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MATERIAL-BASED ASSESSMENT OF IRON AND MANGANESE DISTRIBUTION IN RIVER WATER BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

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Abstract

River water is a widely used source of surface water whose quality requires regular monitoring, particularly for iron (Fe) and manganese (Mn) concentrations, which originate from natural weathering and anthropogenic activities. This study aimed to determine Fe and Mn concentrations in upstream and downstream river water samples using Atomic Absorption Spectrophotometry (AAS) in accordance with SNI 6989.84:2019, to evaluate the reliability of the results through laboratory quality control parameters, and to compare the measured concentrations with the national river water quality standard under Government Regulation No. 22 of 2021. Upstream and downstream samples, together with a laboratory control sample (LCS), a procedural blank, spiked samples, and a certified reference material (CRM), were digested with nitric acid (HNO_3) and analyzed using flame AAS. The Fe and Mn calibration curves showed coefficients of determination (R^2) of 0.9978 and 0.9971, respectively, both exceeding the SNI 6989.84:2019 acceptance criterion of $R^2 \geq 0.995$, and quality control samples met laboratory acceptance criteria. Fe concentrations ranged from 0.1557–0.1594 mg/L upstream and 0.4588–0.4638 mg/L downstream, while Mn was not detected upstream and ranged from 0.5882–0.5915 mg/L downstream. Upstream water met the Class I standard for both parameters, whereas downstream concentrations of both Fe and Mn exceeded the standard. Fe and Mn concentrations increased markedly from upstream to downstream, most plausibly reflecting anthropogenic activities downstream, indicating that downstream water quality requires closer environmental management attention.

Keywords: River Water; Iron (Fe); Manganese (Mn); Atomic Absorption Spectrophotometry (AAS); Water Quality Standard

Abstrak

Air sungai merupakan salah satu sumber air permukaan yang banyak dimanfaatkan sehingga kualitasnya perlu dipantau secara berkala, terutama konsentrasi besi (Fe) dan mangan (Mn) yang berasal dari proses pelapukan alami maupun aktivitas antropogenik. Penelitian ini bertujuan menentukan konsentrasi Fe dan Mn pada air sungai bagian hulu dan hilir menggunakan Spektrofotometri Serapan Atom (SSA) sesuai SNI 6989.84:2019, mengevaluasi keandalan hasil analisis melalui parameter kontrol kualitas laboratorium, serta membandingkan konsentrasi terukur dengan baku mutu air sungai nasional menurut Peraturan Pemerintah Nomor 22 Tahun 2021. Sampel air hulu dan hilir, bersama laboratory control sample (LCS), blangko prosedural, sampel spike, dan certified reference material (CRM), didestruksi dengan asam nitrat (HNO_3) dan dianalisis menggunakan SSA nyala. Kurva kalibrasi Fe dan Mn menunjukkan koefisien determinasi (R^2) berturut-turut sebesar 0,9978 dan 0,9971, keduanya melampaui kriteria penerimaan SNI 6989.84:2019 ($R^2 \geq 0,995$), dan sampel kontrol kualitas memenuhi kriteria penerimaan laboratorium. Konsentrasi Fe berkisar 0,1557–0,1594 mg/L di hulu dan 0,4588–0,4638 mg/L di hilir, sedangkan Mn tidak terdeteksi di hulu dan berkisar 0,5882–0,5915 mg/L di hilir. Kualitas air hulu memenuhi baku mutu Kelas I untuk kedua parameter, sedangkan konsentrasi Fe dan Mn di hilir melampaui baku mutu tersebut. Peningkatan konsentrasi Fe dan Mn yang nyata dari hulu ke hilir kemungkinan besar mencerminkan pengaruh aktivitas antropogenik di sepanjang bagian hilir, yang menunjukkan bahwa kualitas air di hilir memerlukan perhatian pengelolaan lingkungan yang lebih ketat.

Kata Kunci: Air Sungai; Besi (Fe); Mangan (Mn); Spektrofotometri Serapan Atom (SSA); Baku Mutu Air

1. Introduction

Water is among the most essential natural resources supporting human life and a wide range of industrial and agricultural activities. River water is among the most heavily used sources of surface water because of its abundance and accessibility, yet its quality is strongly influenced by the surrounding environment, through both natural processes and anthropogenic pressures such as industry, mining, agriculture, and settlement (Fachrudin & Rismawati, 2026; Fadlillah et al., 2023). Increasing metal loads in a river system as a result of such activities can degrade water quality and threaten both ecosystem integrity and public health once concentrations exceed established standards (Fadlillah et al., 2023).

