BS 192: Chemistry Lab Report Experiment 1A: Calibration of Volumetric Glassware

Akshit Chhabra 24110026, Andhale Advait Bapurao 24110039, Rayan Talukder 24110294, Rhythem Soni 24110296, Roshia Shweta 24110304

***Abstract* — The calibration of volumetric glassware is a crucial procedure in analytical chemistry to ensure the accuracy and precision of volume measurements. This experiment involved the calibration of three different volumetric glassware: a volumetric flask, a pipette and a test tube. The calibration is performed to ensure precision and minimize the errors due to imperfections of the glassware. We measure the mass of the dry glassware and the mass of the glassware after addition of water. Subtracting the latter from the former gives us the mass of the water present in the glassware. To calculate the volume held by the glassware, we divide that mass of water by the density of water at a temperature of 30° C to account for the surrounding temperature. We took the readings for each glassware thrice and calculated the standard deviations of each of the glassware. We obtained the standard deviations as:** $0.049\ mL$ **for the volumetric flask,** $0.107\ mL$ **for the pipette and** $0.014\ mL$ **for the test tube. In addition to this, we also prepared a NaCl solution of the given molarity.**

***Index Terms* - Volumetric Calibration, Analytical Chemistry, Volumetric Glassware, Water Density and Error Analysis calibration**

I. Introduction

We often use glass tools in various experiments to measure the amount of substances essential for experiments. Calibrating glassware is a standard practice in laboratory science to ensure accurate and reliable measurements. Calibration helps identify and correct these discrepancies, providing precise measurements. This experiment tests the accuracy of three essential tools used in chemistry: volumetric flasks, pipettes, and measuring cylinders. These instruments should have precise volume measurements for gravimetric and titrimetric analysis. It is important to have careful calibration to minimize errors, as improper calibration can degrade the quality of the entire analysis.

This experiment calibrates each instrument with distilled water. We measure the mass of empty glassware and then measure the mass when the water is filled into it. We obtain the mass of water each instrument contains by subtracting the empty mass from the filled mass and then converting it to volume using the known density of water at the calibration temperature. We then compare the volume measured with the instrument's specified volume to verify its accuracy. This helps us to establish any discrepancies and correct them for future use.

II. Materials and Methods

A. Materials Required

1) Volumetric Flask (100ml)
2) Pipette (10ml)
3) Test Tube (Unknown Volume)
4) Beaker (50ml)
5) Rubber Bulb
6) Distilled Water
7) Wash Bottle
8) Analytical Balance
9) Dropper
10) Tissue Papers
11) Safety Glasses
12) Gloves

B. Methods

- Volumetric Flask:
  1) Tare the analytical balance and ensure that it displays the value 0.
  2) Place the empty volumetric flask into the analytical balance and note down its mass.
  3) Fill the volumetric flask with distilled water using the wash bottle and dropper until the lower meniscus of the water reaches the mark.
  4) Now, place the flask on the analytical balance and note down its mass.
  5) Empty the volumetric flask and completely dry it.
  6) Repeat steps 1 to 5 (except step 2) twice to obtain three readings of the mass of the filled volumetric flask.
- Pipette:
  1) Tare the analytical balance and ensure that it displays the value 0.
  2) Place the empty beaker into the analytical balance and note down its mass.
  3) Attach the rubber bulb onto the pipette and fill the pipette with distilled water until the lower meniscus of water reaches the mark.

4) Drain all the water from the pipette into the empty beaker.
5) Now, place the beaker on the analytical balance and note down its mass.
6) Empty the beaker and completely dry it.
7) Repeat steps 1 to 6 (except step 2) twice to obtain three readings of the mass of the filled beaker.

- Test Tube:
  1) Tare the analytical balance and ensure that it displays the value 0.
  2) Place the empty volumetric flask into the analytical balance and note down its mass
  3) Fill the test tube with distilled water using the wash bottle and dropper until the lower meniscus of the water reaches the mark.
  4) Pour all the water from the test tube into the empty beaker.
  5) Now, place the beaker on the analytical balance and note down its mass.
  6) Empty the beaker and completely dry it.
  7) Repeat steps 1 to 6 (except step 2) twice to obtain three readings of the mass of the filled beaker.

III. Results

A. Data Used for Calculations

- All the readings were taken at a room temperature of around 30° C.
- The density of distilled water used for the calculations at a room temperature of 30° C was $0.9956502\ g/mL$.

