An Investigation into the Optimal Crystallisation Condition for Synthesis and Metal Ions Adsorption Capacity of Zeolite A

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Abstract

In light of the recent lead water incident in Hong Kong, our team is inspired to delve into the research of water treatment methods, which led us to the family of chemicals named zeolites. We decided to focus on Linde Type A Zeolite owing to its relative synthesis simplicity, and versatile applications.

This paper investigates optimal conditions for Zeolite A synthesis, in the scope of the effect of crystallisation temperature and duration on cation adsorption efficiency of Zeolite A by titrating 25 cm$^3$ Ca(NO$_3$)$_2$(aq) after 0.250g zeolite introduction with Na$_2$EDTA(aq). Same applies for Pb(NO$_3$)$_2$(aq) and various water samples. For 25 cm$^3$ Fe$_2$(SO$_4$_3)(aq) and CuSO$_4$(aq) solutions, 40 cm$^3$ 0.1M NaOH(aq) was used in gravimetric analysis of precipitate and back titration of solution against 0.1 M HCl(aq). These results were then compared to trials without zeolite introduction.

We found the optimum crystallisation temperature and duration to be 115 °C and 300 minutes respectively, which has a Ca$^{2+}$ adsorption efficiency of 58.09% (±2%) and 59.72% (±2%) respectively. We also found that Zeolite A is effective in adsorbing Fe$^{3+}$, Ca$^{2+}$, Pb$^{2+}$ but not Cu$^{2+}$. Lastly, the applicability of Zeolite A in water treatment is evaluated.
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Introduction

Background

In 2015, a widespread panic arose in Hong Kong revolving lead-tainted water in household water, which may lead to adverse health effects in consumers of said water, especially children. In light of the widespread concern, we decided to look into different methods of decontaminating lead water; during this, we came across a type of chemical named zeolite which is used industrially for a multitude of purposes including removal of metal ions in aqueous media. This sparked our interest towards this particular mineral. Therefore, in our investigation we have set out to synthesise our own zeolite, and investigate its ability in removing metal ions from water, which we hope will help us learn more on how the chemical can be applied to solve similar issues in the future.

Chemicals

Zeolite A

Zeolites are aluminosilicate minerals commonly used as molecular sieves, commercial adsorbents and catalysts. Formed from interlinked SiO$_4$ and AlO$_4$ tetrahedra, zeolite frameworks are microporous, having very small pores dimensions of under 15 angstroms (Å) which are comparable to small molecules such as H$_2$O, CO$_2$ and NO$_2$. Different types of zeolites are created when the ratio of SiO$_4$ and AlO$_4$ and/or the framework structure is changed.

The ion exchange capacity of zeolites stems from its structure. The above diagram shows a SiO$_4$ tetrahedral. The 4- charge on the tetrahedral is due to a 1- charge on each of the O atoms. When all SiO$_4$ tetrahedra are bonded to each other as is the case with silicon dioxide, the overall structure is not electrically charged. However, since there is an extra 1- charge in AlO$_4$ tetrahedra due to Al$^{3+}$ having one less positive charge than Si$^{4+}$, when the tetrahedra bond to each other, the 1- charge from O atoms is neutralised while the 1- charge from the Al atom is not accounted for. Hence, the framework must include one 1+ charge balancing cation in the framework pores, for example Na$^+$, for every AlO$_4$ tetrahedra in the framework. While Na$^+$ is a common charge balancing cation, it is not the only possible cation species. Some zeolites can hold other cations such as K$^+$ and Ca$^{2+}$ or even NH$_4^+$ as can be the case in clinoptilolite. Provided the cation size and charge is suitable and there is no net change in framework charge, those framework cations can be exchanged with those existing in the solution to be processed. Due to its size selectivity and highly uniform pore structure, zeolites are commonly used to sort molecules based on their sizes.

Metals other than silicon and aluminium can be introduced during synthesis for specific purposes as well. For example, the ZSM-5 type zeolite can be synthesised with boron, iron or titanium as framework materials (these are named [B]-ZSM-5, [Fe]-ZSM-5 and [Ti]-ZSM-5 respectively). This shows the impressive flexibility of zeolites that can be synthesised with different purposes in mind, from water purification to being a vital material in the petrochemical industry. (IZA Synthesis Commission)
Zeolites can be found naturally in small amounts, formed when volcanic rocks and ash layers react with alkaline groundwater, such as the zeolite type known as clinoptilolite, commonly found around the shores of the Aegean Sea. Zeolites are also industrially synthesised through crystallising silica-alumina gels slowly in the presence of alkalis and organic templates, for example the aforementioned Faujasite (Zeolite Y).

Chemical Structure

The type of zeolite that we will be synthesizing is Zeolite A with its framework named Linde Type A (LTA) by the International Zeolite Association. It has the chemical formula $\text{Na}_{12}(\text{AlO}_2)_{12}(\text{SiO}_2)_{12} \cdot 27\text{H}_2\text{O}$, which gives it a molar mass of 2191.26 g mol$^{-1}$.

Graphical representation of Zeolite A (Takehito, Yasuo)

While the Al: Si ratio can be adjusted relatively flexibly with the use of different reaction stoichiometries, or even with post-synthesis modification, the ratio is ultimately limited by Lowenstein’s Rule which states that whenever “two tetrahedra are linked by one oxygen bridge, the centre of only one of them can be occupied by aluminium; the other centre must be occupied by silicon or by another small ion of electrovalence 4 or more”. This rule is based on the fact that Al-O bonds (≈1.8 Å) are longer than Si-O bonds (≈1.6 Å); therefore, it is more energetically unfavourable to form an Al-O-Al bond. Consequently, zeolites cannot have an Al: Si ratio of more than 1:1. (Szostak)

Current Applications

Zeolites are widely used as ion-exchange beds in domestic and commercial water purification and/or softening, and other applications. Commercially, Zeolite A is generally used in household detergents to reduce the water hardness by exchanging the Ca-ions for Na-ions, though we shall observe whether its application can expand beyond Ca-ions. In chemistry, zeolites are used to isolate molecules or ions as zeolite frameworks are only porous to chemical species of certain sizes and shapes. Zeolites are also widely used as catalysts and sorbents; for example, Zeolite Y is commonly used in petroleum refineries for converting high boiling point, high molecular weight hydrocarbons...
into more commercially valuable gasoline and other products through Fluid Catalytic Cracking (FCC), while studies have shown zeolites to be a promising sorbent of mercury in industrial pollution. Zeolites' regular pore structure and adjustable acidity make them highly active in a large variety of reactions.

**Selection for Further Investigation**

We have decided to base our report on this specific type of Zeolite A as we believe it strikes the best balance between ease of synthesis, safety and range of applications. While many other types of zeolite exist, those often require molecules termed “organic templates” in their synthesis. For example, in the synthesis of high alumina ZSM-5 zeolite, a tetrapropylammonium hydroxide solution is required. These quaternary ammonium cations have considerably adverse health effects and are therefore difficult to handle in high school labs. Other zeolites require extended synthesis durations and advanced machinery - for example, Faujasite zeolites require centrifuges in its product recovery which is rarely if ever installed in high school laboratories. In addition, the synthesis of zeolites, again such as Faujasite, often requires extended incubation periods. The applicability of the type of zeolite is also a major concern, as a zeolite synthesised specifically as mercury sorbent for example will have very limited applications that we in a high school laboratory can test. After considering all our options, we decided on the synthesis of Zeolite A with LTA structure.

**EDTA**

EDTA, full name Ethylenediaminetetraacetic acid, is a type of hexadentate ligand and chelating agent. This means that EDTA is able to form a bond with a single central metal ion, allowing EDTA to remove metal ions from a solution. This property is instrumental in complexometric titration, which is used in the investigation (Sinex, S. A.). A typical divalent metal ion reacts with Na₂EDTA as detailed in the following equation:

\[ M^{2+} + Na₂EDTA \rightarrow MEDTA + 2Na \]

Since the zeolite A we are synthesising has Na⁺ as its exchangeable ions, we have chosen disodium EDTA in particular to account for that – any increase in [Na⁺] due to ion exchange will not affect titre volume.

**Eriochrome Black T**

Eriochrome Black T is a type of indicator that used in complexometric titration. It forms a complex with metal ions to give a pink colour; if it is not complexed with any metal ions, the colour is blue. It works by the following equation: (M refers to any metal, x refers to any charge) (Eriochrome Black-T)

\[ In-M \rightarrow In^{2+}M^{x+} \]

(Pink) (Blue)

Eriochrome Black T only works between pH 7 - 11. Since EDTA is slightly acidic, it is common practice to add an Ammonia Solution as buffer to allow for the indicator to work. Moreover, Eriochrome Black
T solutions are usually unstable, and may lose colour due to air oxidation. Hence it is mixed with NaCl to provide stability as an indicator. (Eriochrome Black-T)

In combination with EDTA, a complexometric titration with Eriochrome Black T could be used to find out the amount of metal ion in a solution. (Eriochrome Black-T)

12 Green Chemistry Principles

Green Chemistry, also known as Sustainable Chemistry, is a philosophical ideal designed to minimise, and even eliminate, environmental degradation caused by generation of hazardous substances. In order to determine the optimal synthesis conditions for Zeolite A, the 12 Green Chemistry Principles, proposed by Anastas and Warner (1998), are taken into consideration. In particular, 5 principles out of the 12 are identified to be closely related to this research. The 5 principles are explained as follows (EPA, OCSPP et al.):

1. Prevent waste: Design chemical syntheses to prevent waste.

4. Design safer chemicals and products: Design chemical products that are fully effective yet have little or no toxicity.

6. Increase energy efficiency: Design chemical syntheses using the least amount of energy.

12. Minimise the potential for accidents: Design chemicals and their physical forms (solid, liquid, or gas) to minimise the potential for chemical accidents including explosions, fires, and releases to the environment.

Principle 1 forms the basis of this research. This research aims to find the most efficient method and condition for Zeolite A synthesis to produce the most amount. It is hoped that by finding the optimal synthesis condition, the adsorption efficiency of Zeolite produced can be maximised and reduce the waste of chemicals used in synthesis. In light of the possible applications of Zeolite aforementioned, this research also satisfies Green Chemistry Principle 4 as we investigate into the possibility of using non-toxic Zeolite to produce clean water through purification. Principle 6 will be addressed in our Oven Temperature and Duration trials as we aim to identify the optimal conditions for Zeolite synthesis, with the aim to minimise the time and energy used during synthesis, thus achieving the highest energy efficiency. Also, by doing so, we are addressing Principle 12, as the duration of synthesis is minimised for better efficiency, the potential risks in heating and synthesis will also be minimised.
Hypothoses
In response to the three aforementioned objectives, three hypotheses have been drafted as follows:

H₁: The optimal temperature of crystallisation for zeolite synthesis is 99.25°C
H₂: The optimal time of crystallisation for zeolite synthesis is between 180-240 minutes (3-4 hours)
H₃: There is a negative correlation between charge density of metal and zeolite adsorption efficiency, the smaller the radius the more efficient.

H₁ and H₂ are justified in the sense that they are common practice among the general scientific community (see synthesis conditions in comparative literature), while H₃ is justified based on our knowledge of atomic structure (see atomic structure on metal ion effects in further research).

Variables

H₁: The optimal temperature of crystallisation for zeolite synthesis is 99.25°C.

Independent Variable:
1. Crystallisation Temperature
   The synthesis of zeolite depends on the Crystallisation Temperature.

It was measured through setting the temperature of the oven at 70.0°C, 80.0°C, 90.0°C, 100.0°C, 115.0°C, 130.0°C respectively.

Constant Variable:
1. Crystallisation Duration
   The synthesis of zeolite depends on the crystallisation duration as well. The crystallisation duration is set at 3 hours.

2. Charge Density
   The adsorption of metal ions by zeolite depends on the charge density of the type of metal ion. The charge density is fixed by using Calcium Nitrate (with Ca²⁺ ion).

H₂: The optimal time of crystallisation for zeolite synthesis is between 180-240 minutes (3-4 hours)

Independent Variable:
1. Crystallisation Duration
   The synthesis of zeolite depends on the crystallisation duration. The crystallisation duration is set at increments of 60 minutes (or 1 hour), 180 minutes (or 3 hours), 300 minutes (or 5 hours), 420 minutes (or 7 hours) and 540 (or 9 hours).

Constant Variable:
1. Crystallisation Temperature
   The synthesis of zeolite depends on the Crystallisation Temperature. The temperature is set at 90.0°C Celsius.
2. Charge density
The adsorption of metal ions by zeolite depends on the charge density of the type of metal ion. The charge density is fixed by using Calcium Nitrate (with Ca\(^{2+}\) ion).

**H\(_0\): There is a negative correlation between charge density of metal and zeolite adsorption efficiency, the smaller the radius the more efficient.**

*Independent Variable:*

1. Charge density:
The adsorption of metal ions by zeolite depends on the charge density of the type of metal ion. The charge density is set by using calcium nitrate (with Ca\(^{2+}\) ion), lead(II) nitrate, copper(II) sulphate and iron(III) sulphate with their respective charge densities.

*Constant Variable:*

1. Crystallisation Temperature
The synthesis of zeolite depends on the Crystallisation Temperature. The temperature is set at 90° Celsius.

2. Crystallisation Duration
The synthesis of zeolite depends on the crystallisation duration as well. The crystallisation duration is set at 3 hours.

*Dependent Variable for all trials:*

1. Adsorption effectiveness (of Ca\(^{2+}\) ions)
The adsorption effectiveness of the Calcium ion is determined through the amount of EDTA used in a complexometric titration, the lower the EDTA titre volume, the more the amount of Calcium ion adsorbed by Zeolite. The concentration of the initial and final concentration can be calculated by the EDTA titre volume.

The adsorption efficiency is calculated with the following equation:

\[
\frac{\text{final conc.} - \text{initial conc.}}{\text{initial conc.}} \ast 100\%
\]

*Constant Variable for all trials:*

1. Contact time between zeolite and metal ions.
When zeolite is in contact with metal ions, it undergoes adsorption. In order to prevent difference in adsorption between trials, the contact time between zeolite and metal ions is fixed at 15 minutes.

2. Stirring rate
The stirring rate affects the adsorption of metal ions because it affects the collision between zeolite and metal ions. Hence the stirring rate is set constant at 1000 rpm.

3. Amount of zeolite added
The amount of zeolite affects the adsorption of metal ions hence it is kept constant at 0.250 g.

4. Amount of metal salt solution
The amount of metal ion affects the calculation of adsorption efficiency. Hence, the volume and concentration is set at 25.00 cm\(^3\) and 0.01M for all metal salt solutions.
Zeolite A Synthesis

*Synthesis procedures from (Methodology Titration of Zinc with EDTA)*

1. **Apparatus**
   - 250 cm$^3$ glass beaker
   - 50 cm$^3$ measuring cylinder (±0.5 cm$^3$)
   - 150 cm$^3$ Polypropylene bottle x3, with cap
   - Large watch glass x3
   - Oven x1 (±0.1 °C)
   - Electronic balance (±0.001 g)
   - Buchner funnel
   - 250 cm$^3$ conical flask

2. **Chemicals**
   - Deionised water
   - Sodium hydroxide NaOH
   - Sodium aluminate NaO$_2$: Al$_2$O$_3$: 3H$_2$O
   - Sodium metasilicate Na$_2$SiO$_3$: 5H$_2$O

3. **Procedures**
   a. **Preparation**
      1. In a 250 cm$^3$ glass beaker, mix 80 cm$^3$ water and 0.723g sodium hydroxide until completely dissolved.
      2. Divide into two equal volumes (i.e. 40 cm$^3$ each) in two polypropylene bottles (bottles A and B)
      3. In bottle A, add 8.258g sodium aluminate, cap bottle and mix until completely dissolved.
      4. In bottle B, add 15.480g sodium metasilicate, cap bottle and mix until completely dissolved.
      5. Pour B into A (silicate into aluminate) quickly.
      6. Cap and mix until homogenised.
   b. **Crystallisation**
      1. Vessel: bottle B is used.
      2. Temperature: 99.0 °C
      3. Time: 3 hours
      4. Agitation: no agitation required
   c. **Recovery**
      1. Take bottle from oven and leave to cool to room temperature (below 30 °C)
2. Set up a vacuum filtration with the Buchner funnel and 250 cm$^3$ conical flask. (Refer to Image 8 in Appendix)
3. Pour bottle contents into Buchner funnel for product recovery.
4. Transfer recovered solids from the filter paper to a watch glass.
5. Dry product watch glass in oven at 90.0 °C for 4 hours.

