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Environmentally Friendly Synthesis of Ferric Chloride Coagulant from Scrap Iron for the Reduction of ion ferro and Total Dissolved Solids

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Abstract
The decline in groundwater quality, particularly in terms of TDS and Fe^{2+} levels, is a major concern in clean water supply. This study aims to synthesize and evaluate the effectiveness of $FeCl_3$ coagulant synthesized from scrap iron waste from a lathe workshop in reducing Total Dissolved Solids (TDS) and Fe^{2+} levels in borehole water. $FeCl_3$ was synthesized by reacting scrap iron waste with a 32% HCl solution. Water samples were tested for TDS and Fe^{2+} levels before and after treatment using gravimetric and UV-Vis spectrophotometric methods. The $FeCl_3$ coagulant doses used were 0.1%, 0.2%, and 0.3%. The results showed that the 0.3% dose provided the highest effectiveness in reducing Fe^{2+} levels by 80.10%, while the reduction in TDS reached 36.05% at the same dose. All doses successfully reduced Fe^{2+} levels to below the threshold set by Ministry of Health Regulation No. 2 of 2023 (<0.2 mg/l). This study demonstrates that $FeCl_3$ coagulant derived from iron waste is effective and has the potential to serve as an economical and environmentally friendly alternative for water treatment.
Keywords: *coagulant; fe^{2+}; ferric chloride; total dissolved solids; well water*

Abstrak
Penurunan kualitas air tanah, terutama kandungan TDS dan Fe^{2+}, menjadi perhatian dalam penyediaan air bersih. Penelitian ini bertujuan mensintesis mengkaji efektivitas koagulan $FeCl_3$ hasil sintesis dari limbah besi bengkel bubut dalam menurunkan kadar Total Dissolved Solids (TDS) dan Fe^{2+} pada air sumur bor. Sintesis $FeCl_3$ dilakukan dengan mereaksikan limbah scrap besi dan larutan HCl 32%. Sampel air diuji kadar TDS dan Fe^{2+} sebelum dan sesudah perlakuan menggunakan metode gravimetri dan spektrofotometri UV-Vis. Variasi dosis koagulan $FeCl_3$ yang digunakan adalah 0,1%, 0,2%, dan 0,3%. Hasil menunjukkan bahwa dosis 0,3% memberikan efektivitas tertinggi dalam menurunkan kadar Fe^{2+} sebesar 80,10%, sedangkan penurunan TDS mencapai efektivitas 36,05% pada dosis yang sama. Seluruh dosis berhasil menurunkan kadar Fe^{2+} hingga di bawah ambang batas Permenkes No. 2 Tahun 2023 (<0,2 mg/l). Penelitian ini membuktikan bahwa koagulan $FeCl_3$ dari limbah besi efektif dan berpotensi sebagai alternatif pengolahan air yang ekonomis dan ramah lingkungan.
Kata Kunci: *air sumur; ferri klorida; fe^{2+}; koagulasi; total padatan terlarut*

1. Introduction

Water is a vital source of life for all living things. In modern life, water plays a crucial role as a parameter of environmental balance and is essential in various sectors [1]. Water quality scarcity occurs when water fails to meet quality standards, even if the quantity is sufficient, thereby posing health risks [2]. Hamidah et al. (2020) found that TDS levels in dug well water in Cirebon Regency reached 381–421 mg/L, exceeding the Ministry of Health regulation limit. Riyanto et al. (2021) also reported that iron (Fe) levels in well water in Jenar Lor Village reached 0.689–1.941 ppm, exceeding the threshold limit.

One important parameter of water quality is TDS, which reflects the amount of dissolved substances such as minerals, salts, heavy metals, and organic matter. High TDS levels can cause water to appear cloudy and yellowish [3], [4]. TDS measurements can be performed using the gravimetric method, which is accurate and sensitive down to 0.0001 grams [14].

Groundwater generally contains Fe^{2+} and Mn, which are characterized by a color change to brownish-yellow upon contact with air, produce an odor, and leave stains. The maximum Fe^{2+} concentration in water according to Ministry of Health Regulation No. 2 of 2023 is 0.2 mg/L.

