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Optimization of Solid Phase Extraction Using Chelating Disk for Cr(III) and Fe(III) Concentration in Water Samples by ICP-AES Analysis

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ABSTRACT

Purpose of the study: This study aims to optimize the solid phase extraction method using a chelating disk for the concentration of Cr(III) and Fe(III) metals in water samples, determine the optimum concentration conditions, evaluate the percent recovery, and examine the effect of the presence of alkali and alkaline earth metal matrices on the effectiveness of separation and analysis using ICP-AES.

Methodology: The study used an experimental method with solid-phase extraction based on 3M Empore Chelating Disk chelating disks, Varian 720 ES ICP-AES instrument analysis, Eppendorf micropipettes, InoLab pH meters, and OHAUS analytical balances. Standard chemicals from Merck were used for solution preparation. The stages included sample preparation, pH optimization and HNO3 concentration, recovery test, RAL statistical analysis, F test, and BNT test.

Main Findings: The optimum concentration condition was obtained at pH 5.5 with HNO3 concentration of 2 M. The recovery percentage of Cr(III) reached 98.9% and Fe(III) was 99.1%. The concentration test showed a recovery of 97–100% at various concentrations and sample volumes. In the matrix test, the recovery of Cr(III) and Fe(III) was 99.0% and 99.6%, respectively, while alkali and alkaline earth ions showed low recovery so they did not interfere with the adsorption process of the target metal.

Novelty/Originality of this study: This research provides new insights into the selectivity of chelating disks under complex matrix conditions and expands the application of solid-phase extraction for more efficient and accurate ICP-AES-based trace metal analysis.

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1. INTRODUCTION

Heavy metal pollution in aquatic environments is a critical issue that has received widespread attention due to its impact on human health and ecosystem stability [1], [2]. Metals such as chromium and iron are commonly found in industrial waste from the metal plating, textile, leather tanning, and mining industries [3], [4]. In water, these metals can accumulate and potentially cause toxic effects if concentrations exceed established thresholds

[5][6]. Chromium in certain forms is known to be toxic and can trigger physiological disorders in living organisms. Therefore, monitoring Cr(III) and Fe(III) levels in water samples is a crucial step in environmental quality control.

Chromium and iron are metal elements that play different roles in environmental systems [7]. Iron is an essential element required by living organisms in certain amounts, but at high concentrations it can cause changes in water quality and disrupt biological processes [8], [9]. Meanwhile, chromium, particularly in certain ionic forms, can have negative impacts when accumulated in aquatic systems [10], [11]. The presence of these two metals in aquatic environments is generally at trace concentrations, making them difficult to detect directly without pretreatment. These conditions require sensitive and selective analytical methods to obtain accurate data.

One widely used metal analysis technique is Inductively Coupled Plasma–Atomic Emission Spectrometry (ICP-AES) [12], [13]. This technique offers advantages such as high sensitivity, simultaneous multi-element analysis capabilities, and a wide linearity range. ICP-AES has been widely applied for metal analysis in various environmental matrices, including surface water, groundwater, and industrial wastewater [14], [15]. However, for metal analysis at very low concentrations, the direct use of ICP-AES often faces limitations due to low analyte concentrations and matrix interference. Therefore, a preprocessing step in the form of concentration is required before measurement.

Solid-phase extraction (SPE), a widely used concentration method for trace metal analysis, offers several advantages over liquid-liquid extraction methods, including less solvent use, a simpler procedure, shorter analysis time, and improved separation capabilities [16], [17]. One effective SPE medium for metal ion separation is a chelating disk, a disk containing a complexing group that selectively binds metal ions [18], [19]. The interaction between the chelating group and the metal ions allows the metal to be retained on the disk surface, which can then be eluted using specific solvents [20]. The use of chelating disks offers significant opportunities to improve the efficiency of metal concentration prior to instrumental analysis.

The success of a solid-phase extraction process is significantly influenced by several operational parameters, such as sample pH, eluent type and concentration, and the presence of interfering ions in the matrix [21], [22]. The pH value influences the form of metal speciation and the ability of the active groups on the chelating disk to interact with target ions. The concentration of nitric acid as an eluent also determines the effectiveness of metal ion release from the disk surface after the adsorption process [23], [24]. Furthermore, the presence of alkali and alkaline earth metal ions in water samples can create adsorption competition, affecting analyte recovery efficiency [25], [26]. Therefore, optimizing these parameters is essential to achieve optimal concentration conditions.

Several previous studies have reported the use of solid-phase extraction for heavy metal concentration, but most have focused on the analysis of a single metal or used conventional sorbents with limited selectivity [27], [28]. Research related to the use of chelating disks for the simultaneous concentration of Cr(III) and Fe(III), particularly evaluating the influence of alkali and alkaline earth ion matrices, is still relatively limited. Furthermore, many studies have not systematically examined the relationship between sample pH, HNO3 concentration, and the recovery efficiency of these two metals. This research gap highlights the need to develop a more comprehensive method for metal analysis in complex water matrices. The novelty of this research lies in the integrated optimization of concentration parameters using a chelating disk and evaluation of its performance under matrix conditions that mimic real-world environmental samples.

The urgency of this research is based on the need for an accurate, efficient, and applicable analytical method for water quality monitoring. A method capable of providing high recovery under complex matrix conditions is essential to support environmental monitoring and enforcement of regulations related to heavy metal pollution. Developing an optimal concentration procedure will also increase the sensitivity of ICP-AES measurements in detecting trace metals. Based on this, this study aims to determine the optimum sample pH and HNO3 concentration for concentrating Cr(III) and Fe(III), the percent recovery of Cr(III) and Fe(III) from the concentration under optimal conditions, and the percent recovery of Cr(III) and Fe(III) in the presence of alkali and alkaline earth metal matrices. The results of this study are expected to contribute scientifically to the development of solid-phase extraction-based heavy metal analysis methods using a chelating disk.

