|The Effect of pH on Crystal Growth|

2024 – Crystal Investigation

Kodithuwakku, Venuki

Introduction:

Aluminium Potassium Sulphate Dodecahydrate ($KAl(SO_4)_2 \cdot 12H_2O$), or Alum, is a chemical compound that is slightly acidic with a pH of between 3.0 and 3.5 in a 10% solution. This year, I am investigating the effects of pH in a crystal growing solution. Since Alum is a weak acid, and the distilled water has a pH of around 7.0 (neutral), then the dissolution is acidic meaning that the crystal was grown in acidic conditions of around 3.0 to 4.0 (assuming that it is a 10% concentrated solution, in which my former experiments have been 50 g -70 g in 500 mL – 600 mL of distilled water and around 10% -13% concentration). Last year, I added sodium chloride (NaCl) which was also a very strong bonded compound with a pH of exactly 7. This is due to the bonding of the strong acidic hydrochloric acid (HCl) with a pH of 0 in a 1 Molar solution (6.022×10^{23} atoms/ Litre) and the strong basic sodium hydroxide (NaOH) with a pH of 14 in a 1 Molar solution which equates to a pH of 7.0.

Alum is the name for a group of chemical compounds, specifically double sulphate salts that consists of a trivalent cation (triple positive charge) and a monovalent cation (one positive charge). The general formula for alum compounds is $XAl(SO_4)_2 \cdot 12H_2O$, where X is the monovalent cation. In the alum compound, Aluminium Potassium Sulphate Dodecahydrate, $KAl(SO_4)_2 \cdot 12H_2O$, K^+ (potassium) represents the monovalent cation, Al^{3+} (aluminium) represents the trivalent cation, $(SO_4)_2$ (sulphate) represents the polyatomic ion (two or more atoms that are covalently bonded and have a net charge, in which this case is 2-). The dodecahydrate -$12H_2O$ (12 molecules of water) helps the crystal maintain its physical properties including its octahedral shape. *Hydrogen and oxygen are also known for supporting the crystalline structure of the alum crystal and affect the reactivity of the compound, hence explaining the importance of having hydrates.* The K^+ monovalent cation and the Al^{3+} trivalent cation balances the negative charges from the two sulphate $(SO_4)_2$ ions. The sulphate anions are part of the structure of the compound and affect the solubility and reactivity of the compound as well.

When alum potassium is dissolved in water, the alum dissociates with its constituent ions. Through a process known as hydrolysis, the aluminium ions react with water, yielding two products: precipitate aluminium hydroxide and hydronium ions (one water molecule and a hydrogen cation which yields H_3O^+, a hydronium ion), which increases the acidity of the water. This shows that alum crystals grown in normal conditions are grown in acidic conditions. The balanced chemical equation below shows the reaction with a trivalent aluminium cation and six molecules of water yields aluminium hydroxide and three molecules of hydronium ions.

$Al^{3+}(aq) + 6H_2O(l) \rightleftharpoons Al(OH)_3(s) + 3H_3O^+ (aq)$

Therefore, adding an acid will not result in a visible reaction as the solution is already acidic. If a base is added to the water, the hydroxide ions from it will react the hydronium ions to form water, which reduces the concentration of hydronium ions. This reduces acidity and increase alkalinity, making the solution basic if adequate base is added.