This study uses $FeCl_3$ as a coagulant, synthesized from iron scrap from a lathe workshop as an effort to utilize waste (Hasanah, 2019). Iron scrap waste has a high Fe content and has the potential to serve as a raw material for metal-based coagulants, which are effective in precipitating colloidal particles in water [5], [6], [7]. Ferric chloride was obtained by reacting scrap iron with 37% HCl [8], [9]. This study focused on the treatment of borehole water to reduce TDS and Fe^{2+} levels using an economical and environmentally friendly approach, so that the water quality meets community standards.

2. Material and Methods

Materials and Equipment

The equipment used in this study included a Shimadzu UV-1800 UV-Vis spectrophotometer; a Mediatech pH meter; 100-ml and 250-ml Pyrex Iwaki beakers; a 100-ml Pyrex Iwaki measuring cylinder; 1.0-ml, 2.0-ml, 5.0 ml; 10.0 ml; 25 ml (Pyrex Iwaki); measuring pipettes (Pyrex Iwaki); Ohaus analytical balance; spray bottle; Duran desiccator; Thermo magnetic stirrer; Pyrex Iwaki watch glasses; Memmert oven; and desiccator.

The materials used in this study were borehole water samples; demineralized water; Merck $C_{12}H_8N_2$ powder; Merck NH_2OH powder; Merck $FeSO_4 \cdot 7H_2O$ p.a. powder; Whatman No. 42 filter paper; $FeCl_3$ coagulant; and Whatman No. 1 filter paper.

Preparation of $FeCl_3$ Coagulants and Qualitative Testing

A total of 50 grams of scrap iron pieces were placed in a 2000-ml beaker, and then 32% HCl solution was added until the entire surface of the scrap iron was submerged. The solution was covered with a watch glass and heated at 60°C for 2 hours to accelerate the dissolution reaction. After heating, the solution was left in a refrigerator for one day until $FeCl_3$ crystals formed. The resulting crystals were then filtered using Whatman No. 42 filter paper and dried in a desiccator. Next, the resulting $FeCl_3$ crystals were qualitatively tested to confirm the presence of Fe^{3+} and Cl^- ions. The Fe^{3+} test was performed by adding the crystal solution to a $K_4[Fe(CN)_6]$ solution, which produced a Prussian blue precipitate, and to KCNS, which produced a blood-red color. Meanwhile, the Cl^- test was performed by adding the crystal solution to $AgNO_3$ and $Pb(NO_3)_2$ solutions, which each formed a white precipitate [10], [11].

Generation of a Standard Series of Fe

A Fe^{2+} stock solution (50 mg Fe/100 mL) was prepared by dissolving 0.02489 g of $FeSO_4 \cdot 7H_2O$ in 50 mL of demineralized water to which 2 mL of concentrated H_2SO_4 had been carefully added, then diluting it to the mark on a 100-ml volumetric flask . Standard solutions with concentrations of 0–0.3 mg/L were prepared by pipetting 0–15 mL of the stock solution into a 50-mL volumetric flask, then adding 5 mL of phenanthroline and 5 mL of acetate buffer, diluting to the mark, homogenizing, and allowing to stand for 10 minutes before measuring the absorbance using a spectrophotometer. Measurements were performed twice [12].

Determination of Fe^{2+} Concentration by UV-Vis Spectrophotometry Before and After Treatment

The determination of Fe^{2+} concentration was conducted in two stages: before and after the addition of the $FeCl_3$ coagulant. In the stage prior to coagulant addition, 25 mL of the sample was pipetted into a 50-mL volumetric flask, followed by the addition of 5 mL of phenanthroline solution and 5 mL of acetate buffer solution. The solution was diluted with demineralized water to the mark, homogenized, and allowed to stand for 10 minutes. After that, the absorbance of the solution was measured using a spectrophotometer at the maximum wavelength, and the measurement was performed twice [13].