B. Notations Used

- The i^{th} volume reading has been taken to be V_i.
- The error of the i^{th} volume reading has been taken to be ΔV_i.
- The total number of readings has been taken to be N.
- The mean volume has been taken to be μ.
- The standard deviation in the volume has been taken to be σ.

C. Formulae Used

- To calculate the volume:

$$Volume = \frac{Mass}{Density} \tag{1}$$

- The mean μ is calculated as:

$$\mu = \frac{\sum_{i=1}^{N} V_i}{N} \tag{2}$$

- Error in the i_{th} volume reading:

$$\Delta V_i = |V_i - \mu| \tag{3}$$

- To find the standard deviation:

$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N} (\Delta V_i)^2} \tag{4}$$

D. Readings

Table 1 shows the readings of the dry apparatus and the three readings taken for each glassware.

TABLE I: Table of readings noted

Volumetric Glassware	mass of Dry Apparatus	mass of Apparatus Filled With Water
Volumetric Flask (100 mL)	68.108 g (volumetric flask)	167.347 g
		167.435 g
		167.461 g
Pipette	28.366 g (beaker)	38.184 g
		38.403 g
		38.418 g
Test Tube (Unknown)	28.366 g (beaker)	32.023 g
		32.016 g
		32.048 g

E. Calculations

Table 2 shows the values of masss of distilled water calculated after subtracting the dry apparatus' mass from the filled apparatus' mass, the volumes calculated using the formula in equation (1), and the corresponding average volume calculated for each glassware using the formula in equation (2).

TABLE II: Table of volumes of water calculated

Volumetric Glassware	mass of Water in the Volumetric Glassware	Volume of Water (in mL)	Average Volume of Water
Volumetric Flask (100 mL)	99.239 g	99.673	99.741 mL
	99.327 g	99.761	
	99.353 g	99.788	
Pipette	9.818 g	9.861	10.013 mL
	10.037 g	10.081	
	10.052 g	10.096	
Test Tube (Unknown)	3.657 g	3.673	3.679 mL
	3.650 g	3.666	
	3.682 g	3.698	

1) Volumetric Flask:

Calculating the absolute error in volume,

$$\Delta V_1 = |V_1 - \mu| = 0.068\ mL$$

$$\Delta V_2 = |V_2 - \mu| = 0.020\ mL$$

$$\Delta V_3 = |V_3 - \mu| = 0.047\ mL$$

Calculating the standard deviation in volume using the formula in equation (4), we obtain:

$$\sigma = 0.049\ mL$$

Therefore, we obtained a percentage error of:

$$0.049\ \%$$

2) Pipette:

Calculating the absolute error in volume,

$$\Delta V_1 = |\mu - V_1| = 0.152\ mL$$
$$\Delta V_2 = |\mu - V_2| = 0.068\ mL$$
$$\Delta V_3 = |\mu - V_3| = 0.083\ mL$$

Calculating the standard deviation in volume using the formula in equation (4), we obtain:

$$\sigma = 0.107\ mL$$

Therefore, we obtained a percentage error of:

$$1.07\ \%$$

3) Test Tube:

Calculating the absolute error in volume,

$$\Delta V_1 = |\mu - V_1| = 0.006\ mL$$
$$\Delta V_2 = |\mu - V_2| = 0.013\ mL$$
$$\Delta V_3 = |\mu - V_3| = 0.019\ mL$$

Calculating the standard deviation in volume using the formula in equation (4), we obtain:

$$\sigma = 0.014\ mL$$

Therefore, we obtained a percentage error of:

$$0.381\ \%$$

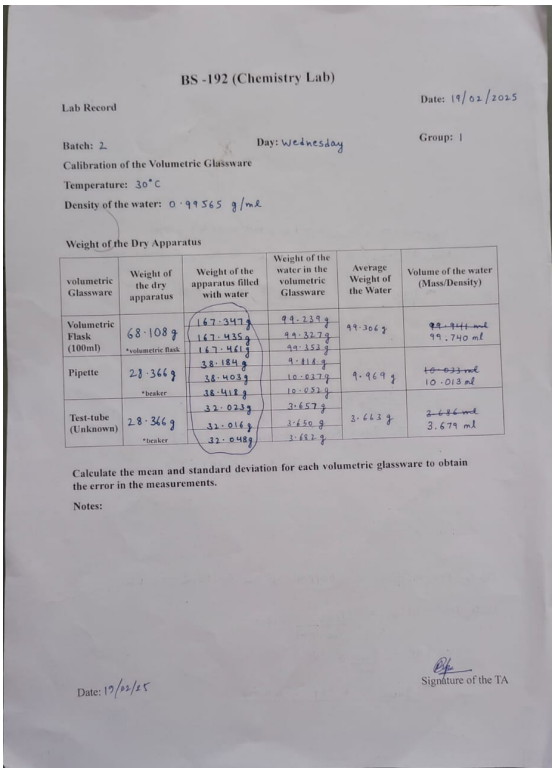
IV. Conclusion

In this experiment, we calibrated volumetric glassware such as a volumetric flask, a pipette, and a test tube using distilled water at room temperature(30° C). Calibration is crucial for ensuring the accuracy of measurements in precise methods like weighing (gravimetric analysis) and determining chemical concentrations (titrimetric analysis). This step is necessary because reliable measurements are key to getting correct results in any analytical procedure.

We obtained the standard deviations as: $0.049\ mL$ for the volumetric flask, $0.107\ mL$ for the pipette and $0.014\ mL$ for the test tube, which indicates high precision in our measurements. We experienced the highest percentage error (1.07%) when calibrating the pipette. This could be since it was difficult to fill the pipette to the exact level required using the rubber bulb.

The values that we got after calculating the results were reasonably close to the claimed values. Small deviations from the advertised values were expected due to human error while experimenting, manufacturing defects and temperature differences. That said, time to time calibration is required to make sure that lab equipment is in good working order.

V. Images of Readings



BS -192 (Chemistry Lab)

Lab Record — Date: 19/02/2025

Batch: 2 — Day: Wednesday — Group: 1

Calibration of the Volumetric Glassware

Temperature: 30°C

Density of the water: 0.99565 g/mL

Weight of the Dry Apparatus

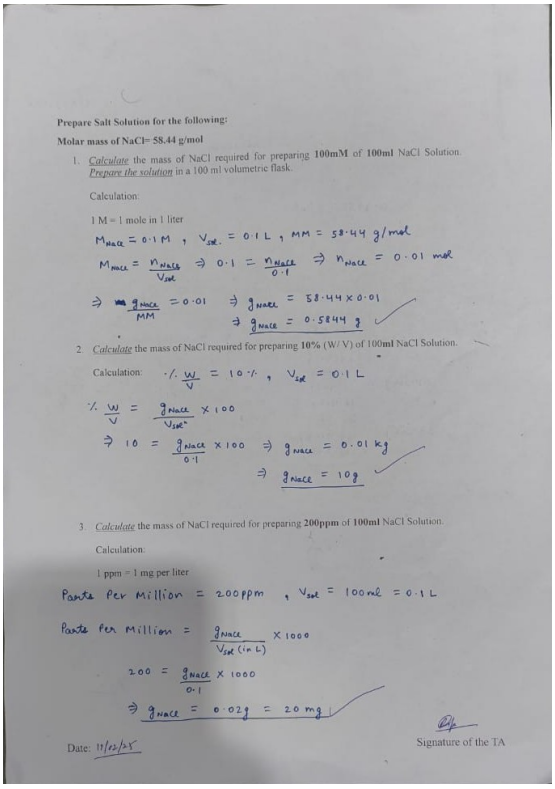
volumetric Glassware	Weight of the dry apparatus	Weight of the apparatus filled with water	Weight of the water in the volumetric Glassware	Average Weight of the Water	Volume of the water (Mass/Density)
Volumetric Flask (100ml)	68.108 g *volumetric flask	167.347 g 167.435 g 167.461 g	99.239 g 99.327 g 99.353 g	99.306 g	~~99.944 ml~~ 99.740 ml
Pipette	28.366 g *beaker	38.184 g 38.403 g 38.418 g	9.818 g 10.037 g 10.052 g	9.969 g	~~10.033 ml~~ 10.013 ml
Test-tube (Unknown)	28.366 g *beaker	32.023 g 32.016 g 32.048 g	3.657 g 3.650 g 3.682 g	3.663 g	~~3.686 ml~~ 3.679 ml

Calculate the mean and standard deviation for each volumetric glassware to obtain the error in the measurements.

Notes:

Date: 19/02/25 — Signature of the TA

Fig. 1: Observation Sheet Signed by TA



Prepare Salt Solution for the following:

Molar mass of NaCl= 58.44 g/mol

1. *Calculate* the mass of NaCl required for preparing **100mM** of **100ml** NaCl Solution. *Prepare the solution* in a 100 ml volumetric flask.

