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Analysis of Chromium, Nickel, Zinc, and Manganese in Wastewater Matrices Using ICP-OES at PT Medialab Indonesia

Analisis Kromium, Nikel, Seng, dan Mangan dalam Matriks Air Limbah Menggunakan ICP-OES di PT Medialab Indonesia

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ABSTRACT

Industrial wastewater from metal-related industries commonly contains heavy metals such as chromium (Cr), manganese (Mn), nickel (Ni), and zinc (Zn), which pose significant risks to aquatic ecosystems and human health if discharged without adequate treatment. This study determined total concentration of Cr, Mn, Ni, and Zn in inlet (untreated) and outlet (treated) wastewater matrices using Inductively Coupled Plasma–Optical Emission Spectroscopy (ICP-OES) at PT Medialab Indonesia, Cikarang Barat, and to evaluate the efficiency of the wastewater treatment process in accordance with Indonesian Minister of Environment Regulation No 5/2014 (Appendix 47). Wastewater samples were subjected to acid digestion using HNO_3 and HCl prior to ICP-OES analysis. Quality assurance and quality control (QA/QC) parameters including calibration linearity (R^2), percent relative percent difference (%RPD), and percent recovery (%REC) were rigorously evaluated. All calibration curves exhibited excellent linearity with R^2 values of 0.9996–0.9998. Analytical precision was confirmed with %RPD values ranging from 0.66% to 6.42%, and matrix spike recoveries fell within the acceptable range of 87.91%–98.62%. Total metal concentrations in the inlet average were 0.0218 mg/L (Cr), 0.1354 mg/L (Mn), 0.0486 mg/L (Ni), and 0.8233 mg/L (Zn). Following the treatment, removal efficiencies of 84.2% for Cr, 93.7% for Mn, and 87.2% for Zn were achieved. All outlet concentrations complied fully with applicable regulatory limits, confirming that the ICP-OES method supported by rigorous QA/QC procedures is reliable and effective for total metal monitoring in complex industrial wastewater matrices.

Keywords: ICP-OES, heavy metals, wastewater analysis, QA/QC, treatment efficiency.

ABSTRAK

Air limbah industri dari sektor pengolahan logam umumnya mengandung logam berat seperti kromium (Cr), mangan (Mn), nikel (Ni), dan seng (Zn) yang dapat menimbulkan risiko signifikan terhadap ekosistem akuatik dan kesehatan manusia apabila dibuang tanpa pengolahan yang memadai. Penelitian ini bertujuan untuk menentukan konsentrasi total logam dalam matriks air limbah inlet (sebelum pengolahan) dan outlet (setelah pengolahan) menggunakan *Inductively Coupled Plasma–Optical Emission Spectroscopy (ICP-OES)* di PT Medialab Indonesia, Cikarang Barat, serta mengevaluasi efisiensi proses pengolahan air limbah sesuai Peraturan Menteri Lingkungan Hidup No. 5 Tahun 2014, Lampiran 47. Sampel air limbah dilakukan destruksi asam menggunakan HNO_3 dan HCl sebelum dianalisis dengan ICP-OES. Parameter quality assurance and quality control (QA/QC) meliputi linearitas kalibrasi (R^2), persen perbedaan relatif (%RPD), dan persen perolehan kembali (%REC). Semua kurva kalibrasi menunjukkan linearitas yang sangat baik dengan nilai R^2 = 0,9996–0,9998. Presisi analitik dikonfirmasi dengan nilai %RPD berkisar 0,66%–6,42%, dan perolehan kembali matriks spike berada dalam rentang 87,91%–98,62%. Konsentrasi total logam pada inlet rata-rata adalah 0,0218 mg/L (Cr), 0,1354 mg/L (Mn), 0,0486 mg/L (Ni), dan 0,8233 mg/L (Zn). Setelah pengolahan, efisiensi penyisihan yang dicapai adalah 84,2% untuk Cr, 93,7% untuk Mn, dan 87,2% untuk Zn. Seluruh konsentrasi outlet memenuhi baku mutu yang berlaku, membuktikan bahwa metode ICP-OES yang didukung oleh prosedur QA/QC yang ketat

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merupakan pendekatan yang andal dan efektif untuk pemantauan logam total dalam matriks air limbah industri yang kompleks.

