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Which Is More Reliable? ICP-OES vs AAS for Chromium and Lead Analysis in Heavy Metal Monitoring

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ABSTRACT

Purpose of the study: This study aims to compare the accuracy and reliability of Inductively Coupled Plasma Optical Emission Spectroscopy and Atomic Absorption Spectrometry in analyzing heavy metal contents specifically lead (Pb) and chromium (Cr) in textile wastewater.

Methodology: The study employed a descriptive experimental method using water samples collected from a river suspected of contamination by textile waste. Tools used include AAS (APHA.322 B-2005), ICP OES (IK/LEL-ITS/ICP), and various laboratory glassware. Sample preparation followed SNI 6989.57:2008, and analytical validation included precision, accuracy, linearity, LOD, and LOQ testing.

Main Findings: Both methods showed acceptable validation values, with linear regression coefficients (R^2) above 0.995. ICP OES and AAS demonstrated good precision (%RSD $\leq$ 2%) and recovery rates between 93%–105%. However, significant differences were found between the two instruments ($p = 0.012$). Pb and Cr concentrations at discharge points exceeded regulatory limits.

Novelty/Originality of this study: This study presents a comparative evaluation of ICP OES and AAS in the context of environmental monitoring, providing essential insights into their suitability for detecting heavy metals in complex wastewater matrices.

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1. INTRODUCTION

The development of industrial technology is currently experiencing very rapid progress [1]-[3]. All of these achievements are aimed at improving the quality of human life. However, behind this progress, there are negative risks that need to be considered. One of the impacts that often arises is environmental pollution due to industrial waste [4]-[6]. This waste can have a negative impact on the ecosystem and human health.

Various industrial sectors are growing rapidly, including the textile industry which has great potential to continue to be developed [7], [8]. However, this industry also produces liquid waste as a by-product that has the potential to pollute the environment. Liquid waste from the textile industry contains complex chemical compounds [9]-[11]. If not treated properly, this waste can pollute the surrounding waters. This raises concerns about the long-term impact on the environment and living things.

The dyes used in the textile industry are aromatic compounds that are difficult to decompose naturally. In addition, this waste often contains heavy metals such as lead (Pb) and chromium (Cr) [12]-[14]. The content of these hazardous materials can accumulate in aquatic ecosystems in a short time [15], [16]. This pollution can disrupt the health of aquatic organisms and damage the balance of the ecosystem [17]-[19]. If left unchecked, this can threaten the survival of living things in the surrounding area.

Liquid waste that is not properly processed can pollute water sources used by the community. Water contaminated with heavy metals is very dangerous if consumed in the long term [20]-[22]. In addition to having an impact on humans, this pollution also threatens the lives of aquatic organisms such as fish and aquatic plants [15], [23], [24]. Polluted water also experiences changes in color and odor, which indicate the presence of toxic compounds [25]-[27]. Therefore, textile waste pollution is a serious problem that needs to be addressed immediately.

Environmental damage is often caused by irresponsible human behavior [28]-[30]. Actions such as littering are one of the main factors of pollution. In the view of religion, damage to the earth is the result of human actions that are negligent and beyond limits. This is emphasized in holy verses that remind humans not to cause damage. Therefore, environmental awareness needs to be instilled so that damage does not continue [31], [32].

Heavy metals are one of the important parameters in measuring the level of environmental pollution [33], [34]. Metals such as Pb and Cr are often found in industrial waste, especially from the coloring and dyeing process [12], [35]. These substances are very dangerous if they exceed the specified threshold. Therefore, it is necessary to analyze the levels of heavy metals to find out how much pollution has occurred. With accurate data, handling actions can be carried out precisely.

Analysis of heavy metal levels can be carried out using several methods, such as AAS and ICP OES. The AAS method has advantages in sensitivity and cost efficiency, but is limited in its analytical range [36], [37]. On the other hand, ICP OES is able to analyze various metals simultaneously with a lower detection limit [38], [39]. The use of these two methods can provide more comprehensive results. This study was conducted to determine the levels of Pb and Cr in textile waste using these two methods.

Both previous studies, namely by Hall et al. [40] and Deshwal et al. [41], both focused on the comparison of metal analysis techniques using spectroscopic instruments such as AAS, ICP-OES, and ICP-MS, but in different contexts and sample matrices—namely human blood and liver and cheese products. Hall et al. [40]evaluated the accuracy and consistency of zinc measurements in various biological matrices, while Deshwal et al. [41] compared the effectiveness of digestion methods in mineral analysis in food products. Although both provide important information on the performance of ICP-OES and AAS methods, no study has specifically compared the reliability of these two techniques in analyzing hazardous heavy metals such as chromium and lead in the context of environmental pollution monitoring. Therefore, the current study fills the gap by focusing on the reliability of chromium and lead analysis using ICP-OES and AAS for heavy metal monitoring purposes, especially in the environmental context, and provides a relevant evaluation of the practical application of both methods under different laboratory conditions.