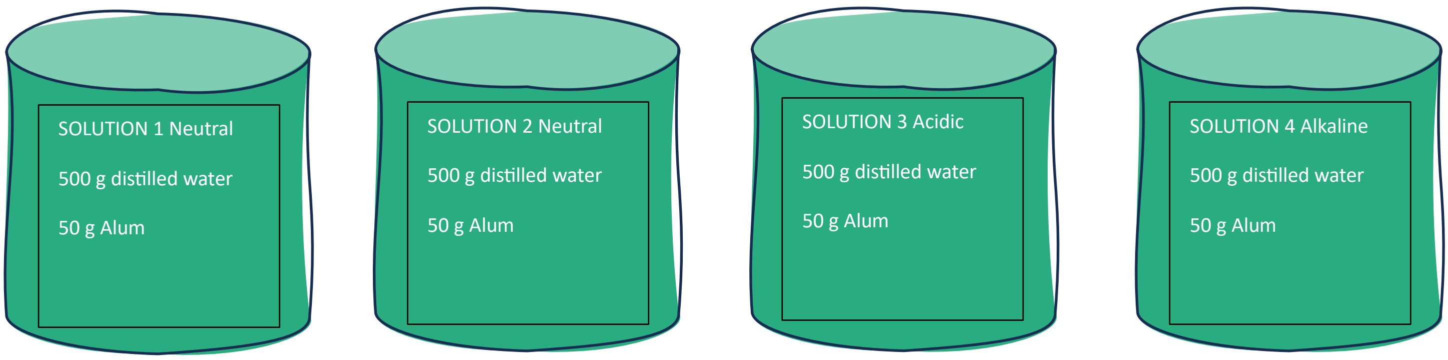
I was determined to find out if a typical alum crystal grows best in an acidic or an alkaline condition comparative to typical crystal growing conditions (pH) based on the background information above. The acidity and alkalinity of the aqaueous solution also determined the concentration of I also wanted to find out how hydroxide ions (hydrogen and oxygen) affect a crystal. This year, I am focusing on better clarity and dimensions.

Hypothesis:

I. I hypothesise that pH has an effect on the physical properties of a crystal.

II. I hypothesise that the Alum crystal that is grown in an acidic condition will have more clarity and smoother dimensions than Alum crystals grown in alkaline and normal crystal growing conditions because normal crystal growing conditions are acidic showing that Alum crystals have better physical properties when there is a greater presence of hydrogen ions (relative to the acidity of the solution).

How will I perform this?



Risks:

The chemical compounds I will be using are known as common laboratory compounds, yet they pose very severe safety hazards if not handled properly. I have safely and sensibly performed these safety measures and have carefully inspected the MSDS (Material Safety Data Sheet) for both sodium hydroxide and hydrochloric acid. The links to these sheets are available below:

1. Hydrochloric Acid: Microsoft Word - 0011.doc (chemsupply.com.au)
2. Sodium Hydroxide: Crystal Reports ActiveX Designer - MSDS_9.RPT (chemsupply.com.au)

Additionally, I have filled out the OSA Risk Assessment Form and performed all safety protocols; wearing mask, eye protection, gloves, hair tied back and maintaining a safe distance from the chemicals. Supervision was incorporated and the two chemicals to less than a molarity of 1.

Materials:

General Materials:

4 × 65 g (260 g) Aluminium Potassium Sulphate Dodecahydrate

1 × Hydrochloric Acid (HCl)

1 × Sodium Hydroxide (NaHO)

4 × Glass Beakers (labelled for convenience)

5 × 500 mL (2.5 L) distilled water

1 × Plastic Container

1 × Weighing Boat

1 × Stirring utensil

1 × Kitchen Scale

1 × Fishing Line

1 × Scissors

4 × Ruler

Method:

Seed Crystallisation:

Materials:

1. *500 mL Distilled water*
2. *LABELLED glass Beaker*
3. *Plastic Container*
4. *Kitchen Scale*
5. *Alum*
6. *Weighing Boat*
7. *Utensils – wooden spoon*

1. Retrieve your beaker and distilled water.
2. Measure out 500 mL of the distilled water using a plastic container and kitchen scale.
3. Pour the contents of the plastic container into a beaker.
4. Measure out 50 g of Alum with the weighing boat and kitchen scale and pour it into the beaker.
5. Mix thoroughly until you cannot manually dissolve anymore Alum.
6. Place the beaker on a stove and heat it to a mild temperature.
7. Grab your spoon and stir the beaker thoroughly until all the Alum has been dissolved.
8. Turn the stove off then set the beaker aside to rest and cool.

9. Get a plastic container and carefully pour the dissolution of the beaker into it, allowing it to settle and nucleate into seed crystals.

Crystallisation:

Materials: ***FOR 1 CRYSTAL***

1. *LABELLED beaker*
2. *Fishing Line*
3. *Scissors*
4. *Seed crystals*
5. *Distilled water*
6. *Ruler*
7. *Alum*
8. *Weighing Boat*
9. *Kitchen Scale*

1. Get your seed crystal, fishing line and scissors.
2. Snipping the fishing line with some scissors to a length of around 20 – 30cm and tightly tie it around the seed crystal.
3. Retrieve the ruler and tie around it the other end of the fishing line.
4. Grab your beaker and distilled water.
5. Tip the contents of the distilled water into the beaker and measure 500 mL of distilled water.
6. Measure 65 grams of Alum using a kitchen scale and weighing boat.
7. Carefully pour the 65 grams of Alum into the beaker and stir with
8. Take the wooden knife that is tied to the fishing line and gently put it over the edge of the beaker, allowing the seed crystal to be placed in the water which will nucleate into a larger seed crystal.
9. Allow the crystal to settle in a controlled environment.