4. Notes
To save time, a large reserve solution of sodium metasilicate was prepared with a 500 cm$^3$ polypropylene bottle first with 400 cm$^3$ deionised water and 4.338 g sodium hydroxide, then with 185.76 g sodium metasilicate added. The bottle is then capped and shaken manually until completely dissolved. For each synthesis batch, 40 cm$^3$ of this solution is added into the sodium aluminate solution in its bottle, effectively covering steps 4 and 5 of part (a).

Sodium aluminate solution was not treated in this way as it was discovered that the solution forms significant pieces of white insoluble solids as time passes, hence not allowing any attempt at creating such a reserve solution: first-hand experience indicates this sodium aluminate solution must be prepared fresh.

Investigating Ion Exchange Capacity of Zeolite A with Different Metal Salt Solutions

Part I: Preparation of Required Chemicals
1. 0.01M metal salt solution
   a. From 0.1M metal salt solution (dilution)
      i. Chemicals: 0.1M metal salt solution, deionised water
      ii. Apparatus:
         1. 10.00 (±0.01) cm$^3$ micropipette
         2. 250 (±0.15) cm$^3$ volumetric flask
      iii. Procedures:
         1. Measure 25.00 cm$^3$ of 0.1M metal salt solution with the micropipette into the volumetric flask.
         2. Add deionised water into the flask until the solution level reaches the graduation line.
         3. Swirl thoroughly.
   b. From metal salt solid
      i. Chemicals: X g metal salt solution, deionised water
      ii. Apparatus:
         1. Electronic balance (±0.001 g)
         2. Spatula
         3. 250 (±0.15) cm$^3$ volumetric flask
         4. 100 cm$^3$ beaker
      iii. Procedures:
         1. Calculate value of X according to this equation:
         4. $X = 0.0025 \times \text{(formula mass of salt)}$
         1. Measure X g of salt on the electronic balance.
2. Fill two thirds of the 100 cm$^3$ beaker with deionised water. Dissolve the solid salt into beaker and stir until completely dissolved.
3. Pour this solution into the 250 cm$^3$ volumetric flask. Rinse the beaker with deionised water; pour these volumes of water into the flask. Rinse until solution level reaches the neck of the flask.
4. Carefully add more deionised water into the flask until the solution level reaches the graduation line.
5. Swirl thoroughly.

2. Disodium EDTA solution
   a. Preparation
      i. Chemicals: 33.822 g Na$_2$EDTA(s), deionised water
      ii. Apparatus:
         1. Electronic balance (±0.001 g)
         2. Spatula
         3. Weighing boat
         4. 1000 (±0.40) cm$^3$ volumetric flask
         5. 600 cm$^3$ beaker
      iii. Procedures:
         1. Measure 33.822 g of Na$_2$EDTA(s) on the electronic balance.
         2. Fill two thirds of the 600 cm$^3$ beaker with deionised water. Dissolve the solid salt into beaker and stir until completely dissolved.
         3. Pour this solution into the 1000 cm$^3$ volumetric flask. Rinse the beaker with deionised water; pour these volumes of water into the flask. Rinse until solution level reaches the neck of the flask.

3. NH$_3$/NH$_4$Cl buffer solution (pH = 10)
   a. Chemicals: 0.84M ammonia solution, NH$_4$Cl(s), deionised water
   b. Apparatus:
      i. Watch glass
      ii. Electronic balance (±0.001 g)
      iii. 50.0 (±0.5) cm$^3$ measuring cylinder x2
      iv. 250 cm$^3$ beaker
      v. Sealable bottle
   c. Procedures:
      i. Measure 50.0 cm$^3$ deionised water with a measuring cylinder and pour into 250 mL beaker
      ii. Weigh 0.400 g NH$_4$Cl(s), put into beaker and stir until dissolved
      iii. Measure 50.0 cm$^3$ 0.84M ammonia solution with the other measuring cylinder. Should 1M ammonia solution be used, measure 42.0 cm$^3$ of this solution and add deionised water up to 50.0 cm$^3$.
      iv. Pour beaker and cylinder contents into sealable bottle. Mix thoroughly.

4. Eriochrome black T indicator
   a. Dry powder form:
      i. Chemicals: 0.100g Eriochrome black T (EBT) powder, 10.000g NaCl(s)
      ii. Apparatus:
         1. Mortar and pestle
2. Electronic balance (±0.001 g)
3. Watch glass x2
4. Sealable bottle

iii. Procedures:
1. Grind and mix 0.100 g EBT powder with 10.000 g NaCl(s).
2. Use 0.040 g of this solid mixture for each titration trial.
3. Put the rest of the powder in the sealable bottle for later use.

**Part II: Complexometric Titration**

1. **Objective:** Determine mole/concentration of metal cation in aqueous solution

2. **Chemicals**
   a. 0.01 M metal salt solution, 25.00 cm$^3$
   b. 0.01 M disodium EDTA solution
   c. NH$_3$/NH$_4$Cl buffer solution (pH = 10) (Buffer)
   d. Eriochrome black T indicator mixture (from Part I (4)) (Indicator)

3. **Apparatus**
   a. Watch glass
   b. Spatula
   c. Electronic balance (±0.001 g)
   d. 1000 (±1) µL micropipette (1 ±0.001 cm$^3$)
   e. 10.00 (±0.01) cm$^3$ micropipette
   f. 100 cm$^3$ conical flask
   g. 50.0 (±0.05) cm$^3$ burette
   h. Funnel
   i. Stand and clamp

4. **Procedures**
   a. Pipette 25.00 cm$^3$ analyte solution into 100 cm$^3$ conical flask
   b. Add 2000 µL (i.e. 2 cm$^3$) of Buffer to the conical flask
   c. Add 0.040 g of Indicator to the conical flask
   d. Swirl everything in the conical flask well
   e. Fill the burette with 0.01 M Na$_2$EDTA
   f. Titrate contents of conical flask against 0.01 M disodium EDTA solution (refer to Image 7 in Appendix)

5. **Notes**
   a. Colour change: **reddish purple → blue** (without hints of red)
   b. Each molecule of disodium EDTA can take up two positive charges. According to equation:

$$\text{Ca}^{2+} + \text{Na}_2\text{EDTA} \rightarrow \text{Ca(EDTA)} + 2\text{Na}^+$$

   a. Zeolite A can theoretically (stoichiometrically) take up a maximum of 12 moles of 1+ cation per mole
   i. From the paper hyperlinked below, it has been calculated that one mole of Z(A) can take up about 12.05 mol of Na (Munthali, Kabwadza-Corner, Johan, & Matsue)
Part III: Z(A) introduction

1. Objective: “Sweep up” some of the metal cations in analyte solution
2. Chemicals
   a. 0.01M metal salt solution, 100.0 cm³
   b. 0.250g Zeolite A (i.e. Z(A))
3. Apparatus
   a. Electronic balance (±0.001 g)
   b. 50.0 (±0.5) cm³ measuring cylinder
   c. 250 cm³ beaker
   d. 250 cm³ conical flask
   e. Magnetic stir bar
   f. Hot plate with magnetic stirrer function
   g. Filter paper
   h. Funnel
4. Procedures
   a. Measure 100.0 cm³ of 0.01M metal salt solution with measuring cylinder
   b. Pour 100.0 cm³ metal salt solution in a 250 cm³ beaker
   c. Measure with electronic balance and put 0.250g Z(A) into the beaker
   d. Stir with magnetic stirrer at 1000 rpm for 15 min.
   e. Filter out Z(A) with funnel, filter paper and conical flask by gravity filtration (refer to image 9 in Appendix)

Note: The purpose of filtering out Z(A) is to quench (stop) the exchange of metal ions, through removing the reactant.

Part IV: Complexometric titration of “swept up” analyte solution

1. Objective: Compare titration results of Part I and III
2. Chemicals, Apparatus, Procedures:
   a. Repeat Part I with analyte solution obtained in Part III.

Part V: Special Methodology for Fe₂(SO₄)₃ and CuSO₄ metal salt solution

1. Objective: Determine mole/concentration of metal cation in aqueous solution
2. Chemicals
   a. 1.999 g Fe₂(SO₄)₃ (anhydrous) solid
   b. 0.1 M CuSO₄ solution
   c. 0.1 M NaOH solution
   d. 0.1 M HCl solution
   e. 0.250 g zeolite solid
   f. Deionised water
3. Apparatus
   a. Watch glass
   b. Spatula
   c. 500 cm³ volumetric flask
   d. 250 cm³ beaker
      i. Electronic balance (±0.001 g)
   e. 10.00 (±0.01) cm³ micropipette
f. 100 cm$^3$ conical flask

4. Procedures

   a. Making 0.01M metal salt solution
      
         i. Dissolve 1.999 g Fe$_2$(SO$_4$)$_3$ in 500 cm$^3$ water in a volumetric flask. Swirl thoroughly.
         
      b. Gravimetric Analysis and Titration without zeolite introduction
         
         i. Take 25 cm$^3$ of the solution in (a). Add 40 cm$^3$ of 0.1M NaOH and stir until no precipitate forms. Allow solution to settle.
         
         ii. Thoroughly wet a piece of filter paper and measure its mass.
         
         iii. Filtrate the settled solution by vacuum filtration using the filter paper in the previous step.
         
         iv. Weigh the filter paper in the previous step and record the new mass.
         
         v. Titrate the filtered solution with 0.1M HCl to determine excess NaOH in solution.

   c. Gravimetric Analysis and Titration with zeolite introduction
      
      i. In a 250 cm$^3$ beaker, mix 100 cm$^3$ of the solution in (a) with 0.250 g zeolite. Stir magnetically for 15 minutes.
      
      ii. Filtrate the solution in the previous step to remove the zeolite through vacuum filtration.
      
      iii. Repeat all steps detailed in part b, instead with filtered solution from previous step.
Data Processing

Preparation of Chemicals

Qualitative Data

The Disodium EDTA solution we used was prepared by dissolving solid granules, which are white in colour into deionised water. The Eriochrome black T indicator was prepared using EBT black powder and NaCl(s) powder, which are purplish black and white in colour respectively; After preparation, the Disodium EDTA solution and NH₃/NH₄Cl buffer was clear colourless and the Eriochrome black T indicator was in purple powder form.

Zeolite A synthesis

Qualitative Data

During preparation, the sodium hydroxide was measured in the form of white pellets. When mixed with deionised water, the resulting NaOH solution is clear colourless.

Bottle A, to which white powder form of sodium aluminate was added, was warm during dissolution of the powder and a milky solution which turned clear with a slight yellow tint was seen inside the bottle. A clear solution was seen inside Bottle B, to which sodium metasilicicate in white powder form was added, and the bottle was cold during dissolution of the powder. When solution from bottle B was poured into bottle A and shaken rapidly, the solution felt slightly warm and a milky viscous solution was observed.

After retrieving the bottle from the oven, a clear distinction between white precipitate and clear colourless supernatant is observed. After filtering the mixture, a white paste collects on the filter paper, which is scraped out and put into the oven for drying. After drying the paste in the oven, the paste turned into a brittle white solid.

Metal ions Adsorption (Adsorption)

Qualitative Data

For non-Coloured Solutions

*Since the same observations can be applied to other colourless metal ions solutions, that is Ca(NO₃)₂ and Pb(NO₃)₂ solution, and water sample solutions, that is seawater, tap water and deionised water samples. For smoother presentation, only the observations in trials using Ca(NO₃)₂ solution is presented.

The Ca(NO₃)₂ solution is originally clear and colourless. After the addition of zeolite, and stirring, the solution became cloudy and white in colour. White precipitate, presumably white zeolite particulate, could be observed on the bottom of the beaker. After filtering the solution using gravity filtration, the solution became clearer.

For coloured Solutions

Fe₂(SO₄)₃ is a clear yellow solution. When the solution is introduced with zeolite, stirred and vacuum filtrated, the zeolite that was separated are observed to have a yellowish tinge. Both batches of non-zeolite treated (original) and zeolite treated solutions are still yellow in colour. Upon the
addition of NaOH solution, both solutions start to form orange-brown suspension, which after 2-3 minutes of settling resulted in a flask containing orange brown precipitate at bottom, and with clear and colourless supernatant (Refer to image 4 in appendix).

CuSO₄ is a clear light blue solution. When the solution is introduced with zeolite, stirred and vacuum filtrated, the zeolite that was separated are observed to have a bluish tinge. Both batches of non-zeolite treated (original) and zeolite treated solutions are still blue in colour. Upon the addition of NaOH solution, both solutions start to form dark blue suspension, which after 2-3 minutes of settling resulted in a flask containing dark blue suspension, and with clear colourless supernatant. Note that the zeolite treated trial has precipitates of a heterogeneous colour, comprising of milky blue and light blue, which may be understood as the unfilterable impurities such as unfilterable zeolite particulates. (Refer to image 6 in appendix)

**Volumetric Analysis**

**Qualitative Data**

The Ca(NO₃)₂ solution without zeolite is originally colourless and clear, while that with zeolite is slightly milky. The solution without zeolite stayed colourless and that with zeolite stayed slightly milky after the addition of the pH buffer. Both solutions turned reddish purple after the addition of EBT indicator. After a sufficient volume of Na₂EDTA solution is added, the solutions turned blue, signalling the endpoint of the titration process. (Refer to image 1 in appendix for endpoint colour)

For the pre-titration colours of other substances, a similar red tinge is present in Pb(NO₃)₂ solution and seawater; tap water solution starts with blueish purple, while deionised water started off with blue colour, that is the endpoint has already been reached.

**Quantitative Data**

There are in total 3 parts in our volumetric analysis of the Zeolite A from our synthesis.

1. Oven Temperature and crystallisation
2. Hours in Oven
3. Applications of Zeolite A
   a. Volumetric Analysis
   b. Back Titration Analysis and Gravimetric Analysis

**Part 1 - Oven Temperature and Crystallisation Temperature**

**Blank trial**

Two sets of titration were performed on two different days to investigate into the effect of crystallisation temperature on the rate of crystallisation of Zeolite A. The rate of crystallisation is reflected by the effectiveness of Zeolite A in adsorbing Ca²⁺ ion.

Blank trials were performed on each day of titration in order to quantify potential systematic errors in experimental design due to the uncertainty in the denaturalisation of Ca(NO₃)₂ solution.
Table 1. Values recorded during blank titration on January 25.

<table>
<thead>
<tr>
<th>Trial # on January 25</th>
<th>Initial Volume (± 0.05 cm³)</th>
<th>Final Volume (± 0.05 cm³)</th>
<th>Volume of Na₂EDTA(aq) Used (± 0.10 cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trial 1</td>
<td>0.00</td>
<td>17.70</td>
<td>17.70</td>
</tr>
<tr>
<td>Trial 2</td>
<td>0.00</td>
<td>17.40</td>
<td>17.40</td>
</tr>
<tr>
<td>Trial 3</td>
<td>0.00</td>
<td>17.60</td>
<td>17.60</td>
</tr>
<tr>
<td>Trial 4</td>
<td>17.60</td>
<td>37.40</td>
<td>19.80</td>
</tr>
<tr>
<td>Trial 5</td>
<td>0.00</td>
<td>19.80</td>
<td>19.80</td>
</tr>
<tr>
<td>Trial 6</td>
<td>0.00</td>
<td>20.10</td>
<td>20.10</td>
</tr>
<tr>
<td>Trial 7</td>
<td>0.00</td>
<td>20.40</td>
<td>20.40</td>
</tr>
<tr>
<td>Trial 8</td>
<td>0.00</td>
<td>19.90</td>
<td>19.90</td>
</tr>
</tbody>
</table>

Table 2. Values recorded blank titration on February 18.