Iron (Fe) and manganese (Mn) are two parameters commonly used to assess river-water quality. Both elements occur naturally through rock and soil weathering, while elevated concentrations may also be associated with industrial effluents, mining drainage, agricultural runoff, domestic wastewater, and other anthropogenic inputs. Reliable quantitative determination is therefore important for identifying spatial changes in river-water quality and assessing compliance with applicable regulatory standards.

Several studies conducted on Indonesian rivers have reported a tendency for metal concentrations to increase from upstream toward downstream sections, reflecting the cumulative influence of human activities along river courses (Elfidasari et al., 2019; Fachrudin & Rismawati, 2026; Fadlillah et al., 2023; Wardhani et al., 2024). A similar upstream–downstream concentration gradient has also been reported outside Indonesia, including in the Tembi River, Iran, downstream of a sewage-discharge point (Shanbehzadeh et al., 2014). In the present study, the term "metal concentration" is used because the measured parameter is concentration rather than mass flux.
Accurate determination of Fe and Mn requires an analytical method with adequate sensitivity, selectivity, and precision. Atomic Absorption Spectrophotometry (AAS) is widely used for quantitative metal determination in environmental samples because it provides suitable analytical sensitivity and precision (Fishman & Downs, 1966; Matusiewicz et al., 1991; Yebra, 2012). In Indonesia, SNI 6989.84:2019 provides the reference procedure for flame-AAS determination of dissolved and total metals in water and wastewater (Badan Standardisasi Nasional, 2019). In this study, AAS performance was evaluated using calibration linearity together with procedural blanks, laboratory control samples (LCS), certified reference material (CRM), and duplicate analyses before the measured concentrations were compared with the applicable Class I river-water standard.

This study aimed to: (1) determine Fe and Mn concentrations in upstream and downstream river water; (2) assess the reliability of the analytical results using laboratory quality-control indicators; and (3) compare the measured concentrations with the Class I river-water quality standard established under Government Regulation No. 22 of 2021.

2. Methodology

This study used a descriptive quantitative, laboratory-based design to determine Fe and Mn concentrations in river water sampled from an upstream and a downstream location along the same river. The analytical workflow consisted of sample collection, acid digestion, flame-AAS measurement, calibration assessment, laboratory quality control, duplicate analysis, and comparison with the applicable regulatory standard.

2.1. Study design and sampling

Each section (upstream and downstream) was sampled in simplo and duplo (single and duplicate). The duplicate measurements were used to assess analytical precision by calculating the relative percent difference (RPD) between paired results.

2.2. Reagents and standards

Nitric acid (HNO_3) was used for sample digestion. Standard stock solutions of Fe and Mn (1,000 mg/L) were used to prepare working standards for calibration curve construction, covering the concentration range of 0–10 mg/L for Fe (0, 0.3, 0.5, 1, 2, 5, and 10 mg/L) and 0–5 mg/L for Mn (0, 0.1, 0.5, 1, 2, and 5 mg/L). A laboratory control sample (LCS), a procedural blank, spiked

sample, and a certified reference material (CRM P360-500) were analyzed alongside the river water samples as part of the quality control programme.

2.3. Sample preparation

Upstream and downstream water samples, together with the LCS solution, procedural blank, and spiked samples, were digested with HNO_3 in accordance with SNI 6989.84:2019 (Badan Standardisasi Nasional, 2019) prior to instrumental measurement. The same preparation sequence was applied to QC materials to assess potential contamination and analytical recovery.

2.4. Instrumentation

Fe and Mn concentrations were measured using flame Atomic Absorption Spectrophotometry (AAS), with absorbance recorded at wavelengths of 248.3 nm for Fe and 279.5 nm for Mn, corresponding to the optimum wavelengths specified in SNI 6989.84:2019 (Badan Standardisasi Nasional, 2019).

2.5. Calibration and quality control

A calibration curve was constructed for each metal by plotting absorbance against known standard concentrations, and the linear regression equation and coefficient of determination (R^2) were calculated. In accordance with SNI 6989.84:2019, a calibration curve is considered acceptable when $R^2 \geq 0.995$ (Badan Standardisasi Nasional, 2019). Quality-control performance was evaluated using procedural blanks, LCS recovery, CRM results against the stated acceptance range, spiked samples, and duplicate precision. LCS recovery was calculated as measured concentration divided by nominal concentration × 100. Duplicate precision was expressed as relative percent difference (RPD) using $RPD = |C1 - C2| / [(C1 + C2)/2] \times 100$.

2.6. Data analysis

Measured Fe and Mn concentrations in the upstream and downstream samples were compared descriptively and evaluated against the Class I river-water quality standard under Government Regulation No. 22 of 2021 (Fe: 0.3 mg/L; Mn: 0.1 mg/L) (Pemerintah Republik Indonesia, 2021). The upstream–downstream difference was interpreted as a spatial concentration gradient; possible sources were discussed cautiously because no source-tracking or discharge-specific measurements were performed.