GROWING TIPS:

1. It is recommended that you reheat and add more Alum to the crystal every fortnight so that the Alum supply (to be nucleated) does not run out. This also stimulates a slight rapid growth since crystals grow better in warm temperatures. However, note that every time you do this, TAKE THE SEED CRYSTAL OUT. Otherwise, the seed crystal will dissolve along with the other Alum. Reheat the beaker so the Alum on the bottom will dissolve and add some more alum for a longer growth duration.
2. Keep the crystal in a dark, quiet yet warm place to grow such as a cupboard.
3. Make sure the crystal is in a controlled environment with controlled variables such as temperature, humidity, sound, and brightness.

How to Calibrate a pH tester:

Calibrating your pH tester is essential to get accurate results from your pH tester every time. Follow these eight simple steps to calibrate your pH tester:

Materials:

1. *pH tester*
2. *Distilled water*
3. *Buffer pH 7.0*
4. *Buffer pH 4.0*
5. *(Depending on your pH tester, you may also have a buffer with pH 10.0)*

1. Put your pH tester to calibrate mode. You may have to hold the on/off button until the screen says ‘Cal’, or you may need to press a certain button. Make sure you read the instruction manual to know how to operate your pH tester.
2. When the pH tester says ‘Cal’, it should also state a pH number which will either be 7.0 or 4.0 *OR* 10.0 if you have a more advanced pH tester.
3. Get the buffer that it corresponds to, open the cap of the pH tester, and carefully dip the pH tester electrode into the buffer solution, until the pH tester gives you a signal to stop.
4. Before showing you the nest buffer to dip the solution into.
5. Before you dip the pH tester into the second buffer, make sure you quickly rinse the electrode of the pH tester in distilled water.
6. Dip your pH tester into the second. If you have a three-point tester, rinse the electrode in water until the third buffer is directed.
7. The pH tester should say ‘Sto’ for stop on the screen when the calibration is over and testing can be performed. Likewise, the pH tester will give you a signal.

Logbook & Observations:

Date	Observations & Notes	Pictures/Additional Information
Day 1: 24/04/2024	Today, I started my crystallisation experiment. I retrieved four beakers and made a dissolution with the Alum. I heated the solution to increase the solubility, so the Alum was more dissolved. Additionally, I added in diluted drops of hydrochloric acid in one through an acid dropper in one beaker, and sodium hydroxide in the other. The other two beakers have no added compounds except the Alum. While adding in the sodium hydroxide, I noticed that there were translucent swirls in the solution which was haphazard. However, I believed there is an immediate chemical reaction between the Alum and sodium hydroxide yielding a precipitate: Aluminium hydroxide. As I mixed the sodium hydroxide, the whole solution became translucent which made my new prediction more credible. Whilst performing the whole experiment, I calibrated my pH tester and continuously measured the pH level of all four beaker while taking pictures. Solution 1: Neutral – pH ±0.2 2.6 Solution 2: Neutral – pH ±0.2 2.5 Solution 3: Acidic – pH ±0.2 2.0 (picture portrays pH of 2.1) Solution 4: Alkaline – pH ±0.2 3.7	
Day 2: 25/04/2024	This morning, I checked my four solutions and found that the seed crystals ON EVERY BEAKER had fallen onto the bottom of the beaker and had slightly dissolved hence the decreased size. Also, in Solution 4, I noticed that there was a white precipitate on the bottom of the beaker. Since this was the sodium hydroxide solution, I believed that there *was* a chemical reaction and that the white precipitate was Aluminium hydroxide.	

