spectrophotometer. The sample absorbance measurement results were then substituted into the predetermined regression line equation. The absorbance and concentration data for the Kelay River water sample solution are presented in the following table.

Table 2. Results of calculations of lead (Pb) levels in Kelay River water samples using Atomic Absorption Spectrophotometry

No	Sample Code	Requirements (mg/l)	Absorbance (A)	Lead Content (mg/l)	Information
1	A		-0.0030	< 0.001	Normal
2	B		0.0001	< 0.001	Normal
3	C	0.01	-0.0004	< 0.001	Normal
4	D		0.0027	0.0773	Exceeding
5	E		0.0021	0.0634	Exceeding

The absorbance values obtained from Kelay River water samples show variations in spectroscopic responses reflecting differences in Pb content at each sampling point. Some samples showed very small absorbance values, even approaching zero, indicating that the number of Pb atoms in the gas phase was at a very low level or below the method's limit of quantification. Conversely, samples with higher absorbance values produced significantly measurable Pb concentrations after being substituted into the regression equation. This difference indicates that the spectroscopic response is highly dependent on the number of Pb atoms capable of absorbing radiation under the same atomization conditions. Thus, these data not only show variations in Pb levels but also reflect the sensitivity and ability of the method to distinguish Pb concentrations at different levels in environmental samples.

3.5. Determination of Detection Limit

The detection limit is obtained through statistical data of the regression line equation Y = 0.02636x + 0.00066, with the calculation formula:

$$SD = \sqrt{\frac{\sum\{(Y-Y)^2}{n-2}} \quad LOD = \frac{3\,x\,S}{S}$$

so that the detection limit value obtained was 0.01098 ppm.

The detection limit value obtained of 0.01098 ppm indicates the method's ability to detect the presence of Pb at very low concentrations in the sample. This value reflects the minimum limit at which the absorbance signal can still be significantly distinguished from the background signal. This detection ability is greatly influenced by signal stability and low interference from instrument noise. The lower the detection limit value, the higher the method's sensitivity in identifying the presence of Pb at trace levels. Thus, the obtained value indicates that the atomic absorption spectrophotometry method used is sensitive enough for environmental water quality monitoring applications.

The calibration curve results show that the relationship between Pb concentration and absorbance values follows a very good linear pattern within the tested concentration range [43], [44]. This linearity indicates that the interaction between electromagnetic radiation and Pb atoms occurs consistently according to the principles of atomic absorption. The slope of the curve reflects the sensitivity of the method, where small changes in concentration produce detectable signal changes [45]. The intercept close to zero indicates that the contribution of background signals is relatively small and therefore does not interfere with measurement accuracy. Thus, the obtained calibration curve provides a solid basis for spectroscopic-based quantitative analysis.

Interpretation of the spectroscopic signal indicates that the measured absorbance originates from the number of Pb atoms in the gas phase, capable of absorbing radiation at specific wavelengths. The atomization process in the flame allows the formation of free atoms that interact directly with the radiation, resulting in a signal proportional to the concentration [46], [47]. The stability of the signal observed during the measurement indicates that the instrument's operating conditions are optimal. This is important because signal fluctuations can cause uncertainty in the analysis results. Therefore, the stability of the spectroscopic response is a key indicator in assessing the quality of the method used.

The obtained detection limit values indicate that the method has good capability in detecting Pb at low concentrations. This limit is determined by the ratio between the analytical signal and noise, which reflects the overall quality of the measurement system. A relatively small value indicates that interference from the background and the instrument can be effectively minimized. This detection capability is crucial in environmental analysis, where heavy metal concentrations are often at trace levels. Therefore, the method used is reliable for sensitive water quality monitoring.

Sample measurement results show significant variations in Pb concentrations between sampling points. Samples with absorbance values close to zero indicate that Pb concentrations are below the limit of quantification and cannot be accurately determined [48], [49]. Conversely, samples with higher absorbance values indicate the presence of significantly measurable Pb levels. This difference reflects variations in environmental conditions and the possibility of local pollution sources. Therefore, spectroscopic analysis not only provides quantitative data but also provides an overview of the distribution of pollutants in the aquatic system.

