

## **Nitrogen Removal by Ammonia Stripping**

**What is the effect of initial concentration of  $\text{NH}_3$  on the final concentration of  $\text{NH}_3$  after treatment through an air stripping column?**

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## INTRODUCTION

### *Research Question*

What is the effect of initial concentration of  $\text{NH}_3$  on the final concentration of  $\text{NH}_3$  after treatment through an air stripping column?

### *Relevance of Ammonia in Wastewater Treatment*

Wastewater treatment plays an invaluable role in processing and integrating the nitrogen at levels that humans produce back into the environment. Humans have an enormous impact of the global nitrogen cycle by the production of wastewater, burning of fossil fuels, and creation and use of fertilizers (Canfield 195). When excessive amounts of nitrogen (in various forms) are present in surface and groundwater, there are significant negative implications to the environment (Scheible et al. 12). Wastewater is the most common cause of artificial eutrophication, defined as human-caused “excessive plant growth and/or algae “blooms” resulting from over-fertilization” (Scheible et al. 13), caused in large part by entry of large quantities of ammonia into bodies of water. These high levels of ammonia can be converted into plant nutrients, notably nitrates, creating algae blooms that reduce the dissolved oxygen levels and kill aquatic life (Lund 372). Ammonia is also toxic to both aquatic and terrestrial life as the concentration of free molecular ammonia ( $\text{NH}_3$ ) increases. (Scheible et al. 14).

ACT

## METHODOLOGY

### *Background*

As an overview, this experiment will be designed to measure the effectiveness of ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) removal by treatment through an air stripping column. This is done by measuring the concentration of ammonium hydroxide in a solution before and after being treated. Solutions of specific concentration are prepared, then heated to shift equilibrium to produce a removable form of ammonia as ammonia gas ( $\text{NH}_3$ ), then treated through a 'air stripping column', a common physical process used in wastewater management to remove excess nitrogen. A back titration will be used to measure both pre- and post-treatment samples' concentration of ammonium hydroxide.

I have been particularly interested in some of the intricacies of wastewater treatment after researching some of the science and history behind my local, murky saltwater harbor where I live. I read in the local paper that since 1986, the wastewater treatment facility for the entire town is located less than a mile away from the harbor. The effluent has since reached the groundwater, and scientists have measured a significant increase in nitrogen levels and growth of algae in harbor since the 90s. Since 2005, after upgrades were put into place, nitrogen levels have decreased from an initial 20-30 mg/l to 3 mg/l, leading me to research the details and methods for removing nitrogen from wastewater effluent (Geib).

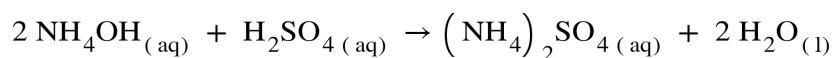
## *Design & Chemistry*

In this experiment, I will investigate the research question: “What is the effect of initial concentration of  $\text{NH}_3$  on the final concentration of  $\text{NH}_3$  after treatment through an air stripping column?”. To answer this, I will investigate the relationship between the initial concentration of ammonium hydroxide solution to the final concentration of  $\text{NH}_3$  with regard to the ammonia removal efficiency, the percentage of removed ammonia. There are five initial concentrations of ammonium hydroxide;  $0.020 \text{ mol dm}^{-3}$ ,  $0.015 \text{ mol dm}^{-3}$ ,  $0.010 \text{ mol dm}^{-3}$ ,  $0.005 \text{ mol dm}^{-3}$ , and  $0.00143 \text{ mol dm}^{-3}$ . The dependant variable is the concentration of ammonium hydroxide measured in  $\text{mol dm}^{-3}$  (from  $0.00143 \text{ mol dm}^{-3}$  -  $0.020 \text{ mol dm}^{-3}$ ) after it has been through the ammonia stripping process. The independent variable is the starting concentration of ammonium hydroxide in  $\text{mol dm}^{-3}$ , before going through the ammonia stripping process. Variables that must be kept constant are the temperature of solutions at the start of ammonia stripping, in  $^{\circ}\text{C}$ , from  $20^{\circ}\text{C}$  -  $80^{\circ}\text{C}$  and the volume of air forced into the tower will be a constant  $15 \text{ dm}^3\text{s}^{-1}$ . The required concentrations are prepared in batches to limit uncertainty, and all titrations are carried out at a room temperature of  $20^{\circ}\text{C}$ . The processing of samples requires the same conditions for multiple trials, so the stripping column is left to dry and rinsed with distilled water three times before processing each trial.

In designing this experiment, two main systems had to be researched and developed. The first was the method to measure the ammonia concentration within a given sample. The second was to construct an air stripping column. Afterwords a procedure can be developed for the purposes of answering the research question.

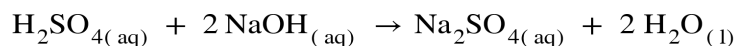
Due to restrictions on cost and availability, this experiment uses a simple acid base back titration to measure ammonia concentration. Unlike a direct titration, a back titration requires the addition of excess titrant to the solution, followed by a titration to ‘remove’ the excess titrant from solution to determine how much is in excess. To measure for ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) a weak base, sulfuric acid ( $\text{H}_2\text{SO}_4$ ), a strong acid, is added in excess to create ammonium sulfate ( $(\text{NH}_4)_2\text{SO}_4$ ) and water, as outlined in equation 2.

*Equation 2 Balanced equation for the reaction between ammonium hydroxide and sulfuric acid.*



A known quantity of sulfuric acid is added in excess such that all ammonium hydroxide present is removed. After the reaction has completed, sodium hydroxide ( $\text{NaOH}$ ), a strong base, is used to titrate the remaining volume of sulfuric acid, as outlined in equation 3.