	SOLUTION: • For a start, I will retrieve new seed crystals and tie it better and tighter to the fishing line to prevent this from occurring in the first place. I will try to tie it twice to stabilize the crystal so to prevent it from a precarious position. • Additionally, I realized that if the Aluminium hydroxide was present at the bottom of the beaker where the small crystals grew then there would be Aluminium hydroxide on my final crystal. I may have to extract the precipitate by decanting it since the solution was heterogeneous, meaning that the solution is a mixture. • If I do decant Solution 4, then I may have to add more Alum since the Alum at the bottom of the beaker with the Aluminium hydroxide got separated with the distilled water. I also grew some more seed crystals since the ones I am growing were made a few months ago. I am also making it for my brother to do his crystal investigation.	
Day 8 01/05/2024 10:20 PM	Today, I removed the previous crystals and added some of the new seed crystals which I had grown a few days ago. I put the seed crystals into all 4 solutions. However, I did not check and re-do the pH as I wanted the crystals to settle in before the change of pH. The precipitate on the bottom of solution 4 (Sodium Hydroxide) which I thought was Aluminium Hydroxide, had substantially decreased.	
Day 10 03/05/2024 2:15 PM	Today, I noticed that the crystal's fishing line was slightly less visible meaning that a new layer of crystals had grown on top of it. Therefore, I took the crystals out and altered the pH. Because I did not have proper lab storing apparatus, I assumed that I had to apply more of the hydrochloric acid and sodium hydroxide to yield the similar pH as last time. Solution 1: Neutral – pH ±0.2 2.9 Solution 2: Neutral – pH ±0.2 2.7 Solution 3: Acidic – pH ±0.2 1.9 Solution 4: Alkaline – pH ±0.2 3.7	
Day 11 04/05/2024 3:30 PM	After yesterday's pH adjustment, I noticed significant growth in the Acidic crystal. This also supports my hypothesis since the Alum crystal grown in acidic conditions showed a better growth rate.	
Day 19 11/05/2024 CLARITY CHECK	Today, I checked the clarity of my crystals: Solution 1: Neutral – quite clear yet still translucent parts Solution 2: Neutral – more clarity than Solution 1 and a more defined octahedron Solution 3: Acidic – due to its substantial growth, the Acidic crystal had the least clarity. I was most impressed with the clarity of the Alkaline crystal, and it also was growing according to my hypothesis. There are also some smaller crystals growing on the string. Solution 4: Alkaline – very slow growth, yet extremely clear.	*Top Left – Solution 1* *Top Right – Solution 2* *Bottom Left – Solution 3* *Bottom Right – Solution 4*

<table>
<tr><td></td><td></td><td></td></tr>
<tr><td>Day 20
12/05/2024
3:00 PM
QUALITY CHECK</td><td>The day after the clarity check is the quality check that makes sure that my crystals are according to OSA's judging rubric. I will be judging three qualities of the crystals:
<table>
<tr><td>QUALITY</td><td>Neutral 1</td><td>Neutral 2</td><td>Acidic 3</td><td>Alkaline 4</td></tr>
<tr><td>Clarity</td><td>Moderate</td><td>Moderate</td><td>Good</td><td>Excellent</td></tr>
<tr><td>Smoothness/Regularity</td><td>Rigid edges</td><td>Uneven edges</td><td>Straight yet uneven edges</td><td>Uneven edges</td></tr>
<tr><td>Shape</td><td>Flat corners</td><td>Flat corners</td><td>Flat corners</td><td>Flat corners</td></tr>
</table>
</td><td></td></tr>
<tr><td>Day 47
08/06/2024
9:54 PM
UPDATE</td><td>Today, I heated up my solutions.</td><td></td></tr>
<tr><td>Day 88
19/07/2024
CONCLUDE EXPERIMENT</td><td>Today, I concluded the experiment. I collected the crystals, cleaned out all the beakers, stored all materials and wrote my conclusion and results.</td><td></td></tr>
</table>

Conclusion:

Crystal	Solution 1 – Neutral	Solution 2 – Neutral	Solution 3 – Acidic	Solution 4 – Alkaline (basic)
Mass	11.16 g	9.25 g	14.94 g	24.07 g
Clarity (measured by light diffusion)	Mild Scattered light	Mild Scattered light	Excellent clarity, overall focused and sharp light beam	Least clarity, very scattered light
Dimension Quality	Octahedral shape with smooth edges however some edges are uneven yet smooth	Defined octahedral shape with smooth edges however with flat vertices	Defined octahedral shape with smooth edges and flat vertices, faces also resemble equilateral triangles	Large defined octahedral shape that has smooth and the sharpest edges however vertices are flat but quite uniform structure
Significant Observations/FINAL PICTURES				