The presence of other components in the sample matrix can affect the spectroscopic response generated during the measurement [50], [51]. Other ions can interfere with the atomization process or affect the efficiency of radiation absorption by Pb atoms. In addition, suspended particles and complex compounds can cause changes in the measured signal intensity. The influence of this matrix can cause deviations between measured and true values [52], [53]. Therefore, understanding matrix effects is crucial to ensure accurate and representative analysis results.

The precision and accuracy of the method demonstrate that the analysis system has a good level of reliability in producing consistent data that is close to the true value [54], [55]. A low relative standard deviation indicates that variation between measurements can be well controlled [56], [57]. A recovery value approaching 100% indicates that the method does not experience significant analyte loss during the analysis process. This indicates that the method has good validity for quantitative Pb analysis. Thus, the combination of precision and accuracy strengthens confidence in the results obtained.

Overall, the results of this study indicate that the atomic absorption spectrophotometry method has good analytical performance for determining Pb in river water samples. The method's reliability is supported by high linearity, good sensitivity, and adequate signal stability. Furthermore, evaluation of analytical parameters indicates that the method is capable of producing accurate and precise results [58], [59]. The spectroscopic approach used not only enables quantification but also provides insight into the characteristics of the resulting signal [60], [61]. Thus, this method can be used as an effective tool in spectroscopy-based environmental analysis.

This study provides important contributions to environmental monitoring by demonstrating that atomic absorption spectrophotometry can produce reliable and sensitive data for detecting trace levels of lead (Pb) in river water. The findings offer practical implications for environmental agencies in assessing water quality and identifying potential pollution sources, particularly in areas affected by mining activities [62], [63]. Furthermore, the integration of spectroscopic signal evaluation and analytical validation strengthens the scientific basis for improving the accuracy and interpretability of environmental analysis methods [64], [65].

However, this study has several limitations. The use of judgment sampling may limit the representativeness of the sampling locations, potentially affecting the generalization of the results [66], [67]. In

addition, matrix interference from other dissolved substances in river water may influence the spectroscopic response, despite efforts to control analytical conditions [68], [69]. The study also focuses only on Pb without considering other heavy metals that may coexist and contribute to environmental risk [70], [71]. Future research is recommended to apply more comprehensive sampling strategies, include multi-element analysis, and explore advanced techniques to minimize matrix effects and enhance analytical performance.

4. CONCLUSION

Based on the results of the study, it can be concluded that lead (Pb) was successfully detected in Kelay River water samples, with concentrations of 0.0773 mg/L and 0.0634 mg/L found in samples D and E, respectively, exceeding the acceptable limits, while samples A, B, and C showed concentrations below 0.01 mg/L. Furthermore, the atomic absorption spectrophotometry method demonstrated excellent analytical performance, indicated by high linearity (R = 0.99831), good sensitivity, and a low detection limit of 0.01098 ppm. The stable spectroscopic response confirms that the method is reliable for quantitative Pb determination in environmental water samples. Overall, this study highlights that the integration of spectroscopic evaluation and analytical validation provides not only accurate quantitative results but also strengthens the reliability and interpretability of environmental analysis. Future research is recommended to expand the scope of analysis by including multiple heavy metals and applying more representative sampling techniques across broader spatial and temporal scales. In addition, the use of advanced analytical methods with higher sensitivity and selectivity is suggested to minimize matrix interference and to enhance the accuracy and reliability of environmental monitoring results.

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AUTHOR CONTRIBUTIONS

Conceptualization, R.N.A. and E.L.; Methodology, R.N.A.; Software, R.N.A.; Validation, E.L. and A.M.; Formal Analysis, R.N.A.; Investigation, R.N.A.; Resources, E.L.; Data Curation, R.N.A.; Writing – Original Draft Preparation, R.N.A.; Writing – Review & Editing, E.L. and A.M.; Visualization, R.N.A.; Supervision, A.M.; Project Administration, E.L.; Funding Acquisition, E.L.

CONFLICTS OF INTEREST

The authors declare no conflict of interest.

USE OF ARTIFICIAL INTELLIGENCE (AI)-ASSISTED TECHNOLOGY

The authors declare that no artificial intelligence (AI) tools were used in the generation, analysis, or writing of this manuscript. All aspects of the research, including data collection, interpretation, and manuscript preparation, were carried out entirely by the authors without the assistance of AI-based technologies.

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