<table>
<thead>
<tr>
<th>Trial # on February 18</th>
<th>Initial Volume (± 0.05 cm³)</th>
<th>Final Volume (± 0.05 cm³)</th>
<th>Volume of Na₂EDTA(aq) Used (± 0.10 cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trial 1</td>
<td>0.00</td>
<td>22.60</td>
<td>22.60</td>
</tr>
<tr>
<td>Trial 2</td>
<td>0.40</td>
<td>21.70</td>
<td>21.30</td>
</tr>
<tr>
<td>Trial 3</td>
<td>21.70</td>
<td>43.70</td>
<td>22.00</td>
</tr>
<tr>
<td>Trial 4</td>
<td>0.30</td>
<td>20.80</td>
<td>20.50</td>
</tr>
</tbody>
</table>

Sample Calculation for titration volumes

For Trial 1 on February 18,

\[
Volume \ of \ Na_2EDTA \ used = Final - Initial \ Volume \\
= (22.60 \pm 0.05) - (0.00 \pm 0.05) \\
= 22.60 \pm 0.10 \text{cm}^3
\]

\[
Average = \frac{\sum[Volume \ of \ Na_2EDTA \ used]}{number \ of \ trials} \\
= \frac{22.60 + 21.30 + 22.00 + 20.50}{4} \pm \frac{0.10 \times 4}{4} \\
= 21.60 \pm 0.10 \text{cm}^3
\]

Table 3. Calculated average values of Na₂EDTA used in both blank trials.

<table>
<thead>
<tr>
<th># Blank trial (Date of blank trial)</th>
<th>Average Volume of Na₂EDTA(aq) Used</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank trial 1 (January 25)</td>
<td>19.09 ± 0.10 cm³</td>
</tr>
<tr>
<td>Blank trial 2 (February 18)</td>
<td>21.60 ± 0.10 cm³</td>
</tr>
</tbody>
</table>

With the two average volumes of Na₂EDTA used in blank trials on both days, the difference of which can be taken, such that the results obtained from two different days can be calibrated to avoid any influence caused by the difference in time of titration.
**Sample Calculation for Difference between Average Volume**

\[
\text{Difference} = \text{Trial 1} - \text{Trial 2}
\]

\[
= (19.09 \pm 0.10) - (21.60 \pm 0.10)
\]

\[
= -2.51 \pm 0.20 \text{ cm}^3
\]

**Increments trial**

In the titration to investigate the effect of crystallisation temperature and the crystallisation of Zeolite A, the Zeolite A prepared in 70.0°C, 80.0°C, 90.0°C, 100.0°C, 115.0°C and 130.0°C are titrated and the respective results are presented in Table 4.

Table 4. Values recorded during titration on January 25 in investigation of crystallisation temperature and crystallisation. (Raw initial and final EDTA volumes are not reported.)

<table>
<thead>
<tr>
<th>Temperature (±0.1°C)</th>
<th>70.0</th>
<th>80.0</th>
<th>90.0</th>
<th>100.0</th>
<th>115.0</th>
<th>130.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume of Na₂EDTA(aq) Used (±0.10cm³)</td>
<td>8.70</td>
<td>8.80</td>
<td>20.10</td>
<td>16.10</td>
<td>8.20</td>
<td>8.40</td>
</tr>
<tr>
<td></td>
<td>8.70</td>
<td>8.50</td>
<td>18.20</td>
<td>14.60</td>
<td>7.80</td>
<td>8.40</td>
</tr>
<tr>
<td></td>
<td>8.40</td>
<td>8.40</td>
<td>19.15</td>
<td>15.35</td>
<td>8.00</td>
<td>8.33</td>
</tr>
</tbody>
</table>

From Table 4, it was significant that the results of the Zeolite A prepared in 90.0°C and 100.0°C were outliers from the rest of the increments, each at around 8 to 8.60. We traced the problem to insufficient oven time for crystallisation due to constraints in our laboratory’s open hours; thus, a new batch of Zeolite A were re-made for 90.0°C and 100.0°C. The second sets of Zeolite A were tested and titration was performed on February 18 with the results presented in Table 5.

Table 5. Values recorded during titration on February 18 in investigation of crystallisation temperature and crystallisation.

<table>
<thead>
<tr>
<th>Temperature (±0.1°C)</th>
<th>70.0</th>
<th>80.0</th>
<th>90.0</th>
<th>100.0</th>
<th>115.0</th>
<th>130.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume of Na₂EDTA(aq) Used (±0.10cm³)</td>
<td>11.20</td>
<td>10.80</td>
<td>10.80</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>11.20</td>
<td>11.00</td>
<td>11.20</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>10.90</td>
<td>11.00</td>
<td>10.80</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average Volume of Na₂EDTA(aq) Used (±0.10cm³)</td>
<td>11.10</td>
<td>10.93</td>
<td>10.93</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

From evaluating Table 4 and Table 5, it is seen that the performance of the control set of Zeolite A prepared in 70.0°C varied on February 18 when compared to that on January 25. As such calibration by blank trial is necessary for a fair comparison between temperature increments.

**Sample Calculation for Difference between 70.0°C Trials**

\[
\text{Difference} = \text{Average[70.0°C on Jan 25]} - \text{Average[70.0°C on Feb 18]}
\]

\[
= (8.60 \pm 0.10) - (11.0 \pm 0.10)
\]

\[
= -2.50 \pm 0.20 \text{ cm}^3
\]
In fact, by comparing the similar values of the difference between the average volumes of Na₂EDTA used in blank trials on both days (i.e. -2.51±0.20cm³) with the difference between the average volume of Na₂EDTA used in 70.0°C trials (i.e. -2.50±0.20cm³), it is feasible to use the difference between the blank trials to calibrate the titration results (for 90.0°C and 100.0°C increments) obtained on February 18. As such, the data values are presented in Table 6 after calibration.

Table 6. Final titration results in investigation of crystallisation temperature and crystallisation after calibration.

<table>
<thead>
<tr>
<th>Temperature (±0.1°C)</th>
<th>70.0*</th>
<th>80.0</th>
<th>90.0</th>
<th>100.0</th>
<th>115.0</th>
<th>130.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average Volume of Na₂EDTA(aq) Used on Jan 25 (±0.10cm³)</td>
<td>8.60</td>
<td>8.57</td>
<td>19.15</td>
<td>15.35</td>
<td>8.00</td>
<td>8.33</td>
</tr>
<tr>
<td>Average Volume of Na₂EDTA(aq) Used on Feb 18 (±0.10cm³)</td>
<td>11.10</td>
<td>10.93</td>
<td>10.93</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Calibration unit</td>
<td>-2.51 ± 0.20cm³</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average Volume of Na₂EDTA(aq) Used (±0.10)</td>
<td>8.60 (±0.10)</td>
<td>8.57 (±0.10)</td>
<td>8.42 (±0.30)</td>
<td>8.42 (±0.30)</td>
<td>8.00 (±0.10)</td>
<td>8.33 (±0.10)</td>
</tr>
<tr>
<td>% Uncertainty</td>
<td>1.16%</td>
<td>1.17%</td>
<td>3.56%</td>
<td>3.56%</td>
<td>1.25%</td>
<td>1.20%</td>
</tr>
</tbody>
</table>

*The data for 70.0 °C is not calibrated, hence the data from the first trial is taken.

Sample calculation for Calibration value for 90.0°C Trial

Calibrated value for 90.0°C = Original value + Calibration unit

= (10.93 ± 0.10) + (-2.51 ± 0.20)

= 8.42 ± 0.30 cm³

= 8.42 ± 3.56%

Percentage uncertainty of 70.0 °C

\[
\frac{0.10}{8.60} \times 100\% = 1.16\%
\]
Interpretation of Figure 1

Figure 1 graphs titre volume of Na$_2$EDTA used against crystallisation temperature. From the parabolic nature of the line of best fit, it is concluded that crystallisation temperature and cation adsorption has a parabolic relationship: cation adsorption increases from 70.0 °C, peaks at 115.0 °C and begins decreasing as crystallisation temperature rises to 130.0 °C. Since the number of moles of Ca$^{2+}$ ions adsorbed by the zeolite framework is inversely related to the titre volume in the graph, it is inferred that the adsorption efficiency and capacity of zeolites are optimum at 115.0 °C. The axis of symmetry of the parabolic line of best fit is at $x = 122.081$ which suggests the temperature at which a minimum titre volume is needed, and hence the crystallisation temperature at which zeolite adsorption is maximum, is roughly 122 °C. There is a deviation between trend line optimum and experimental optimum, as the $R^2$ value of 0.6363 isn’t of the highest confidence.

From the error bars of the graph, the random error produced in this experiment is relatively uniform except for the increased random error due to calibration measures employed in the data points for a crystallisation temperature of both 90.0 °C and 100.0 °C. Additionally, the $R^2$ value of the line of best fit is 0.6363 which suggests a correlation, but not a particularly strong correlation.
Part 2 – Oven Time and Crystallisation Duration
Additionally, two sets of syntheses and titration were performed on the aforementioned two different days to investigate the effect of time of batch in oven (“crystallisation duration”) on crystallisation completeness of Zeolite A. The completeness of crystallisation is reflected in the batch’s effectiveness in adsorbing Ca\(^{2+}\) ions.

As with Part 1, blank trials were performed on both days to quantify and allow for accounting of potential systematic errors. This data is presented in Tables 1, 2 and 3 of Part 1.

**Hours in Oven Trial**

Table 7. Values recorded during titration on January 25 in investigation of crystallisation duration and crystallisation.

| Crystallisation duration (±1 min) | 60 (1 hr) *
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume of Na(_2)EDTA(aq) Used (±0.10 cm(^3)) *</td>
<td>25.30</td>
</tr>
<tr>
<td></td>
<td>25.20</td>
</tr>
<tr>
<td></td>
<td>25.70</td>
</tr>
<tr>
<td>Average Volume of Na(_2)EDTA(aq) Used (±0.10 cm(^3))</td>
<td>25.40</td>
</tr>
</tbody>
</table>

*It is noted that for the 60-minute (1 hr) batch, the colour of solution at 25.30 cm\(^3\) titre volume is still purple. However, the still-purple solution slowly turns blue when left as is within 15 minutes. The same holds true for the two other trials: the purple solution in the conical flask of each trial eventually turns blue. This may be due to the incomplete zeolite framework disintegrating: particularly, it may be due to Al\(^{3+}\) ions being broken from the framework and being chelated by EDTA. Since this process takes time, this explains why 15 minutes must elapse before the solution turns blue.

Table 8. Values recorded during titration on February 18 in investigation of crystallisation duration

<table>
<thead>
<tr>
<th>Crystallisation Duration (±1 min)</th>
<th>180 (3 hr)</th>
<th>300 (5 hr)</th>
<th>420 (7 hr)</th>
<th>540 (9 hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume of Na(_2)EDTA(aq) Used (±0.10 cm(^3))</td>
<td>10.80</td>
<td>10.20</td>
<td>18.80</td>
<td>21.80</td>
</tr>
<tr>
<td></td>
<td>11.00</td>
<td>10.30</td>
<td>18.50</td>
<td>21.55</td>
</tr>
<tr>
<td></td>
<td>11.00</td>
<td>10.10</td>
<td>18.20</td>
<td>21.60</td>
</tr>
<tr>
<td>Average Volume of Na(_2)EDTA(aq) Used (±0.10 cm(^3))</td>
<td>10.93</td>
<td>10.20</td>
<td>18.50</td>
<td>21.65</td>
</tr>
</tbody>
</table>

**Sample Calibration for average titration volumes**

For 300 min oven time batch,

\[
\text{Average} = \frac{\sum \text{[Volume of Na}_2\text{EDTA used]}}{\text{number of trials}}
\]

\[
= \frac{10.20 + 10.30 + 10.10}{3} \pm \frac{0.10 \times 3}{3}
\]

\[
= 10.20 \pm 0.10 \text{ cm}^3
\]

As noted previously, the deviation of data in volume of Na\(_2\)EDTA used in the 70.0 °C and blank trials necessitate a calibration procedure for data from these two dates to be compared on fair grounds. An identical calibration procedure has been done for the raw data in Table 7 and 8 and is presented accordingly in Table 9.
Table 9. Final titration results in investigation of crystallisation duration and completeness of crystallisation after calibration.

<table>
<thead>
<tr>
<th>Crystallisation Duration (±1 min)</th>
<th>60 (1hr)*</th>
<th>180 (3 hr)</th>
<th>300 (5 hr)</th>
<th>420 (7 hr)</th>
<th>540 (9 hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average Volume of Na$_2$EDTA(aq) Used on Jan 25 (±0.10cm$^3$)</td>
<td>25.40</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average Volume of Na$_2$EDTA(aq) Used on Feb 18 (±0.10cm$^3$)</td>
<td></td>
<td>10.93</td>
<td>10.20</td>
<td>18.50</td>
<td>21.65</td>
</tr>
<tr>
<td>Calibration unit è</td>
<td>-2.51 ± 0.20cm$^3$</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average Volume of Na$_2$EDTA(aq) Used</td>
<td>25.40 (±0.10)</td>
<td>8.42 (±0.30)</td>
<td>7.69 (±0.30)</td>
<td>15.99 (±0.30)</td>
<td>19.14 (±0.30)</td>
</tr>
<tr>
<td>% Uncertainty</td>
<td>0.39%</td>
<td>2.38%</td>
<td>2.60%</td>
<td>1.25%</td>
<td>1.04%</td>
</tr>
</tbody>
</table>

*Data for 60-minute trial is not calibrated, hence data from the first trial is used.

Figure 2: Scatter plot showing trend of average volume of Na.EDTA used in Table 9.
Interpretation of Figure 2

Figure 2 graphs titre volume of Na₂EDTA used against crystallisation duration. From the parabolic nature of the line of best fit, it is concluded that crystallisation duration and cation adsorption has a parabolic relationship: cation adsorption increases from 60 minutes, peaks at 300 minutes and begins decreasing as crystallisation temperature rises to 540 minutes. Since the number of moles of Ca²⁺ ions adsorbed by the zeolite framework is inversely related to the titre volume in the graph, it is inferred that the adsorption efficiency and capacity of zeolites are optimum at 300 minutes of crystallisation. The axis of symmetry of the parabolic line of best fit is at \( x = 5.14 \) which suggests the duration at which a minimum titre volume is needed, and hence the duration at which zeolite adsorption is at its highest point, is roughly 5 hours and 23 minutes.

The error bars on the graph are not easily visible, which suggests the percentage uncertainty of the experiment trials are rather small; in other words, our trials were relatively precise. In addition, the \( R^2 \) value of the line of best fit is 0.7910 which suggests a fairly strong correlation.

Sample Calibration for 300 min Increment

\[ \text{Calibrated value} = \text{Original value} + \text{Calibration unit} \]

\[ = (10.20 \pm 0.10) + (-2.51 \pm 0.20) \]

\[ = 7.69 \pm 0.30 \text{ cm}^3 \]

\[ = 7.69 \pm 3.90\% \]

For better comparison, the cation adsorption efficiency of zeolites synthesised at different temperatures and crystallisation durations has been calculated and presented in Tables 10 and 11.