*Equation 3 Balanced equation for the reaction between sulfuric acid and sodium hydroxide.*

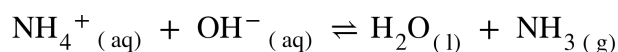


In both steps, phenolphthalein indicator will be used to determine the end point in terms of color change from colorless (acidic) to dark pink (basic) (Amrita).  $0.05 \text{ mol dm}^{-3}$  is determined to be an ideal concentration for both titrants,  $\text{H}_2\text{SO}_4$  and  $\text{NaOH}$ , given the maximum volume of the burette used ( $50 \text{ cm}^3$ ) and a range of concentrations between  $0.020 \text{ mol dm}^{-3}$  and  $0.00143 \text{ mol dm}^{-3}$ , where the volume of  $\text{H}_2\text{SO}_4$  is varied from  $30 \text{ cm}^3$  to  $15 \text{ cm}^3$  (required + excess titrant) and the volume of  $\text{NaOH}$  is varied from at least  $10 \text{ cm}^3$  and at most  $40 \text{ cm}^3$ . This falls within the range of both the burette and the expected concentrations of ammonium hydroxide.

This experiment is based on the equilibrium reaction between ammonium hydroxide, ammonia gas, and water as seen in Equation 1. To address the research question, this experiment

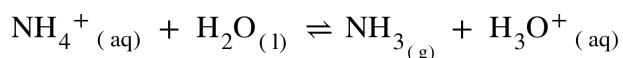
will use heat to increase the amount of ammonia gas present so that a measurable quantity is removed during the stripping process.

*Equation 1 Balanced equation for the reaction between dissociated ammonium hydroxide and ammonia gas and water.*



The equilibrium reaction shown in Equation 1 has an enthalpy of  $\Delta_f H^\circ (0 \text{ K}) = 30.77 \text{ kJ/mol}$ , an endothermic reaction (PubChem). An increase in temperature and pH will shift the equilibrium towards the products, increasing the amount of ammonia gas in solution and decreasing the amount of ammonium overall.

In order to create a procedure that responds to the research question, research was completed to develop an understanding of the professional approach to the functions of ammonia stripping. The process of ammonia stripping is used to reduce a solution's ammonia content by desorption, where ammonia is released from a surface face. Ammonium hydroxide,  $\text{NH}_4\text{OH}$  present in the wastewater, exists in equilibrium with both Ammonia gas  $\text{NH}_3$  and water  $\text{H}_2\text{O}$  according to equation 1. In this case, dissolved ammonium ions are taken out of aqueous solution and form ammonia gas in the presence of air unsaturated with ammonia gas. Ammonia itself exists in equilibrium with ammonium ion:



The ammonia gas is the only substance able to be removed from water by the process of ammonia stripping. The amount of ammonia that is lost is dependant upon the concentration of the dissociated ammonia, the surface area between the solution and the gas, the mass-transfer

coefficient, and the pressure exerted by ammonia in the gas phase (Srinath and Loehr 1939). The amount of ammonium in solution can be represented as a function of pH according to the following equation:

$$\frac{\text{undissociated ammonium}}{\text{total ammoniacal nitrogen}} = \frac{10^{\text{pH}}}{\frac{k_b}{k_w} + 10^{\text{pH}}} = F$$

where  $K_b$  is the ionization constant of aqueous ammonia and  $K_w$  is the ionization constant of water. It has been shown that data indicates that these constants increase with an increase in temperature, so both higher pH values and temperature will shift equilibrium to the gaseous form of ammonia,  $\text{NH}_4$ .

The amount of removable ammonia is able to be increased by increasing the length of time that the air is exposed to the solution, area of contact between the air and the solution, as well as the concentration of ammonium in solution. I will only heat the solution before processing it as raising the pH would interfere with the titration as the phenolphthalein indicator is pH dependant. Raising the temperature to  $80^\circ\text{C}$  will shift equilibrium of Equation 1 to its products, increasing the amount of ammonia gas released and subsequently stripped off. For these reasons, the heating and processing parts of this experiment will have solutions prepared in  $1\text{ dm}^3$  erlenmeyer flasks may be heated to  $80^\circ\text{C}$ , as measured by a thermometer placed in the center of the flask. A stir bar may be necessary to evenly heat the solution. One  $100\text{ cm}^3$  sample is collected just after the sample is heated, and the rest immediately poured into the sample inlet of the air stripping column while the air pump is on. Processed solution is later collected from the outlet with a  $1\text{ dm}^3$  flask.



## HYPOTHESIS

Given that the amount of ammonia capable of being desorbed is dependant upon the length of time the solution is in contact with the air, the area of contact and the concentration of ammonia, I suggest the higher initial concentration of ammonium hydroxide, the more ammonia gas will be removed from solution. I can assume the length of time the solution is in contact with the air as well as the area of contact between the air and solution will remain consistent through all trials, as conditions are kept consistent. The same maximum solution flow rate is reached for each trial and the volume of air pumped into the column remains at  $15 \text{ dm}^3\text{s}^{-1}$ .

## PROCEDURE

### *Materials*

#### **Equipment and glassware**

- (1) Graduated cylinder ( $25 \pm 0.35 \text{ cm}^3$ )
- (1) Graduated cylinder ( $50 \pm 0.52 \text{ cm}^3$ )
- (2) Burettes ( $50 \pm 0.5 \text{ cm}^3$ )
- (5) Volumetric flasks ( $1000 \pm 0.30 \text{ cm}^3$ )
- (2) Volumetric flasks ( $500 \pm 0.15 \text{ cm}^3$ )
- (7) Volumetric flask stoppers
- (2) Erlenmeyer flasks ( $1000 \text{ cm}^3$ )
- (2) Erlenmeyer flask stoppers
- (5) Beakers ( $250 \text{ cm}^3$ )
- (1) Ring stand
- (1) Burette clamps
- (1) Hot plate (w/ stir function)
- (1) Stir bar

#### **Chemicals**

Distilled water

$\text{NH}_4\text{OH}$  (Ammonium hydroxide) ( $14.8 \text{ mol dm}^{-3}$ )

(5)  $1000 \text{ cm}^3$  at 0.020 M, 0.015 M, 0.010 M, 0.005 M, and 0.0014 M.