Kata kunci: ICP-OES, logam berat, analisis air limbah, kromium, mangan, nikel, seng, QA/QC

INTRODUCTION

Industrial activities generate various types of wastewater containing dissolved and suspended contaminants, including heavy metals such as chromium (Cr), zinc (Zn), manganese (Mn), and nickel (Ni). These metals may originate from raw materials, electroplating processes, corrosion of equipment, and other industrial operations (Barakat, 2011; Fu & Wang, 2011; Shrestha et al., 2021). Due to their toxic characteristics, environmental persistence, and potential for bioaccumulation, these metals can pose significant risks to aquatic ecosystems and human health if discharged without adequate treatment (Tchounwou et al., 2012; Jaishankar et al., 2014). Therefore, monitoring total metal concentrations in industrial wastewater is an essential component of environmental protection and sustainable industrial management (Qasem et al., 2021).

In Indonesia, wastewater discharge is regulated under the Regulation of the Minister of Environment Indonesian Minister of Environment Regulation No 5/2014 (Appendix 47). This regulation establishes maximum allowable concentrations for heavy metals in industrial effluent: Cr total ≤ 0.5–1.0 mg/L, Ni ≤ 0.5–1.0 mg/L, Zn ≤ 2.0–5.0 mg/L, and Mn ≤ 2.0 mg/L. These limits serve as mandatory references for regulatory compliance, requiring analytical results to be accurate, precise, and scientifically valid.

To evaluate treatment efficiency, metal concentrations must be determined in both inlet (untreated) and outlet (treated) wastewater matrices. However, wastewater represents a complex analytical matrix due to the presence of dissolved solids, suspended particles, and organic substances that may cause matrix and spectral interferences during instrumental analysis (Todolí & Mermet, 2006).

Inductively Coupled Plasma–Optical Emission Spectroscopy (ICP-OES) is widely used for multi-element metal determination in environmental samples, following standardized procedures such as the Standard Methods for the Examination of Water and Wastewater (American Public Health Association et al., 2017). ICP-OES operates by introducing a liquid sample into a high-temperature argon plasma (6,000–10,000 K), where atoms and ions are excited and emit radiation at element-specific wavelengths (Boss & Fredeen, 2004). The emission intensity (I) is proportional to the analyte concentration (C), following $I = kC$, where k is a proportionality constant, allowing simultaneous determination of Cr, Zn, Mn, and Ni with high sensitivity and wide linear dynamic range (Skoog et al., 2018).

Quality assurance and quality control (QA/QC) parameters were evaluated including precision (%RPD ≤ 20%), accuracy (%Recovery 80–120%), and calibration linearity ($R^2 \geq 0.995$) (American Public Health Association et al., 2017; U.S. Environmental Protection Agency, 1994; Susanto, 2020). At PT Medialab Indonesia, ICP-OES is employed through appropriate sample digestion, calibration using certified reference standards, and rigorous QA/QC implementation to ensure compliance with Indonesian Minister of Environment Regulation No 5/2014 (Appendix 47).

RESEARCH METHODOLOGY

Location

The research was in PT Medialab Indonesia, an accredited environmental testing laboratory located in Cikarang Barat, West Java, Indonesia.

Materials and Reagents

All chemicals were of analytical grade or higher. Deionized water (resistivity ≥ 18.2 MΩ·cm) was used throughout. Reagents included nitric acid (HNO_3) 65% and hydrochloric acid (HCl) 37% (Merck, Germany). Mixed standard solutions for Cr, Mn, Ni, and Zn (100 mg/L) were prepared from certified stock solutions (1000 mg/L). Calibration standards were prepared by serial dilution in 2% HNO_3. The ICP-OES instrument used was an Agilent 5110 (dual view).

Sample Collection and Preservation

Wastewater samples were collected from the inlet (untreated) and outlet (treated) of an industrial wastewater treatment facility following APHA 3120b (2017) (American Public Health Association et al., 2017). Samples were collected in 250 mL polyethylene bottles

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pre-cleaned with 10% HNO_3, immediately acidified with concentrated HNO_3 to pH < 2, and stored at 4 °C. Samples were analyzed within 28 days of collection.