Table 10. Effect of Crystallisation Temperature on Adsorption Efficiency of Ca²⁺ ions

<table>
<thead>
<tr>
<th>Crystallisation Temperature / °C</th>
<th>Mass of zeolite</th>
<th>No. of moles of Ca²⁺ ions exchanged</th>
<th>% Efficiency</th>
<th>% Capacity</th>
<th>% Calibrated Capacity</th>
</tr>
</thead>
<tbody>
<tr>
<td>70.0</td>
<td>0.250 g</td>
<td>0.0001049 (±2.0%)</td>
<td>55.0 (±3%)</td>
<td>15.3 (±2%)</td>
<td>42.0 (±4%)</td>
</tr>
<tr>
<td>80.0</td>
<td>0.250 g</td>
<td>0.0001052 (±1.9%)</td>
<td>55.1 (±3%)</td>
<td>15.4 (±2%)</td>
<td>42.1 (±4%)</td>
</tr>
<tr>
<td>90.0</td>
<td>0.250 g</td>
<td>0.0001067 (±4%)</td>
<td>55.9 (±4%)</td>
<td>15.6 (±4%)</td>
<td>42.7 (±5%)</td>
</tr>
<tr>
<td>100.0</td>
<td>0.250 g</td>
<td>0.0001067 (±4%)</td>
<td>55.9 (±4%)</td>
<td>15.6 (±4%)</td>
<td>42.7 (±5%)</td>
</tr>
<tr>
<td>115.0</td>
<td>0.250 g</td>
<td>0.0001109 (±1.8%)</td>
<td>58.1 (±2%)</td>
<td>16.2 (±2%)</td>
<td>44.4 (±3%)</td>
</tr>
<tr>
<td>130.0</td>
<td>0.250 g</td>
<td>0.0001076 (±1.9%)</td>
<td>56.4 (±2%)</td>
<td>15.7 (±2%)</td>
<td>43.0 (±4%)</td>
</tr>
</tbody>
</table>

Table 11. Effect of Crystallisation Duration on Adsorption Efficiency of Ca²⁺ ions

<table>
<thead>
<tr>
<th>Crystallisation Duration / minutes</th>
<th>No. of moles of Ca²⁺ ions exchanged</th>
<th>% Efficiency</th>
<th>% Capacity</th>
<th>% Calibrated Capacity</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>0.00006310 (±3%)</td>
<td>-33.1 (±3%)</td>
<td>-9.22 (±3%)</td>
<td>-25.2 (±4%)</td>
</tr>
<tr>
<td>180</td>
<td>0.0001067 (±1.9%)</td>
<td>55.9 (±2%)</td>
<td>15.6 (±2%)</td>
<td>42.7 (±4%)</td>
</tr>
<tr>
<td>300</td>
<td>0.0001140 (±1.8%)</td>
<td>59.7 (±2%)</td>
<td>16.7 (±2%)</td>
<td>45.6 (±3%)</td>
</tr>
<tr>
<td>420</td>
<td>0.00003100 (±6%)</td>
<td>16.2 (±7%)</td>
<td>4.53 (±7%)</td>
<td>12.4 (±8%)</td>
</tr>
<tr>
<td>540</td>
<td>-5.000 x 10⁻⁷ (±400%)</td>
<td>-0.260 (±400%)</td>
<td>-0.0730 (±400%)</td>
<td>-0.200 (±400%)</td>
</tr>
</tbody>
</table>
Sample calculation of adsorption efficiency of 70.0°C zeolite

Difference in Vol. Na₂EDTA used = 19.09 (±0.10) − 8.60 (±0.10) = 10.49 (±0.20) cm³

Difference in No. of moles of Na₂EDTA = \( \frac{10.49}{1000} \times 0.01 = 0.0001049 \) mol

\[ \text{% Uncertainty} = \left| \left( \frac{0.20}{10.49} \right) + \left( \frac{0.001}{33.822} + \frac{0.40}{1000} \right) \right| \times 100\% = 2.0\% \]

No. of moles of Ca²⁺ exchanged = Difference in No. of moles of Na₂EDTA = 0.0001049 (±1.950%) mol

Original number of moles of Ca²⁺ without zeolite = \( \frac{19.09}{1000} \times 0.01 = 0.0001909 \) mol

\[ \text{% Uncertainty} = \left| \left( \frac{0.10}{19.09} \right) + \left( \frac{0.001}{33.822} + \frac{0.40}{1000} \right) \right| \times 100\% = 0.57\% \]

Adsorption Efficiency = \( \frac{0.0001049}{0.0001909} \times 100\% = 55.0\% \) (3 s.f.)

\[ \text{% Uncertainty} = 2.0\% + 0.57\% = 3\% \]

Sample Calculation of Adsorption Capacity of 70.0°C Zeolite

Adsorption Capacity = \( \frac{0.0001049}{2191.26 \times 2} \times 100\% = 15.3\% \)

\[ \text{% Uncertainty} = \left| \frac{0.20}{10.49} + \frac{0.001}{0.250} \right| \times 100\% = 2\% \]

Theoretical Maximum Ca²⁺ Adsorption of 0.250g Zeolite A

\[ = \text{Mol of zeolite A} \times \left( \frac{12}{\text{charge of cation}} \right) \]

\[ = \left( \frac{0.250}{2191.26} \right) \times \left( \frac{12}{2} \right) = 6.85 \times 10^{-4} \text{ mol of Ca}^{2+} \]

Theoretical Maximum Adsorption of Ca²⁺ in analyte as a percentage of maximum adsorption

\[ = \frac{\text{Initial mole of Ca}^{2+} \text{ present in analyte}}{\text{Theoretical Maximum Ca}^{2+} \text{ Uptake Efficiency of 0.250g zeolite A}} \times 100\% \]

\[ = \frac{0.01 \times \left( \frac{25.0}{1000} \right)}{6.85 \times 10^{-4}} \times 100\% = 36.5\% \]

Calibrated Capacity = \[ \frac{\text{Experimental Uptake Capacity}}{\text{Theoretical Maximum Capacity of Ca}^{2+} \text{ in solution}} \times 100\% \]

\[ = \frac{15.32\%}{36.5\%} \times 100\% = 42.0\% \]

Percentage Uncertainty of Calibrated Capacity

\[ = \left| \frac{0.20}{10.49} + \frac{0.001}{0.250} + \left( \frac{0.10}{19.09} \right) + \left( \frac{0.001}{33.822} + \frac{0.40}{1000} \right) + \frac{0.001}{0.250} \right| \times 100\% = 4\% \]
Part 3 – Applications of Zeolite A

**Titration Analysis (For Colourless Solutions)**

To test the application of our synthesised Zeolite A, the batch prepared at 90.0°C for 3 hours were put to test in a series of titration with various water sources and various species of metallic compounds (in respective nitrate aqueous solution form). In this part, the tested water source and metallic compounds are in colourless aqueous form, thus allowing us to use complexometric titration procedures, as above, for data collection. For other coloured aqueous solutions, the acid-base back-titration method is used (refer to *Back Titration Analysis* part for more information). The results collected from respective titrations are shown as in Table 12 and Table 13.

### Table 12. Results of complexometric titrations of various treated water sources.

<table>
<thead>
<tr>
<th>Water source</th>
<th>Deionised water</th>
<th>Tap water</th>
<th>Sea water</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Without Zeolite A</td>
<td>With Zeolite A</td>
<td>Without Zeolite A</td>
</tr>
<tr>
<td>Initial Volume (± 0.05 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Final Volume (± 0.05 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Volume of Na$_2$EDTA(aq) Used (± 0.10 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>1.00</td>
</tr>
</tbody>
</table>

**Trial 1**

<table>
<thead>
<tr>
<th>Water source</th>
<th>Deionised water</th>
<th>Tap water</th>
<th>Sea water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial Volume (± 0.05 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Final Volume (± 0.05 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Volume of Na$_2$EDTA(aq) Used (± 0.10 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>1.00</td>
</tr>
</tbody>
</table>

**Trial 2**

<table>
<thead>
<tr>
<th>Water source</th>
<th>Deionised water</th>
<th>Tap water</th>
<th>Sea water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial Volume (± 0.05 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Final Volume (± 0.05 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Volume of Na$_2$EDTA(aq) Used (± 0.10 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>1.00</td>
</tr>
</tbody>
</table>

**Trial 3**

<table>
<thead>
<tr>
<th>Water source</th>
<th>Deionised water</th>
<th>Tap water</th>
<th>Sea water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial Volume (± 0.05 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Final Volume (± 0.05 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Volume of Na$_2$EDTA(aq) Used (± 0.10 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>1.00</td>
</tr>
</tbody>
</table>

**Average**

<table>
<thead>
<tr>
<th>Water source</th>
<th>Deionised water</th>
<th>Tap water</th>
<th>Sea water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume of Na$_2$EDTA(aq) Used (± 0.10 cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>1.00</td>
</tr>
</tbody>
</table>
Table 13. Results of complexometric titrations of various treated metal salt solutions.

<table>
<thead>
<tr>
<th>Metal Salt Solution</th>
<th>Pb(NO$_3$)$_2$</th>
<th>Ca(NO$_3$)$_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Without Zeolite A</td>
<td>With Zeolite A</td>
</tr>
<tr>
<td>Trial 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial Volume</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Final Volume</td>
<td>27.40</td>
<td>8.50</td>
</tr>
<tr>
<td>Volume of Na$_2$EDTA(aq) Used</td>
<td>27.40</td>
<td>8.50</td>
</tr>
<tr>
<td>Trial 2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial Volume</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Final Volume</td>
<td>27.30</td>
<td>8.90</td>
</tr>
<tr>
<td>Volume of Na$_2$EDTA(aq) Used</td>
<td>27.30</td>
<td>8.90</td>
</tr>
<tr>
<td>Trial 3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial Volume</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Final Volume</td>
<td>27.00</td>
<td>8.40</td>
</tr>
<tr>
<td>Volume of Na$_2$EDTA(aq) Used</td>
<td>27.00</td>
<td>8.40</td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume of Na$_2$EDTA(aq) Used</td>
<td>27.20</td>
<td>8.60</td>
</tr>
</tbody>
</table>

Gravimetric Analysis (For Coloured Solutions)

It is difficult and impractical to perform volumetric analysis on coloured solutions. As such, gravimetric analysis is performed before back titration analysis to detect whether there is a change in precipitate mass after zeolite treatment. This is to make sure there is in fact an observable and meaningful purpose for performing back titration analysis on respective solutions.

Qualitative Data

1. Fe$_2$(SO$_4$)$_3$ solution

Following our mentioned methodology, the resultant mixture presumably of Na$_2$SO$_4$ solution, Fe(OH)$_3$ precipitate, and excess NaOH solution is vacuum filtrated by filter paper. After filtration for 10 minutes, the clear solutions have mostly entered the Buchner flask, appearing as a clear and colourless solution. Left on the filter paper is a large mass of orange brown solid, which is taken out
and weighed. The mass of the filter paper containing the precipitate subtracted by the mass of filter papers are understood as the mass of Fe(OH)$_3$ precipitate. It should be noted that the trials of zeolite-treated solutions are distinguishably lighter in colour, taking a slight yellow tinge, while non-zeolite treated trials are brownish in colour. (Refer to image 3 in appendix)

2. CuSO$_4$ solution

Resultant mixture presumably of Na$_2$SO$_4$ solution, Cu(OH)$_2$ precipitate, and excess NaOH solution is vacuum filtrated by filter paper. After filtration for 10 minutes, the clear solutions have mostly entered the Buchner flask, appearing as a clear and colourless solution. Left on the filter paper is a large mass of dark blue solid, which is taken out and weighed. The mass of the filter paper containing the precipitate subtracted by the mass of filter papers are understood as the mass of Cu(OH)$_2$ precipitate. Both non-zeolite treated and zeolite treated trials are indistinguishable in colour by qualitative observation. (Refer to image 6 in appendix)

Quantitative Data

<table>
<thead>
<tr>
<th>Trial</th>
<th>Mass of Object / g</th>
<th>Fe$_2$(SO$_4$)$_3$ w/out Zeolite</th>
<th>Fe$_2$(SO$_4$)$_3$ with Zeolite</th>
<th>CuSO$_4$ w/out Zeolite</th>
<th>CuSO$_4$ with Zeolite</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Precipitate with filter papers</td>
<td>6.272</td>
<td>5.378</td>
<td>11.156</td>
<td>13.374</td>
</tr>
<tr>
<td></td>
<td>Precipitate only</td>
<td>1.859</td>
<td>0.965</td>
<td>6.743</td>
<td>8.961</td>
</tr>
<tr>
<td>2</td>
<td>Precipitate with filter papers</td>
<td>6.334</td>
<td>5.363</td>
<td>11.861</td>
<td>11.884</td>
</tr>
<tr>
<td></td>
<td>Precipitate only</td>
<td>1.921</td>
<td>0.950</td>
<td>7.448</td>
<td>7.471</td>
</tr>
<tr>
<td>3</td>
<td>Precipitate with filter papers</td>
<td>5.984</td>
<td>5.374</td>
<td>12.328</td>
<td>12.630</td>
</tr>
<tr>
<td></td>
<td>Precipitate only</td>
<td>1.571</td>
<td>0.961</td>
<td>7.915</td>
<td>8.217</td>
</tr>
<tr>
<td>Average mass of precipitate</td>
<td>1.784</td>
<td>0.959</td>
<td>7.369</td>
<td>8.216</td>
<td></td>
</tr>
</tbody>
</table>

Notes
1. Mass of filter papers (dry bottom + wet used in funnel) = 1.136g + 32.77g = 4.413g
2. Mass of Precipitate only = Mass of precipitate with filter papers – Mass of filter papers

As seen from Table 14, there is mass change of the precipitate of the solution, signifying that there is an observable effect of the Zeolite being added to the solution. Hence, back titration analysis is performed to further quantify the effects of Zeolite in the respective solutions.

Back Titration Analysis (For Coloured Solutions)

Qualitative Data
For trials on all back titrations on coloured ions, the resulting clear and colourless solution collected under the Buchner funnel is used as analyte in back titration for excess NaOH solution. Methyl orange indicator is added to analyte, giving rise to a yellowish colour. Back titration is performed using HCl solution as titrant. Upon reaching endpoint, the analyte solutions appeared to have a red tinge.
Quantitative Data

To test for coloured metallic compounds in aqueous solution form, back titration analysis is taken and the results is taken and shown in Table 15.

Table 15. Results of back titrations with Zeolite A and various metallic compounds.

<table>
<thead>
<tr>
<th>Trial</th>
<th>Metallic compounds</th>
<th>Fe$_2$(SO$_4$)$_3$</th>
<th>CuSO$_4$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Without Zeolite A</td>
<td>With Zeolite A</td>
</tr>
<tr>
<td>Trial 1</td>
<td>Initial Volume</td>
<td>0.70</td>
<td>0.00</td>
</tr>
<tr>
<td></td>
<td>(± 0.05 cm$^3$)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Final Volume</td>
<td>27.20</td>
<td>32.80</td>
</tr>
<tr>
<td></td>
<td>(± 0.05 cm$^3$)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Volume of HCl(aq) titrated = NaOH(aq) excess after filtration (± 0.10 cm$^3$)</td>
<td>26.50</td>
<td>32.80</td>
</tr>
<tr>
<td></td>
<td>Volume of NaOH(aq) actually used in precipitation with metal salt: (40.00 cm$^3$ – excess) (± 0.20 cm$^3$)</td>
<td>13.50</td>
<td>7.20</td>
</tr>
<tr>
<td>Trial 2</td>
<td>Initial Volume</td>
<td>1.10</td>
<td>0.50</td>
</tr>
<tr>
<td></td>
<td>(± 0.05 cm$^3$)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Final Volume</td>
<td>27.30</td>
<td>34.90</td>
</tr>
<tr>
<td></td>
<td>(± 0.05 cm$^3$)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Volume of HCl(aq) titrated = NaOH(aq) excess after filtration (± 0.10 cm$^3$)</td>
<td>26.20</td>
<td>34.40</td>
</tr>
<tr>
<td></td>
<td>Volume of NaOH(aq) actually used in precipitation with metal salt: (40.00 cm$^3$ – excess) (± 0.20 cm$^3$)</td>
<td>13.80</td>
<td>5.60</td>
</tr>
<tr>
<td>Trial 3</td>
<td>Initial Volume</td>
<td>0.70</td>
<td>0.50</td>
</tr>
<tr>
<td></td>
<td>(± 0.05 cm$^3$)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Final Volume</td>
<td>27.40</td>
<td>32.50</td>
</tr>
<tr>
<td></td>
<td>(± 0.05 cm$^3$)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Volume of HCl(aq) titrated = NaOH(aq) excess after filtration (± 0.10 cm$^3$)</td>
<td>26.70</td>
<td>32.00</td>
</tr>
<tr>
<td></td>
<td>Volume of NaOH(aq) actually used in precipitation with metal salt: (40.00 cm$^3$ – excess) (± 0.20 cm$^3$)</td>
<td>13.30</td>
<td>8.00</td>
</tr>
<tr>
<td>Average</td>
<td>Volume of NaOH(aq) used (± 0.20 cm$^3$)</td>
<td><strong>13.53</strong></td>
<td><strong>6.93</strong></td>
</tr>
</tbody>
</table>
It should be understood (in Table 15) that the volume of HCl\(_{\text{(aq)}}\) in the back titration represents the excess NaOH\(_{\text{(aq)}}\) in the clear and colourless solution after filtration and supernatant of precipitation reaction due to 1:1 mole ratio in neutralisation. Thus in order to deduced the amount of metal ion present in metal solution, deducting the total amount of NaOH\(_{\text{(aq)}}\) used from the excess NaOH\(_{\text{(aq)}}\) volume will yield the amount of OH\(^-\) used in complete precipitation of metal salts. By converting the mole ratio of OH\(^-\) with respective metal salts: 2 times for Cu(OH)\(_2\)\(_{\text{(aq)}}\) and 3 times for Fe(OH)\(_3\)\(_{\text{(aq)}}\), the correct quantity of metal ions consumed is found. This method can reflect the difference of zeolite treated and non-zeolite treated metal solutions with the different tire volumes, reflecting the adsorptive efficacy of Zeolite A on such metal ions.