In the stage following the addition of the $FeCl_3$ coagulant, three beakers were each filled with 100 mL of sample, and then a pinch of calcium bicarbonate and the synthesized $FeCl_3$ coagulant were added at mass concentrations of 0.1%, 0.2%, and 0.3%. The mixture was stirred at 120 rpm for 2 minutes, followed by 20 rpm for 20 minutes. After that, the solution was allowed to stand for 1 hour, then filtered, and the filtrate was collected. A total of 25 mL of the filtrate was transferred to a 50-mL volumetric flask, to which 5 mL of phenanthroline solution and 5 mL of acetate buffer were added, then diluted to the mark. The solution was homogenized and allowed to stand for 10 minutes before its absorbance was measured using a spectrophotometer. The measurement is performed twice, and the absorbance of the filtrate is recorded at the maximum wavelength [12].

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Determination of TDS Levels by Gravimetric Analysis Before and After Treatment

The determination of Total Dissolved Solids (TDS) began by preparing a porcelain dish that was heated to 180 ± 2°C for one hour, cooled in a desiccator, and weighed until a constant weight (W_0) was obtained [14]. Before adding the $FeCl_3$ coagulant, the well water sample was filtered after homogenization, then 25 ml of the filtrate was placed in a dish and evaporated to dryness. The dish was then reheated, cooled, and weighed until a stable weight (W_1) was reached, after which the TDS value was calculated and the test was repeated twice. After adding $FeCl_3$ coagulant at varying concentrations of 0.1%, 0.2%, and 0.3% along with calcium bicarbonate, the samples were stirred (120 rpm for 2 minutes and 20 rpm for 20 minutes), allowed to stand for 1 hour, and then filtered. A 25-ml filtrate was evaporated, dried in an oven, cooled, weighed until a constant weight was reached (W_1), and the TDS value was calculated from the weight difference; the measurement was performed twice to ensure accuracy [14].

Data Analysis

The data obtained from the study were analyzed descriptively by comparing the Fe^{2+} and TDS levels in well water before and after treatment with the coagulant $FeCl_3$. The formula for calculating the iron concentration is as follows:

Iron concentration (mg/L):

$y = bx + a$

where:

y = absorbance value of the sample
x = concentration of the standard solution (mg/L)
b = slope
a = intercept

The formula for calculating total dissolved solids (TDS) is:

$$\text{TDS (mg/L)} = \frac{(W1-W0)\ X\ 1000}{V}$$

where:

W0 = the constant weight of the empty beaker after equilibration
W1 = the constant weight of the beaker containing the total dissolved solids after equilibration
V = the volume of the test sample in milliliters
1000 = the conversion factor from milliliters to liters

The formula for calculating the Relative Percent Difference (RPD) is:

$$\%\ \text{RPD} = \frac{Measurement\ Results - Duplikat\ Pengukuran}{(Measurement\ Results + Duplicate\ Measurements)/2}\ x\ 100\%$$

Duplicate Measurement formula for calculating the percentage of effectiveness is:

$$\%\ \text{effectiveness} = \frac{C\ before - C\ after}{C\ before}\ x100\%$$

where:

C before = concentration before treatment
C after = concentration after treatment

3. Results and Discussion

Synthesis and Qualitative Testing of $FeCl_3$ Coagulant from Iron Waste

The $FeCl_3$ coagulant synthesized from iron waste from a lathe shop was reacted with 32% hydrochloric acid, producing $FeCl_3$ as shown in **Figure 1**.

Fig. 1: The process of producing $FeCl_3$ coagulant from scrap iron from a lathe workshop

The resulting crystals are yellowish-green in color. This result is consistent with the study by Almeida & Schneider, who produced $FeCl_3$ coagulant from iron ore waste (Almeida & Schneider, 2020). The reaction for the formation of $FeCl_3$ from iron waste and hydrochloric acid is as follows:

$Fe + 2HCl \rightarrow Fe^{2+} + 2Cl^- + H_2\uparrow$

$Fe^{3+} + 3Cl^- \rightarrow FeCl_3$ [15]

The crystals obtained from the study were then qualitatively tested to identify the presence of Fe^{3+} and Cl^- ions, as shown in **Table 1**.