Acid Digestion Procedure

Total metal digestion was performed following EPA Method 3015A (U.S. Environmental Protection Agency, 2007). A 25.0 mL aliquot of the homogenized sample was transferred to a digestion vessel. Five mL of concentrated HNO_3 and 2 mL of concentrated HCl were added, and the mixture was heated at 95 °C until the volume was reduced to 5–10 mL. After cooling, the digest was filtered through Whatman No. 42 filter paper and diluted to 25 mL with deionized water. A preparation blank was processed identically using deionized water.

ICP-OES Analysis

Calibration curves were prepared from five standard concentrations (0.1, 0.5, 1.0, 2.0, and 5.0 mg/L) in 2% HNO_3. Table 1 summarizes the ICP-OES operating conditions. The analytical sequence included: initial calibration blank, calibration standards, initial calibration verification (ICV), preparation blank, samples (simplo and duplo), matrix spikes, continuing calibration verification (CCV), and continuing calibration blank (American Public Health Association et al., 2017; U.S. Environmental Protection Agency, 1994).

Table 1. ICP-OES Operating Conditions

Parameter	Value / Specification
Instrument	Agilent 5110 ICP-OES
RF Power	1.2–1.5 kW
Plasma Gas (Ar)	12.0 L/min
Nebulizer Gas	0.7 L/min
View Mode	Axial/Radial (dual view)
Wavelengths (nm)	Cr: 206.550; Mn: 257.610; Ni: 230.299; Zn: 202.548
Replicates	3 per sample

QA/QC Parameters

Precision was assessed as percent relative percent difference (%RPD) between simplo and duplo analyses (acceptance criterion: ≤20%). Accuracy was evaluated using matrix spike analyses (spike concentration ≈1.0 mg/L per metal; acceptance criterion: 80–120% recovery). Calibration verification standards (ICV and CCV at 0.10 mg/L) were required to be within ±10% of the nominal value. Blank correction was applied as appropriate.

RESULTS AND DISCUSSION

Calibration Linearity

Calibration curves for Cr, Mn, Ni, and Zn were constructed using five standard concentrations (0.1–5.0 mg/L). Table 2 summarizes the calibration parameters. All four metals exhibited excellent linearity with R^2 values of 0.9996–0.9998, all exceeding the acceptance criterion of $R^2 \geq 0.995$ (American Public Health Association et al., 2017; U.S. Environmental Protection Agency, 1994). The high R^2 values confirm that the instrument response was linear throughout the entire calibration range, supporting the reliability of subsequent sample quantification (Boss & Fredeen, 2004; Skoog et al., 2018). The strong linear response also indicates that the operating conditions — including RF power, plasma gas flow, and nebulizer pressure — were properly optimized. Those are because we have reached the optimum condition

Table 2. Calibration Parameters for Cr, Mn, Ni, and Zn by ICP-OES

Metal	Wavelength (nm)	Slope (b)	Intercept (a)	R	R^2
Cr	206.55	6,439.0	−113.8	0.999	**0.999**
Mn	257.61	269,383	−1,536.9	0.999	**0.999**
Ni	230.29	14,875.9	−58.6	0.999	**0.999**
Zn	202.54	80,854.9	1,870.7	0.999	**0.999**

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QA/QC Results

The preparation blank showed negligible concentrations for all target metals (Cr: −0.0006 mg/L; Mn: 0.0055 mg/L; Ni: −0.0003 mg/L; Zn: 0.0621 mg/L), confirming that the digestion reagents did not introduce significant contamination. ICV recoveries were: Cr 102.6%, Mn 104.4%, Ni 98.7%, and Zn 92.8%; CCV recoveries were: Cr 102.3%, Mn 104.1%, Ni 98.9%, and Zn 91.7%. All values fell within ±10% of the nominal concentration, confirming instrument stability throughout the run.

Table 3 shows %RPD values for all metals and matrices. All %RPD values were well within the ≤20% acceptance criterion, ranging from 0.66% to 6.42% (American Public Health Association et al., 2017; U.S. Environmental Protection Agency, 1994). The highest %RPD was for Cr in the inlet (6.42%), still considerably below the threshold. The low %RPD values demonstrate excellent agreement between simplo and duplo measurements, confirming high analytical precision and reproducibility (Susanto, 2020). The good precision is consistent with the stability of the ICP-OES instrument and the homogeneity of the digested sample solutions (Boss & Fredeen, 2004; Skoog et al., 2018).

