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Analysis of Ca^{2+} and Fe^{2+} Ions in Ngade Water and Tolire Lake Water Ternate, North Maluku

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ABSTRACT

This study aims to obtain optimal and valid conditions for the analysis of inorganic ions Ca^{2+} and heavy metal Fe2+ and the comparison between inorganic ions Ca^{2+} and heavy metal ions Fe^{2+} contained in the water of Lake Ngade and Lake Tolire, using ion chromatography and UV-Vis spectrophotometry. This type of research is experimental laboratory. Based on the results of the research, the concentrations obtained from Ca^{2+} ions and Ca^{2+} ions are for the highest concentration of Ca^{2+} ions in Lake Ngade water at 1.259 ppm, Big Tolire Lake water at 3.410 ppm and Small Tolire Lake water at 6.154 ppm. Meanwhile, the highest concentration of Fe^{2+} ions is in Lake Ngade water at 5.606 ppm, Big Tolire Lake water at 5.500 ppm, and Small Tolire Lake water at 5.379 ppm.

Keywords: Ca^{2+}, Fe^{2+}, Ion chromatography, Ngade lake, Tolire lake

ABSTRAK

Penelitian ini bertujuan untuk mendapatkan kondisi yang optimal dan valid untuk analisis ion anorganik Ca^{2+} dan logam berat Fe^{2+} dan perbandingan antara ion anorganik Ca^{2+} dan ion logam berat Fe^{2+} yang terkandung dalam air Danau Ngade dan Danau Tolire, menggunakan kromatografi ion dan spektrofotometri UV-Vis. Jenis penelitian ini eksperimental laboratorium. Berdasarkan hasil penelitian konsentrasi yang didapatkan dari ion Ca^{2+} dan ion Fe^{2+} yaitu untuk konsentrasi ion Ca^{2+} tertinggi berada pada air Danau Ngade sebesar 1.259 ppm, air Danau Tolire Besar 3.410 ppm dan air Danau Tolire Kecil 6.154 ppm. Sedangkan untuk konsentrasi ion Fe^{2+} tertinggi berada pada air Danau Ngade sebesar 5.606 ppm, air Danau Tolire Besar 5.500 ppm, dan air Danau Tolire Kecil sebesar 5.379 ppm.

Kata kunci: Ca^{2+}, Fe^{2+}, Kromatografi ion, Danau Ngade, Danau Tolire

INTRODUCTION

Ngade and Tolire lake (large and small) are located on Ternate Island, North Maluku Province. The two lakes are located at the foot of Mount Gamalama, the highest active volcano in North Maluku Province and very close to the center of Ternate City. The existence of Ngade Lake and Tolire Lake is multifunctional, namely ecological, economic, educational, socio-cultural and religious functions, such as recreation, research, fish farming and as a source of raw water. The utilization of Ngade's Lake clean water as a source of clean water has been carried out to meet the needs of the community's water but has not been utilized optimally due to the lack of further research that examines the quality and quantity of Ngade's Lake water to determine the feasibility of water as a source of raw water, besides that Ngade Lake does not has a river cycle

that drains its water into the sea, as well as the Big and Small Lakes of Tolire as far as observations have been made, the water has not been properly utilized as a source of raw water.

Ngade Lake and Tolire Lake often get buildup of chemical materials as a result of the eruption of Mount Gamalama which can cause the water to become cloudy and turn black or yellow which can disrupt the ecosystems of the two lakes. There is also plastic waste as well as household waste from the people living around Ngade and Tolire Lake who still throw garbage not far from the lake, which during the rainy season the waste will be carried by the water and into the lake. The eruption of Mount Gamalama and the people's bad habits in maintaining the aesthetics of the lake greatly affect the condition of the lake, there is concern that there will be dangerous heavy metal pollution in the Ngade and Tolire lakes. From a toxicological point of view, calsium (Na) alkali metal and heavy metal iron (Fe) are metals that are needed by living organisms, but these metals in excessive concentrations can cause poisoning effects. Iron metal (Fe) is a type of environmental pollutant usually found in waters, for the content of iron metal in waters can be found scientifically, usually in smaller amounts (Mu *et al*., 2017). However, there are factors that increase iron metal caused by community activities around the waters, for example mountain eruptions, garbage, domestic industries and others can increase concentrations that need to be avoided. Meanwhile, calcium (Ca) is very important to neutralize acidic compounds. This acid compound when the concentration is above the quality standard has a negative effect on plants. The structure of calcium is very important in agricultural soil, because there is a complex adsorption of Ca^{2+} ions in groundwater which must be quite high (Amin, 2016). The researchers have conducted research on liquid waste for the concentration of Fe^{2+} in human water and other living things under certain conditions it requires iron (Fe) as a nutrient but excessive levels need to be avoided (Gierl *et al*., 2022; Eisele & Gabby, 2014; Ondigo *et al*., 2013).