3. Results and Discussion

3.1. Calibration curve and method linearity

Table 1 and Table 2 present the absorbance readings obtained across the working concentration range for Fe and Mn, respectively.

Table 1. *Calibration data for iron (Fe)*

Concentration (mg/L)	Absorbance
0	-0.0005
0.3	0.0239
0.5	0.0398
1	0.0800
2	0.1560
5	0.3621
10	0.6657

Regression equation: $y = 0.0666x + 0.0107$; $R^2 = 0.9978$.

Table 2. *Calibration data for manganese (Mn)*

Concentration (mg/L)	Absorbance
0	-0.0006
0.1	0.0233
0.5	0.1106
1	0.2053
2	0.3281
5	0.8628

Regression equation: y = 0.1696x + 0.0119; R² = 0.9971.

Linear regression of the Fe calibration data yielded the equation y = 0.0666x + 0.0107, with a coefficient of determination (R^2) of 0.9978, while the Mn calibration data yielded y = 0.1696x + 0.0119, with R^2 = 0.9971. Both values exceed the SNI 6989.84:2019 acceptance criterion of $R^2 \geq 0.995$ (Badan Standardisasi Nasional, 2019), indicating an excellent linear relationship between absorbance and concentration across the tested range. Although the coefficient of determination is widely used as a practical indicator of calibration linearity, it should be interpreted together with the overall regression behaviour, since R^2 alone does not fully characterize deviations from linearity across an analytical range (Moosavi & Ghassabian, 2018). Nonetheless, the R^2 values obtained for both Fe and Mn, together with the consistent and monotonic increase in absorbance with concentration, support the suitability of the calibration curves for quantifying Fe and Mn in the river water samples analyzed.

3.2. Laboratory quality control

Tables 3 and 4 summarize the quality control results for Fe and Mn analysis, respectively, comprising the procedural blank, laboratory control sample (LCS), certified reference material (CRM), simplo and duplo measurements of the upstream and downstream samples, and the spiked-sample results.

Table 3. *Quality control data for iron (Fe)*

No.	Sample Code	Absorbance (y)	Concentration (x) (mg/L)	True Concentration (mg/L)	QC Acceptance Limits (µg/L)
1	Blank	-0.0002	-0.1601	-0.1601	-
2	LCS 5 mg/L	0.3442	5.0054	5.0054	-
3	CRM P360-500	0.1625	2.2795	2.2795	1,910-2,340
4	Simplo upstream	0.0211	0.1594	0.1594	-
5	Duplo upstream	0.0209	0.1557	0.1557	-
6	Spike upstream 2 mg/L	0.1650	2.3167	2.3167	-
7	Simplo downstream	0.0414	0.4638	0.4638	-
8	Duplo downstream	0.0411	0.4588	0.4588	-
9	Spike downstream 2 mg/L	0.1433	1.9916	1.9916	-

QC = quality control.

Table 4. *Quality control data for manganese (Mn)*

No.	Sample Code	Absorbance (y)	Concentration (x) (mg/L)	True Concentration (mg/L)	QC Acceptance Limits (µg/L)
1	Blank	-0.0006	-0.0737	-0.0737	-
2	LCS 2 mg/L	0.3332	1.8948	1.8948	-
3	CRM P360-500	0.1455	0.7877	0.7877	691-820
4	Simplo upstream	0.0099	-0.0114	-0.0114	-
5	Duplo upstream	0.0100	-0.0114	-0.0114	-
6	Spike upstream 2 mg/L	0.4649	2.6712	2.6712	-
7	Simplo downstream	0.1122	0.5915	0.5915	-
8	Duplo downstream	0.1116	0.5882	0.5882	-
9	Spike downstream 2 mg/L	0.3627	2.0686	2.0686	-

QC = quality control.

For Fe, the procedural blank yielded an absorbance close to zero, indicating negligible contamination during sample preparation and analysis. The Fe LCS at a nominal concentration of 5 mg/L returned 5.0054 mg/L, corresponding to 100.11% recovery. The Fe CRM (P360-500) returned 2.2795 mg/L, within its stated acceptance range of 1.910–2.340 mg/L. Duplicate RPD values were 2.35% upstream and 1.08% downstream. For Mn, the blank was close to zero, the 2 mg/L LCS returned 1.8948 mg/L (94.74% recovery), and the CRM returned 0.7877 mg/L within its acceptance range of 0.691–0.820 mg/L. Mn duplicate RPD was 0.00% upstream and 0.56% downstream. These quantitative QC indicators provide direct evidence of calibration and measurement reproducibility for the reported Fe and Mn concentrations.