2. RESEARCH METHOD

2.1. Research Materials

The materials used in this study included Merck FeCl3·6H2O, CrCl3·6H2O, MgSO4·7H2O, KNO3, NaNO3, and CaSO4·2H2O solids. In addition, 65% nitric acid (HNO3) solution, 100% acetic acid (CH3COOH), and 25% ammonium hydroxide (NH4OH) solution were also used, all of which were obtained from Merck, Germany. Other supporting materials were distilled water and 3M Empore Chelating Disk chelating discs with a diameter of 1 cm which were used as solid-phase extraction media. All of these materials were used without further purification treatment.

2.2. Research Tools

The equipment used in this study included Eppendorf micropipettes with a volume range of 100–1000 µL and 500–5000 µL, a set of vacuum devices, droppers, beakers with a capacity of 100 mL and 250 mL, and a glass funnel. In addition, iron tongs, a container, a glass stirrer, tweezers, sample bottles, and a spray bottle were also used as supporting equipment during the research process. Measurements of acidity were carried out using an InoLab pH meter, while material weighing used an OHAUS analytical balance. The main instrument for metal content analysis was the Varian 720 ES ICP-AES equipped with a set of 5 mL plastic syringes.

2.3. Research Stages

This research phase began with material preparation to ensure all solutions and reagents were ready for use according to analysis requirements. Next, a chelating disk column was prepared as the solid-phase extraction medium used in the metal concentration process. A calibration curve was then determined to obtain a linear relationship between the concentration of the standard solution and the instrument response, which served as the basis for analyte quantification [29], [30]. The next stage was parameter optimization, which included determining the optimum sample pH and the optimum HNO3 concentration as the eluent in the metal desorption process. After obtaining optimum conditions, the study continued with a concentration test of Cr(III) and Fe(III) using the chelating disk. The next stage was testing the percent recovery of Cr(III) and Fe(III) in the presence of interfering ion matrices to evaluate the effect of alkali and alkaline earth metals on concentration efficiency. All measurement data were then analyzed to determine the optimal concentration conditions, evaluate method effectiveness, and assess the performance of the solid-phase extraction method in metal analysis using ICP-AES.

2.4. How the Research Works

The study began with the preparation of stock solutions of Cr(III), Fe(III), Mg(II), K(I), Na(I), and Ca(II) with a concentration of 1000 mg/L made from the respective solids using 1 M HNO3 solvent. Next, pH adjusting solutions were prepared, namely pH 3 from 1 M HNO3 and pH 4–7 from a mixture of CH3COOH and NH4OH. A 1 cm diameter chelating disk was installed in a 5 mL plastic syringe coated with fiber on both sides, then assembled with a vacuum system. The chelating disk column was conditioned through washing with distilled water, pH adjustment using a buffer solution, flowing a sample mixture of Cr(III) and Fe(III), and elution using HNO3. The concentrated eluate was then collected and analyzed using ICP-AES. A calibration curve was made from a standard solution of a mixture of Cr(III) and Fe(III) at several concentration variations to obtain a linear relationship between concentration and emission intensity. Parameter optimization was carried out by varying the pH of the sample (3–7) and the concentration of HNO3 (0.5–3 M) to determine the optimum concentration conditions. After the optimum conditions were obtained, a concentration test was carried out using variations in the concentration and volume of the metal mixture sample. Furthermore, a recovery test was carried out by adding a matrix of Na(I), K(I), Mg(II), and Ca(II) ions to evaluate the effect of interfering ions on concentration efficiency. All analysis results were calculated in the form of percent recovery to assess the performance of the solid phase extraction method using a chelating disk.

2.5. Data Analysis

The research data were analyzed quantitatively through the calculation of linear regression equations and correlation coefficients (R^2) to construct calibration curves for Cr(III) and Fe(III) metals based on the relationship between standard concentrations and emission intensities measured using ICP-AES. The average value of each measurement was calculated from the results of repetitions to obtain representative data. The effectiveness of concentration was evaluated through the calculation of percent recovery by comparing the metal concentrations before and after the adsorption process, as well as the determination of the theoretical concentration factor based on the ratio of sample volume to eluent volume [31], [32]. In the matrix test, the percent removal and matrix recovery were calculated to determine the effect of interfering ions on the target metal adsorption process. Furthermore, the data on sample pH optimization and eluent concentration were analyzed statistically using the Completely Randomized Design method at a 95% confidence level through the F test. If there was a significant difference between treatments, then it was continued with the Least Significant Difference (LSD) test to determine the best treatment.

3. RESULTS AND DISCUSSION

In this study, the concentration of Cr(III) and Fe(III) metals and the separation of alkali and alkaline earth matrices were carried out using the solid phase extraction method with Chelating disk adsorbent. In general, this study consists of several stages, namely parameter optimization which includes optimization of the pH of Cr(III) and Fe(III) metal samples and HNO3 concentration, Cr(III) and Fe(III) metal concentration test, and Cr(III) and Fe(III) metal recovery test in the presence of a matrix.

3.1. Parameter Optimization

3.1.1. Optimization of pH of Cr(III) and Fe(III) Metal Samples

In the sample pH optimization stage, the adsorption process of Cr(III) and Fe(III) metals was carried out using a chelating disk containing an active iminodiacetate (IDA) group. The pH value is an important parameter because it greatly affects the ability of metal ions to bind to the IDA group when the sample solution is flowed through the column. In this study, optimization was carried out with variations in pH 3, 4, 5, 5.5, 6, and 7. The IDA group acts as a complexing ligand that binds Cr(III) and Fe(III) ions through the formation of chelate compounds. Before the binding process takes place, the IDA group first undergoes deprotonation by releasing H^+ ions at certain pH conditions. The results of observations on the effect of pH on the adsorption of Cr(III) and Fe(III) are then presented in the form of a relationship curve between sample pH and percent recovery in Figures 1 and 2.