So far, several methods have been used to determine the concentration of Ca^{2+} and Fe^{2+} ions such as coagulation and adsorption in water but at a high cost and takes a long time (Ngteni *et al*., 2020; Mu *et al*., 2017; Tiwow *et al*., 2017; Zheng *et al*., 2020; Kyomugasho *et al*., 2017). Therefore the analysis of Ca^{2+} and Fe^{2+} and ions in Ngade and Tolire Lake uses the analytical method with a UV-Vis spectrophotometer and chromatography. Ion is simpler and doesn't cost a lot. For the chromatographic method, the main principle in this method is based on the interaction of positive and negative charges between specific molecules and the matrix inside the chromatographic column (Coskun, 2016; Acikara, 2013; Sulistyani *et al*., 2019). While UV-Vis spectrophotometry has the advantage of analyzing the amount of organic and inorganic substances which is carried out quickly and precisely. Apart from that, this UV-Vis spectrophotometer instrument also obtains very accurate results, where the numbers are read directly recorded by the detector and printed in the form of numbers or graphs that have been regressed (Rohman, *et al*., 2021).

With the above description, it is necessary to carry out research on the analysis of Ca^{2+} ions and Fe^{2+} ions in the water of Ngade and Tolire Lake on Ternate Island using ion chromatography (Ca^{2+}) and UV-Vis spectrophotometry (Fe^{2+}) to determine the concentration of Ca^{2+} ions and Fe^{2+} ions.

METHODOLOGY

The research method used is laboratory experimental research. In this research, will be conducted to obtain the analysis of inorganic ions Ca^{2+} and heavy metal Fe^{2+} in Lake Ngade and Lake Tolire by Ion Chromatography and UV-Vis Spectrophotometry. This research took place in Ngade Lake, South Ternate District and Tolire Lake, both large and small, in Ternate Island

District. Sample testing was carried out at the UPT Laboratory of Basic and Integrated Laboratories, Khairun University in June-August 2022.

1. Equipment Used

Compact 761 Metrohm Ion Chromatography (Switzerland) which is also equipped with a Metrosep C2-150 type cation separator column used for analysis, 50 ml beaker, 10 mL volumetric flask, 100 mL volumetric flask, watch glass, analytical balance, UV-Vis spectrophotometer, Spatula, sample bottle, funnel, dropper, 5 ml volume pipette and 10 ml volume pipette.

2. Materials Used

Cation resin, Ngade and Tolire Lake water, aquades, $FeCl_2.6H_2O$, $CaCl_2.2H_2O$, $Na_2S_2O_3$ (sodium thiosulfate), acetate buffer pH 4.5 (sodium acetate trihydrate and glacial acid), acetone and 1,10-phenanthroline.

3. Sample Preparation

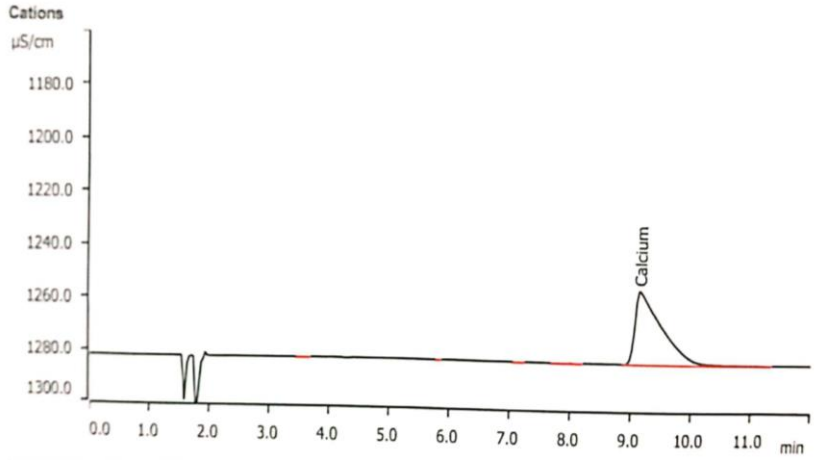
Sample preparation was carried out from various types of lake water samples to be collected from various locations. There are 3 types of lake water collected: Ngade lake water, large tolire lake and small tolire lake. The three types of lake water are taken from the city of Ternate, North Maluku. Sampling was done randomly. The purpose of this sampling is to collect the volume of a body of water to be studied with the smallest possible amount (Flanagan *et al*., 2021). The sample was taken using a glass bottle that had been rinsed with distilled water and dried. The samples that have been taken are then stored in a refrigerator at 4^{o}C to avoid contamination.