$\text{NaOH}$  (Sodium hydroxide) ( $1 \text{ mol dm}^{-3}$ )

(1) 500 cm<sup>3</sup> at 0.05 M

H<sub>2</sub>SO<sub>4</sub> (Sulfuric Acid) (1 mol dm<sup>-3</sup>)

(1) 500 cm<sup>3</sup> at 0.05 M

Phenolphthalein indicator (50 cm<sup>3</sup>)

### **Other**

PVC Pipe with appropriate end cap (150 cm in length, 5 cm diameter)

Large ring stand

(2) Hot plates that can serve as magnetic stirrers

(2) Stir bars

Thermometer

Gravel rocks, each ~5 cm<sup>3</sup> in volume (Approx. 10 dm<sup>3</sup>)

(1) Air pump (10 dm<sup>3</sup>s<sup>-1</sup>)

(1) Drill

(1) 5mm drill bit

(1) 10mm drill bit

### *Apparatus*

I constructed a stripping tower by placing the PVC tube on a large ring stand and capping off one of the ends with a cap with a hole in the bottom serving as the solution outlet. A small hole in the side, close to the bottom of the tube serves as the air pump inlet. Gravel is poured into the tube and serves as the packing material within the aeration column. The apparatus is seen in Figure 1.

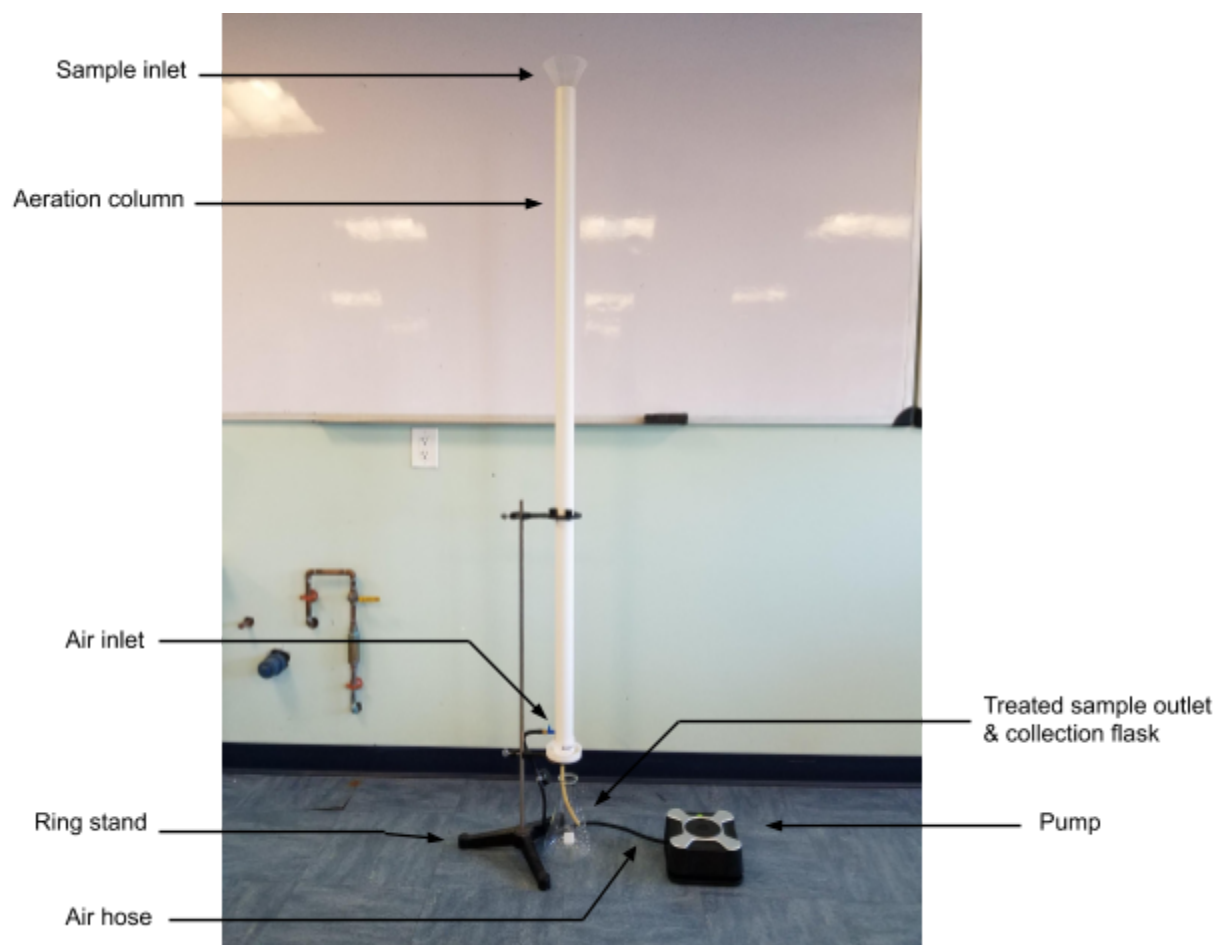


Figure 1 Laboratory setup of aeration stripping column.

## *Experiment*

### Titration

- 1) Place two burettes on a ring stand along with a magnetic stirrer set to a low stir bar setting.
- 2) Fill the burettes with 50 cm<sup>3</sup> of 0.5 mol dm<sup>-3</sup> H<sub>2</sub>SO<sub>4</sub> into one and 50 cm<sup>3</sup> 0.5 mol dm<sup>-3</sup> NaOH into the other.
- 3) Measure out 100 cm<sup>3</sup> from the volumetric flask prepared with 0.020 mol dm<sup>-3</sup> and pour it into a 250 cm<sup>3</sup> beaker.
- 4) Add ~4 drops of phenolphthalein indicator to the beaker so that it reaches a dark pink. Place a stir bar into the beaker.
- 5) Begin by adding 30 cm<sup>3</sup> of 0.05 mol dm<sup>-3</sup> H<sub>2</sub>SO<sub>4</sub> to the beaker using the burette. The solution will become colorless.
- 6) Use the second burette with the NaOH titrant and begin to titrate. Stop adding NaOH when the color of the solution returns to a dark pink. This is the end point.
- 7) Record the volume in cm<sup>3</sup> of NaOH required to reach the stopping point.
- 8) Repeat steps 1-7 as outlined above for each concentration. There are three steps in titrations for each concentration.
  - a) Record an initial sample (as outlined above).
  - b) Record 5 control samples. Step 3 will use a sample taken from the 125 cm<sup>3</sup> flask that contains a sample that was only heated. This was collected in the heating and processing steps mentioned in *methodology*.