3.3. Fe and Mn concentrations in upstream and downstream river water

Based on the analytical results, Fe concentrations in the upstream section of the river ranged from 0.1557–0.1594 mg/L, while concentrations in the downstream section increased to 0.4588–0.4638 mg/L. Mn was not detected in the upstream section; the measured value of approximately −0.0114 mg/L was below the method's practical quantification capability and was therefore treated as a non-detect rather than a true negative concentration. Downstream Mn concentrations ranged from 0.5882–0.5915 mg/L. Table 5 summarizes these results in comparison with the Class I water-quality standard under Government Regulation No. 22 of 2021.

Table 5. *Summary of Fe and Mn concentrations in upstream and downstream river water compared with the Class I standard*

Parameter	Upstream (mg/L)	Downstream (mg/L)	Class I Standard (mg/L)[a]	Downstream Status
Iron (Fe)	0.1557-0.1594	0.4588-0.4638	0.3	Exceeds standard
Manganese (Mn)	Not detected (≈ -0.0114)	0.5882-0.5915	0.1	Exceeds standard

[a] *Class I river water quality standard under Government Regulation No. 22 of 2021 (Pemerintah Republik Indonesia, 2021).*

Upstream Fe concentrations remained below the Class I standard (0.3 mg/L), while Mn was not detected at the upstream location and therefore was not interpreted as a positive concentration. Downstream concentrations of both Fe and Mn exceeded the Class I standard (Fe: 0.3 mg/L; Mn: 0.1 mg/L), indicating that the downstream sampling location did not meet the applicable Class I criterion for these two parameters.

The increase in Fe and Mn concentrations from the upstream to the downstream section demonstrates a spatial concentration gradient and is consistent with additional metal inputs occurring along the river reach. Plausible contributors include industrial effluents, mining

drainage, agricultural runoff, domestic wastewater, and corrosion or disturbance of iron- and manganese-bearing materials. However, the present dataset does not establish which specific source is responsible. Confirmation of source contributions would require additional evidence such as source-specific discharge measurements, land-use information, hydrological data, or more spatially resolved sampling. Therefore, the downstream enrichment is interpreted cautiously as being consistent with anthropogenic influence rather than as proof of a specific pollution source.

3.4. Implications for water quality management

The elevated downstream Fe concentration exceeded the Class I water-quality criterion and is relevant to environmental monitoring because elevated Fe can contribute to discoloration, sediment formation, metallic taste, and corrosion-related problems in water systems. The downstream Mn concentration also exceeded the Class I criterion. Because this study did not include exposure assessment, dose estimation, or health-outcome data, it does not constitute a human-health risk assessment. The findings instead support continued monitoring, investigation of possible upstream/downstream inputs, and management actions aimed at maintaining compliance with applicable water-quality standards.

The calibration curves for Fe and Mn exhibited acceptable linearity (R^2 = 0.9978 and 0.9971, respectively), exceeding the SNI 6989.84:2019 criterion. Quantitative QC results further supported the analytical performance: Fe and Mn LCS recoveries were 100.11% and 94.74%, respectively, while duplicate RPD values were ≤2.35%. CRM results were within the stated acceptance ranges for both metals. Fe concentrations ranged from 0.1557–0.1594 mg/L upstream and 0.4588–0.4638 mg/L downstream, while Mn was not detected upstream and ranged from 0.5882–0.5915 mg/L downstream. The upstream–downstream concentration gradient and regulatory comparison demonstrate higher Fe and Mn concentrations downstream. These findings are consistent with additional anthropogenic influence along the river reach, but the present study does not identify specific contamination sources. Continued monitoring, source investigation, and compliance management are therefore warranted.

The calibration curves for Fe and Mn in this study exhibited excellent linearity (R^2 = 0.9978 and 0.9971, respectively), exceeding the SNI 6989.84:2019 acceptance criterion, and the associated quality control results (blank, LCS, CRM, and duplicate analyses) indicated adequate accuracy and precision for the flame AAS method applied. Fe concentrations ranged from 0.1557–0.1594 mg/L upstream and 0.4588–0.4638 mg/L downstream, while Mn was undetected upstream and ranged from 0.5882–0.5915 mg/L downstream. Upstream water quality met the Class I standard under Government Regulation No. 22 of 2021 for both parameters, whereas downstream Fe and Mn concentrations exceeded this standard. The marked increase in Fe and Mn concentrations from upstream to downstream, consistent with patterns reported for other rivers subject to anthropogenic pressure, indicates a decline in downstream water quality that warrants closer environmental monitoring and management attention.

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