Table 1. Qualitative tests for Fe^{3+} and Cl^- in the crystals obtained from the study

Type of test	Type of reagent used	Results
Fe^{3+}	Potassium ferrocyanide solution, $K_4[Fe(CN)_6]$	Prussian blue deposits
	Potassium cyanide solution, KCNS	Blood-red deposits
Cl^-	Silver nitrate solution, $AgNO_3$	White sediment
	Lead nitrate solution, $Pb(NO_3)_2$	White sediment

Table 1 confirms that the crystals synthesized from scrap iron and hydrochloric acid are ferric chloride, $FeCl_3$. A qualitative test for Fe^{3+} was carried out by dissolving the crystals obtained in a test tube and then reacting the solution with $K_4[Fe(CN)_6]$, resulting in the formation of a Prussian blue precipitate, as shown in **Figure 2**.

Fig. 2: Prussian blue-coloured deposits

The reaction that takes place is:

$4Fe^{3+} + 3K_4[Fe(CN)_6] \rightarrow Fe_4[Fe(CN)_6]_3\downarrow$ Prussian blue

The second qualitative test for Fe^{3+} was carried out by dissolving the crystals obtained from the study in a test tube, then adding a solution of KCNS, which resulted in the formation of a blood-red precipitate, as shown in **Figure 3**.

Fig. 3: Blood-red deposits

The reaction that takes place is:

$Fe^{3+} + 3KCNS \rightarrow 3K^+ + Fe[(CNS)_3]\downarrow_{\text{blood-red}}$

The test for the presence of Cl^- ions was conducted by reacting the crystal solution with a silver nitrate ($AgNO_3$) solution, resulting in the formation of a white precipitate, as shown in **Figure 4**.

Fig. 4: White sediment

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The reaction that occurs is:

$$Cl^- + AgNO_3 \rightarrow AgCl\downarrow_{white} + NO_3^-$$

Another method for testing for Cl^- anions involves reacting a crystal solution with a $Pb(NO_3)_2$ solution, resulting in the formation of a white precipitate, as shown in **Figure 5**.

Fig. 5: White sediment

The reaction that takes place is:

$$2Cl^- + Pb(NO_3)_2 \rightarrow 2NO_3^- + PbCl_2\downarrow_{white}$$

Maximum Wavelength and Linear Regression Curve

The Fe^{2+} concentration was determined using a calibration curve based on six graded standard solutions (0–0.3 mg/L) prepared from a 1 mg/L Fe stock solution. Absorbance was measured using a UV-Vis spectrophotometer at wavelengths ranging from 450 to 600 nm, and the maximum absorbance was observed at 510 nm, as shown in **Figure 6** and **Table 2**.

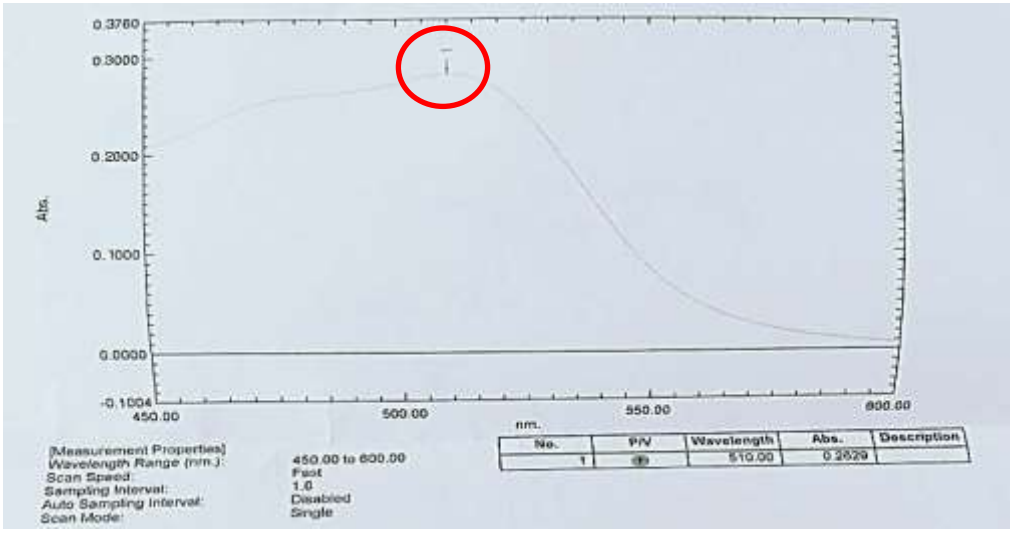

Fig. 6: Maximum Wavelength

Table 2. Absorbance measurements of the Fe^{2+} standard series solutions

Concentration (mg/l)	Absorbance I	Absorbance II	Average
0	0.000	0.000	0.000
0.05	0.044	0.045	0.045
0.1	0.099	0.097	0.098
0,.15	0.153	0.155	0.154
0.2	0.203	0.203	0.203
0.25	0.262	0.264	0.263
0.3	0.324	0.325	0.325

From **Table 2**, a standard calibration curve for Fe^{2+} can be plotted as shown in **Figure 6**.