- c) Record 5 post-treated samples. This was the end, processed solution collected in the heating and processing steps mentioned in *methodology*.

### Safety Measures

Ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) is a corrosive chemical and acutely toxic. Considering the high concentration of the stock ammonium hydroxide solution at  $14.8 \text{ mol dm}^{-3}$ , Protective gloves, clothing and goggles must be worn when diluting it to  $0.5 \text{ mol dm}^{-3}$ .

Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) is a corrosive chemical and can cause severe skin burns and eye damage. Protective gloves, clothing and eye and/or face protection is required.

Sodium hydroxide ( $\text{NaOH}$ ) is a corrosive chemical and can cause severe skin and eye damage. Protective gloves, clothing and eye protection must be worn.

The samples of ammonium hydroxide, sulfuric acid and sodium hydroxide can all be flushed as they are neutralized within the acid-base titration and are in dilute concentrations.

### RESULTS

The results shown below contain raw data from all back titrations of each of the prepared  $\text{NH}_4\text{OH}$  concentrations. Table 1.1 contains the data for the volume of  $0.05 \text{ mol dm}^{-3}$   $\text{NaOH}$  used for each of the five post-treatment trials. Table 1.2 and 1.3 contains the data for the volume of  $0.05 \text{ mol dm}^{-3}$   $\text{NaOH}$  as well as the volume of  $0.5 \text{ mol dm}^{-3}$   $\text{H}_2\text{SO}_4$  for the control titrations and pretreatment titration trials respectively. It is important to distinguish the ‘control trials’ as titration data collected before anything has been done to the samples, from the ‘pretreatment titration trials’, which is titration data from samples collected after the solutions were heated, but before undergoing ammonia stripping.

**Table 1.1** A table of data for the volume of  $0.5 \text{ mol dm}^{-3}$  NaOH for each of the five *post-treatment trials*.

Initial concentration ( $\text{NH}_4\text{OH}$ , $\text{mol dm}^{-3}$ )	Trial 1 : Volume of $0.05 \text{ mol dm}^{-3}$ NaOH ( $\text{cm}^3$ ) $\pm$ $0.03 \text{ cm}^3$	Trial 2: Volume of $0.05 \text{ mol dm}^{-3}$ NaOH ( $\text{cm}^3$ ) $\pm$ $0.03 \text{ cm}^3$	Trial 3: Volume of $0.05 \text{ mol dm}^{-3}$ NaOH ( $\text{cm}^3$ ) $\pm$ $0.03 \text{ cm}^3$	Trial 4: Volume of $0.05 \text{ mol dm}^{-3}$ NaOH ( $\text{cm}^3$ ) $\pm$ $0.03 \text{ cm}^3$	Trial 5: Volume of $0.05 \text{ mol dm}^{-3}$ NaOH ( $\text{cm}^3$ ) $\pm$ $0.03 \text{ cm}^3$
0.020	28.84	29.63	29.67	29.49	29.86
0.015	23.21	22.91	23.00	22.68	22.71
0.010	20.68	21.51	21.80	21.76	21.48
0.005	15.61	16.20	16.67	16.12	16.90

**Table 1.2** A table of data for the volume of  $0.05 \text{ mol dm}^{-3}$  NaOH as well as the volume of  $0.05 \text{ mol dm}^{-3}$   $\text{H}_2\text{SO}_4$  for the *control trials*.

Initial trial concentration ( $\text{NH}_4\text{OH}$ , $\text{mol dm}^{-3}$ )	Volume of $0.05 \text{ mol dm}^{-3}$ $\text{H}_2\text{SO}_4$ ( $\text{cm}^3$ ) $\pm$ $0.25 \text{ cm}^3$	Volume of $0.05 \text{ mol dm}^{-3}$ NaOH ( $\text{cm}^3$ ) $\pm$ $0.25 \text{ cm}^3$
0.020	30.00	15.31
0.015	25.00	20.63
0.010	20.00	20.21
0.005	15.00	14.84

**Table 1.3** A table of data for the volume of  $0.5 \text{ mol dm}^{-3}$  NaOH as well as the volume of  $0.5 \text{ mol dm}^{-3}$   $\text{H}_2\text{SO}_4$  for the *pretreatment titration trials*.

Initial trial concentration ( $\text{NH}_4\text{OH}$ , $\text{mol dm}^{-3}$ )	Volume of $0.05 \text{ mol dm}^{-3}$ $\text{H}_2\text{SO}_4$ ( $\text{cm}^3$ ) $\pm$	Volume of $0.05 \text{ mol dm}^{-3}$ NaOH ( $\text{cm}^3$ ) $\pm$
0.020	30.00	16.30
0.015	25.00	20.19
0.010	20.00	16.20
0.005	15.00	19.21

It is important to note that the  $0.00143 \text{ mol dm}^{-3}$   $\text{NH}_4\text{OH}$  trial failed to produce useful results. The phenolphthalein indicator, when added, did not change from colorless to pink. After retrying the trial with a new solution mixed, the same thing happened again. This suggests that the concentration was below the limit of detection, and this methodology is unable to determine the concentration of  $\text{NH}_4\text{OH}$  for solutions with concentrations of  $\text{NH}_4\text{OH}$  between 0 and  $0.00143 \text{ mol dm}^{-3}$ .

### *Data processing*

To show how this data is processed, I will first present an example calculation to determine both uncertainty and the end concentration of one example trial, taken from the control data. This specific trial is control trial for the  $0.020 \text{ mol dm}^{-3}$  concentration group, as seen in [Table 1.2](#).