The difference between titre volume with and without Zeolite A treatment in each titration and back titration was calculated and presented in Table 16 for further comparison and discussion. Also, the calculated cation adsorption efficiency and capacity of Zeolite A of respective ions is taken and shown in Table 17.

Table 16. Calculated values for the difference before and after adding Zeolite A.

<table>
<thead>
<tr>
<th></th>
<th>Average Volume without Zeolite A (± x cm(^3))</th>
<th>Average Volume with Zeolite A (± x cm(^3))</th>
<th>Difference (± 2x cm(^3))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titration with 0.01M Na(_2)EDTA (x=0.10)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Deionised water</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Tap water</td>
<td>1.00</td>
<td>0.52</td>
<td>0.48</td>
</tr>
<tr>
<td>Sea water</td>
<td>34.00</td>
<td>33.50</td>
<td>0.50</td>
</tr>
<tr>
<td>Pb(NO(_3))(_2)</td>
<td>27.20</td>
<td>8.60</td>
<td>18.60</td>
</tr>
<tr>
<td>Ca(NO(_3))(_2)</td>
<td>21.97</td>
<td>10.93</td>
<td>11.04</td>
</tr>
<tr>
<td>Back Titration with 0.1M HCl (x=0.20)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fe(_2)(SO(_4))(_3)</td>
<td>13.53</td>
<td>6.93</td>
<td>6.60</td>
</tr>
<tr>
<td>CuSO(_4)</td>
<td>38.43</td>
<td>38.47</td>
<td>-0.03</td>
</tr>
</tbody>
</table>

Table 17. Calculated cation adsorption efficiency of Zeolite A

<table>
<thead>
<tr>
<th></th>
<th>% Adsorption Efficiency</th>
<th>% Adsorption Capacity*</th>
<th>Calibrated % Adsorption Capacity*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deionised water</td>
<td>0% (±0%)</td>
<td>0% (±0%)</td>
<td>0% (±0%)</td>
</tr>
<tr>
<td>Tap water</td>
<td>48.0% (±50%)</td>
<td>0.701% (±40%)</td>
<td>1.92% (±40%)</td>
</tr>
<tr>
<td>Sea water</td>
<td>1.47% (±40%)</td>
<td>0.730% (±40%)</td>
<td>2.00% (±40%)</td>
</tr>
<tr>
<td>Pb(NO(_3))(_2)</td>
<td>68.4% (±1.4%)</td>
<td>27.2% (±2%)</td>
<td>74.4% (±3%)</td>
</tr>
<tr>
<td>Ca(NO(_3))(_2)</td>
<td>50.3% (±2%)</td>
<td>16.1% (±2%)</td>
<td>44.2% (±3%)</td>
</tr>
<tr>
<td>Fe(_2)(SO(_4))(_3)</td>
<td>48.8% (±1.5%)</td>
<td>24.1% (±6%)</td>
<td>48.8% (±7%)</td>
</tr>
<tr>
<td>CuSO(_4)</td>
<td>-0.10% (±1000%)</td>
<td>-0.0438% (±1000%)</td>
<td>-0.120% (±1000%)</td>
</tr>
</tbody>
</table>
Sample Calculation of Adsorption Efficiency for Calcium Nitrate

\[
\% \text{ Uptake Efficiency} = \left( \frac{[\text{Final}] - [\text{Initial}]}{[\text{Initial}]} \right) \times 100\% \\
= \left( \frac{21.97 - 10.93}{21.97} \right) \times 100\%
\]

\[
= 50.3\% \pm 2\%
\]

\[
\% \text{ Uncertainty} = \left| \frac{0.10 + 0.10}{21.97} - \frac{10.93}{21.97} \right| \times 100\% = 2\%
\]

Calculation of Percentage Uncertainty of Adsorption Efficiency and Capacity for Iron Sulphate

\[
\% \text{ Uncertainty of Uncalibrated \% Capacity} = \left( \frac{0.20 + 0.20}{13.53 - 6.93} + \frac{0.001}{0.250} \right) \times 100\% = 6\% (1 \text{ s.f.})
\]

\[
\% \text{ Uncertainty of Calibrated \% Capacity} = \left( \frac{0.20 + 0.20}{13.53 - 6.93} + \frac{0.001}{0.250} + \frac{0.01 + 0.01 + 0.01}{1.999} + \frac{0.01 + 0.01 + 0.01}{25.00} + \frac{0.001}{0.250} \right) \times 100\%
\]

\[
= 7\% (1 \text{ s.f.})
\]

Calculation of Adsorption Efficiency and Capacity for Copper Sulphate

\[
\% \text{ Uncertainty of Uncalibrated \% Capacity} = \left| \frac{0.20 + 0.20}{38.43 - 38.47} + \frac{0.001}{0.250} \right| \times 100\%
\]

\[
= 1000\% (1 \text{ s.f.})
\]

\[
\% \text{ Uncertainty of Calibrated \% Capacity} = \left| \frac{0.20 + 0.20}{38.43 - 38.47} + \frac{0.001}{0.250} + \frac{0.01 + 0.01 + 0.01}{10.00} + \frac{0.01 + 0.01 + 0.01}{25.00} + \frac{0.001}{0.250} \right| \times 100\%
\]

\[
= 1000\% (1 \text{ s.f.})
\]

*For Sample Calculations of adsorption capacity of colourless metal salt solutions, refer to that in Tables 10 and 11.*
Interpretation of Part 3

It is noteworthy to state that data from gravimetric analysis and titrimetric analysis goes hand in hand, in terms of consistency and reflecting the adsorption efficacy of zeolite on that particular metal ion. Thus both data approaches should be equally considered and in fact understood in a self-complimentary manner.

On the basis that colourless and coloured solutions are measured using two separate methods, the data from two parts are therefore not directly compatible in terms of comparison. No trend of sorts can be plotted against the metal we have investigated due to many extraneous variables (see further research).

However, what that can be interpreted from the various changes in volume is the versatility of Zeolite A, with Zeolite A being able to adsorb numerous metal ions of different charge densities (which is a function of the ion’s charge and ionic radius) ranging from 1.33 Å of Pb$^{2+}$ ion and 1.14 Å of Ca$^{2+}$ ion; and not being able to adsorb Cu$^{2+}$ with 0.87 Å (see ‘Atomic structure on metal ion adsorption’ in further research). This gives insight on the functionality of zeolite, and its application in real life.

It should also be commented that the slight change in copper sulphate titre volume with and without zeolite of -0.03cm$^3$ is negligible; the random error of instrumental uncertainties primarily contributed to this difference. The fact that the percentage uncertainty for copper sulphate titre volume’s percentage efficiencies and capacities is 1000% reflects the magnitude of uncertainties in face of such a small calculated value.
Refined Trial - Modification of Synthesis

Table 18. Data collected from Refined Trial Volumetric Analysis

<table>
<thead>
<tr>
<th></th>
<th>Temperature</th>
<th>115.0 °C</th>
<th>Duration</th>
<th>330 minutes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trial no.</td>
<td></td>
<td></td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Initial Volume of Na₂EDTA / cm³</td>
<td></td>
<td></td>
<td>1.10</td>
<td>14.50</td>
</tr>
<tr>
<td>Final Volume of Na₂EDTA / cm³</td>
<td></td>
<td></td>
<td>14.50</td>
<td>27.50</td>
</tr>
<tr>
<td>Volume of Na₂EDTA used / cm³</td>
<td></td>
<td></td>
<td>13.40</td>
<td>13.00</td>
</tr>
<tr>
<td>Average Volume of Na₂EDTA used / cm³</td>
<td></td>
<td></td>
<td></td>
<td>13.07</td>
</tr>
<tr>
<td>Adsorption Efficiency</td>
<td></td>
<td></td>
<td></td>
<td>39.5%</td>
</tr>
<tr>
<td>Uncalibrated Adsorption Capacity</td>
<td></td>
<td></td>
<td></td>
<td>12.5%</td>
</tr>
<tr>
<td>Calibrated Adsorption Capacity</td>
<td></td>
<td></td>
<td></td>
<td>34.1%</td>
</tr>
</tbody>
</table>

Discussion of Refined Trial

The average volume of Na₂EDTA titrant is 13.07 cm³. Compared to the previously determined titre volumes at optimum temperature and time, which is 8.00 cm³ and 7.62 cm³ respectively, this is an unexpectedly large value. Since average Na₂EDTA titre volume is inversely related to adsorption efficiency of the zeolite batch, this data shows that our refined trial, which should have a titre volume lower than 7.62 cm³, is not “refined” at all.

We believe this is due to the “overcooking” of our zeolite batch. We have stated previously that crystallisation temperature determines rate of crystallisation of zeolite A, and crystallisation duration determines crystallisation “reaction” duration. Since zeolite A is a metastable phase of the reaction mixture, the crystallisation duration has to be optimum in order to prevent any more side products which are more stable than zeolite A to form. As at the time we didn’t account for this factor, our zeolite can be concluded as “overcooked”, i.e. it overshot the optimum duration respective to its crystallisation temperature (relative optimum). Therefore, its adsorption effectiveness is less than the said relative optimum.
Data Analysis

General Analysis

From Table 10, it can be seen that the calibrated adsorption capacity of our synthesised zeolites did not exceed 50%. This is a rather low experimental capacity, which can be attributed to two primary factors.

Firstly, we assumed the zeolite framework to be perfect, but in reality, there are very often defects in the framework which may trap some Na\(^+\) ions in their framework cages; in other words, these Na\(^+\) sites are inaccessible to the adsorbate solution. In addition, “Zeolite A has twelve [exchangeable ions] per unit cell, eight of which are ... easily identified by X-ray crystallography” (Sherry 1969). This led Whitehead to suggest “sites located in denser, smaller cages are much less accessible” (2000). If X-ray crystallography could not readily identify the remaining four exchangeable ion sites, it should not be surprising that these sites are rarely accessible and hence seldom participate in ion exchange processes. These inaccessible sites therefore do not participate in the ion exchange process. This causes our adsorption capacity calculations to overstate the said capacity, and contributes to the apparently low cation adsorption ability of the synthesised zeolites.

Secondly, we assumed the complete movement of ions to and from the zeolite framework will be completed by the time we removed the zeolite through filtration. In reality, the diffusion kinetics of cations into and away from the zeolite framework can be rather slow. This implies that even if a perfect zeolite framework has been synthesised, without sufficient exposure duration there will still be adsorbate cations in solution and charge balancing ions within the framework. While we conducted a set of trials which concluded the difference between cations exchanged within a duration of 10 and 20 minutes remains negligible, the same negligible degree of change cannot be guaranteed for longer durations such as 20 minutes and 2 hours. This explains the positive readings of titre volume even after zeolite introduction into the adsorbate solution; in addition, since our adsorption capacity calculations unrealistically assumes complete adsorbate cation adsorption, this overstates the actual maximum adsorption capacity given a 15-minute exposure time, and contributes to the apparently low cation adsorption ability of the synthesised zeolites.

The duration of the batch of materials in the oven is related to the available time for crystallisation that the batch has; the crystallisation of Zeolite A is at its heart a chemical kinetics reaction: its reaction rate is dependent on temperature and the amount of product crystal formed is dependent on duration of reaction.

However, this does not mean the higher the crystallisation temperature, the higher the rate of crystallisation, due to a phenomenon named polymorphism, which refers to the ability of a compound to exist in several physical forms. It can be considered to be a wider definition of allotropy, which is the existence of two or more different physical forms of a chemical element. (Dictionary.com). At different synthesis conditions, different forms of zeolite, which may affect the framework structural integrity of our desired Zeolite A product. We suspect that at lower than optimum temperatures, our product framework integrity will be subpar, decreasing the total number of accessible Na\(^+\) sites; meanwhile at higher than optimum temperatures, the formation of side products other than Zeolite A will become increasingly energetically favourable, which also decreases the total number of accessible Na\(^+\) sites. Our data as presented in Figure 1, discovered the optimum temperature to be...
around 122 °C, where any deviation from this temperature results in a decrease in cation adsorption, which supports our assertion.

Crystallisation duration is also a major determining factor of cation adsorption efficiency or capacity. Ostwald’s Step Rule (van Santen) states that “if a reaction can result in several products, it is not the most stable with the least amount of energy that is initially obtained, but the least stable state, lying nearest in free energy to the original state”. This rule has major implications when applied to the data in our crystallisation duration trials as presented in Table 11 and Figure 2. Since any deviation from the optimum crystallisation duration of roughly 5 hours result in a decrease in adsorption capacity / efficiency, taking Ostwald’s Rule into account, this means that in the hours leading up to the 5-hour mark, our batch is not simply molecules and other chemical species waiting around to be integrated into the zeolite framework; rather, these raw materials have been significantly more active than anticipated, forming different side products that may not even remotely possess the cation exchange capacity that is the prominent feature of zeolites: for example, we suspect formation of amorphous sodium aluminosilicate is prominent especially during early stages of the crystallisation: since an amorphous solid lacks the long range order that a crystal has, therefore the former has a more positive entropy level than the previous one, resulting in its Gibbs free energy being closer to our original synthesis mixture, which means our assumption that the product is entirely defect or impurity free is highly unrealistic. This means our theoretical exchange capacity calculations have overstated the value – the true value should in fact be lower than the current theoretical value, but since we have no method of determining percentage composition of chemical species in a mixture in a high school laboratory setting, we could only offer our analysis of the data. On the contrary, when more than 5 hours have been allowed for the crystallisation to occur, more stable products with a lower entropy level than Zeolite A may be present, which may not have the desired and as easily accessible Na⁺ sites as Zeolite A, which could explain the reason behind decreased adsorption capacity, which supports our assertion that there exists an optimum crystallisation duration for the synthesis of Zeolite A.

While we have not been able to suggest a conclusive trend that relates charge density of metal cations and their adsorption effectiveness by zeolite, we have nonetheless been able to show the versatility of zeolite A in adsorbing different metal cations which are mostly divalent (with the exception of trivalent Fe³⁺) with similar ionic radii. This versatility is one of the primary reasons behind the wide applicability of zeolite A. We have also shown that the zeolite A adsorption effectiveness for Pb²⁺ is higher than that of Ca²⁺, where our theory is two-fold: firstly, we theorise that since the bond between exchangeable ions and the framework itself is predominantly ionic, the increase ionic radius given a fixed space for the exchange site results in a shorter ionic bond length and hence an increased ionic bond strength between Pb²⁺ and the zeolite framework than that of Ca²⁺. Secondly as illustrated by poor copper adsorption by zeolite, the charge density of the cation also comes into play in determining the cation’s zeolite adsorption effectiveness. For comparison, Li⁺ ion is an ion that literature established is only poorly adsorbed by zeolite frameworks as its high charge density forms a compact hydration sphere that requires a significant amount of energy to be “shed”. A similar principle can be applied to the Cu²⁺ ion: since it has a charge of 2+ and an ionic radius of 0.87 Å compared to that of lithium’s 1+ and 0.9 Å, this means Cu²⁺ has an even higher charge density than Li⁺, which explains the lack of copper adsorption by zeolite in our trials.
Significance of Results

The discovery of an optimum crystallisation temperature and duration is significant for the application of Zeolite A as the material can now be synthesised with an optimised temperature range and crystallisation duration which increases the product’s adsorption capacity and efficiency. This in turn makes the production and consumption of the zeolite more efficient, hence reducing the production costs of Zeolite A. The results closely tie into Principle 6 of the 12 Green Chemistry Principles. By optimizing the crystallisation temperature and duration of the synthesis, this thereby reduces excess energy used and thus increases energy efficiency.

Moreover, from all 3 parts, it is observed that the addition of Zeolite has adsorbed metal ions. This signifies that Zeolite will be able to treat waste water or polluted water. This helps reduce potential toxicity of water and thus Principle 4 is addressed as the introduction of Zeolite to waste water has shown great significance in benefits for the environment.