This study has high novelty and urgency because it specifically compares the reliability of two most commonly used spectroscopic methods, namely ICP-OES and AAS, in analyzing hazardous heavy metals such as chromium and lead in the context of environmental monitoring. Although both methods have been widely used individually in various studies, comparative studies that focus on the effectiveness and accuracy of heavy metal analysis results under similar laboratory conditions are still very limited. Amid the increasing global concern about heavy metal pollution that is harmful to human health and ecosystems, this study is very important to help laboratories, government agencies, and environmental monitoring agencies in choosing the most efficient, accurate, and appropriate analytical method with available resources.

based on the explanation above, This study aims to compare the accuracy and reliability of Inductively Coupled Plasma Optical Emission Spectroscopy and Atomic Absorption Spectrometry in analyzing heavy metal contents specifically lead (Pb) and chromium (Cr) in textile wastewater.

2. RESEARCH METHOD

Analysis of chromium and lead levels in textile waste was carried out quantitatively using ICP OES and AAS as standard methods. This study used a descriptive experimental method with samples taken from rivers suspected of being polluted by textile waste. Samples were tested quantitatively to identify Lead and Chromium levels and method validation was carried out. The tools needed in this study were pipettes, one 1000 mL Iwaki brand measuring flask; one 100 mL Iwaki brand measuring flask; one 210-Lc type analytical balance; IKA RW 20 Digital Hotplate; two 5 mL and 500 mL Iwaki brand measuring cylinders; AAS (APHA.322 B-2005); ICP OES (IK/LEL-ITS/ICP). The materials needed in this study included concentrated HNO3 (PT. Bratachem); ± 1000 mL aquadest (Merck); ± 1500 mL river water sample; Pb(NO3)2 powder (Merck); Cr(NO3)2.9H2O (Merck).

The method of sampling and determining the location of the sample is based on SNI 6989.57:2008 concerning the method of surface water sampling. Sampling is carried out using a simple sampling tool in the form of a plastic bucket equipped with a long-handled plastic dipper rope or using a regular bottle directly on the condition that the sampling tool is made of a material that does not affect the sample; easy to wash from previous sample marks; samples are easy to transfer into a container without any remaining suspended material in it; easy and safe to carry; the capacity of the tool is in accordance with the purpose of the test. The selection of this simple sampling tool was chosen because the condition of the river is not too shallow and can be reached with a simple sampling tool. Data analysis is the most decisive step in a study, because data analysis functions to conclude the results of the study. The samples obtained were analyzed using Precision, Accuracy, LOD, LOQ, Linearity Tests and then the t-test was carried out.

3. RESULTS AND DISCUSSION

3.1. Sample Preparation

The sampling method is the initial stage of a study [42], [43]. The sampling technique is based on SNI 6989.57:2008 concerning the method of surface water sampling. The sample used is river water polluted by textile industry waste in Bedali Lawang and taken as much as 1 liter at each point.

The first sample is at the point before the river is polluted by waste as a comparison of river water that has not been polluted by textile waste, the second sample is taken close to the waste outlet point so that the highest concentration of polluted waste is obtained and the concentration of waste pollution entering the river water is obtained, the third sample is taken at the confluence of 2 rivers so that the effect of increasing river water volume discharge can be known, the fourth sample is taken at a distance of 250 meters after the 3rd sample so that the distance the waste can pollute the river can be known and the fifth sample is taken in river water that enters residents' rice fields so that the presence of heavy metals in the flowing water can be known.

Sample preparation was carried out using the wet destruction method, the initial step taken was to take a 1000 mL waste sample and add H2SO4 and HNO3 as a destructor with a ratio of HNO3: H2SO4 (2:1). The selection of this combination destructor was because in the previous experiment the use of HNO3 destructor produced samples that were less clear and when reading using the instrument was not good, then the combination destructor HNO3: H2SO4 (2:1) was used. The use of this combination of destruction solutions was chosen based on research by Very et al. (2014) which showed that HNO3: H2SO4 (2:1) had better linearity and absorbance reading values. From the addition of HNO3: H2SO4, the following reaction results were obtained:

Pb metal: Pb(CH2O)3+HNO3 + H2SO4→Pb(NO3)3 + 3CO2 + NO+SO4+4H2O 2NO + O2 → 2NO2 Cr metal: Cr(CH2O)3 + HNO3 → Cr(NO3)3 + CO2 + NO + H2O 2NO + O2 → 2NO2 Pb(CH2O)3 / Cr(CH2O)2 is decomposed (oxidized) by nitric acid (HNO3).