This investigation uses a back titration where  $\text{H}_2\text{SO}_4$  in excess is initially reacted with the  $\text{NH}_4\text{OH}$ .  $\text{NaOH}$  is then added to remove the remaining  $\text{H}_2\text{SO}_4$  in solution. The molarity of  $\text{NH}_4\text{OH}$  can then be deduced. First,  $\frac{1}{2}$  the volume of  $\text{NaOH}$  must be subtracted from the volume of  $\text{H}_2\text{SO}_4$  used in the second part of the back titration to deduce the amount of excess  $\text{H}_2\text{SO}_4$  that has not reacted with  $\text{NH}_4\text{OH}$ . These values can be subtracted given that the concentration of  $\text{NaOH}$  and  $\text{H}_2\text{SO}_4$  are  $0.05 \text{ mol dm}^{-3}$ . The molar ratio of  $\text{H}_2\text{SO}_4$  to  $\text{NaOH}$  is 1:2. For this sample calculation, it is:

$$\begin{aligned} & (30.0 \text{ cm}^3 \pm 0.25) - \frac{1}{2}(16.3 \text{ cm}^3 \pm 0.25) \\ & = 21.85 \text{ cm}^3 \pm 0.375 \text{ reacted H}_2\text{SO}_4 \end{aligned}$$

Determination of the moles of  $\text{H}_2\text{SO}_4$  that reacts with  $\text{NH}_4\text{OH}$ :

$$\begin{aligned} 0.05 \text{ mol dm}^{-3} \pm 0.002 \text{ mol dm}^{-3} &= \frac{x \text{ mol}}{0.02185 \text{ dm}^3 \pm 0.000375 \text{ dm}^3} \\ x &= 1.0925 \cdot 10^{-3} \text{ mols} \pm 2.312 \cdot 10^{-5} \text{ H}_2\text{SO}_4 \end{aligned}$$



H<sub>2</sub>SO<sub>4</sub> and NH<sub>4</sub>OH are in a molar ratio of 1:2 as seen in [Equation 2](#), so to determine the concentration of NH<sub>4</sub>OH, convert the moles of H<sub>2</sub>SO<sub>4</sub> to moles of NH<sub>4</sub>OH by multiplying the moles of H<sub>2</sub>SO<sub>4</sub> by 2. Solve for concentration by dividing the moles of NH<sub>4</sub>OH by 0.1 dm<sup>3</sup>, the sample volume used for titration.

$$\begin{aligned}
 & 2\left(1.0925 \cdot 10^{-3} \text{ mol} \pm 2.312 \cdot 10^{-5} \text{ H}_2\text{SO}_4\right) \\
 & = 2.185 \cdot 10^{-3} \text{ mol} \pm 4.624 \cdot 10^{-5} \text{ NH}_4\text{OH} \\
 \text{concentration} & = \frac{2.185 \cdot 10^{-3} \text{ mol} \pm 4.624 \cdot 10^{-5}}{0.1 \text{ dm}^3} \\
 \text{concentration} & = 2.185 \cdot 10^{-2} \text{ mol dm}^{-3} \pm 4.624 \cdot 10^{-4} \text{ NH}_4\text{OH}
 \end{aligned}$$

To assess the effect of initial ammonia concentration on the extent of ammonia removal, this investigation will calculate a percentage of ammonia removed. To calculate the **ammonia removal efficiency**, divide the initial pretreated concentration of ammonium hydroxide in mol dm<sup>-3</sup> over the final treated concentration of ammonium hydroxide in mol dm<sup>-3</sup>, and subtract it from one.

### *Uncertainty*

- H<sub>2</sub>SO<sub>4</sub> - Measured by burette and diluted into a 500 cm<sup>3</sup> volumetric flask.  $\pm 0.25 \text{ cm}^3$
- NaOH - Measured by burette and diluted into a 500 cm<sup>3</sup> volumetric flask.  $\pm 0.25 \text{ cm}^3$
- NH<sub>4</sub>OH - Diluted from 14.8 mol dm<sup>-3</sup>, measured by burette and diluted into a 1 dm<sup>3</sup> volumetric flask.  $\pm 1.17 \text{ cm}^3$
- Concentration of NH<sub>4</sub>OH - Calculations as worked out above result in an uncertainty of  $\pm 4.624 \cdot 10^{-4} \text{ mol dm}^{-3}$ .

### *Calculated results*

These calculations are completed for all trials. Table 2.1 contains the calculated concentrations of ammonium hydroxide for all trials. Tables are organized by concentration, beginning with 0.020 mol dm<sup>-3</sup>. [Table 2.2](#) contains both the initial (known) pretreatment

concentration of  $\text{NH}_4\text{OH}$  ( $\text{mol dm}^{-3}$ ) for each solution and the average post-treatment concentration of  $\text{NH}_4\text{OH}$  ( $\text{mol dm}^{-3}$ ). Results for all four trials' **ammonia removal efficiency** is listed in Table 3.

*Table 2.1 Concentrations of  $\text{NH}_4\text{OH}$  ( $\text{mol dm}^{-3} \pm 4.624\text{E-}4 \text{ mol dm}^{-3}$ ) for post-treatment samples of 0.020, 0.015, 0.010 and 0.005  $\text{mol dm}^{-3} \text{NH}_4\text{OH}$ .*

Initial concentration $\text{NH}_4\text{OH}$ $\text{mol dm}^{-3}$ , $\pm 0.00117 \text{ M}$	Post treatment Trial 1 - $\text{NH}_4\text{OH}$ ( $\text{mol dm}^{-3}$ ) $\pm 4.624\text{E-}4 \text{ M}$	Post treatment Trial 2 - $\text{NH}_4\text{OH}$ ( $\text{mol dm}^{-3}$ ) $\pm 4.624\text{E-}4 \text{ M}$	Post treatment Trial 3 - $\text{NH}_4\text{OH}$ ( $\text{mol dm}^{-3}$ ) $\pm 4.624\text{E-}4 \text{ M}$	Post treatment Trial 4 - $\text{NH}_4\text{OH}$ ( $\text{mol dm}^{-3}$ ) $\pm 4.624\text{E-}4 \text{ M}$	Post treatment Trial 5 - $\text{NH}_4\text{OH}$ ( $\text{mol dm}^{-3}$ ) $\pm 4.624\text{E-}4 \text{ M}$
0.020	0.1558	0.01519	0.01517	0.01526	0.01507
0.015	0.01340	0.01360	0.01350	0.01366	0.01365
0.010	0.00966	0.00925	0.00910	0.00962	0.00926
0.005	0.00720	0.00690	0.00667	0.00694	0.00655