RESULTS AND DISCUSSION

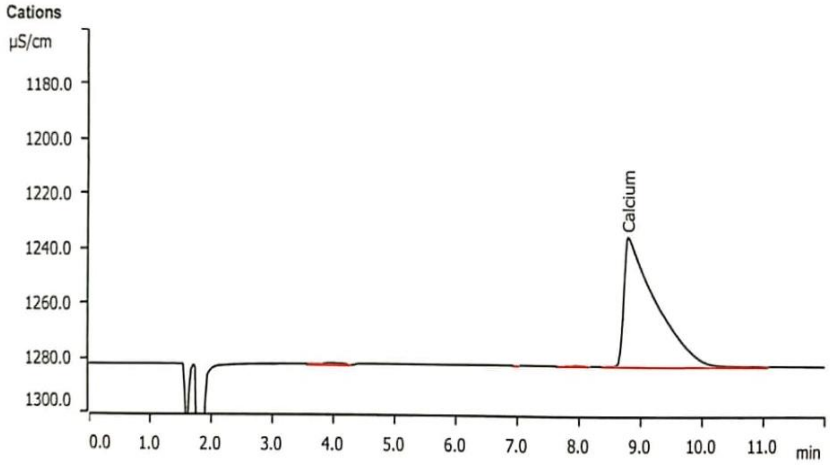
Based on the research that has been carried out to test the inorganic ions of calcium and iron heavy metal ions with samples of Lake Ngade Water, Big Tolire Lake and Small Tolire Lake on Ternate Island, the following results were obtained calcium (Ca^{2+}) inorganic ion test, metal ion test iron (Fe), inorganic ion Ca^{2+} test with ion chromatography and Fe^{2+} metal ion test with UV-Vis spectrophotometry.

Calcium (Ca^{2+}) Inorganic Ion Test

The results of the inorganic calcium ion test as shown in Figure 1, table 1 and table 2.



(a)



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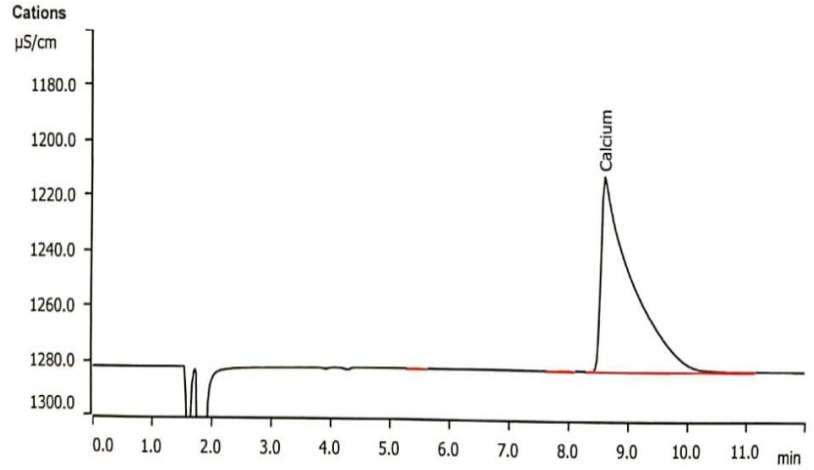
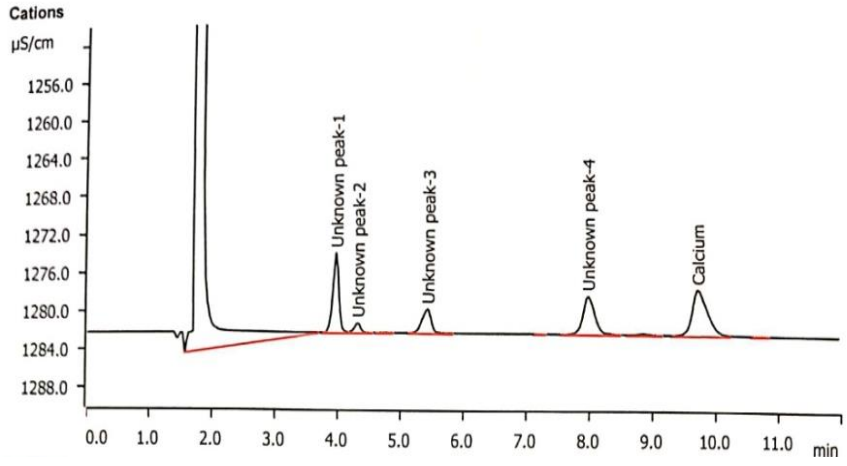


(c)

Figure 1. Standard solution chromatogram graph with: (a) concentration of 10 ppm, (b) concentration of 20 ppm, and (c) concentration of 30 ppm.