Table 3. Precision (%RPD) of Duplicate Analyses

Metal	Inlet S (mg/L)	Inlet D (mg/L)	Inlet Avg (mg/L)	Inlet %RPD	Outlet %RPD
Cr	0.0211	0.0225	0.0218	6.42	2.90
Mn	0.1348	0.1359	0.1354	0.81	4.71
Ni	0.0498	0.0473	0.0486	5.15	1.40
Zn	0.8179	0.8287	0.8233	1.31	0.66

Table 4 presents matrix spike recoveries. All recoveries fell within the acceptable 80–120% range, confirming satisfactory method accuracy. Inlet spike recoveries ranged from 95.77% (Cr) to 98.62% (Ni). Outlet spike recoveries ranged from 87.91% (Ni) to 95.45% (Mn). The slightly lower outlet recoveries may reflect residual matrix effects from dissolved organic matter or competing ions in the treated effluent (Todolí & Mermet, 2006; Skoog et al., 2018). All values remained within the acceptable window, validating the methods suitability for accurate quantification (Susanto, 2020).

Table 4. Percent Recovery (%Rec) of Matrix Spike Analyses

Metal	Matrix	C Spike+Sample (mg/L)	%Recovery
Cr	Inlet	0.9788	95.77%
Mn	Inlet	1.1100	97.52%
Ni	Inlet	1.0360	98.62%
Cr	Outlet	0.8892	88.85%
Mn	Outlet	0.9628	95.45%
Ni	Outlet	0.9359	87.91%
Zn	Outlet	0.9948	88.94%

Total Metal Concentrations

Total metal concentrations in inlet and outlet wastewater are summarized in Table 5. In the inlet, zinc exhibited the highest concentration (0.823 mg/L), which may be attributed to the use of zinc compounds in surface treatment and electroplating processes (Barakat, 2011; Shrestha et al., 2021). This relatively elevated Zn concentration reflects the common occurrence of zinc in industrial effluents from metal finishing operations, originating from process chemicals, plating baths, and rinse waters (Fu & Wang, 2011; Qasem et al., 2021).

Manganese in the inlet (0.135 mg/L) was within moderate levels, while chromium (0.022 mg/L) and nickel (0.049 mg/L) were both present at relatively low concentrations (Tchounwou et al., 2012). The low Cr concentration suggests that hexavalent chromium, if present, had likely been partially reduced prior to the sampling point, or that the industrial process generates predominantly low-Cr effluent (Sharma et al., 2020). In the outlet, all metal concentrations were substantially reduced compared to the inlet. An exception was observed for nickel, which exhibited a

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marginal apparent increase from 0.049 mg/L (inlet) to 0.057 mg/L (outlet), discussed further in Section 3.5.

Table 5. Total Metal Concentrations in Inlet and Outlet Wastewater

Metal	Inlet S (mg/L)	Inlet D (mg/L)	Inlet Avg (mg/L)	Outlet Avg (mg/L)
Cr	0.0211	0.0225	0.0218	0.0035
Mn	0.1348	0.1359	0.1354	0.0085
Ni	0.0498	0.0473	0.0486	0.0572
Zn	0.8179	0.8287	0.8233	0.1058

Wastewater Treatment Efficiency

The efficiency of the wastewater treatment plant in removing each metal was evaluated using $E(\%) = [(C_{inlet} - C_{outlet}) / C_{inlet}] \times 100\%$. Table 6 presents the results.

The treatment plant demonstrated high removal efficiency for manganese (93.7%), zinc (87.2%), and chromium (84.2%) in Indonesian Minister of Environment Regulation No 5/2014 (Appendix 47). The excellent removal of Mn is consistent with the effectiveness of conventional physicochemical treatment processes such as coagulation, flocculation, sedimentation, and filtration, which are particularly efficient at removing manganese through oxidation and precipitation as MnO_2 and $Mn(OH)_2$ (Barakat, 2011; Shrestha et al., 2021). The high zinc removal (87.2%) suggests that the treatment system effectively precipitates $Zn(OH)_2$ or adsorbs zinc onto floc surfaces (Fu & Wang, 2011; Qasem et al., 2021).