*Table 2.2 Average post-treatment concentration of  $\text{NH}_4\text{OH}$  ( $\text{mol dm}^{-3} \pm 4.624\text{E-}4 \text{ M}$ ) of each trial*

Average of trials 1-5 for 0.020 $\text{mol dm}^{-3} \text{NH}_4\text{OH}$ ( $\text{mol dm}^{-3}$ ) $\pm 4.624\text{E-}4 \text{ M}$	Average of trials 1-5 for 0.015 $\text{mol dm}^{-3} \text{NH}_4\text{OH}$ ( $\text{mol dm}^{-3}$ ) $\pm 4.624\text{E-}4 \text{ M}$	Average of trials 1-5 for 0.010 $\text{mol dm}^{-3} \text{NH}_4\text{OH}$ ( $\text{mol dm}^{-3}$ ) $\pm 4.624\text{E-}4 \text{ M}$	Average of trials 1-5 for 0.005 $\text{mol dm}^{-3} \text{NH}_4\text{OH}$ ( $\text{mol dm}^{-3}$ ) $\pm 4.624\text{E-}4 \text{ M}$
0.0153	0.0135	0.0094	0.0069

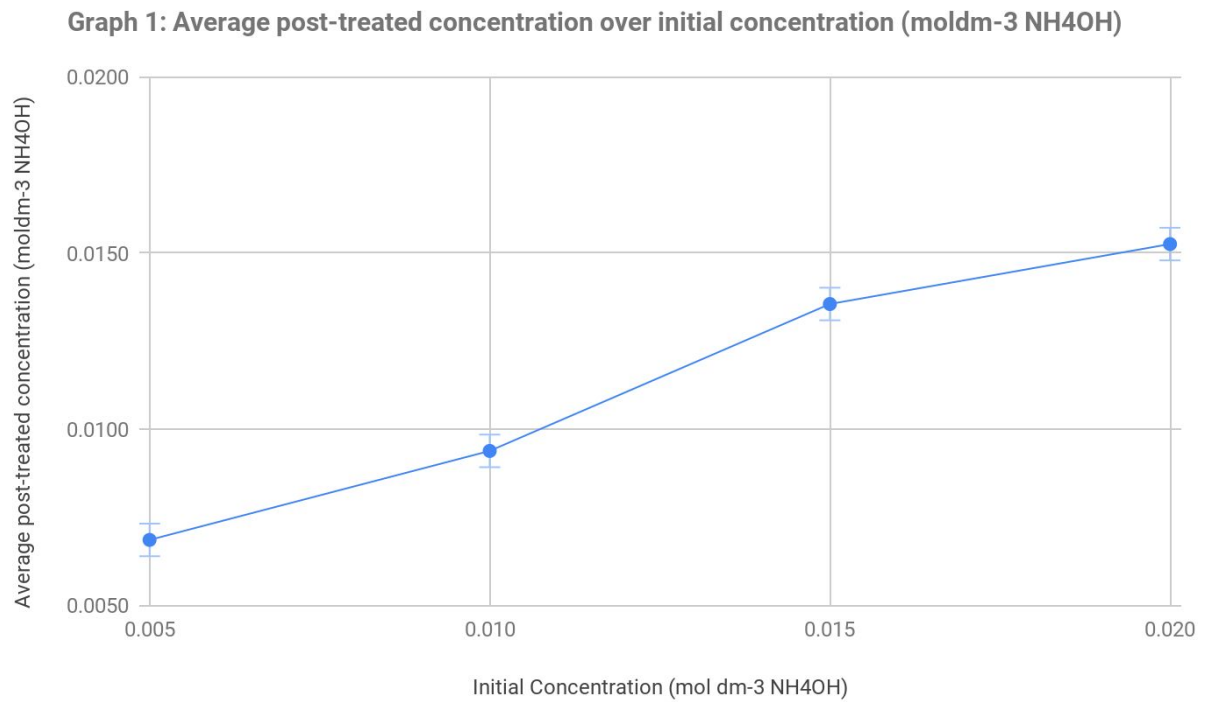
*Table 3 Post-treatment ammonia removal efficiency for each initial concentration of  $\text{NH}_4\text{OH}$*

0.020 $\text{mol dm}^{-3}$ - Removal efficiency of $\text{NH}_4\text{OH}$	0.015 $\text{mol dm}^{-3}$ - Removal efficiency of $\text{NH}_4\text{OH}$	0.010 $\text{mol dm}^{-3}$ - Removal efficiency of $\text{NH}_4\text{OH}$	0.005 $\text{mol dm}^{-3}$ - Removal efficiency of $\text{NH}_4\text{OH}$
23.75 %	9.67 %	6.23 %	-37.00 %