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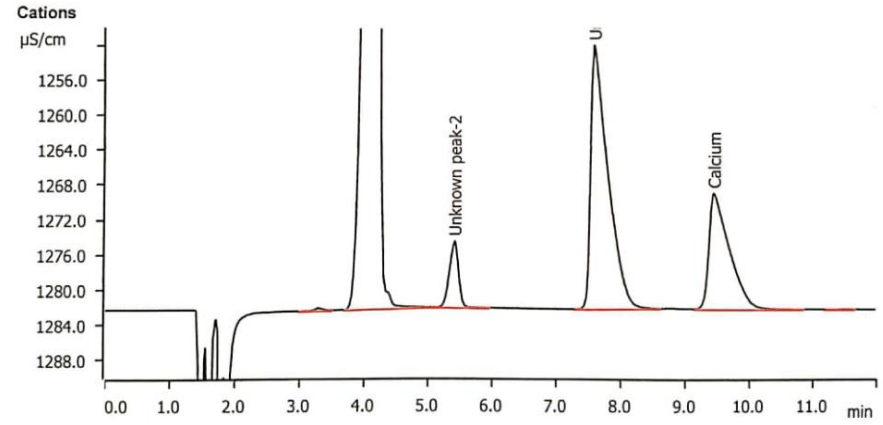

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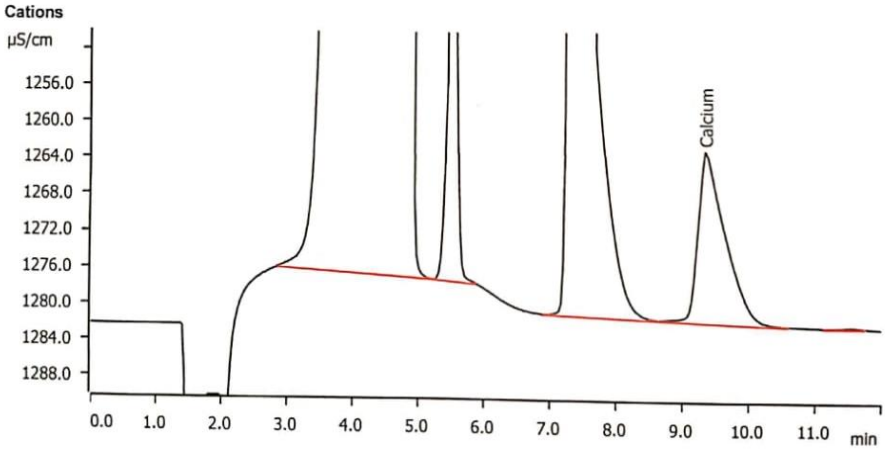


(c)

Figure 2. Sample chromatogram (a) lake ngade water, (b) large tolire lake water and (c) small tolire lake water

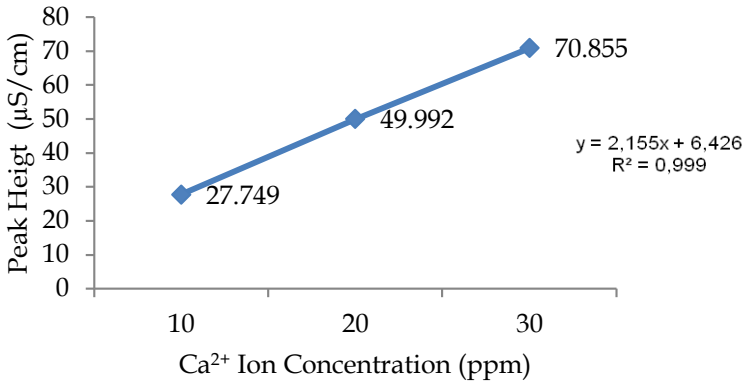
Table 1. Recapitulation of analysis results of Ca^{2+} ion standard

Ion	Standard Solution (ppm)	Retention time (min)	Signal (µS/cm)	
			Peak area	Peak height
Ca^{2+}	10	9.113	14.0004	27.749
	20	8.788	28.0517	46.992
	30	8.598	41.8789	70.855

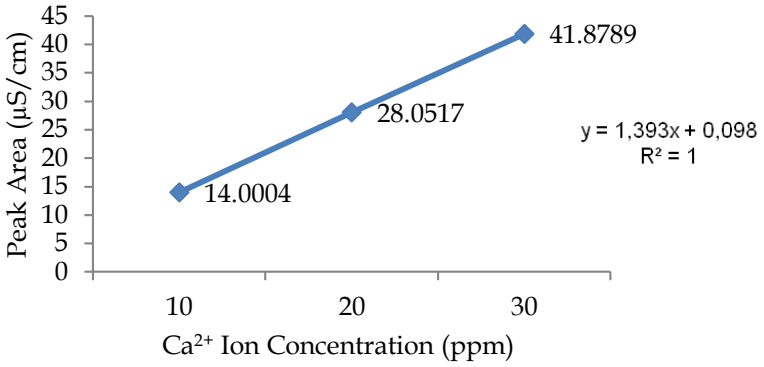
Table 2. Recapitulation of analysis results of Ca^{2+} ion concentration in samples