Furthermore, as seen in Figure 1 and 2, the efficiency of adsorption of metal ions will be reduced if the synthesis conditions were not optimised, in other words, some Zeolite and chemicals used for synthesis will be put to waste. The optimum crystallisation temperature and duration found in this research can be referenced to reduce the potential wastage of chemicals used in future synthesis of Zeolite A. As such, Principles 1 is addressed.
Comparative literature

Drawing comparison from other sources that also study the synthesis and application of Zeolite A, 5 different sources has been used to reinforce and validate our experimental findings, with “Introduction” (Flanigen, Broach & Wilson, 2010), “Linde Type-A zeolite synthesis and effect of crystallisation on its surface acidity” (Khan, Reza, Razzaque, & Alam, 2010), “Synthesis and characterization of Zeolites from sodium aluminosilicate solution” (Ibrahim, 2007), and “Synthesis of Zeolite A: A Review” (Petrov & Michalev, 2012) being papers with findings on the synthesis conditions of Zeolite A; while papers on the application of Zeolite A on different metals include “Sorption of Lead and Cadmium Cations on Natural and Manganese-Modified Zeolite” (Anielak & Schmidt, 2010), and “The Application of Synthetic Zeolites for the Removal of Heavy Metals from Contaminated Effluents” (Kate Whitehead, 2000).

Synthesis conditions

Conditions for zeolite synthesis will first be discussed. Our experimental data suggests that a crystallisation temperature of 115°C for a constant crystallisation duration of 180 minutes (3 hours) is the optimal condition for the synthesis of Linde type A Zeolite. Studies such as by (Flanigen, Broach & Wilson, 2010), (Khan, Reza, Razzaque, & Alam, 2010) and (Ibrahim, 2007) all suggest that the optimum temperature for Zeolite A synthesis is at 100°C, where the study on “Linde Type-A zeolite synthesis and effect of crystallisation on its surface acidity” has specifically delved into various synthesis conditions, with a constant time of 4 hours and with temperatures from 80°C to 160°C being tested at 20°C intervals, with surface acidity as their method of measurement of zeolite quality. Therefore, a deviation of 15°C from the literature value is found. However, given the different crystallisation duration (3 hours for our temperature trials vs 4 hours in literature) and considering the slightly different synthesis methodology, it can be understood that our data for effect of temperature investigation is rather accurate with only 15% deviation from literature and is therefore valid.

For our investigation into the optimum time for zeolite synthesis, our results concluded that for a crystallisation temperature of 90.0°C, 5 hours is the optimal time for crystallisation for the synthesis of Zeolite A. However, from “Linde Type-A zeolite synthesis and effect of crystallisation on its surface acidity”, a rather different result has been obtained, where they have found a direct relationship between crystallisation duration and effectiveness of zeolite (in terms of surface acidity), where “The acidity increased sharply until 5 h and then gradually increased to prolong time.” This means the longer the crystallisation duration the better quality of zeolite and higher efficiency in metal adsorption, suggesting there is no optimum. However, another paper by Petrov and Michalev “Synthesis of Zeolite A: A Review” refutes the idea of a direct relationship and suggests that there is an optimum temperature: “Reaction time. Crystallisation parameter must be adjusted to minimise the production of the other phases while also minimizing the time needed to obtain the desired crystalline phase.” This implies that the optimal time is the minimum time needed to obtain the desired crystalline phase, that is the cubic/tetrahedral crystals of Zeolite A, and that there exists a notion of optimal time. This suggests that our experiment is indeed valid and is proven in a wider scientific community to a certain extent. Moreover, another important piece of information is on the minimum time required to crystallise and obtain functional zeolite. In “Linde Type-A zeolite synthesis and effect of crystallisation on its surface acidity”, it is mentioned that:
“At the crystallisation duration 1 h, the surface acidity is found to be 0.55 meq/g which is almost the same as of the amorphous alumino-silicate catalyst. This finding reveals that the nucleation of the crystals does not start within 1 h. The activation time for crystallisation is 2 h. The surface acidity starts increasing after 2 h of crystallisation duration.”

This suggests that any crystallisation duration below 2 hours will most likely results in dysfunctional Zeolite and an incomplete framework, which provides explanation and insight to why our 1 hour batch has a surprisingly high titre volume (25.40 cm$^3$), higher than blank titration of 21.60 cm$^3$ of 18$^{th}$ Feb blank titration, and explains why zeolite of this batch has ‘disintegrated’ as all solution turns blue given enough time when added with calcium nitrate and indicator, regardless of the addition of EDTA. Thus it is seen that even on minute details, our experimental results are relevant and coherent to that of the scientific community.

Our experimentation on synthesis conditions can therefore be deemed scientifically valid and generally accurate.

**Metal Ions**

One of the many uses of zeolite is to decontaminate water via metal ion adsorption, due to its ion exchange properties. This induced us to investigate on the adsorption of different metal ions. In our investigation, we tested for metal ion adsorption of zeolite with the following metal ions. Lead (II) ion, Copper (II) ion, Calcium ion, Iron (III) ion. The results were that there was metal ion adsorption in Lead (II) ion, Calcium ion, Iron (III) ion. However, for Copper (II) ions, there was no huge change in metal ion. (The results are reflected by the volume of EDTA used).

To compare, other studies also mostly indicate a positive result in the adsorption of metal ions. In a paper published by Anna M. Anielak and Rafal Schmidt, it was held that:

“Our work shows that active masses modified with manganese that are used to remove Mn(II) and Fe(II) can also remove the heavy metals Pb and Cd. The study was conducted for solutions with a concentration of cation 100 mg/L and demonstrated the high correlation between hydroxo-complexes and their potential, and the process of removing investigated metals (Pb and Cd). (Anielak & Schmidt, 2010)

This states that the active masses, zeolite, can remove heavy metals Pb and Cd; on top of that, in another research by Kate Whitehead, it was stated:

**Allen et al. monitored the adsorption of trace levels of heavy metals (100 μg/l of zinc, copper, cadmium, and nickel and 50 μg/l) whilst assessing hydrolysis effects, giving the following selectivity series: Pb$^{2+}$>Cd$^{2+}$>Zn$^{2+}$>Cu$^{2+}$>Ni$^{2+}$ (Allen 1983)**

This tells us that these Zeolite adsorption includes these metals, with Lead having a high selectivity in being adsorption. This Is concurrent with our results regarding lead. As for copper (II) ions, again referring to the paper by Allen et al, it is seen that Copper (II) ion is low on the selectivity series. Perhaps we did not allow for enough time in stirring or contact time for zeolite in order to allow for it
to be adsorbed into zeolite, thus explaining the small change in our data taken. In the same paper, it was stated:

The zeolite frameworks are dissolving and possibly re-precipitating as new aluminosilicate products due to the pH-correction and hydrolysis processes, calcium complexes may be adsorbed onto these degradation products, and therefore removed from solution.

This means that calcium complexes are adsorbed by zeolite, thus giving support to our results that calcium content decreased after contact with zeolite.

As for iron, an investigation on the effect of natural zeolite on water treatment, they found out that “Fe$^{2+}$ and Fe$^{3+}$ ratio results in sorption of iron ions and iron oxihydroxides on the surface and in the pores of clinoptilolite.” (Margeta, Logar, Šiljeg and Farkaš) And also referencing the previous paper, from Anna M. Ainelak, we see that zeolite has been used for the adsorption of iron ions (although this is for Iron (II) ion. This coheres with our findings that presence of Iron (III) ions greatly reduces after Zeolite adsorption.

To sum up, other research supports our results for Lead (II) ion, Iron (III) ion, Calcium ion. For copper ion, it may be due to its low order in the activity series its high charge density which its strongly attracted hydration sphere may have prevented Zeolite A adsorption. The other papers also used other types of zeolite, explaining our negative result using Zeolite A. In looking at other research papers, it seems that more metal ions could have been tested in order to further our research, such as cadmium and Nickel. However, the conclusion is that our research is deemed accurate and valid in terms of zeolite application in adsorption of metal ions.
Conclusion

With reference to the above comparison with contemporary literature, we can see that our results are similar to other researchers.

To recapitulate, our results indicate for the synthesis of zeolite, in terms of crystallisation temperature, the optimum temperature was determined by the experiment to be at 115°C Celsius, however, after interpolation, the optimum temperature is determined to be at 122°C Celsius.

For oven time, the crystallisation duration was determined by the experiment to be at 5 hours (or 300 minutes), whereas interpolation determined the optimum temperature is 5 hours, 23 minutes (or 323 minutes).

In determining the reliability of data of these two findings, the percentage uncertainty and $R^2$ value has to be considered. In the results from the determination of optimum temperature of synthesis condition of zeolite, the percentage uncertainty is relatively low, all of them within the range of ±1.16% and ±3.56. Moreover, the error bars for the optimum temperature, 115°C Celsius, does not overlap. This means that there is significant difference between the increments, and that 115°C Celsius is indeed the optimum temperature. For the interpolated value, it is calculated with the equation of the best fit line. As an indication of the reliability of the best fit line and hence reliability, the $R^2$ value is used. The $R^2$ value of the best fit line in Temperature is 0.6363, although this $R^2$ value is not high, but bearing in mind that the interpolated value of 122°Celsius is close to the experimental value, the interpolated value could serve as a reference point as to where the optimum temperature is.

For the determination of the crystallisation temperature, again the percentage uncertainty is very low it is only between ±0.39% and ±2.60%. This means that the deviation is small and the results are generally reliable. Although the error bars do overlap each other, we can still clearly see zeolite is most effective with a crystallisation time somewhere near 3 to 5 hours. In terms of the interpolated data, again, the reliability of it will determine on the $R^2$ value since it is calculated with the equation of the best fit line. The $R^2$ value of the equation is 0.791, which is high, this shows that the interpolated data is high and that the optimum time is 5 hours, 23 minutes. The low percentage uncertainty and high $R^2$ value allows us to accept the data.

As to the application of zeolite, specifically, the adsorption of metal ions, no notable conclusion could be gained from the comparison of deionised water, tap water and seawater. However, we found out that zeolite is able to adsorption (adsorb) various metal ions (of different ionic charge, and ionic radius). This ranges from being able to adsorb lead (II) ion, of 1.33 Å and 2+ charge of and calcium ion, of 1.14 Å with 2+ charge, Zeolite is able to adsorb Iron (III) ion, with a charge of 3+, but to a much smaller extent than that of the others. Zeolite is not able to adsorb copper (II) ion with 0.87 Å with 2+ charge.

As to the reliability of these results, only the percentage uncertainty is of concern. In general, the percentage uncertainty is small, for adsorption of Lead (II) ion it is ±3%, for adsorption of Calcium ion, it is also ±3%, for Iron it is ±4%. The small percentage uncertainty allows us to accept our data.

The results we obtained allowed us accept our hypotheses 1 and 2 with slight variations. Instead of the optimal temperature of crystallisation for zeolite synthesis is 99.25°C”, it is between
115°C and 122°C and the optimal time of crystallisation for zeolite synthesis is instead of being between 180-240 minutes, it is between 300 and 323 minutes. However due to the data received, it we reject Hypothesis 3, “There is a negative correlation between charge density of metal and zeolite adsorption efficiency, the smaller the radius the more efficient.”. This may be due to other factors affecting the adsorption of metal ions by zeolite.

As to hypothesis 3, it is partially accepted. In the results we have obtained, in prima facie understanding, there is no discernible relationship. Hence we cannot accept the hypothesis that “There is a negative correlation between charge density of metal and zeolite adsorption efficiency, the smaller the radius the more efficient.”. However, in our further investigation done, after plotting a graph, there is indeed a sort of negative correlation. For the same ionic charge but different charge density with the percentage adsorption by zeolite (percentage adsorption is used to represent adsorption efficiency), there is negative correlation. On top of that, there are only three data points on the graph with one outlier (Iron (III) ion). This makes the relationship seen in the graph unreliable, and hence there is only a weak correlation between the independent and dependent variable. This means we cannot fully accept hypothesis 3. (Please refer to further investigation part for more details.)

With these results, we have found out how to best synthesise Zeolite A, and what types of metal ions can be adsorbed by this type of zeolite. This allows us to produce a type of zeolite that could be used as a decontaminator for polluted water. However, on the other hand, we have also found out that presence of extraneous variables may interfere with adsorption, especially in deionised water, tap water and seawater. This may mean that the water zeolite is added to has to be appropriate for decontamination.

Extension (Conclusion for refined trial):

As in extension, a refined trial, with the optimum crystallisation temperature and duration used was done. The volume of EDTA used in the refined trial was calculated with the mean of 13.07 cm³. However, at 115°C, at 3 hours (the optimum in the investigation of optimum in temperature), the volume of EDTA used was (8.00 ± 0.10) cm³. Whereas at 5 hours, at 90°C (the optimum in the investigation of optimum in duration), the EDTA used was (7.69 ± 0.30) cm³. Since 13.07 > 8.00 > 7.69, and the less EDTA used, the more adsorption by zeolite, this tells us that the adsorption efficiency in the refined trial was actually lower than that during investigation, and is less efficient than expected. To conclude this, perhaps the optimum of crystallisation conditions is relative to each other. That is, the optimum crystallisation temperature depends on the crystallisation duration, and the optimum crystallisation duration depends on the temperature. Perhaps the real optimum crystallisation should be found by doing an investigation of optimum duration for each temperature increment, and the optimum can be found via comparing the optimum of optimums.
Evaluation

Three aspects of our experimental results are to be discussed, namely: validity, reliability and accuracy.

Validity

Validity of data refers to whether data collected is relevant to experimental objectives. In this broad scoped experiment for the synthesis of Zeolite A, we have three experimental objectives we intended to investigate:

1. Investigate effect of crystallisation temperature on zeolite synthesis
2. Investigate effect of crystallisation duration on zeolite synthesis
3. Investigate zeolite adsorption efficiency of various metal ions

The experiment data we have collected are relevant to our experimental objectives in this case:

Regarding the first experimental objective, the first section of our experiment has been designed specifically to investigate the different temperatures of crystallisation of zeolite has on the quantity and quality of zeolite, which we assume to be perfectly functional due to lack of characterisation methods, as measured by the different titre volume of EDTA by titrating with 25 cm$^3$ 0.01M of Ca(NO$_3$)$_2$(aq), with which from the temperature of 70.0 to 130.0 °C with constant time of 3 hours. The optimal temperature for crystallisation is found to be 115°C with a titre volume of 8.42 cm$^3$.

Regarding the second experimental objective, the second section of our experiment has been designed to investigate the different time of crystallisation of zeolite through baking in oven for 90°C degrees has on the quantity and quality of functional zeolite measured by titre volume of EDTA, ranging from 1 to 9 hours. From the experiment the optimal time for crystallisation is found to be 5 hours, with a titre volume of 7.69 cm$^3$.

Regarding the third experimental objective, the third section of our experiment has been designed to apply our refined zeolite into testing various metal salts and real life substances, in order to investigate Zeolite A’s adsorption efficiency. Efficiency is affected by the charge density of the metal ion, measured either as the volume of EDTA used in titration or the amount of HCl$_{(aq)}$ consumed in NaOH$_{(aq)}$ back titration (two different methods, one for transparent solutions, another for coloured solutions; refer to Methodology for details). From the experiment results, we found that zeolite has different adsorption quantity for different metal ions. However, the only constraint here is that there may be too many variables affecting the results such as ionic radius, charge, number of valence electrons of ion, so that although different Zeolite adsorption efficiencies are found for different metal ions, the exact trend and reason for which causes these behaviour is not conclusively found in our experiment, i.e. a difference in efficiency is found but a trend cannot be established. The 2 methodologies are versatile in measuring various metal ions: the EDTA method for measuring group 1 and 2 metals where the ions are colourless and does not form precipitate easily, while the precipitation back titration method can be used for transition metals where titration with colour is hardly visible but metal ions form insoluble salts easily. To find trends, more extensive experimentation is required (see Further Research).
As all three of the experimental objectives are successfully investigated, the experiment and investigation are therefore valid.

**Reliability/Precision**

Reliability of data refers to whether experimental data is repeatedly obtainable, and is based on the precision (distribution of) experimental results from random error. They are affected by imprecision of instruments, steps in methodology, and quantity of trials.

**Strengths:**
1. Procedures in the synthesis of zeolite and application testing has been optimised, i.e. minimal steps, such that the accumulation of uncertainty of instruments and random is minimal, increasing precision.
2. The use of precise digital instruments such as electronic balance (±0.001 g), 10 cm$^3$ micropipette (±0.1cm$^3$) allows lesser accumulation of instrumental uncertainty, increasing precision of data.

**Limitations:**
1. There is a lack of trials for experimental data collection, such that there is imprecision of the calculated mean (mean is calculated with only 3 data points in temperature section, 3 trials in time section, 3 trials in application testing), i.e. the data points may not reflect the ‘true value’.
2. Trials of zeolite synthesis has been done in two separate occasions, one in January, and in February, thus the February results has to be calibrated against January's data, leading to increase in uncertainty and imprecision.