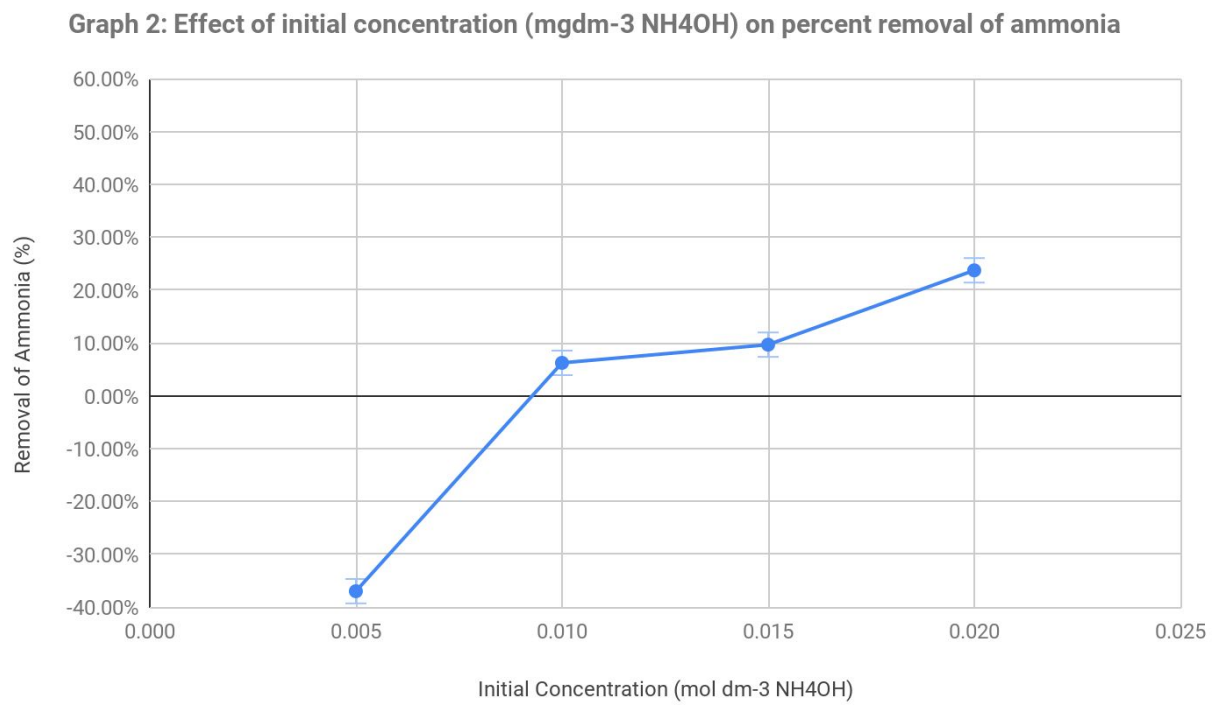
*Table 4 Pre-treatment ammonia removal efficiency for each initial concentration of  $\text{NH}_4\text{OH}$*

0.020 $\text{mol dm}^{-3}$ - Removal efficiency of $\text{NH}_4\text{OH}$	0.015 $\text{mol dm}^{-3}$ - Removal efficiency of $\text{NH}_4\text{OH}$	0.010 $\text{mol dm}^{-3}$ - Removal efficiency of $\text{NH}_4\text{OH}$	0.005 $\text{mol dm}^{-3}$ - Removal efficiency of $\text{NH}_4\text{OH}$
-2.27 %	1.48 %	16.85 %	-40.50 %

Graph 1

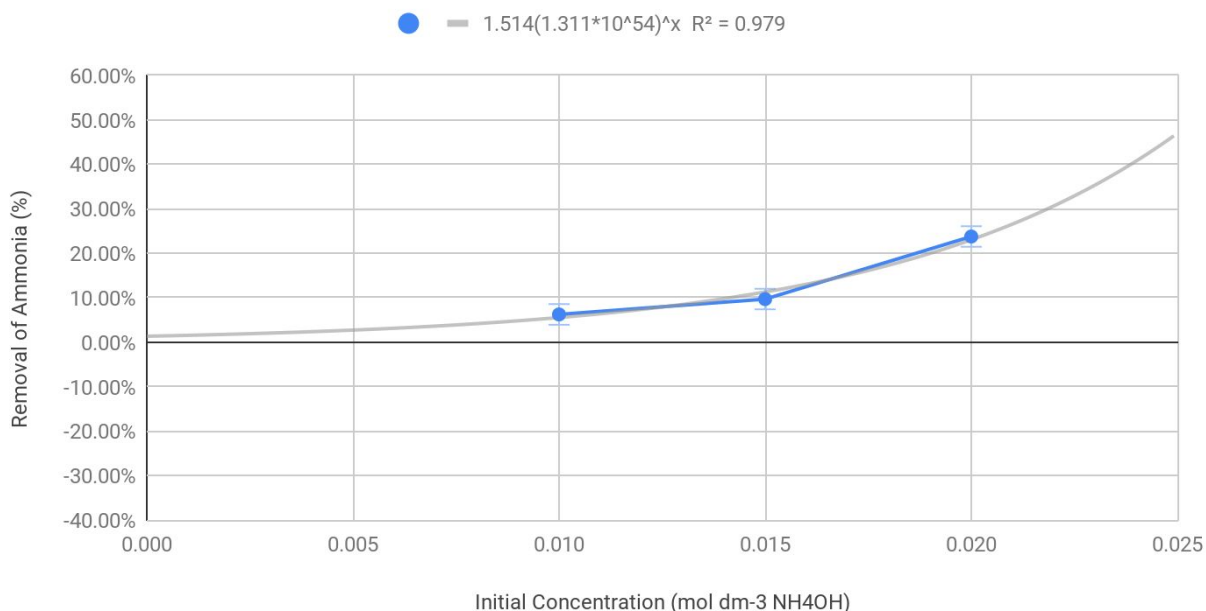


Graph 2



### Graph 3

**Graph 3: Effect of initial concentration (mgdm<sup>-3</sup> NH<sub>4</sub>OH) on percent removal of ammonia (ommiting 175 mg dm<sup>-3</sup> trial)**



### CONCLUSION

The research question for this investigation is: How effective is the method of ammonia stripping in removing different concentrations of ammonical nitrogen in wastewater?

One of the goals of this procedure was to determine the efficacy of my procedure. To achieve this, I recorded control and pretreated trials. Control trials help to determine the efficacy of the back titration. The uncertainty of mixing the solutions was determined to be  $\pm 1.17 \text{ cm}^3$  for all 5 concentrations. The result of the calculated value of the concentration of  $\text{NH}_4\text{OH}$  ( $\text{mol dm}^{-3}$ ) for the control titration are presented [Table 1.2](#). Both the concentrations of  $0.020 \text{ mol dm}^{-3}$  and  $0.010 \text{ mol dm}^{-3}$  resulted in a difference of  $+ 0.002 \text{ mol dm}^{-3} \text{ NH}_4\text{OH}$  compared to their respective actual concentrations. This, paired with the fact that the  $0.020 \text{ mol dm}^{-3}$  trial resulted in  $+ .0004 \text{ mol dm}^{-3}$  when compared to the actual concentration indicates that there was a positive systematic error in either the measurement of dilution of the solutions, or a positive systematic error in the back titration.