Sample	Signal (µS/cm)	
	Peak area	Peak height
Ngade Lake	1.3577	4.835
Big Tolire Lake	4.8489	13.239
Small Tolire Lake	8.6701	18.636

Below is a graphic image of the concentration and sample chromatograms and a table of analytical conditions in determining cations using ion chromatography techniques.



(a)



(b)

Figure 3. Standard calibration curve of Ca^{2+} ion sample:
(a) peak height and, (b) peak area

Based on Figure 3 calibration curve, the standard Ca^{2+} ion sample above has a value (R^2) = 0.999 for Peak Heigt, while for Peak Area the value (R^2) = 1. Therefore, the researchers took the value (R^2) of the peak area as the data to be used because when In research, researchers made a standard sample solution well and did not experience errors so that the results were good (Tran et al., 2017) and below are the results of the calculation of the peak heagt and peak area values for each sample (Lake Ngade, Big Tolire Lake and Small Tolire Lake) show in table 3.

Table 3. Result of recapitulation of Ca^{2+} ion concentration in sample based on peak height and peak area data

Sample	Ca^{2+} Ion Concentration (ppm)
1.259	1.259
3.410	3.410
6.154	6.154

Table 4. Analytical conditions in the determination of cations using ion chromatography techniques.

Parameter	Analysis conditions
Eluent composition	6MmHNO$_3$
Separation column	Metrocep C4-150
Detector type	conductivity
Eluent water velocity	0.800 Ml/min
Injector volume	10 μL

Metal ion test Iron (Fe)

Calculation of the concentration of iron ion (Fe) 100 ppm using the graph of the results of UV-Vis spectrophotometry. The results of the 100 ppm Iron ion test using a standard solution can be seen in table 5.

Table 5. Absorbance value of standard solution

Concentration (ppm)	Absorbance (nm)
1	0.905
3	1.225
5	1.438
7	1.714

From table 5 above, the Fe^{2+} calibration curve is obtained as shown in Figure 4.

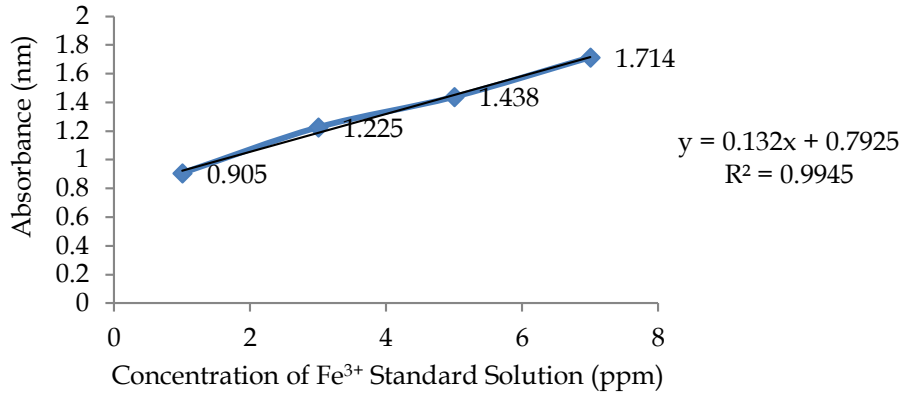


Figure 4. Fe Calibration Curve Fe^{2+}

From the results of Figure 4 obtained the equation:

$$y = 0.132x + 0.792$$

The following are the measurement results for each sample (Ngade Lake, Bigi Tolire Lake and Small Tolire Lake) shown on table 6.

Table 6. Concentration of Fe^{2+} ions in each sample (lake ngade, big tolire lake and small tolire lake)

Sample	Absorbance (nm)	Fe^{2+} Ion Concentration (ppm)
Ngade Lake Water	0.052	5.606
Big Tolire Lake Water	0.066	5.500
Small Tolire Lake Water	0.082	5.379

Sample preparation was carried out from various types of lake water samples to be collected from various locations. There are 3 types of lake water collected: Ngade lake, big tolire lake and small tolire lake. The three types of lake water are taken from the city of Ternate, North Maluku. Sampling was done randomly. The purpose of this sampling is to collect the volume of a body of water to be studied with the smallest possible amount (Ma *et al.*, 2022). The sample was taken using a glass bottle that had been rinsed with distilled water and dried. The samples that have been taken are then stored in a refrigerator at 4^{o}C to avoid contamination.