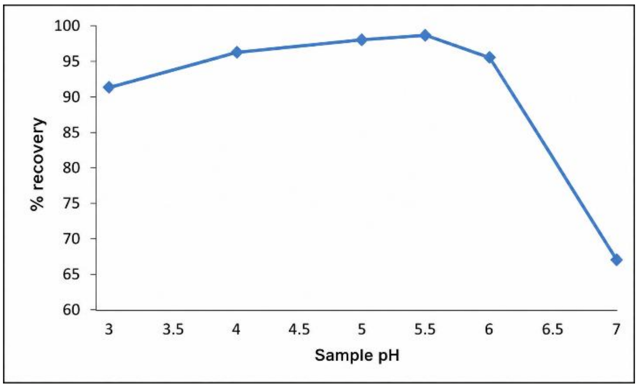

Figure 1. Relationship curve between pH Cr(III) and %recovery

Based on the results of the F test analysis using a completely randomized design (CRD), it was found that pH variations had a significant effect on the adsorption of Cr(III) metal on the chelating disk. This was indicated by the calculated F value being greater than the F table, so there was a significant difference between pH treatments. Therefore, the analysis was continued with the Least Significant Difference (LSD) test to determine the differences between treatments more specifically. The LSD test results showed that pH had a significant effect on the formation of complexes between Cr(III) ions and iminodiacetate (IDA) groups on the chelating disk. The recovery values at pH 5.5 and 6 did not show a significant difference, so the optimum pH range for Cr(III) adsorption was within that range. However, in this study, pH 5.5 was chosen as the optimum condition because at higher pH, Cr(III) began to precipitate so that the adsorption process did not take place optimally. Based on Figure 1, Cr(III) adsorption shows an increase in percent recovery in the pH range of 3 to 5.5, then decreases at pH 6 and 7. At pH 3, the amount of metal adsorbed is still relatively low because the IDA group has not been completely deprotonated, resulting in suboptimal metal ion binding capacity. The increase in recovery at pH 5.5 occurs because the IDA group undergoes optimal deprotonation through the release of H^+ ions, forming negatively charged groups that effectively bind Cr(III) ions. This condition allows for the formation of a stable chelate complex between Cr(III) and the active group on the chelating disk.

The decrease in percent recovery at pH 6 and 7 is due to a hydrolysis reaction that triggers the formation of chromium hydroxide precipitates, Cr(OH)3. This precipitate formation reduces the number of free Cr(III) ions that can interact with the IDA group, thus decreasing adsorption efficiency. Theoretically, the very low Ksp value of Cr(OH)3 indicates that this compound readily precipitates at higher pH conditions. Based on theoretical calculations, Cr(III) precipitation begins to occur at a pH of around 6.16. Measurement data show that the Cr(III) recovery percentage at pH 3, 4, 5, 5.5, 6, and 7 is 91%; 96.4%; 98.4%; 98.9%; 95.4%; and 66.6%, respectively. Thus, based on the results of ICP-AES measurements and statistical analysis, the optimum pH for Cr(III) adsorption is determined at pH 5.5.

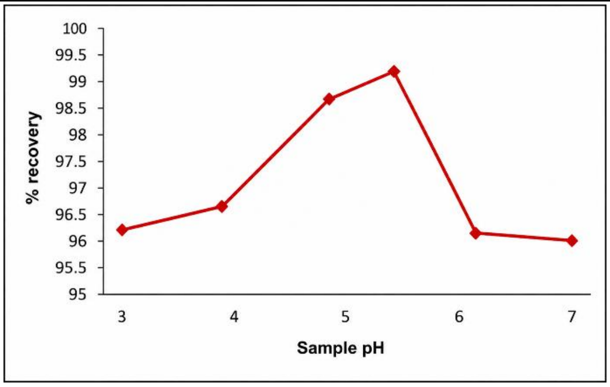

Figure 2. Relationship curve between Fe(III) pH and % recovery

Based on the results of the F-test at a 5% significance level as per Appendix L.5.1, it was found that pH variation had no significant effect on Fe(III) adsorption on the chelating disk. This is indicated by the calculated F value being smaller than the F table, thus concluding that Fe(III) ions can still be adsorbed well in the pH range of 3–7. Nevertheless, this study determined pH 5.5 as the optimum condition because it produced the highest recovery percentage, at 99.1%.

Based on Figure 2, Fe(III) adsorption showed an increase in recovery percentage in the pH range of 3 to 5.5, from 96.2% to 99.1%. After passing the optimum pH, recovery percentage decreased at pH 6 and 7 to 96.1% and 96.0%, respectively. This pattern indicates that the Fe(III) adsorption process is most effective at pH 5.5. Under these conditions, the iminodiacetate (IDA) group undergoes optimal deprotonation, transforming both carboxylate groups into negatively charged forms capable of strong binding with Fe(III) ions. This interaction results in the formation of a stable chelate complex and increases the efficiency of metal adsorption on the chelating disk surface.

The decrease in recovery percentage at pH 6 and 7 is thought to be due to a hydrolysis reaction that triggers the formation of iron hydroxide precipitates, Fe(OH)3. This precipitate formation reduces the number of free Fe(III) ions available to interact with the IDA group, resulting in a less than optimal adsorption process. Theoretically, based on the Ksp value of Fe(OH)3 of 3.8×10^{-38}, precipitation begins to occur at a pH of around 6.54. If the pH is further increased, the likelihood of precipitate formation increases, thus further decreasing adsorption efficiency. In general, the adsorption capacity of the IDA group for Cr(III) and Fe(III) metals is in the pH range of 4–6, with optimum stability at pH 5.5. Based on the results in Figure 1 and Figure 2, it can be concluded that the optimum conditions for adsorption of both metals on the chelating disk were obtained at pH 5.5.

3.1.2. Optimization of HNO3 Concentration

In this optimization stage, the desorption process of Cr(III) and Fe(III) metals is influenced by the concentration of HNO3 solution used as an eluent. The selection of HNO3 is based on its properties as a strong acid that is effective in releasing metal ions from the active group of the chelating disk without causing significant interference to the analysis process using ICP-AES. The desorption mechanism occurs when the H^+ ion from HNO3 interacts with the deprotonated carboxylate group ($CH2COO^-$) on the iminodiacetate (IDA) group, so that the group is re-protonated to CH2COOH. This process causes the metal ion previously bound to the IDA group to be released from the surface of the chelating disk. The reaction mechanism is shown in Figure 3.