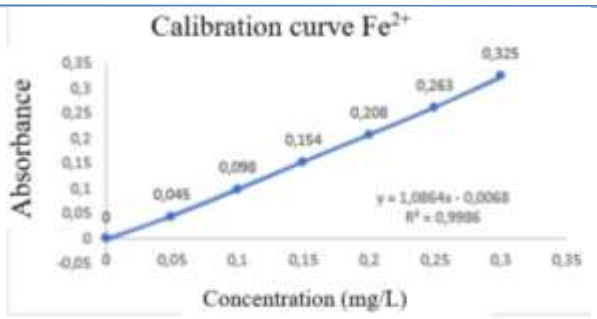

Fig. 6: Standard calibration curve for Fe^{2+}

Figure 6 shows that the standard calibration curve for Fe^{2+} was obtained using the linear regression equation y = 1.0864x – 0.0068, with an R^2 value of 0.9986.

The Fe^{2+} concentration in the well water samples was calculated using the equation y = 1.0864x – 0.0068. The results of the Fe^{2+} concentration analysis in the initial samples are shown in **Table 3**.

Table 3. Results of the calculation of the initial Fe^{2+} concentration in well water samples

No.	Sample	Initial Fe^{2+} concentration (mg/l)	Average level (mg/l)	% RPD	Quality standard (mg/l)
1	AI	0.1821	0.1816	0.51%	0.2
2	AII	0.1811			
3	BI	0.1499	0.1489	1.23%	
4	BII	0.1480			
5	CI	0.2419	0.2419	0%	
6	CII	0.2419			

Table 3 shows that well water sample A, with an average Fe^{2+} concentration of 0.1816 mg/L, and well water sample B, with an average Fe^{2+} concentration of 0.1489 mg/L, meet the Fe^{2+} quality standard of < 0.2 mg/L as stipulated in Indonesian Ministry of Health Regulation No. 2 of 2023 (Indonesian Ministry of Health, 2023). Well water sample C, with an average Fe^{2+} concentration of 0.2419 mg/L, exceeds the quality standard limit of 0.2 mg/L. Well water C requires a reduction in Fe^{2+} levels using $FeCl_3$ coagulant synthesized from lathe workshop waste.

Reduction of Fe^{2+} levels using $FeCl_3$ as a coagulant from scrap iron

The reduction in Fe^{2+} concentration was achieved using $FeCl_3$ coagulant synthesized from scrap iron at concentrations of 0.1%, 0.2%, and 0.3%, with each treatment conducted in duplicate. The results of the Fe^{2+} concentration reduction are shown in **Table 3** and **Figure 7**.

Table 3. Results of Fe^{2+} concentration reduction using $FeCl_3$ as a coagulant from scrap iron

No.	Sample	Final concentration Fe^{2+} (mg/l)	Average level (mg/l)	% RPD	% Effectiveness
1	C (0.1%) I	0.1462	0.1457	0.63 %	39.76 %
2	C (0.1%) II	0.1453			
3	C (0.2%) I	0.0863	0.0859	1.07%	64.50%
4	C (0.2%) II	0.0854			
5	C (0.3%) I	0.0486	0.0481	1.91 %	80.10%
6	C (0.3%) II	0.0477			