Chromium removal of 84.2% is satisfactory given the already low inlet concentration (0.022 mg/L), indicating that coagulation and settling stages effectively removed chromium species (Blöcher et al., 2003; Shrestha et al., 2021). High removal efficiencies for chromium have been widely reported in physicochemical treatment systems, particularly when Cr(III) is the dominant species, as it readily precipitates as $Cr(OH)_3$ at neutral to mildly alkaline pH (Sharma et al., 2020).

The apparent negative removal efficiency for nickel (−17.8%) warrants careful interpretation. Both inlet and outlet nickel concentrations (0.049 and 0.057 mg/L, respectively) are at near-trace levels, well below the regulatory limit of 1.0 mg/L (Kementerian Lingkungan Hidup dan Kehutanan, 2014). At such low concentrations, measurement uncertainty from the ICP-OES instrument, minor adsorption–desorption effects on treatment vessel walls, or leaching of trace nickel from pipeline materials could account for the slight apparent increase (Todolí & Mermet, 2006; Skoog et al., 2018). The absolute difference of only 0.008 mg/L is within the range of analytical uncertainty (Boss & Fredeen, 2004; U.S. Environmental Protection Agency, 1994). This result reflects inherent measurement variability at near-detection-limit concentrations rather than a true treatment failure (Susanto, 2020).

Table 6. Metal Removal Efficiency of the Wastewater Treatment Plant

Metal	Inlet Avg (mg/L)	Outlet Avg (mg/L)	Removal Efficiency (%)
Cr	0.0218	0.0035	84.2
Mn	0.1354	0.0085	93.7
Ni	0.0486	0.0572	−17.8*
Zn	0.8233	0.1058	87.2

**Apparent negative removal; attributable to measurement uncertainty at near-trace levels.*

Regulatory Compliance

The outlet metal concentrations were compared against the maximum allowable limits stipulated in Indonesian Minister of Environment Regulation No 5/2014 (Appendix 47). Table 7 presents the compliance assessment. The outlet wastewater fully complied with all applicable regulatory limits. Chromium in the outlet (0.0035 mg/L) was 143 times lower than the maximum allowable concentration of 0.5 mg/L, while manganese (0.0085 mg/L) was 235 times below its 2.0 mg/L limit. Nickel (0.0572 mg/L) and zinc (0.1058 mg/L) were also far below their respective limits of 1.0 and 5.0 mg/L. The substantial safety margins confirm that the treatment plant is performing at a level significantly exceeding the minimum required by current regulations.

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Table 7. Compliance of Outlet Concentrations with Indonesian Minister of Environment Regulation No 5/2014 (Appendix 47)

Metal	Outlet (mg/L)	Max. Limit (mg/L)	Safety Margin	Status
Cr	0.0035	0.5	143×	**COMPLIANT**
Mn	0.0085	2.0	235×	**COMPLIANT**
Ni	0.0572	1.0	17.5×	**COMPLIANT**
Zn	0.1058	5.0	47.3×	**COMPLIANT**

CONCLUSION

This study validated ICP-OES as an accurate and reliable method for determining concentration of Cr, Mn, Ni, and Zn in industrial wastewater at PT Medialab Indonesia. Calibration curves achieved excellent linearity (R^2 = 0.999–0.999), confirming instrument suitability. QA/QC parameters demonstrated strong performance: %RPD values of 0.66–6.42% and matrix spike recoveries of 87.91–98.62% all met acceptance criteria.

Zinc was the dominant inlet metal (0.823 mg/L), consistent with electroplating operations. The treatment plant achieved high removal efficiencies for Mn (93.7%), Zn (87.2%), and Cr (84.2%). The marginal apparent increase in Ni is attributable to measurement uncertainty near detection limits rather than a true treatment failure. All outlet concentrations complied with Indonesian Minister of Environment Regulation No 5/2014 (Appendix 47) with substantial safety margins, particularly for Cr (143×) and Mn (235× below respective limits). Continued application of this ICP-OES method with rigorous acid digestion and QA/QC protocols is strongly recommended to support ongoing environmental compliance, early anomaly detection, and sustainable industrial operations.

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