Inorganic Ion Ca^{2+} Test With Ion Chromatography

To test the inorganic calcium ion, start by weighing 0.3675 grams of $CaCl_2.2H_2O$ crystals using an analytical balance. The crystals were put into a 100 mL volumetric flask, distilled water was added to the mark and homogenized. The solvent used is water solvent (aquades) because it is easy to obtain and this solvent is easily soluble in ionic compounds (Fatimah *et al.*, 2022), (Setyawati *et al.*, 2020) and (Sonia *et al.*, 2022)**.** Next, the stock solution was taken and then made some standard solutions such as 10, 20 and 30 ppm and put them in a 100 mL volumetric flask. The standard solution is taken and then put into the ion chromatography system. The concentration of the three cations can be seen in Figure 5.

Figure 5. Fe standard solution

Based on Figure 5 quantitative analysis of the three standard solutions based on different plots of calibration curve methods. Further treatment for the sample, before the sample is injected into the ion chromatography system, the sample is allowed to reach room temperature.

Figure 6. Samples (a) lake ngade water (b) big tolire lake water and,
(c) small tolire lake water

The sample is taken and then injected into an empty bottle, after which a hose is inserted so that it can mix with the eluent and go together to the separator column. The cations are then measured by a conductivity detector and the results of the sample analysis can be seen in Figure below.

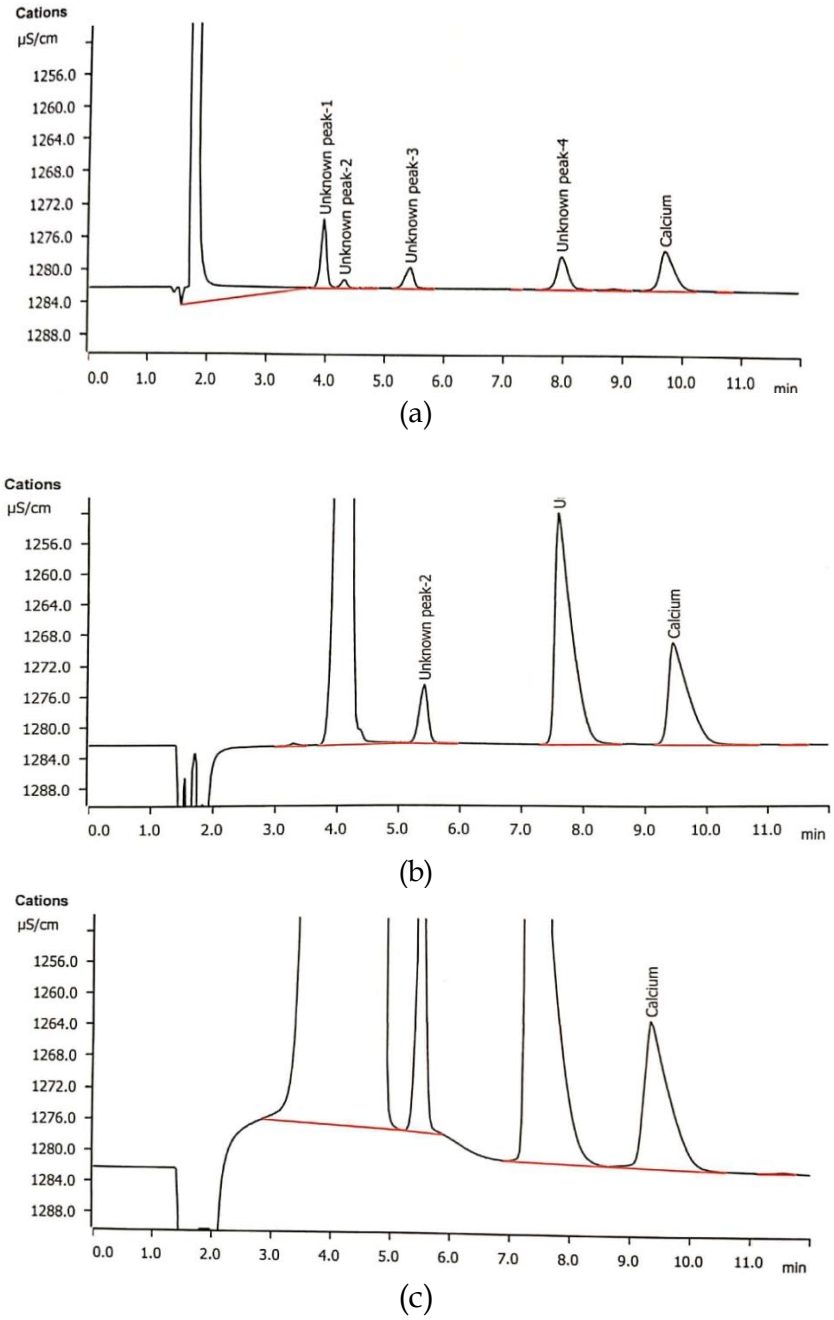


Figure 7. Sample chromatogram (a) lake ngade water, (b) big tolire lake water, and (c) small tolire lake water