Secondly, I also investigated the change in concentration that was experienced by the trials after they had been heated, but before they were treated within the stripping tower. This will enable us to determine the amount of ammonia hydroxide that was lost to the heating process instead of the stripping tower. These results can be seen in [Table 4](#). A positive systematic error persists within the  $0.020 \text{ mol dm}^{-3}$  trial, while the  $0.005 \text{ mol dm}^{-3}$  also appears to have  $+0.0025 \text{ mg dm}^{-3}$  above the known concentration. Given that no trial was significantly below the known concentration, the heating of the solutions indicates no significant ammonium hydroxide loss. This shows that the ammonia lost during the ammonia stripping treatment is the only major contributing factor to the measurement of ammonium hydroxide loss throughout this method.

By looking at the graphs obtained in the previous section, it is possible to answer to the effectiveness of ammonia stripping procedure and as well as the research question. [Graph 2](#) indicates the effect of the initial concentration of ammonium hydroxide  $\text{NH}_4\text{OH}$ , on the percentage of removed ammonia from solution. There is a distinct upwards trend indicating that the greater initial concentration of  $\text{NH}_4\text{OH}$ , the greater ammonia removal efficiency. The biggest at  $700 \text{ mg dm}^{-3}$  at 23.75%. This increases gradually, as indicated by the proximity of the  $0.010 \text{ mol dm}^{-3}$  and  $0.015 \text{ mol dm}^{-3}$  concentrations, differing only by 3.44 %. However, the ammonia removal efficiency of the trial  $175 \text{ mg dm}^{-3}$  is an outlier at -37.00 %, indicating that the concentration of ammonia increased through that trial, an impossibility. As [Table 4](#) indicates, the removal efficiency of the  $0.005 \text{ mol dm}^{-3}$  trial was -40.5 %, so this outlier was already inaccurate before being processed. Therefore, it is likely caused by an initial error in mixing the solution, where a much lower concentration was used. Disregarding the  $0.005 \text{ mol dm}^{-3}$  trials, an exponential relationship of the equation  $y = 1.514(1.311 * 10^{58})^x$  is indicated by the line of best fit shown in [Graph 3](#). The  $r^2$  value for this graph is 0.979, indicating a strong relationship that remains within calculated uncertainty. This equation is limited by the range of data, as this exponential relationship would suggest a concentration greater than  $0.0313 \text{ mol dm}^{-3} \text{ NH}_4\text{OH}$  would strip more than 100 % of the ammonia.

### Limitations and unresolved questions

Limitations to this investigation include the additional investigation of factors that contribute to the concentration of ammonia in solution, outlined in 'methodology', caused by both word and procedure limitations. Access to methods of measuring ammonia concentration limited this investigation to only consider the contribution of temperature when shifting equilibrium, as it was the only factor increasing the amount of gaseous ammonia available to be removed. Raising the pH was considered to elicit more extractable ammonia gas, and has proven to raise the ammonia removal efficiency to over 90 % (Alam and Hossain 210) but was ruled out due to interference with phenolphthalein indicator being a pH-based. This investigation has been sufficient only to respond to the research question, providing a relationship between the two outlined factors. The outlier mentioned above may have been caused by its proximity to the lower limit of detection, where the phenolphthalein indicator may not be reliable.

### Further Investigation

With access to a built stripping tower, many more investigations could take advantage of this. The effect of packing material, pH, rate at which air is moved through the tower and height of packing material could all be tested to find their effect on ammonia removal to find an ideal combination of factors that could increase efficiency of ammonia removal and/or decrease price or materials. Finding the lower limit of detection (LLD) of the concentration of ammonium hydroxide for the back titration in this experiment would further assess values and limitations of this procedure and would aid in preventing inaccurate data as a result of exceedingly low concentrations.

### EVALUATION

There were many problems in the development of this experiment. The experiment required a method to measure the ammonium ion concentration after being processed. I considered multiple ways of doing this, including a purpose-built ammonium ion probe and titration testing. Due to the cost limitations, I chose to do an acid-base titration, preventing me

from my initial plan of altering the pH of the solution to increase the amount of ammonia gas in equilibrium, which would have increased the amount of ammonium removed. Unfortunately, testing at a specific concentration did not elicit correct results. The pH indicator I first used, methyl orange, had a pH range that was insufficient to the  $\text{NH}_4\text{OH}$  and  $\text{H}_2\text{SO}_4$  neutralization. To remedy both problems, I decided to do a back titration which would allow the first neutralization to occur, however also uses a secondary titration between  $\text{H}_2\text{SO}_4$  (a strong acid) and  $\text{NaOH}$  (a strong base) for measurement purposes. Measuring the pH with litmus paper, I determined phenolphthalein would better suit the range of pH between these two chemicals. Determining the concentration of ammonia to test also proved difficult as I began testing different concentrations. Given that raw wastewater typically has ammonium concentrations of  $6.25 \cdot 10^{-4} \text{ mol dm}^{-3}$  to  $9.13 \cdot 10^{-4} \text{ mol dm}^{-3}$  (Reeves 1895), I initially chose a range of 5 concentrations to test, from  $0.0005 \text{ mol dm}^{-3}$  to  $0.005 \text{ mol dm}^{-3}$ . My trial at  $0.00143 \text{ mol dm}^{-3}$  did not work, as there was no clear end point. I determined this trial to have such a low concentration that it was below the limit of detection. However, subsequent concentrations between  $0.005 \text{ mol dm}^{-3}$  and  $0.020 \text{ mol dm}^{-3}$  did elicit useful results.



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