**Potential improvements:**
1. Do more repeated trials of each procedure in order to reduce the effects of random error and increase the precision of the experiment results, such that results will deviate less than the ‘true value’, increasing reliability of results.
2. Avoid doing experiment in separate temporal conditions.

**Accuracy**

Accuracy of data refers to whether the experimental data collected is close to the true/literature value. It is a result of systematic error, which is influenced by human, methodological, and instrumental (faulty instruments) factors. As this experiment consists of three major parts (temperature, time, application), the analysis of precision of data will be conducted in three separate sections.

**Experimentation of crystallisation temperature on zeolite synthesis**

**Strengths:**
1. Methodology for this section is optimised, with no wrongly planned steps such that accurate measurement can be facilitated.
2. Oven temperature can be accurately determined as it is controlled by inbuilt thermostat.
3. Despite the 6 increment trials span over 2 experiment days (25 January and 18 February), the discrepancies of the data are calibrated through comparing blank trial tests as well as internal justification (see Appendix 2 and 4).
Limitations:
1. Endpoint of titration is determined by human observation and may not reflect the equivalence point of titration.
2. Stands and burette clamps provided by the lab are tilted slightly, which may slightly affect the measurements obtained due to parallax problem of reading tilted burette.
3. Mass of sodium aluminate and sodium metasilicate are slightly unequal, as sodium metasilicate is often clumped into large crystals and finding exactly 15.480g of sodium metasilicate crystals is a far-fetched task. Common practice is to accept to nearest decimal place for practical reasons (except refined trial). As such there might be slight excess of either sodium aluminate or sodium metasilicate in our final product, which to a small extent affects accuracy of results.
4. Some zeolite particulates may have not been completely filtered by gravitational filtration and may have pass through the pores of filter paper

Potential improvements:
1. Spectroscopic approach can be used instead of volumetric approach to quantify results to address the first limitation, where a coloured transition metal salt can be used in place of calcium nitrate, where a spectrophotometer can be used to measure the extent of the change (decrease in absorbance) of a solution after the addition and extraction of zeolite. However, this has not been adopted because of the third limitation, because excess dissolved sodium metasilicate when using zeolite may make the solution slightly cloudy, affecting solution absorbance, resulting in unwanted accuracy. It can be adopted however if exact measurements are used, and if no excess of sodium metasilicate is present.
2. Use newer and less faulty instruments
3. Measure exact mass of each measurement, but chiselling a large sodium metasilicate crystal into exactly 15.480g is very time consuming and impractical.
4. Use filters with finer pores, such as film composite membranes, or advanced filtering methods such as syringe filtration or microfiltration.

Experimentation of crystallisation duration on zeolite synthesis

Strengths:
1. Methodology for this section is optimised, with no wrongly planned steps such that accurate measurement can be facilitated.

Limitations:
1. Crystallisation time cannot be accurately determined as it is affected by the time we put and extract from oven, which is affected by our school timetable and other users of the lab, as we can only take out during non-class periods and queue for other users, making our time of extraction slightly inaccurate. However, all time deviations have been less than 5 minutes.
2. Endpoint of titration is determined by human observation and may not reflect the equivalence point of titration.
3. Stands and burette clamps provided by the lab are tilted slightly, which may slightly affect the measurements obtained due to parallel problem of reading tilted burette.
4. Mass of sodium aluminate and sodium metasilicate are slightly unequal - refer to limitation 3 in section above.
6. Some zeolite particulates may have not been completely filtered by gravitational filtration and may have pass through the pores of filter paper

Potential improvements:
1. Spectroscopic approach as an alternative to volumetric analysis - refer to improvement 1 in section above.
2. Use newer and less faulty instruments
3. Measure exact mass of each measurement - refer to improvement 3 in section above.
4. Use filters with finer pores, such as film composite membranes, or advanced filtering methods such as syringe filter or microfiltration.

Experimentation on Zeolite adsorption efficiency of various metal ions (application testing)

Strengths:
1. Methodology is versatile, EDTA titration method for transparent solutions that cannot form insoluble metal hydroxides, precipitation and NaOH back titration method for coloured transition metal solutions where indicators can’t be clearly seen but forms insoluble metal hydroxide precipitates. This allows for an accurate measurement in each separate case and solution analysed, as each method can accommodate their respective metal solutions with clear results. Difference in adsorption efficiency of different metal ions can be accurately distinguished.

Limitations:
1. Endpoint of titration is determined by human observation and may not reflect the equivalence point of titration, especially for determination of deionised water, tap water and seawater with EDTA (sodium chloride requires this method), as the colour change is from light bluish purple to dark blue, which is often hard to perceive.
2. Stands and burette clamps provided by the lab are tilted slightly, which may slightly affect the measurements obtained due to parallax problem of reading tilted burette.
3. Though the 2 separate methods are versatile, the results may not be accurately comparable, for one is the volume required for complete chelation of metal ions in analyte, the other the volume (deducted from back titration) needed to completely precipitate all of the analysed metal salt in the solution, where they are only accurate in each case.
4. Uncontrollable variables in different metal ions contribute to the efficiency of adsorption, as ionic charge, ionic radius, relativistic effects (such as copper being a noble metal) from the different groups and periods of metals all play to the different efficacy and efficiency of different metal ion adsorption of zeolite

Improvements:
1. Spectroscopic approach as an alternative to volumetric analysis, refer to section above
2. Use newer and less faulty instruments
3. Use more extensive methods to test the various metal ions (but may require currently inaccessible equipment), see further research.

Thus it is seen that for the application testing of different metal ions, we can indeed test and see a difference between which metal salt can be adsorption by zeolite (or cannot), but we cannot
establish a trend based on separate variables, as it is impossible to separate such variables by methods available in a school laboratory setting.

**Refined Trial experimentation**

On the data obtained for refined trial, through comparison with the optimum temperature data (115°C 3 hours constant) having 8.00 cm³, and optimum time data (5 hours with 90°C constant) 7.69 cm³ volume, However the combination of both time and temperature optimum did not result in optimum adsorption efficiency, reflected by titre volume of 13.07 cm³. This trial is valid in a sense that though the results are not expected, as we expect the lowest titre volume, it allowed us to apprehend that optimal conditions is relative, and that thorough testing for every single combination of synthesis condition is needed to find the optimum is relative optimum that is closest to ‘true’ value. This also suggests that our data is rather accurate, because the identification suggests that our optimum conditions deviated from the literature optimum which is true as we the literature temperature is 100°C. The fact that we used three trials also indicates that such a conclusion of relative optimum is indeed valid.

From the various sources of error and aspects of our experimental results, we found that even though we may not have the most accurate nor the most precise results and experimental methods, we do indeed have sufficiently clear results to establish a trend, with which we can justify scientifically. Hence it is safe to say that our data is rather reliable, and valid, to our objectives and in a scientific context.
Further Research

This section aims to elaborate on further investigate topics of Zeolite and to address several conundrums encountered in our experiment.

Spectroscopy

As mentioned in improvements, a spectroscopic approach can always be an alternative to measure the concentration change of metal ions before and after zeolite treatment of temperature and time investigations, using a calibrated spectrophotometer to measure the complementary colour of a certain ion, for example violet colour (~450nm) for an iron sulphate analyte, with the extent of concentration change of the solution measured by decrease in absorbance or increase in transmittance after zeolite treatment. This method would allow accurate quantification of data that would be more accurate than titration which is determined by human perception of colour change.

However, it may not be the most reliable method due to the presence of impurities such as excess sodium metasilicate and unfilterable zeolite particulates (both causes cloudiness and reflects light not uniformly). Removing such impurities may cost much time and manpower if one were to use microfilter to filtrate (time consuming, and not conventionally available in school laboratory) or chiselling sodium metasilicate crystals. However, should such measures be employed spectroscopy will be a viable alternative for volumetric analysis in this experiment; perhaps it will be interesting to compare the data recorded by spectroscopic analysis against volumetric analysis.

Reusability

A question we have encountered during our experiment is: “Are Zeolite reusable and how does reusing affect the adsorption efficiency of Zeolite?”

Throughout the experiment, we have investigated into several key aspects of the synthesis process of zeolite. However, we did not take into the account one of the most promising and unique feature of zeolite - its reusability. Unlike activated carbon filters which require laborious processes to reuse the material, Zeolite is found to be reusable for 22 times in composting process (Zorpas). Theoretically, the adsorption efficiency would decrease as the number of times of reusing increases.

Investigation on the reusability of zeolite will be an interesting extension to our current experiments. Further experiments can be performed on obtaining the optimal number of times of reusing Zeolite in water purification, for it will provide a more complete and thorough profile of the mineral Zeolite.

Identification of the product

From the investigation in sections above, we have often come across the notion that the optimum condition provides the best zeolite, that is zeolite with the least titre volume (more metal ions exchanged, the lesser the remaining in surrounding solution, thus less titrant needed). This is how we measure adsorption efficiency as well; from above sections we also know that efficiency is related to exchange capacity. But this begs the question: What determines the adsorption efficiency of zeolite, does the optimal conditions allow for the largest quantity of zeolite produced, or the best quality
(most capacity), or both? In fact, our current experimental procedures do not provide insight on such a topic, however the direction of quality has been hinted at in one of our pilot tests:

Calcium nitrate solution has been used with Zeolite synthesised with 1 hour. Titration ends when solution turns blue. However, in this batch, all solution turns blue given enough time. This indicates that zeolite quality may be a factor in the adsorption efficiency of metal ions, and of experimental results, as this batch is only crystallised for 1 hour, zeolite may not have formed properly, so that not all of the product have been zeolite, or once was but decomposed into back into sodium aluminate and sodium metasilicate. So, we are faced with another question: How can we be sure that the product after synthesis is purely zeolite?

A method to distinguish our products is needed. Methods such as our regular volumetric analysis or spectroscopy can only indirectly indicate whether our zeolite functions, which is useful. However, if a more direct approach is needed to identify our product, methods such as X-ray diffraction and X-ray crystallography are required to examine the molecular structure of the product zeolite. That however will be rather impractical and out of the scope and resources of this experiment. However, this will be an interesting direction: investigating how the molecular structure of Zeolite affects its chemical properties of chelation.

Nevertheless, identification of our product has been an ongoing conundrum in our experiments and we could only assume that most of our products are completely functional zeolite - at least according to our reference source for synthesis procedure, the percentage yield should be effectively 100% (International Zeolite Association).

**Effect of moisture**

Moisture is a rather surprising topic we came across during experimentation. In our initial synthesis of 90°C and 100°C (18th Feb) we accidentally forgot to place some our zeolite inside the desiccator. Thus our anhydrous Zeolite A became hydrated Zeolite A, which its hygroscopic properties have absorbed water vapour in the atmosphere. With this incident, we decided to investigate whether there would be a difference in adsorption efficiency, i.e. titre volume of this hydrated batch against anhydrous batch of 90°C and 100°C (18th Feb); as expected, our titration with calcium nitrate solution results were vastly different (refer to Appendix 1). It is seen not only that hydrated zeolite has a much lower adsorption efficiency than anhydrous zeolite, less calcium ion adsorption, implies there is more calcium ion in surrounding solution, meaning more EDTA is required for titration: Moistened trials of 90°C taking 17.10 cm³, 100°C taking 19.50 cm³; while dry trials of 90°C taking 10.93 cm³, 100°C taking 10.93 cm³; a 56.45% increase in wet from dry 90°C, and a 78.41% increase in wet from dry 100°C. It is noteworthy to mention that the 19.50 cm³ of hydrated 100°C is similar to that of the blank trial 21.60 cm³, so it is seen that moist conditions for the storage of zeolite is very undesirable for the storage of zeolite, to the extent that it may affect the quality of zeolite. Perhaps this is the manifestation of the principle of serendipity of science.

Our investigation did not consider the effect of moisture in Zeolite affecting the adsorption efficiency. If we could quantify the size of effect, the results would provide a practical indication of the proper method to store Zeolite for future use. Perhaps this question could be of use in future investigation: “How does the moisture content in Zeolite affect its adsorption efficiency?”
Effect of pH

pH is also a variable that affects Zeolite adsorption of metal ions, as seen in literature (Kabwadza-Corner, Johan, & Matsue). The structural stability of most zeolites is dependent on pH - if the pH value of the solution the zeolite is put into solutions of pH < 4, the zeolite structure may begin to disintegrate. The disintegration of zeolite structure is much more evident, and happens on a much larger scale in solutions with a pH value < 3. With the collapse of zeolite structure, the amount of metal ions that can be adsorbed by the framework is significantly less.

We can see the impact of the pH value of the solution on the metal ion adsorption efficiency from reviewing research from outside sources. In Kabwadza-Corner, Johan and Matsue’s investigation, they researched the Pb ion adsorption of various types of zeolites, with varying pH values of the Pb(NO₃)₂ solution (the source of Pb ions). From the data they have obtained, we can see that when the pH of the solution drops, the adsorption of the Pb ions also drops.

Although we have not investigated the effect of pH on the adsorption efficiency of metal ions in our investigation, the effect of pH on metal cation adsorption efficiency is a promising topic of further investigation. pH of a solution can be controlled using a buffer solution and its effect on adsorption efficiency can then be measured through volumetric or spectroscopic methods.

Atomic structure on metal ion adsorption

As we can only find the different of various metal ions on Zeolite adsorption efficiency, but not trends, investigation on various variables on metal ion itself can provide insight.

Ionic radius

Ionic radius is determined by the period (down period larger) and group (across group smaller), where the effective nuclear charge from number of protons (nuclear attraction) and electron repulsion from lower shell and same shell electrons come impacts upon. It plays a role in zeolite adsorption efficiency, as zeolite forms ionic bonds with metal ions. As bond strength is affected by bond length (shorter bond being stronger), which in turns affect the amount of energy required to break the bond and subsequently when released. As such, it is understood that smaller metal ions would form shorter and stronger bond with zeolite, and requiring more energy to break it, while larger metal ions forms longer and weaker bonds with zeolite, less energy required in bond breaking. So it can also be implied that zeolite will be more efficient in exchanging smaller metal ions than large ones and will prioritise exchanging smaller ions over large ones (see competing metal ions below). However, the trend may not be the case as there are other variables.

Ionic charge

Ionic charge is determined by which group the metal ion is (number of valence electrons). Ionic charge affects the strength of the ionic bond between zeolite and metal ion. An ion with a higher ionic charge, i.e. more valence electron will form stronger ionic bond, as there will be more electrostatic attraction (from more electron transferred) between cation(s) and anion(s). This indicates that metals from group 2 will form stronger bonds and is more preferable to be adsorbed by zeolite than group 1, group 3 than group 2, etc. This concept can be combined with ionic radius as a macro concept of charge density (see below)
Another important note on ionic charge is the exchange capacity of zeolite. As each zeolite A can hold 12 positive charges, it means that zeolite of a fixed quantity can only adsorb a fixed number of ions, which 12 group 1 ions, and 6 group 2 ions etc.

Therefore, it is seen that ionic charge does in fact contribute to the extent of adsorption of metal ions by zeolite.

**Charge density**

Charge density is affected by both ionic radius and ionic. When an adsorbate ion enters the zeolite framework, it must “shed” its hydration sphere (Kate Whitehead). As a result, the enthalpy of hydration of different metal cations has a significant effect on its adsorption percentage efficiency. Since enthalpy of hydration is then related to the charge density of the cation, we investigated further by graphing the percentage adsorption efficiency against each cations’ charge densities which is calculated according to the following equation:

\[
\text{Charge Density} \propto k = \frac{q}{r^3}
\]

Figure 3. Charge density of metal ions used in application testing trial on Adsorption % efficiency

Where \( q \) is the charge on the cation and \( r \) is the cation’s ionic radius, while \( k \) is some number directly proportional to the cation’s true charge density. Referring to Figure 3, The resulting trendline is linear, suggesting a negative proportionality between the \( k \) value and the percentage efficiency. There is an exceptionally strong correlation as the \( R^2 \) value is 0.9962 which is very close to 1. This suggests a primary factor in predicting the adsorption percentage efficiency of zeolite A on a divalent metal cation is its charge density provided constant ionic charge – with same ionic charge, the higher the charge density (from increasing ionic radius), the less the percentage efficiency.