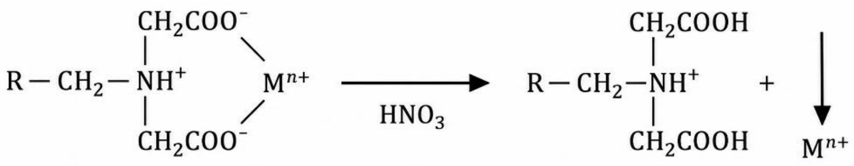

Figure 3. Reaction of the IDA Group with HNO3

In the elution process, H^+ ions from HNO3 replace the position of metal ions bound to the IDA group, so that Cr(III) and Fe(III) metals are desorbed and carried out with the eluent. The desorption eluate was then analyzed using ICP-AES to determine the metal content that was successfully separated from the adsorbent media. Optimization of the HNO3 concentration was carried out to obtain the optimum eluent concentration that was able to desorb the metal maximally, resulting in the highest percent recovery. The variations in HNO3 concentration used in this study were 0.5 M; 1 M; 2 M; and 3 M. The measurement data are presented in the appendix, while the relationship between eluent concentration and percent recovery of Cr(III) and Fe(III) metals is shown in Figures 4 and 5.

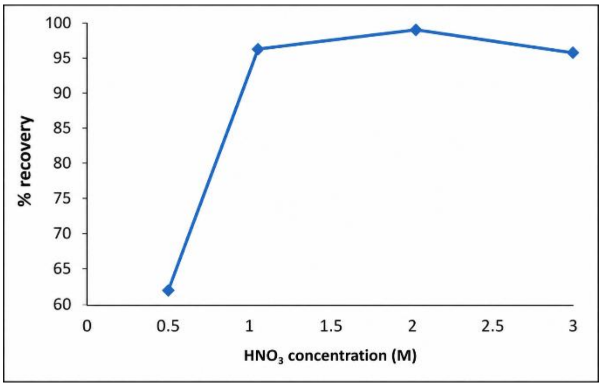

Figure 4. Relationship curve between HNO3 concentration and % recovery of Cr(III)

In Figure 5, it is known that at a concentration of 0.5 M - 2 M there is an increase, while at a concentration of 3 M there is a decrease. At a concentration of 0.5-2 M the % recovery obtained is 62% -99%, while at a concentration of 3 M the % recovery obtained is 95%. At a concentration of 0.5-2 M there is an increase in desorption because HNO3 will protonate the acetyl group (COO-) to form an acetate group (COOH) which results in the chelate bond with the Cr(III) metal ion being released. The higher the concentration of HNO3, the more H+ will replace the Cr(III) metal. At a concentration of 2 M is the most optimum concentration.

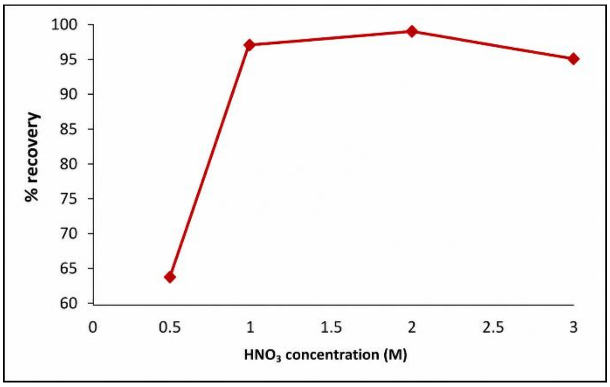

Figure 5. Relationship curve between HNO3 concentration and % recovery of Fe(III)

Based on Figure 5, the Fe(III) recovery percentage increased with increasing HNO3 concentration from 0.5 M to 2 M, from 64.1% to 99.1%. However, at a concentration of 3 M, recovery decreased to 95.0%. This pattern indicates that increasing the eluent concentration to a certain limit can increase the effectiveness of Fe(III) ion desorption from the chelating disk surface, but at excessively high concentrations, measurement efficiency tends to decrease.

The results of statistical analysis using a completely randomized design (CRD) using the F test showed that variations in HNO3 concentration significantly affected the desorption of Cr(III) and Fe(III). This is indicated by the calculated F value being greater than the F table. Further analysis using the Least Significant Difference (LSD) test showed significant differences between treatments, particularly at a concentration of 2 M, which provided optimum results for both metals. Therefore, the HNO3 concentration of 2 M was determined as the optimum concentration for the desorption of Cr(III) and Fe(III).

Increasing the concentration of HNO3 affects the desorption capacity because the higher the acid concentration, the greater the number of H^+ ions available to replace the metal ions bound to the iminodiacetate (IDA) group. This ion exchange process causes the release of metals from the chelating disk surface to be more effective. Based on the research results, at an optimum HNO3 concentration of 2 M, a recovery percentage of 98.9% was obtained for Cr(III) and 99.1% for Fe(III). The decrease in recovery at a concentration of 3 M is thought to be caused by the high concentration of the solution which affects the analysis process using ICP-AES. A solution that is too concentrated can interfere with the nebulization process in the nebulizer, so that the number of aerosol droplets entering the torch is reduced and the detected emission intensity is lower.

3.2. Cr(III) and Fe(III) Metal Concentration Test

The concentration test was conducted to determine whether the Chelating disk could concentrate metals at low concentrations with a large enough sample volume. The variations of the samples were 0.5 mg/L in 10 mL; 0.5 mg/L in 20 mL; 0.05 mg/L in 50 mL; 0.1 mg/L in 100 mL. After being measured using ICP-AES, the % recovery was obtained as shown in Tables 1 and 2.