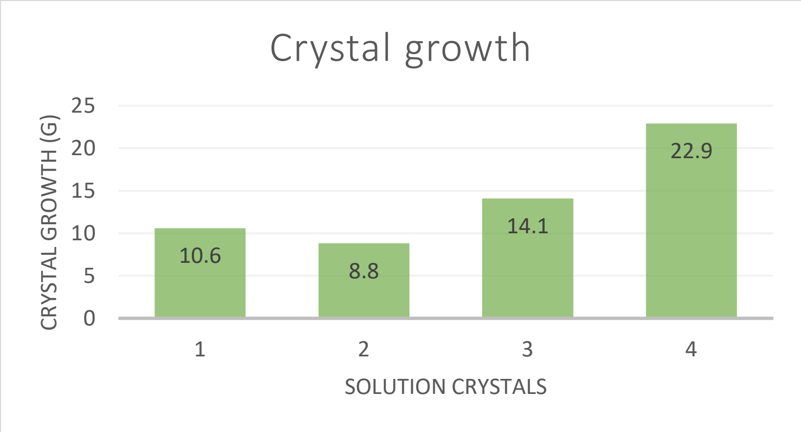
I had two hypothesises:

I. I hypothesise that pH has an effect on the physical properties of a crystal.

II. I hypothesise that the Alum crystal that is grown in an acidic condition will have more clarity and smoother dimensions than Alum crystals grown in alkaline and normal crystal growing conditions because normal crystal growing conditions are acidic showing that Alum crystals have better physical properties when there is a greater presence of hydrogen ions (relative to the acidity of the solution).

In conclusion, the pH of a crystal affects the physical properties of a crystal. The Acidic crystal grew most accordingly to the physical features mentioned in my hypothesis. Therefore, my hypothesis was correct. The alkaline crystal was the largest but that did not mean the crystal would have better clarity and smoother dimensions, as a faster growth rate indicates a decrease in the quality of the crystal and an increase of the size dimensions of the crystal. My hypothesis referred the acidic crystal as having better clarity and smoother dimensions but not a faster growth rate (therefore size), as size is dependent on the growth rate. The alkaline crystal had a higher growth rate, which was not according to my hypothesis.

To prove this are the evident masses from the table above which shows that the acidic crystal weighed 14.94 grams while the alkaline crystal weighed 24.07 grams. Even though the acidic crystal weighed less, it had better clarity. Below is a graph that shows the crystal growth of each crystal (the difference between the masses of each crystal before and after crystallisation). The alkaline crystal has significant growth, consequently decreasing the physical qualities of the crystal.



To conclude the results is the given impact of hydroxide and hydrogen (hydronium ions in this context). Since the acidic crystal had better clarity and acidity is relative to the concentration of hydrogen (hydronium ions) present in the water, I can conclude that the concentration of hydrogen is relative to the clarity of the crystal. Additionally, the alkaline crystal had the least clarity but the fastest growth rate and as alkalinity is relative to the concentration of hydroxide ions, I can conclude that the concentration of hydroxide ions is relative to the alkalinity of the crystal.

Some improvements that could have been implemented throughout, before and after to the crystallisation experiment were adding two other different compounds that could not yield any precipitate or substance when reacting with Alum. I might also consider heating the crystal solution less to maintain the pH without needing to adjust it (temperature affects pH). If I do implement this strategy, the crystals would need to be grown in warm weather temperatures such as summer to ensure the crystals can grow well. Something that would have improved the accuracy of my findings is if all four solutions had the same final mass. As all crystals were given the same amount of alum but two crystals were given additional compounds, this may have changed the accuracy of my findings.

CONCLUSION:

I. The pH *has* an effect on the physical properties of a crystal.

II. An Alum crystal that is grown in an acidic condition *will* have more clarity and smoother dimensions than Alum crystals grown in alkaline and normal crystal growing conditions because normal crystal growing conditions are acidic showing that Alum crystals have better physical properties when there is a greater presence of hydrogen ions (relative to the acidity of the solution).

References:

[fscimage.fishersci.com/msds/19200.htm]

[Alum | Definition, Uses, Formula, & Facts | Britannica]

Acknowledgments:

I would like to thank my parents for helping me buy the necessary equipment and compounds.