Based on Figure 7, it is found that the types of samples that have high Ca^{2+} cations are the water of Small Tolire Lake, Big Tolire Lake and Lake Ngade. The results obtained from the analysis using ion chromatography for water samples were 18.6 s/cm (Small Tolire Lake), 13.2 s/cm (Big

Tolire Lake), and 4.8 s/cm (Lake Ngade). As for the results of the sample chromatogram, namely y = 6.900x - 1.564 and R^2 = 0.984. Ca^{2+} ions were found in the Ngade lake samples because they contain many types of pollution such as domestic, agricultural and industrial wastes around the waters. As it is known that North Maluku is one of the provinces in Indonesia whose pollution levels are increasing because many people who live around the lake area build restaurant and residential industries (Amin, 2020).

Table 7. Results of recapitulation of Ca^{2+} ion concentration in samples based on peak height and peak area data

Sample	Ca^{2+} Ion Concentration (ppm)
Ngade Lake	1.259
Big Tolire Lake	3.410
Small Tolire Lake	6.154

Based on table 7 the concentration of Ca^{2+} ions in the sample has different concentration results from each sample, namely for Lake Ngade water the concentration value is 1.259 ppm, Big Tolire Lake water is 3,410 ppm and Small Tolire Lake is 6.154 ppm. There is a study conducted by (Pujiastuti, C. et al. 2019) that the more cation resin added Ca^{2+} ions which are absorbed into the resin the greater, the more resin, the ability of the resin to absorb ions increases. The longer the contact time the Ca^{2+} ion absorbed is greater and exceeds the other ions. This is in accordance with the concept of ion selectivity, namely in ions that have the same valence ion, the greater the atomic weight of the ion, the higher will be absorbed into the resin (Paper, 2019).

Fe^{2+} Metal Ion Test with UV-Vis Spectrophotometry

The Fe^{2+} metal ion test was started by weighing 0.483 grams of $FeCl_3.6H_2O$ crystals, 0.10 grams of 1,10-phenanthroline, 0.0235 grams of $Na_2S_2O_3$ using a watch glass. Each of these crystals was put into a 100 mL and 50 mL volumetric flask, added with distilled water up to the mark and homogenized. As for the acetate buffer crystal or 3.97 grams of sodium acetate trihydrate, put into a 50 mL volumetric flask, add 5 mL of glacial acetic acid and distilled water to the mark.

Next, a standard solution of 100 ppm was taken by making several concentrations such as 1, 3, 5, and 7 ppm. The standard solutions used in this study were primary and secondary standard solutions. Primary standard solutions are standard solutions made from standard substances with very high purity which are generally supplied by NIST, NIBSC which is used for calibration of standard solutions made, while secondary calibration is a solution whose concentration is determined by reliable analytical methods (Visschedijk et al., 2005). Then added $Na_2S_2O_3$, 1,10-phenanthroline reagent, acetate buffer, acetone and aquade. The existence of this reagent is in order to determine the solution used as an ingredient for a reaction to take place (Pretsch et al., 2020). For a blank solution, it is made by adding reagents and aquades to the limit mark, in the presence of a blank solution, the absorbance can be zeroed on the UV-Vis spectrophotometry tool.

For testing lake water samples, it is done by taking samples and putting them into a 10 mL volumetric flask and then adding the reagent to each volumetric flask containing the sample. Then the standard solution and sample were measured using UV-Vis spectrophotometry with a wavelength of 509 nm. The test results of each concentration can be seen in the figure 8.

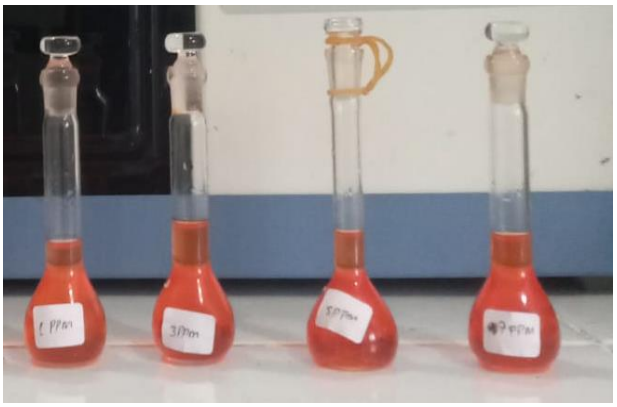


Figure 8. Test results of standard solutions with concentrations of 1, 3, 5, and 7 ppm

From Figure 8, the results experience a color change from faded pink to dark pink which is different for each concentration because the higher the concentration, the more colorful or concentrated the solution is as stated (Maskan, 2006; Wang *et al*., 2021; Abdel-lateef *et al*., 2022). For the measurement of iron in water can be done using spectrophotometry to determine the absorbance value. The color change was due to the addition of phenanthroline reagent which made the standard test solution red orange or dark pink according to research (Sriram *et al*., 2017; Siddiqui *et al*., 2022).