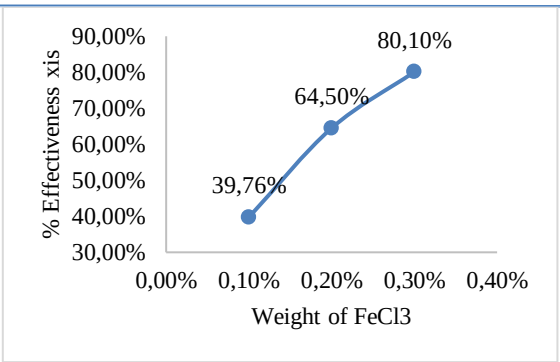

Fig. 7: Graph of $FeCl_3$ weight vs. % Effectiveness of Fe^{2+} reduction

The decrease in Fe^{2+} concentration following the addition of the coagulant $FeCl_3$ can be explained by the simultaneous mechanisms of oxidation and coagulation-precipitation occurring in the solution. When $FeCl_3$ dissolves in water, Fe^{3+} ions undergo hydrolysis to form hydroxide species such as $Fe(OH)^{2+}$, $Fe(OH)_2^+$, and ultimately an amorphous $Fe(OH)_3$ precipitate with a high surface area. Fe^{2+} ions present in the solution can be oxidized to Fe^{3+} by dissolved oxygen, particularly under neutral to basic pH conditions. The resulting Fe^{3+} then participates in the formation of insoluble $Fe(OH)_3$ flocs [16]. Consequently, the concentration of dissolved Fe^{2+} decreases because some of it is converted to Fe^{3+} and subsequently precipitates as iron hydroxide. Additionally, the formed $Fe(OH)_3$ flocs possess high adsorption capacity, enabling them to bind metal ions and colloidal particles through mechanisms such as sweep flocculation, surface adsorption, and charge neutralization. This process causes Fe^{2+} ions, which were originally in the dissolved phase, to transfer to the solid phase and be separated via sedimentation [17].

Increasing the $FeCl_3$ dose generally improves the efficiency of Fe^{2+} removal until the optimum dose is reached. At an adequate dose, the amount of $Fe(OH)_3$ formed increases, providing more active sites for adsorbing and coagulating metal ions. However, if the $FeCl_3$ dose is too high, particle restabilization may occur due to an excess of positive charge, so that removal efficiency no longer increases significantly. Recent studies indicate that coagulation using $FeCl_3$ is highly effective in removing various metal ions through the formation of stable, compact iron hydroxide flocs [11]. The effectiveness of the process is significantly influenced by pH, coagulant dose, mixing time, and initial metal concentration. Therefore, the observed decrease in Fe^{2+} concentration following the addition of $FeCl_3$ indicates that the dominant mechanism occurring is the oxidation of Fe^{2+} to Fe^{3+}, followed by the formation of $Fe(OH)_3$ precipitates and the binding of metal ions into the floc structure, thereby significantly reducing the concentration of dissolved Fe^{2+}[18].

Reduction of TDS Levels Using $FeCl_3$ as a Coagulant in Scrap Iron

The results of the initial TDS analysis of three well water samples using the gravimetric method are presented in **Table 4**.

Table 4. Results of TDS analysis of initial well water samples

No.	Sample code	Sample	Initial concentration (mg/l)	Average concentration	% RPD	Quality standard (mg/l)
1	C1	AI	348	344	2.33%	< 300
2	C2	AII	340			
3	C3	BI	244	240	3.33%	
4	C4	BII	236			
5	C5	CI	288	284	2.82%	
6	C6	CII	280			

Table 4 shows that the TDS level in Sample A averaged 344 mg/L, exceeding the quality standard set by Ministry of Health Regulation No. 2 of 2023 (300 mg/L), and thus did not meet water quality standards. To reduce the TDS, $FeCl_3$ coagulant derived from scrap iron was added at varying concentrations of 0.1%, 0.2%, and 0.3%. The results of the TDS reduction using $FeCl_3$ are shown in **Table 5** and **Figure 6**.