This can be the reason which copper ions has not been adsorbed by zeolite. Lithium 1+ ion is an ion that cannot be adsorbed by zeolite, the reason being its high charge density (1+ charge, 0.9Å) from its compact hydration sphere. Similar can be applied to the copper (II) ion, as it has an ionic radius
of 0.87Å and ionic charge of 2+, which has a higher charge density than lithium ion (smaller ionic radius and higher charge). Thus its lack of adsorption by zeolite can be understood.

However, there are severe limitations in the derivation of this conclusion. Firstly, there is a significant outlier which is the percentage efficiency of Fe\(^{3+}\) ion. Granted the two data points cannot be fully compared in that the methodologies to determine its percentage efficiency are entirely different; however, this is still a conundrum in investigating the effect of charge in charge density on adsorption efficiency – a glaring indicator that the crude theory proposed above is still far from completion. Secondly, there are only three independent variables investigated which is far from the minimum number of said variables required to draw a reasonably legitimate conclusion: 3 types of metal ions for 2+ charge (Fe\(^{2+}\), Ca\(^{2+}\), Pb\(^{2+}\)) and 1 for 3+ charge (Fe\(^{3+}\)). Given our time and material constraints we were unable to perform more trials on divalent ions of different ionic radii; however, given the trend is promising, we highly suggest further research into the effect of charge density on adsorption percentage efficiency of zeolite A. We also suggest investigating the respective trends for monovalent and trivalent ions.

**Oxidative potential**

Oxidative potential is the tendency for an ion to lose electrons (metals ions have relatively high oxidative potential) This has a certain effect on zeolite adsorption efficiency, because the likeliness of losing electrons determines the tendency to form an ionic bond and the priority compared to other metals to form a bond. The higher the oxidative potential, the more likely it will form ionic bonds compared to other metals that have lower oxidative potential, provided their ionic charge is the same and ionic size is similar. This effect however is often overshadowed by the two former factors. This effect may provide direction on the following problem: priorities of metal ions in zeolite adsorption, elaborated below.

**Other types of zeolite**

On top of Zeolite A with sodium as exchangeable ion, there are other types of zeolites and various viable exchangeable ions to choose that can be further investigated. Zeolites with other exchangeable ions such as potassium may provide an interesting direction, as increased ionic radius from having an extra shell of electrons may well affect the overall charge stability of zeolite. It may even be interesting to compare on the difference of using potassium chemicals in place of sodium, i.e. potassium hydroxide, potassium aluminate, potassium metasilicate has on the results of synthesis.

Readings from other sources also shows that zeolites can be modified with other metal ions in order to change the functionality and characteristics of zeolite such as how zeolite modified by manganese can allow it to adsorption heavy metal ions such as Cadmium and increase its efficiency in adsorbing lead: “Our work shows that active masses modified with manganese that are used to remove Mn(II) and Fe(II) can also remove the heavy metals Pb and Cd.” in ‘Sorption of Lead and Cadmium Cations on Natural and Manganese-Modified Zeolite’. Another example is how iron modified zeolites can increase its affinity in adsorption of arsenic oxyanions, “the common property of all iron modified zeolites is a high increase in sorption capacity for arsenic oxi-anions present in
water solutions.” in “Natural Zeolites in Water Treatment – How Effective is Their Use” (Margeta, Logar & Farkaš).

Though these are other types of zeolite, the significance of modifying zeolites to increase its chemical properties and versatility in application is very intriguing and fascinating nonetheless, and perhaps it may give insight into investigating on how to modify Zeolite A with other metal ions to increase its adsorption capacity for copper (II) ions. But modification of zeolite is only effective to a certain extent, where in order to access other functions other types of zeolites may be needed.

Apart from Zeolite A, there are other types of zeolites which can be investigated, where they all have different synthesis conditions and different functions due to their different crystalline structures. Most notable examples are: cubic zeolite Y (Faujasite) for catalytic cracking, trigonal Chabazite that can adsorb smaller metal ions, and monoclinic heulandite that can act as an oxygen concentrator (International Mineralogical Association). Other types of zeolites such as natural zeolite, zeolite B, zeolite chabazite, zeolite H, zeolite P exist, and they may have other purpose such as in industry, medical frontiers or simply just as gemstones, but such is only a few examples among a sea of 232 zeolites, according to the “Database of Zeolite structures” (Baerlocher, McCusker and Olson). Whether natural or synthesised it mayhap be a meaningful endeavour in investigating the various unique zeolite frameworks.

**Final Note**

Despite several limitations, this research proposes a sound and justified conclusion in the optimal synthesis condition and the metal ions adsorption efficiency of Zeolite A. It should be noted that there is still a lot more to be researched on the topic for Zeolite. In respect of Green Chemistry and other significances of Zeolite A, we hope to see that the results from this research can be put to practical use in the near future, as everyone in our research team hopes to contribute to the society using our chemistry knowledge.
Appendix – Raw Data

Appendix 1 – Test for Effect of Moisture

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Appendix 2 - Internal justification between two days

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Appendix 3 - Stirring Duration

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### Appendix 4 – Blank Trial

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</tr>
<tr>
<td>Trial 3</td>
<td>21.70</td>
<td>43.70</td>
<td>22.00</td>
</tr>
<tr>
<td>Trial 4</td>
<td>0.30</td>
<td>20.80</td>
<td>20.50</td>
</tr>
<tr>
<td><strong>Average</strong></td>
<td><strong>5.60</strong></td>
<td><strong>27.20</strong></td>
<td><strong>21.60</strong></td>
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</table>
### Appendix 5 – Raw Data for Oven Temperature Trials

#### 25 January

<table>
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<th>Temperature</th>
<th>70.0</th>
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<th>90.0</th>
<th>100.0</th>
<th>115.0</th>
<th>130.0</th>
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</thead>
<tbody>
<tr>
<td>Trial no.</td>
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<td>2</td>
<td>3</td>
<td>1</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>Initial Volume</td>
<td>17.40</td>
<td>0.00</td>
<td>8.70</td>
<td>17.10</td>
<td>26.10</td>
<td>34.10</td>
</tr>
<tr>
<td>Final Volume</td>
<td>26.10</td>
<td>8.70</td>
<td>17.10</td>
<td>25.90</td>
<td>34.60</td>
<td>43.30</td>
</tr>
<tr>
<td>Volume Used</td>
<td>8.70</td>
<td>8.70</td>
<td>8.40</td>
<td>8.80</td>
<td>8.50</td>
<td>9.20</td>
</tr>
<tr>
<td>Average Volume</td>
<td>8.60</td>
<td>8.83</td>
<td>19.15</td>
<td>15.35</td>
<td>8.00</td>
<td>8.33</td>
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#### 8 February

<table>
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<th>Temperature</th>
<th>70.0</th>
<th>80.0</th>
<th>90.0</th>
<th>100.0</th>
<th>115.0</th>
<th>130.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trial no.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>1</td>
<td>2</td>
<td>3</td>
</tr>
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<td>Initial Volume</td>
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<td>23.10</td>
<td>n/a</td>
<td>0.80</td>
<td>10.60</td>
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<tr>
<td>Final Volume</td>
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<td>23.10</td>
<td>34.00</td>
<td>n/a</td>
<td>11.60</td>
<td>21.60</td>
</tr>
<tr>
<td>Volume Used</td>
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<td>11.20</td>
<td>10.90</td>
<td>n/a</td>
<td>10.80</td>
<td>11.00</td>
</tr>
<tr>
<td>Average Volume</td>
<td>11.10</td>
<td>n/a</td>
<td>10.93</td>
<td>10.93</td>
<td>n/a</td>
<td>n/a</td>
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### Appendix 6 – Processed Data for Oven Temperature Trials

<table>
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<th>Temperature</th>
<th>70.0</th>
<th>80.0</th>
<th>90.0</th>
<th>100.0</th>
<th>115.0</th>
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</tr>
</thead>
<tbody>
<tr>
<td>January 25</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume of Na$_2$EDTA(aq) Used</td>
<td>8.70</td>
<td>8.80</td>
<td>20.10</td>
<td>16.10</td>
<td>8.20</td>
<td>8.40</td>
</tr>
<tr>
<td></td>
<td>8.70</td>
<td>8.50</td>
<td>18.20</td>
<td>14.60</td>
<td>7.80</td>
<td>8.40</td>
</tr>
<tr>
<td></td>
<td>8.40</td>
<td>8.40</td>
<td></td>
<td></td>
<td>8.00</td>
<td>8.20</td>
</tr>
<tr>
<td>Average Volume of Na$_2$EDTA(aq) Used</td>
<td>8.60</td>
<td>8.57</td>
<td>19.15</td>
<td>15.35</td>
<td>8.00</td>
<td>8.33</td>
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<tr>
<td>February 18</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume of Na$_2$EDTA(aq) Used</td>
<td>11.20</td>
<td></td>
<td>10.80</td>
<td>10.80</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>11.20</td>
<td></td>
<td>11.00</td>
<td>11.20</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>10.90</td>
<td></td>
<td>11.00</td>
<td>10.80</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average Volume of Na$_2$EDTA(aq) Used</td>
<td>11.10</td>
<td></td>
<td>10.93</td>
<td>10.93</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Calibration unit</td>
<td>-2.51 ± 0.20cm$^3$</td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Average Volume of Na$_2$EDTA(aq) Used

|             | 8.60 (±0.10) | 8.57 (±0.10) | 8.42 (±0.30) | 8.42 (±0.30) | 8.00 (±0.10) | 8.33 (±0.10) |
| % Uncertainty | 1.16%        | 1.17%        | 3.56%        | 3.56%        | 1.25%        | 1.20%        |
## Appendix 7 – Raw Data Oven Hour Trials

<table>
<thead>
<tr>
<th>Minutes</th>
<th>60</th>
<th>180</th>
<th>300</th>
<th>420</th>
<th>540</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Trial no.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial Volume</td>
<td>0.70</td>
<td>0.00</td>
<td>0.00</td>
<td>0.80</td>
<td>10.60</td>
</tr>
<tr>
<td>Final Volume</td>
<td>26.00</td>
<td>25.20</td>
<td>25.70</td>
<td>11.60</td>
<td>21.60</td>
</tr>
<tr>
<td>Volume Used</td>
<td>25.30</td>
<td>25.20</td>
<td>25.70</td>
<td>10.80</td>
<td>11.00</td>
</tr>
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<td>Average Volume</td>
<td>25.40</td>
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<td>10.93</td>
<td></td>
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</tbody>
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### Appendix 8 – Calculated Efficiency

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<thead>
<tr>
<th>Temperature (°C)</th>
<th>70.0</th>
<th>80.0</th>
<th>90.0</th>
<th>100.0</th>
<th>115.0</th>
<th>130.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of Zeolite</td>
<td></td>
<td></td>
<td></td>
<td>0.250g</td>
<td></td>
<td></td>
</tr>
<tr>
<td>No. of moles of Ca(NO₃)₂ exchanged</td>
<td>0.0001049 (±2.0%)</td>
<td>0.0001052 (±1.9%)</td>
<td>0.0001067 (±4%)</td>
<td>0.0001067 (±4%)</td>
<td>0.0001109 (±1.8%)</td>
<td>0.0001076 (±1.9%)</td>
</tr>
<tr>
<td>% Efficiency</td>
<td>54.95 (±3%)</td>
<td>55.11 (±3%)</td>
<td>55.89 (±4%)</td>
<td>55.89 (±4%)</td>
<td>58.09 (±2%)</td>
<td>56.36 (±2%)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Minutes</th>
<th>60</th>
<th>180</th>
<th>300</th>
<th>420</th>
<th>540</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of Zeolite</td>
<td></td>
<td></td>
<td></td>
<td>0.250g</td>
<td></td>
</tr>
<tr>
<td>No. of moles of Ca(NO₃)₂ exchanged</td>
<td>-0.0000631 (±3%)</td>
<td>0.0001067 (±1.9%)</td>
<td>0.0001140 (±1.8%)</td>
<td>0.0000310 (±6%)</td>
<td>-0.0000005 (±400%)</td>
</tr>
<tr>
<td>% Efficiency</td>
<td>-33.05 (±3%)</td>
<td>55.89 (±2%)</td>
<td>59.72 (±2%)</td>
<td>16.24 (±7%)</td>
<td>-0.26 (±400%)</td>
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### Appendix 9 – Raw Data for Application Trials

**Volumetric (Titration) Analysis (for non-coloured solutions)**

<table>
<thead>
<tr>
<th>Volume of Na$_2$EDTA(aq) Used (cm$^3$)</th>
<th>Deionised Water</th>
<th>Tap water</th>
<th>Sea water</th>
<th>Pb(NO$_3$)$_2$</th>
<th>Ca(NO$_3$)$_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>w/out Z</td>
<td>with Z</td>
<td>w/out Z</td>
<td>with Z</td>
<td>w/out Z</td>
</tr>
<tr>
<td>Trial</td>
<td>Initial</td>
<td>Final</td>
<td>Change</td>
<td>Initial</td>
<td>Final</td>
</tr>
<tr>
<td>1</td>
<td>0.00</td>
<td>12.70</td>
<td>0.10</td>
<td>0.00</td>
<td>32.90</td>
</tr>
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<td>2</td>
<td>0.00</td>
<td>13.70</td>
<td>0.90</td>
<td>0.00</td>
<td>33.00</td>
</tr>
<tr>
<td>3</td>
<td>0.00</td>
<td>14.90</td>
<td>1.60</td>
<td>0.20</td>
<td>36.40</td>
</tr>
<tr>
<td>Average titrant used</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>36.20</td>
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<tr>
<td>Difference between w and w/out Z</td>
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<td>0.48</td>
<td>0.53</td>
<td>18.63</td>
<td>2.40</td>
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### Appendix 10 – Raw Data from Back Titration Analysis

**Back Titration Analysis (for coloured solutions)**

<table>
<thead>
<tr>
<th>Volume of Na$_2$EDTA(aq) Used (cm$^3$)</th>
<th>Fe$_2$(SO$_4$)$_3$</th>
<th>CuSO$_4$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>w/out Z</td>
<td>with Z</td>
</tr>
<tr>
<td><strong>Trial</strong></td>
<td><strong>Initial</strong></td>
<td>0.70</td>
</tr>
<tr>
<td></td>
<td><strong>Final</strong></td>
<td>27.20</td>
</tr>
<tr>
<td></td>
<td><strong>Change</strong></td>
<td>26.50</td>
</tr>
<tr>
<td>2</td>
<td><strong>Initial</strong></td>
<td>1.10</td>
</tr>
<tr>
<td></td>
<td><strong>Final</strong></td>
<td>27.30</td>
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<tr>
<td></td>
<td><strong>Change</strong></td>
<td>26.20</td>
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<tr>
<td></td>
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## Appendix 11 – Instrument Uncertainty

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<th>Apparatus</th>
<th>Subtype of apparatus (cm$^3$)</th>
<th>Type of Scale</th>
<th>Uncertainty (±cm$^3$)</th>
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<tbody>
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<td>Volumetric Flask</td>
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</tr>
<tr>
<td>250</td>
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<td>analogue</td>
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<td>0.4</td>
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<td>digital</td>
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<tr>
<td>Oven thermostat</td>
<td></td>
<td>digital</td>
<td>0.1 °C</td>
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</tbody>
</table>
Appendix – Images

Image 1 – Ca(NO3)2 with Zeolite solution after volumetric analysis

Image 2 – Tap water with Zeolite solution after volumetric analysis
Optimal Synthesis Condition and Adsorption Capacity of Zeolite A

Image 3 – Gravimetric Analysis of filtered Fe(OH)₃(s) precipitate: Zeolite treated (Yellow) and non-Zeolite treated (brown)

Image 4 – Fe(OH)₃ precipitate, Na₂SO₄(aq), and excess NaOH(aq) mixture after settling
Image 5 - Gravitational filtration of Zeolite treated CuSO4 solution

Image 6 - Zeolite treated Cu(OH)2 precipitate, Na2SO4(aq), and excess NaOH(aq) mixture after settling
Image 7 - Titration setup

- Burette Clamp
- 50 cm³ Burette
- 100 cm³ conical flask
- Stand
Image 8 - Vacuum filtration setup

- Büchner funnel with filter paper
- Stopper
- Rubber tubing connected to aspirator on tap
- Büchner flask

Image 9 - Gravity filtration setup

- Funnel with filter paper
- 250 cm³ conical flask
References


University of Canterbury, College of Science. (2017). Determination of Total Calcium and Magnesium Ion Concentration.