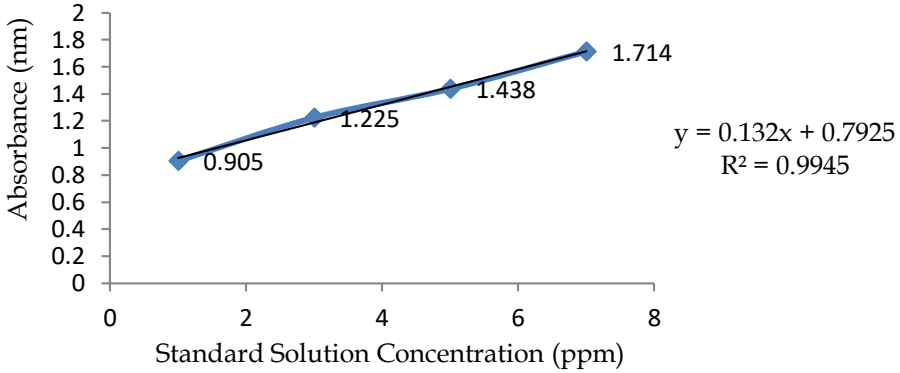


Figure 9. Calibration Curve Graph

Based on Figure 9 can show that the test results have a fairly good linearity. This is shown by the coefficient value (R^2) of 0.994. In addition, there are researchers (Onyelowe *et al*., 2022) who conducted the same study having a coefficient value (R^2) of 0.9606 for Fe metal. In other words, the greater the metal content, the greater the light absorbance value (Rahmani *et al*., 2018).

Furthermore, testing samples that only add a reagent solution, so you can see the test results as shown on figure 10 and table 8.

Figure 10. Sample test

From Figure 10, it can be seen that the color became cloudy for each sample the same due to the addition of reagents. Below is a table to determine the Fe content in the sample.

Table 8. Concentration of Fe ions in each sample (lake ngade water, big tolire lake water and small tolire lake water)

Sample	Absorbance	Fe^{2+} Ion Concentration (ppm)
Ngade Lake Water	0.052	5.606
Big Tolire Lake Water	0.066	5.500
Small Tolire Lake Water	0.082	5.379

Based on table 8. above, it can be seen that the water containing more Fe is Small Tolire Lake, followed by Big Tolire Lake and the last one is Lake Ngade. Meanwhile, if calculated using the formula y = ax + b, then the largest value is Lake Ngade, then followed by Big Tolire Lake and the last is Small Tolire Lake.

CONCLUSION

Based on the results of research and discussion obtained from inorganic ions Ca^{2+} and heavy metal ions Fe^{2+}, it can be concluded that: Analysis of Ca^{2+} ions has 2 data obtained using ion chromatography, the data in question are peak height and peak area. The data (peak height) obtained results from the three water samples, namely Lake Ngade water at 4.835 S/cm, Big Tolire Lake water water 13.239 S/cm, and Small Tolire Lake water water 18.636 S/cm. As for the data (peak area) the results from the three water samples are Lake Ngade water of 14.0004 S/cm, Big Tolire Lake water water is 28.0517 S/cm, and Small Tolire Lake water is 8.6701 S/cm. Then analysis of Fe^{2+} absorbance values obtained through measurements using UV-Vis spectrophotometry from the three water samples, namely Lake Ngade water 0.052 nm, Big Tolire Lake water 0.066 nm and Small Tolire Lake water 0.082 nm. Furthermore, the results of the standard calibration curve of the sample obtained through data on the concentration of Ca^{2+} peak area in each sample of Lake Ngade water is 1.259 ppm, Big Tolire Lake water is 3.410 ppm and Small Tolire Lake water is 6.154 ppm. calculation of the concentration, the results of the three water samples are for Lake Ngade water of 5.606 ppm, Big Tolire Lake water for 5.500 ppm, and Small Tolire Lake water for water for 5.379 ppm. From the results obtained above, this indicates that the method used is correct, because the results obtained are close to the theoretical results and also the values obtained from the analysis of inorganic ions Ca^{2+} and heavy metal ions Fe^{2+} some are larger and some are small depending on the measurement and calculation.

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