Table 1. Cr(III) Concentration Test Data

C Early (mg/L)	Vsample (mL)	V eluent (mL)	Concentration Level	C end (mg/L)	% recovery
0.5	10	10	1	0.5	100
0.5	20	10	2	0.99	99
0.05	50	10	5	0.246	98.4
0.1	100	10	10	0.976	97.6

Table 2. Fe(III) Concentration Test Data

C Early (mg/L)	Vsample (mL)	V eluent (mL)	Concentration Level	C end (mg/L)	% recovery
0.5	10	10	1	0.5	100
0.5	20	10	2	0.99	100
0.05	50	10	5	0.249	99.6
0.1	100	10	10	0.978	97.8

Based on the data above, it can be seen that if the concentration of Cr(III) and Fe(III) metals is lower, the sample volume required is greater so that a larger concentration factor is obtained. This is the basic principle of the concentration technique. From the results of the table above, it can be concluded that the Chelating disk can bind metals well even with low concentrations and quite large volumes. When applied to waters, a relatively large sample volume is required at very low metal concentrations, so that the metal concentration can be measured precisely after concentration with the Chelating disk.

3.3. Recovery Test of Cr(III) and Fe(III) Metals in the Presence of Matrix

In this study, the sample matrices examined were Na(I), K(I), Ca(II), and Mg(II) because they are generally found in natural water and can interfere with the detection of Cr(III) and Fe(III) during ICP-AES measurements. Alkali and alkaline earth metals have low ionization energies, making them more readily ionized in the argon plasma of ICP-AES than other metals. In large quantities, ionization of these alkali and alkaline earth metals can suppress the ionization of other metals (e.g., transition metals) in the plasma, leading to measurement errors. Furthermore, high concentrations of alkali and alkaline earth metals can damage the ICP-AES optics. Therefore, the alkali and alkaline earth metals must be separated before measuring transition metals, which are present in much lower concentrations.

The analysis process was carried out by adding the following matrixes: Na(I) 20 mg/L; Ca(II) 50 mg/L; K(I) 10 mg/L; Mg(II) 15 mg/L in a mixture of Cr(III) and Fe(III) samples with concentrations of 0.5 ppm each. In the matrix sample, the % matrix recovery is calculated to determine how much alkali metal is still left in the Chelating disk, the smaller the % matrix recovery, the better. The calculation of the % matrix removal is intended to determine how much matrix has been lost, and the greater the amount produced, the better. From the results obtained, it is known that relatively little alkali metal is bound in the Chelating disk. The data are shown in Table 3.

Table 3. Recovery Test Data on Metal Ions and Matrix

No	Logam	%recovery	%removal
1	Cr (III)	99	1
2	Fe (III)	99.6	0.4
3	K	2.74	97.26
4	Mg (II)	8.107	91.9
5	Na	0.75	99.25
6	Ca (II)	0.0712	99.92

From the data above, the % recovery of metals from the ICP-AES detection results is Cr(III) 99% and Fe(III) metal while in the matrix K 2.740%, Mg(II) 8.107%, Na 0.750%, and Ca(II) 0.0712% then in % removal obtained K 97.26%, Mg(II) 91.893%, Na 99.25%, Ca(II) 99.92%. It can be concluded that the matrix adsorbed in the Chelating disk is only a little because the matrix cannot form chelates with the IDA group while in metals, the IDA group is quite good in forming chelates. Alkali and alkaline earth matrices cannot form chelates on the

Chelating disk because the matrix is more easily solvated with water and rarely complexed so that the IDA group tends to bind to the transition. In the formation of chelate complexes, d orbitals are utilized, whereas these alkalis do not have these orbitals.

When the sample is inserted into a column containing a Chelating disk, Cr(III) and Fe(III) metals will form chelates with the IDA group while Na(I), K(I), Ca(II), and Mg(II) metals will dissolve into ionic states and come out with the solvent. If viewed from the ionization energy, Cr(III) and Fe(III) metals tend to form chelates more easily than alkali metals. This is because Cr(III) and Fe(III) have smaller atomic radii and greater ionization energy than Na(I), K(I), Ca(II), Mg(II) metals. The greater the ionization energy, the easier it is for an element to form chelates and vice versa if the ionization energy is small then it is more difficult to form chelates and tends to dissolve in ionic states. So when measuring the sample, a greater % recovery of Cr(III) and Fe(III) is obtained compared to Na(I), K(I), Ca(II), Mg(II).

The results of this study indicate that the use of iminodiacetate (IDA)-based chelating disks has good adsorption and desorption capabilities for Cr(III) and Fe(III) ions in the solid-phase extraction process. The success of the adsorption process is influenced by the solution pH, as pH determines the degree of deprotonation of the active IDA group in forming complexes with metal ions. In highly acidic conditions, the active group is not optimally deprotonated, resulting in low metal binding capacity. Conversely, at excessively high pH conditions, metal hydroxide precipitates begin to form, reducing the number of free ions in the solution [33], [34]. This indicates that pH regulation is a critical factor in maintaining the stability of metal complexes during the extraction process.

The effectiveness of using HNO3 as an eluent indicates that the metal desorption process from the chelating disk surface is significantly influenced by the concentration of H+ ions in the solution. As the acid concentration increases, up to the optimum level, the protons increase the ability to replace metal ions on the IDA group, resulting in more effective metal release [35]. However, at excessively high concentrations, measurement efficiency decreases due to increased interference with the nebulization process during ICP-AES analysis [36], [37]. These conditions indicate that optimizing eluent concentration is not only crucial for increasing metal recovery but also for maintaining the stability of analytical instrument performance during the measurement process.

The chelating disk's ability to maintain high recovery values at low metal concentrations demonstrates that the solid-phase extraction method has good sensitivity for analyzing trace metals in water samples [38], [39]. The lower the metal concentration in the sample, the larger the sample volume required to increase the concentration factor and optimally detect the metal using ICP-AES. This demonstrates that the developed method has potential for application in environmental quality monitoring, particularly in waters with very low heavy metal content but with the potential to cause toxic impacts if accumulated continuously.

The presence of alkali and alkaline earth ions in the sample did not significantly affect the recovery of Cr(III) and Fe(III). This indicates that the chelating disk has good selectivity for transition metal ions compared to other matrix ions. The IDA group more readily forms complexes with transition metals due to the involvement of d orbitals in the coordination bond formation [40], [41]. Meanwhile, alkali and alkaline earth ions tend to remain in dissolved ion form and are therefore less adsorbed on the disk surface. This selectivity is a crucial advantage in heavy metal analysis because it minimizes matrix interference, which often occurs in environmental samples with complex ionic compositions.