Table 5. Results of TDS reduction using $FeCl_3$ as a coagulant from scrap iron

No.	Sample	Final Fe^{2+} concentration (mg/l)	Average concentration (mg/l)	% RPD	% Effectiveness
1	C (0.1%) I	0.1462	0.1457	0.63 %	39.76 %
2	C (0.1%) II	0.1453			
3	C (0.2%) I	0.0863	0.0859	1.07%	64.50%
4	C (0.2%) II	0.0854			
5	C (0.3%) I	0.0486	0.0481	1.91 %	80.10%
6	C (0.3%) II	0.0477			

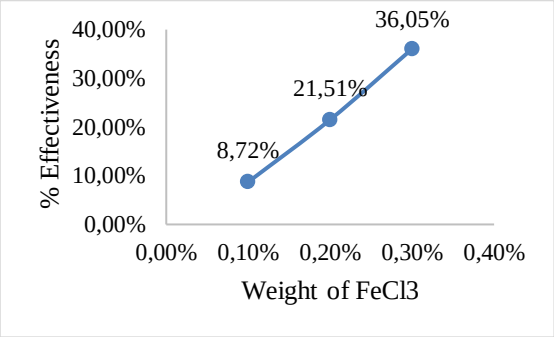

Fig. 6: Graph of $FeCl_3$ weight versus % TDS reduction efficiency

Table 5 and **Figure 6** show that increasing the dose of the $FeCl_3$ coagulant reduces TDS levels. A 0.1% dose can reduce TDS but does not yet meet the quality standard limit of 300 mg/L. At doses of 0.2% and 0.3%, TDS drops to 270 mg/L and 220 mg/L, respectively, which meets water quality standards for hygiene and sanitation. The effectiveness of TDS reduction was 8.72%, 21.51%, and 36.05%, respectively. This reduction is relatively low compared to the study by Esteki et al. (2024), which concluded that $FeCl_3$ is highly effective at reducing turbidity, TSS, and some dissolved organics, but less effective against TDS-constituting ions such as Na^+, Cl^-, and SO_4^{2-}. The addition of $FeCl_3$ in excessive doses or pH adjustment with $NaOH/H_2SO_4$ can increase TDS, especially at high pH (Prathna & Srivastava, 2021).

The reduction in Total Dissolved Solids (TDS) levels following the addition of the coagulant $FeCl_3$ occurs through the hydrolysis of Fe^{3+} ions, which produces various iron hydroxide species, such as $Fe(OH)^{2+}$, $Fe(OH)_2^+$, and $Fe(OH)_3$ precipitates. These species carry a high positive charge, enabling them to neutralize the negative charge of colloidal particles and dissolved compounds in wastewater. In addition to the charge neutralization mechanism, the formed $Fe(OH)_3$ also acts as an adsorbent capable of binding dissolved ions, dissolved organic compounds, phosphates, heavy metals, and other dissolved solid fractions. Subsequently, these particles aggregate to form larger flocs that settle easily, thereby reducing the concentration of dissolved substances in the liquid phase. Various studies indicate that iron-based coagulants, particularly $FeCl_3$, possess a high capacity for removing dissolved contaminants through the mechanisms of sweep flocculation and adsorption by the iron hydroxide flocs formed during the coagulation process [19].

The effectiveness of TDS reduction by $FeCl_3$ is greatly influenced by the coagulant dose, solution pH, mixing time, and wastewater characteristics. At the optimum dosage, an increase in the $FeCl_3$ concentration produces more $Fe(OH)_3$ flocs, thereby increasing the adsorption capacity for dissolved ions and significantly reducing the TDS value. However, excessive dosing can lead to an increase in residual chloride and dissolved iron ions, which may actually increase the final TDS [11]. Therefore, determining the optimal dosage through jar tests is a crucial step in achieving maximum TDS removal efficiency. Recent studies have shown that $FeCl_3$ provides better coagulation performance than several other coagulants in removing dissolved organic compounds, nutrients, and various contaminants that contribute to TDS levels, making it widely used in both industrial and domestic wastewater treatment [20].

4. Conclusion

The conclusion of this study is that iron scrap waste from a lathe workshop can be utilized as a raw material for $FeCl_3$ coagulant through a dissolution process using 32% hydrochloric acid. The presence of Fe^{3+} and Cl^- ions in the synthesized product was confirmed through qualitative testing. The synthesized $FeCl_3$ coagulant was proven effective in reducing Fe^{2+} and TDS levels in dug well water. A dosage of 0.3%

provided the highest effectiveness, with a reduction in Fe^{2+} of up to 80.10% and TDS of up to 36.05%, and met the clean water quality standards based on Indonesian Ministry of Health Regulation No. 2 of 2023.

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