Pb(CH2O)3 / Cr(CH2O)2 is decomposed (oxidized) by nitric acid (HNO3) will produce CO2 and NO3, this gas can increase pressure in the destruction process. Due to the decomposition of organic matter by nitric acid, the element being studied is released from its bond with organic matter, then converted into its salt form into metal (NO3)x which is easily soluble in water [44], [45]. NO gas is produced during the oxidation of organic matter by nitric acid, then the NO gas evaporated from the solution reacts with oxygen to produce NO2 gas, this gas is reabsorbed in the solution. The presence of NO2 gas indicates that the organic matter has been oxidized by nitric acid.

In the next stage, the sample that has been added with the destructor is heated at a temperature of 80°C for 10 hours. This temperature was chosen because in the initial experiment using a temperature of 40°C it lasted for 30 hours. While the use of a temperature of 100°C resulted in damaged or charred samples caused by the carbon in the sample being quickly destroyed and exceeding its boiling point. After destruction was carried out for 10 hours. Samples whose volume has decreased and have begun to contain sediment are separated using filter paper to obtain a clear solution and are ready to be analyzed using AAS and ICP OES. The perfection of destruction is indicated by obtaining a clear solution in the destruction solution which indicates that all existing constituents have been completely dissolved or the breakdown of organic compounds has gone well.

3.2. Preparation of Lead (Pb) and Chromium (Cr) Standard Curves Using AAS and ICP OES

Standard calibration curve is an important part in testing the content of an element in AAS analysis. The standard curve is a curve used to express the relationship between the absorbed radiation beams. Based on the Lambert-Beer law, namely A = a.b.C. Where A is absorbance, a is absorptivity, b is flame thickness and C is concentration. The concentration of standard Pb and Cr solutions for ICP OES used is 0.5 ppm; 1 ppm; 1.5 ppm; 2 ppm and 2.5 ppm. While the dilution is made from a 1000 ppm Pb stock solution then dissolved in H2SO4 solvent, this is done to facilitate the reading of lead metal in the sample. This treatment is also carried out in the preparation of the Cr standard solution.

3.3. Method Validation

Validation of an analytical method is an act of assessing certain parameters, based on laboratory experiments, to prove that these parameters meet the requirements for their use.

3.3.1. Linearity

Linearity is the ability of an analysis method to provide a proportional response to the concentration of the analyte in the sample. Linearity is determined from the correlation coefficient value on the regression curve of the standard solution measurement results. Good linearity will produce a correlation coefficient value close to 1. The correlation coefficient value for verifying this analysis method can be seen on the calibration curve of each metal using the AAS and ICP OES instruments.

Table 1. Values of Pb and Cr regression curve equations using AAS and ICP OES

Instrument	Metal	Regression Curve Equation	Regression Value (R2)
AAS	Pb	y = 0.4375x + 0.0016	0.9975
	Cr	y = 0.4326c + 0.0005	0.9968
ICP OES	Pb	y = 215.45x + 6.381	0.9972
	Cr	y = 3913.6x + 372	0.9972

Based on Table 1, it can be seen that all metals tested meet the acceptance requirements of the US.EPA 2007 Method, namely the R2 value is greater than 0.995. This value means that the AAS and ICP OES instruments are in good condition. From the equation in table 5.1, it can be seen that y is the absorbance and b is the slope, x is the concentration and a is the intercept. Seen from this equation, the greater the absorbance value produced, the greater the concentration value obtained. Absorbance is directly proportional to concentration, so if the concentration is high, the absorbance value will also be high, while if the concentration is low, the absorbance will also be low. The correlation coefficient (r) in the study meets the acceptance.

The results of reading the concentration of Pb and Cr using AAS and ICP OES on samples that have been contaminated and marked * are textile waste with concentrations exceeding the limits permitted by the Minister of State for the Environment and Forestry Kep.02/Men-KLH/1998 concerning guidelines for determining environmental quality standards, namely 1 ppm for Cr compounds and the maximum limit for Pb levels in water itself is 0.5 ppm based on SNI No. 7387:2009. Sample 1 has the lowest concentration of Pb and Cr, this is because sample 1 has not been contaminated by textile waste. While sample 2 has the highest concentration of heavy metals Pb and Cr compared to other samples, sample 2 is where the waste comes out directly and as the river water flows further away from the waste source, the sample concentration decreases, this can be seen from the concentrations of 3, 4, 5 decreasing.