The results of this study align with several previous studies that have shown that solid-phase extraction methods can increase the sensitivity and selectivity of heavy metal analysis at trace concentrations. Research by Badawy et al., [16] explains that SPE techniques offer high efficiency in metal concentration due to the use of fewer solvents and superior separation capabilities compared to conventional extraction methods. Furthermore, research by Ahmad et al., [26] shows that metal preconcentration using a chelating agent-based material can improve the accuracy of trace metal analysis in environmental samples with complex matrices. These findings reinforce the potential for the use of IDA-based chelating disks in this study to be developed as an effective and applicable heavy metal analysis method for water quality monitoring.

This research makes a significant contribution to the development of a more selective, sensitive, and efficient solid-phase extraction-based heavy metal analysis method for environmental analysis. The developed method increases the effectiveness of Cr(III) and Fe(III) concentration at low concentrations, thus supporting more accurate water quality monitoring activities [42], [43]. Furthermore, the use of a chelating disk with high recovery and low matrix interference has the potential to be applied to the analysis of environmental samples with complex ion compositions. Academically, this research also expands the development of ICP-AES applications combined with preconcentration techniques for trace heavy metal analysis in environmental chemistry and instrumental analysis.

This research still has several limitations that require consideration. The study focused only on the analysis of Cr(III) and Fe(III), so the method's effectiveness against other types of heavy metals has not been comprehensively evaluated. Furthermore, the testing used laboratory-made samples with conditioned matrices, which do not fully represent the complexity of natural environmental samples such as river water, industrial wastewater, or groundwater [44], [45]. This study also did not assess the stability of the chelating disk during repeated use or the influence of other instrumental parameters on the sensitivity of ICP-AES. Therefore, further research is needed to test the performance of the method on various types of real environmental samples with more complex matrix conditions.

4. CONCLUSION

Based on the results of the research that has been conducted, it can be concluded that the concentration process of Cr(III) and Fe(III) metals using chelating disks shows optimum conditions at a sample pH of 5.5 with an HNO3 eluent concentration of 2 M. This condition provides the most effective adsorption and desorption results, resulting in the highest percent recovery for both metals. In the concentration test, the percent recovery of Cr(III) and Fe(III) metals was relatively uniform, which was in the range of 97–100%. These results indicate that the chelating disk has high effectiveness in the concentration process of both metals, even though it was carried out at different concentrations and sample volumes. Thus, the solid phase extraction method using chelating disks has been proven to be able to provide good performance in concentrating Cr(III) and Fe(III) metals. In the recovery test with the presence of a matrix, the percent recovery of Cr(III) and Fe(III) metals remained high, at 99.0% and 99.6%, respectively. Meanwhile, the recovery of alkali and alkaline earth metal ions detected was relatively low, namely K(I) of 2.740%, Mg(II) of 8.107%, Na(I) of 0.750%, and Ca(II) of 0.0712%. These results indicate that the presence of alkali and alkaline earth metal matrix does not have a significant effect on the recovery of Cr(III) and Fe(III), so this method has good selectivity for the analysis of target metals in water samples. Further research is recommended to test the solid-phase extraction method using chelating disks on real environmental samples such as river water, industrial wastewater, and groundwater to obtain more applicable method validation in complex matrix conditions. Furthermore, future research can develop the use of other types of adsorbents or complexing groups to increase the selectivity and efficiency of simultaneous concentration of various heavy metals.

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AUTHOR CONTRIBUTIONS

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

CONFLICTS OF INTEREST

The authors declare no conflict of interest.

USE OF ARTIFICIAL INTELLIGENCE (AI)-ASSISTED TECHNOLOGY

Not applicable.

REFERENCES

[1] M. Sharma, R. Kant, A. K. Sharma, and A. K. Sharma, "Exploring the impact of heavy metals toxicity in the aquatic ecosystem," *Int. J. Energy Water Resour.*, vol. 9, no. 1, pp. 267–280, Mar. 2025, doi: 10.1007/s42108-024-00284-1.

[2] D. Piwowarska, E. Kiedrzyńska, and K. Jaszczyszyn, "A global perspective on the nature and fate of heavy metals polluting water ecosystems, and their impact and remediation," *Crit. Rev. Environ. Sci. Technol.*, vol. 54, no. 19, pp. 1436–1458, 2024, doi: 10.1080/10643389.2024.2317112.

[3] M. D. Ahmed, K. M. Maraz, and R. Amin Khan, "Prospects and challenges of chrome tanning: Approach a greener technology in leather industry," *Guigoz. Sci. Rev.*, vol. 7, no. 3, pp. 42–49, 2021, doi: 10.32861/sr.73.42.49.

[4] A. Arti and R. Mehra, "Analysis of heavy metals and toxicity level in the tannery effluent and the environs," *Environ. Monit. Assess.*, vol. 195, no. 5, pp. 554–561, May 2023, doi: 10.1007/s10661-023-11154-4.

[5] G. I. Edo *et al.*, "Environmental persistence, bioaccumulation, and ecotoxicology of heavy metals," *Chem. Ecol.*, vol. 40, no. 3, pp. 322–349, Mar. 2024, doi: 10.1080/02757540.2024.2306839.

[6] M. M. Uddin, M. C. M. Zakeel, J. S. Zavahir, F. M. M. T. Marikar, and I. Jahan, "Heavy metal accumulation in rice and aquatic plants used as human food: A general review," *Toxics*, vol. 9, no. 12, pp. 1–19, 2021, doi: 10.3390/toxics9120360.

[7] J. Bell, X. Ma, T. J. McDonald, C.-H. Huang, and V. K. Sharma, "Overlooked role of chromium(V) and chromium(IV)

in chromium redox reactions of environmental importance," *ACS ES T Water*, vol. 2, no. 6, pp. 932–942, 2022, doi: 10.1021/acsestwater.1c00409.

[8] E. C. Emenike, K. O. Iwuozor, and S. U. Anidiobi, "Heavy metal pollution in aquaculture: Sources, impacts and mitigation techniques," *Biol. Trace Elem. Res.*, vol. 200, no. 10, pp. 4476–4492, Oct. 2022, doi: 10.1007/s12011-021-03037-x.

[9] F. Kordbacheh and & Golnaz Heidari, "Water pollutants and approaches for their removal," *Mater. Chem. Horizons*, vol. 2, no. 2, pp. 139–153, 2023, [Online]. Available: https://mch.du.ac.ir/article_324.html

[10] E. Sawicka, K. Jurkowska, and A. Piwowar, "Chromium (III) and chromium (VI) as important players in the induction of genotoxicity – Current view," *Ann. Agric. Environ. Med.*, vol. 28, no. 1, pp. 1–10, 2021, doi: 10.26444/aaem/118228.

[11] K. Pandey, B. S. Saharan, R. Kumar, D. Jabborova, and J. S. Duhan, "Modern-day green strategies for the removal of chromium from wastewater," *J. Xenobiotics*, vol. 14, no. 4, pp. 1670–1696, 2024, doi: 10.3390/jox14040089.

[12] S. R. Khan, B. Sharma, P. A. Chawla, and R. Bhatia, "Inductively coupled plasma optical emission spectrometry (ICP-OES): A powerful analytical technique for elemental analysis," *Food Anal. Methods*, vol. 15, no. 3, pp. 666–688, Mar. 2022, doi: 10.1007/s12161-021-02148-4.

[13] E. A. Karpukhina, E. A. Vlasova, D. S. Volkov, and M. A. Proskurnin, "Comparative study of sample-preparation techniques for quantitative analysis of the mineral composition of humic substances by inductively coupled plasma atomic emission spectroscopy," *Agronomy*, vol. 11, no. 12, pp. 1–22, 2021, doi: 10.3390/agronomy11122453.

[14] D. P. P. Talware, "Physico-chemical analysis and detection of heavy metals in water through electro plating industries by ICP-AES technique," *Int. J. Res. Appl. Sci. Eng. Technol.*, vol. 9, no. 8, pp. 2244–2247, 2021, doi: 10.22214/ijraset.2021.37756.

[15] D. Yadav, R. Jha, P. Kumar, and P. Singh, "Various Analytical Techniques for Se Determination in Different Matrices," in *Selenium Contamination in Water*, Wiley, 2021, pp. 91–114. doi: 10.1002/9781119693567.ch6.

[16] M. E. I. Badawy, M. A. M. El-Nouby, P. K. Kimani, L. W. Lim, and E. I. Rabea, "A review of the modern principles and applications of solid-phase extraction techniques in chromatographic analysis," *Anal. Sci.*, vol. 38, no. 12, pp. 1457–1487, Dec. 2022, doi: 10.1007/s44211-022-00190-8.

[17] E. Öztürk Er, G. Dalgıç Bozyiğit, Ç. Büyükpınar, and S. Bakırdere, "Magnetic nanoparticles based solid phase extraction methods for the determination of trace elements," *Crit. Rev. Anal. Chem.*, vol. 52, no. 2, pp. 231–249, Feb. 2022, doi: 10.1080/10408347.2020.1797465.

[18] P. Yudaev and E. Chistyakov, "Chelating extractants for metals," *Metals (Basel).*, vol. 12, no. 8, pp. 1–60, 2022, doi: 10.3390/met12081275.

[19] D. A. B. Obukohwo, "Review on processes in liquid-liquid and solid phase extraction," *Int. J. Res. Appl. Sci. Eng. Technol.*, vol. 11, no. 1, pp. 1276–1286, 2023, doi: 10.22214/ijraset.2023.48272.

[20] I. Gulcin and S. H. Alwasel, "Metal Ions , metal chelators and metal chelating assay as antioxidant method," *Processes*, vol. 10, no. 132, pp. 1–16, 2022, doi: 10.3390/pr10010132.

[21] G. J. Maranata, N. O. Surya, and A. N. Hasanah, "Optimising factors affecting solid phase extraction performances of molecular imprinted polymer as recent sample preparation technique," *Heliyon*, vol. 7, no. 1, pp. 1–12, 2021, doi: 10.1016/j.heliyon.2021.e05934.

[22] M. Mahdavijalal, C. Petio, G. Staffilano, R. Mandrioli, and M. Protti, "Innovative solid-phase extraction strategies for improving the advanced chromatographic determination of drugs in challenging biological samples," *Molecules*, vol. 29, no. 10, pp. 1–28, 2024, doi: 10.3390/molecules29102278.

[23] Y. Yang *et al.*, "The adsorption and desorption behavior and mechanism research of cobalt, nickel and copper in nitrite - sulfuric acid system," *Sep. Sci. Technol.*, vol. 57, no. 12, pp. 1848–1859, Aug. 2022, doi: 10.1080/01496395.2021.2021425.

[24] F. Mayer *et al.*, "Best of both worlds: Adsorptive ultrafiltration nanocellulose-hypercrosslinked polymer hybrid membranes for metal ion removal," *Small Sci.*, vol. 4, no. 10, pp. 1–11, 2024, doi: 10.1002/smsc.202400182.

[25] J. Jiang, H. Wang, J. Ren, L. Deng, and D. Che, "Effect of inherent alkali and alkaline earth metals in biochar on adsorption of Pb2+ in aqueous solution: Different roles of Na/Mg/K/Ca," *Sep. Purif. Technol.*, vol. 354, p. 128766, Feb. 2025, doi: 10.1016/j.seppur.2024.128766.

[26] H. Ahmad, F. M. Husain, and R. A. Khan, "Graphene oxide lamellar membrane with enlarged inter-layer spacing for fast preconcentration and determination of trace metal ions," *RSC Adv.*, vol. 11, no. 20, pp. 11889–11899, 2021, doi: 10.1039/d1ra01055g.