3.3.2. Limit of Detection (LOD) and Limit of Quantification (LOQ) of Pb and Cr

The limit of detection (LOD) is the lowest concentration limit of an analyte that can still be detected by an atomic absorption spectrophotometer. While the limit of quantitation (LOQ) is the smallest amount of analyte contained in a sample that can be quantified precisely and accurately (Harmita, 2004). Based on the LOD and LOQ calculations in Appendix 2, the LOD and LOQ results for Pb and Cr compounds using the ICP OES and AAS instruments are obtained in Table 2.

The LOD value of Pb using the AAS and ICP OES instruments obtained were 0.0286 ppm and 0.1853 ppm. Meanwhile, the LOD value of Cr using the AAS and ICP OES instruments obtained were 0.0324 ppm and 0.1447 ppm. This means that the lowest concentration limit is included in the concentration of lead and chromium analyzed using AAS and ICP OES. The LOQ value using the AAS and ICP OES instruments obtained were 0.0953 ppm and 0.6176 ppm. Meanwhile, the LOQ value of Cr using the AAS and ICP OES instruments obtained were 0.1079 ppm and 0.4823ppm. This means that the concentration of the analyte contained in the analyzed sample is included in the concentration range of lead and chromium analyzed using AAS and ICP OES. This LOQ value is an accurate quantitative value because the LOQ itself has been quantified precisely and accurately. The resulting LOD value is always less than the LOQ value.

Table 2. LOD and LOQ for Pb and Cr compounds using ICP OES and AAS instruments

No	Metal	Instrument	LOD (ppm)	LOQ (ppm)
1	Pb	AAS	0.0286	0.0953
2	Cr	AAS	0.0324	0.1079
3	Pb	ICP OES	0.1853	0.6176
4	Cr	ICP OES	0.1447	0.4823

3.2. Precision

Precision is a measure of the closeness of the analysis results obtained from a series of repeated measurements of the same size. One of the techniques used to determine the precision value is the repeatability technique. The precision test (similarity) is determined by the RSD (Relative Standard Deviation) parameter with the formula: (Harmita, 2004). Precision was carried out on 5 different samples with the same treatment and repeated three times.

The results of the precision test of Pb and Cr metals using the AAS and ICP OES instruments showed a %RSD value that met the precision requirements, namely ≤ 2%, which means that the individual sample results and average values have a homogeneous degree of conformity, while the %RSD value of sample 3 on Cr metal showed results that did not meet the requirements, namely 2.6618 and sample 2 Pb metal 2.3846, this is because the spread of individual results shows values that have a fairly large difference so that the spread is not homogeneous, so that in this study the less the difference in sample concentration, the better the %RSD results will be.

Table 3. Precision of Pb and Cr Using AAS

Sample	Metal	%RSD
1	Pb	0.476
	Cr	0.3222
2	Pb	0.4415
	Cr	0.0158
3	Pb	0.3148
	Cr	0.0068
4	Pb	0.189
	Cr	0.07234
5	Pb	0.291
	Cr	0.2063

Table 4. Precision of Pb and Cr Using ICP OES

Sample	Metal	%RSD
1	Pb	0.66789
	Cr	0.7471
2	Pb	2.38462
	Cr	1.0311
3	Pb	1.3787
	Cr	2.6618
4	Pb	1.9117
	Cr	0.1835
5	Pb	0.47692
	Cr	0.7406

3.3. Accuracy

Accuracy is a measure that shows the degree of analysis results with actual levels. In this study, the method used is standard addition, where the sample is analyzed and then a number of analytes (concentration variations in the regression equation) are added to the sample, mixed and analyzed again. The difference between the two results is compared with the actual levels.

Table 5. Results of Pb and Cr Accuracy Calculations Using AAS

Sample	Metal	Analyte Concentration in Sample (ppm)	%Recovery
1	Pb	0.3851	99.5%
	Cr	1.1587	100.54%
2	Pb	0.9249	99.7%
	Cr	4.6836	99.94%
3	Pb	0.8265	100.2%
	Cr	6.043	101.3%
4	Pb	0.8087	100%
	Cr	5.9375	95.43%
5	Pb	0.6902	99.9%
	Cr	2.0297	101.31%

Table 6. Results of Pb and Cr Accuracy Calculations using ICP OES

Sample	Metal	Analyte Concentration in Sample (ppm)	%Recovery
1	Pb	0.936	98.7%
	Cr	1.4663	129.3%
2	Pb	1.979	93.3%
	Cr	38.111	105.27%
3	Pb	1.77	99%

	Cr	38.2415	99.93%
4	Pb	1.750	100%
	Cr	34.9483	96.67%
5	Pb	1.632	99.1%
	Cr	22.7214	98.67%

The steps to carry out the validation analysis of the accuracy method are to add a Pb standard to the sample of 1 ppm as much as 1 mL and add a Cr standard of 1.5 ppm as much as 1 mL to the sample, then read using AAS and ICP OES.