[27] I. Hagarová and L. Nemček, "Analytical application of layered double hydroxides as high-capacity sorbents in dispersive solid phase extraction for the separation and Preconcentration of (Ultra)Trace Heavy Metals," *Crit. Rev. Anal. Chem.*, vol. 54, no. 8, pp. 3114–3127, Nov. 2024, doi: 10.1080/10408347.2023.2227906.

[28] M.-M. Zhao, H.-Z. Wu, X.-K. Deng, R.-N. Yi, and Y. Yang, "The application progress of magnetic solid-phase extraction for heavy metal analysis in food: A mini review," *Anal. Methods*, vol. 16, no. 3, pp. 333–343, 2024, doi: 10.1039/D3AY01617J.

[29] H. Y. Chen and C. Chen, "Evaluation of calibration equations by using regression analysis: An example of chemical analysis," *Sensors*, vol. 22, no. 2, pp. 1–33, 2022, doi: 10.3390/s22020447.

[30] C.-H. Ko, A. B. Tadesse, and A. C. Kabiso, "Spectrochip-based calibration curve modeling (CCM) for rapid and accurate multiple analytes quantification in urinalysis," *Heliyon*, vol. 10, no. 18, pp. 1–21, Sep. 2024, doi: 10.1016/j.heliyon.2024.e37722.

[31] C. Yu *et al.*, "Batch sorption and fixed-bed elution for Pd recovery using stable amine-functionalized melamine sponge," *J. Clean. Prod.*, vol. 337, pp. 1–11, 2022, doi: 10.1016/j.jclepro.2022.130475.

[32] M. A. Akl, A. G. Mostafa, A. S. El-Zeny, and E.-S. R. H. El-Gharkawy, "Design, spectroscopic analysis, DFT calculations, adsorption evaluation, molecular docking, comprehensive in silico and in vitro bioactivity studies of thiocarbohydrazide grafted dialdehyde cellulose nanobiosorbent," *Sci. Rep.*, vol. 15, no. 1, pp. 1–28, Apr. 2025, doi:

10.1038/s41598-025-96525-2.

[33] J. H. Mugumya *et al.*, “Synthesis and theoretical modeling of suitable co-precipitation conditions for producing NMC111 cathode material for lithium-ion batteries,” *Energy and Fuels*, vol. 36, no. 19, pp. 12261–12270, 2022, doi: 10.1021/acs.energyfuels.2c01805.

[34] R. Zahedi and S. J. Mirmohammadi, “Sulfate removal from chemical industries’ wastewater using ettringite precipitation process with recovery of Al(OH)3,” *Appl. Water Sci.*, vol. 12, no. 9, pp. 1–10, 2022, doi: 10.1007/s13201-022-01748-7.

[35] J. Y. Lin *et al.*, “Controllable preparation and performance of bio-based poly(lactic acid-iminodiacetic acid) as sustained-release Pb2+ chelating agent,” *iScience*, vol. 24, no. 6, pp. 1–18, 2021, doi: 10.1016/j.isci.2021.102518.

[36] J.-H. Dong *et al.*, “Development of a portable method for serum lithium measurement based on low-cost miniaturized ultrasonic nebulization coupled with atmospheric-pressure air-sustained discharge,” *Anal. Chem.*, vol. 93, no. 39, pp. 13351–13359, Oct. 2021, doi: 10.1021/acs.analchem.1c03133.

[37] J. Sawicki *et al.*, “Design of experiment approach to optimize high resolution ICP-OES method for biomonitoring of Zn level in human blood samples,” *J. Elem.*, vol. 28, no. 4, pp. 1353–1367, 2023, doi: 10.5601/jelem.2023.28.3.3076.

[38] A. M. Elkhatat *et al.*, “Recent trends of copper detection in water samples,” *Bull. Natl. Res. Cent.*, vol. 45, no. 1, pp. 1–18, 2021, doi: 10.1186/s42269-021-00677-w.

[39] Z. U. Zango *et al.*, “Assessment of soil microplastics: An overview on toxicity, effects on heavy metals adsorption, solid-phase extraction, and detection techniques,” *Sustain. Environ.*, vol. 11, no. 1, pp. 1–41, 2025, doi: 10.1080/27658511.2025.2601424.

[40] T. P. Pabst and P. J. Chirik, “A tutorial on selectivity determination in C(sp2)a ’H oxidative addition of arenes by transition metal complexes,” *Organometallics*, vol. 40, no. 7, pp. 813–831, 2021, doi: 10.1021/acs.organomet.1c00030.

[41] Z.-G. Yang *et al.*, “Recent progress in the synthesis of transition metal nitride catalysts and their applications in electrocatalysis,” *Nanoscale*, vol. 15, no. 28, pp. 11777–11800, 2023, doi: 10.1039/D3NR01607B.

[42] S. N. Zainurin *et al.*, “Advancements in monitoring water quality based on various sensing methods: A systematic review,” *Int. J. Environ. Res. Public Health*, vol. 19, no. 21, pp. 1–21, 2022, doi: 10.3390/ijerph192114080.

[43] S. Paramparambath, K. Oflaz, M. Geetha, M. El-Azazy, A. S. El-Shafie, and K. K. Sadasivuni, “A dual approach to detecting iron ions and analyzing water quality,” *Chem. Africa*, vol. 8, no. 3, pp. 1115–1126, 2025, doi: 10.1007/s42250-024-01177-w.

[44] A. Szarka, L. Vnuková, Z. Keršňáková, N. Viktoryová, and S. Hrouzková, “Contamination with pharmaceuticals in aquatic environment: Focus on analytical methodologies,” *Appl. Sci.*, vol. 14, no. 19, pp. 1–21, 2024, doi: 10.3390/app14198645.

[45] P. Inaudi, O. Abollino, P. Bottoni, R. A. Grifa, and S. Caroli, “The role of spectrochemical techniques for the detection, identification, and quantification of residues and metabolites of pharmaceuticals in the environment: a review of developments in ecopharmacovigilance over the past decade,” *Appl. Spectrosc. Rev.*, vol. 60, no. 9–10, pp. 1086–1155, Nov. 2025, doi: 10.1080/05704928.2025.2507212.