3.4. T-test

The two-sample independent t-test (Independent Sample t-Test) is used to compare the difference between two means of two independent samples assuming the data is normally distributed. In this study, a comparison was made of the use of two different tools between ICP OES and AAS and the data obtained in Appendix 3. From the results obtained, it can be seen that there is a significant difference in the analysis using the AAS and ICP OES instruments. The significance value obtained is 0.012, the value.

The sample preparation procedure and method validation in this study demonstrated a high level of care and precision, which is an important foundation in heavy metal analysis studies. The selection of the wet digestion method with a combination of nitric and sulfuric acids was not only based on previous scientific references, but has also been tested experimentally to ensure its effectiveness in releasing metals from their bonds. This reflects a scientific approach that is adaptive to technical constraints in the laboratory.

The excellence of the experimental design also lies in the application of standard calibration methods and comprehensive validation, including linearity, sensitivity, precision, and accuracy tests. This step shows that the study not only seeks to compare two analytical methods, but also ensures that both tools used are truly metrologically feasible for use in environmental testing. This is important because environmental data-based decision making greatly requires scientifically validated methods.

In addition, the use of statistical tools such as the t-test strengthens the quantitative approach in distinguishing the performance of the two techniques being compared. This shows that researchers are not only assessing based on visual or technical tendencies alone, but also based on measurable scientific significance. In a scientific and practical context, this approach strengthens the credibility of the results and expands the possibility of adoption of these findings by environmental laboratories and regulatory agencies.

Thus, this discussion confirms that the main strength of this study lies not in data acquisition alone, but in the design of a comprehensive analysis method, systematic validation, and an evaluative approach that is able to provide scientific justification for the selection of the most appropriate heavy metal analysis method for the context of wastewater pollution.

This study has a significant impact in the context of environmental monitoring, especially in selecting the most effective analysis method for detecting heavy metals such as lead (Pb) and chromium (Cr) in industrial wastewater. The results and validation carried out can be a reference for environmental laboratories, academics, and government agencies in determining a more appropriate method based on the need for sensitivity, accuracy, and operational efficiency. However, this study also has limitations, including only focusing on two types of heavy metals and one type of environmental sample, namely wastewater from the textile industry. In addition, other limitations lie in the limited scope of the tools tested, without considering advanced analysis methods such as ICP-MS which have higher sensitivity. Other factors such as operational costs, availability of tools in public laboratories, and the influence of complex matrices in samples have also not been thoroughly explored, so the results of this study need to be studied further in different sample contexts and conditions.

4. CONCLUSION

Textile waste contains Pb and Cr metals exceeding the specified limits, namely based on SNI no. 7387:2009 of 0.5 ppm for Pb compounds and 1.5 ppm for Cr compounds based on the decree of the Minister of State for the Environment Kep. 02/Men-KLH/1998 concerning guidelines for determining environmental quality standards. The validation parameter values of the Pb and Cr metal methods using AAS and ICP OES show that the linearity value (r) meets the requirements of ≥ 0.995. The LOD of Pb and Cr AAS is 0.0286 ppm and 0.0324 ppm, while the LOD of ICP OES Pb and Cr is 0.1853 ppm and 0.1447 ppm; LOQ Pb and Cr 0.0953 ppm and 0.1079 ppm while LOQ Pb and Cr metal ICP OES 0.6176 ppm and 0.4823 ppm. The precision of Pb and Cr metal using AAS and ICP OES instruments showed a %RSD value that met the precision requirements, namely $\leq 2\%$, while the %RSD value of sample 3 on Cr metal showed results that did not meet the requirements, namely 2.6618% and sample 2 Pb metal 2.3846. The accuracy of Pb and Cr metal using AAS and ICP OES showed a %recovery value between 93% - 105% with the addition of 1 ppm Pb and 1.5 ppm Cr. There is a significant difference in the analysis using the AAS and ICP OES instruments. The significant value obtained is 0.012, the value. Further research is recommended to expand the types of heavy metals analyzed and involve various types of environmental samples such as soil and sediment. In addition, comparisons with other analytical methods such as ICP-MS or XRF are also needed to provide a more comprehensive picture of the effectiveness and efficiency of each method.

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