Calculation:

1 M = 1 mole in 1 liter

$M_{NaCl} = 0.1\ M$, $V_{sol.} = 0.1\ L$, $MM = 58.44\ g/mol$

$M_{NaCl} = \frac{n_{NaCl}}{V_{sol}} \Rightarrow 0.1 = \frac{n_{NaCl}}{0.1} \Rightarrow n_{NaCl} = 0.01\ mol$

$\Rightarrow \frac{g_{NaCl}}{MM} = 0.01 \Rightarrow g_{NaCl} = 58.44 \times 0.01$

$\Rightarrow g_{NaCl} = 0.5844\ g$

2. *Calculate* the mass of NaCl required for preparing **10%** (W/V) of **100ml** NaCl Solution.

Calculation: $\% \frac{W}{V} = 10\%$, $V_{sol} = 0.1\ L$

$\% \frac{W}{V} = \frac{g_{NaCl}}{V_{sol}} \times 100$

$\Rightarrow 10 = \frac{g_{NaCl}}{0.1} \times 100 \Rightarrow g_{NaCl} = 0.01\ kg$

$\Rightarrow g_{NaCl} = 10\ g$

3. *Calculate* the mass of NaCl required for preparing **200ppm** of **100ml** NaCl Solution.

Calculation:

1 ppm = 1 mg per liter

Parts Per Million = 200ppm, $V_{sol} = 100ml = 0.1\ L$

Parts Per Million = $\frac{g_{NaCl}}{V_{sol\ (in\ L)}} \times 1000$

$200 = \frac{g_{NaCl}}{0.1} \times 1000$

$\Rightarrow g_{NaCl} = 0.02g = 20\ mg$

Date: 19/02/25 — Signature of the TA

Fig. 2: Calculation Sheet Signed by TA

VI. Author Contributions

1) Akshit Chhabra, 24110026
   - Performed the experiment and filled the volumetric glassware with distilled water such that the lower meniscus reaches the mark.
   - Completed the 'Abstract' and 'Author Contributions' sections and compiled, formatted the report on LaTeX.
2) Andhale Advait Bapurao, 24110039
   - Performed the experiment and assisted in transfer of water and drying of apparatus.
   - Completed the 'Conclusion' section.
3) Rayan Talukder, 24110294
   - Performed the experiment and performed all the calculations for difference, mean and standard deviation in the observation sheet.
   - Completed the 'Results' section.
4) Rhythem Soni, 24110296
   - Performed the experiment and assisted in measuring and noting down the readings of the mass of various apparatus.
   - Completed the 'Introduction' section and assisted in the 'Materials and Methods' section.
5) Roshia Shweta, 24110304
   - Performed the experiment and assisted in measuring and noting down the readings of the mass of various apparatus.
   - Assisted in the 'Materials and Methods' section.

References

[1] BS 192 Chemistry Lab Manual

Experiment 1B: Synthesis of Commercial Polymer Nylon 6, 6

Akshit Chhabra 24110026, Andhale Advait Bapurao 24110039, Rayan Talukder 24110294,
Rhythem Soni 24110296, Roshia Shweta 24110304

***Abstract* — In this experiment, we synthesize Nylon 6,6, a commercially significant polyamide polymer though a step-growth poly-condensation reaction between hexamethylene-diamine(HMDA) and adipoyl chloride. We conduct the reaction under both neutral and basic conditions. The reaction was conducted under both basic and neutral conditions. The resulting polymer is then extracted, cleaned, dried, and analyzed. The Fourier Transform Infrared (FTIR) spectra of the reactants and the final polymer are measured to confirm the successful formation of Nylon 6,6 by identification characteristic functional groups. This experiment highlights the fundamental principles of polymer chemistry and the industrial significance of Nylon 6,6 in various industries.**

***Index Terms* - Polymers, Polymer Synthesis, Nylon, Hexamethylenediamine, Adipoyl Chloride, Step-Growth Polymerization, Fourier Transform Infrared Spectroscopy, FTIR**

I. Introduction

Nylon, a widely used polymer, primarily exists in two forms: nylon 6, 6 and nylon 6. Developed in the 1930s, these two variants share many similarities. Nylon 6, 6 was first synthesized in the USA by Wallace Carothers at DuPont. It is known for its strength, durability and chemical resistance and hence has become an essential material in textile and plastic manufacturing. It is produced through a self-growth poly-condensation reaction between hexamethylenediamine and either adipoyl chloride or adipic acid, resulting in a high molecular weight polymer.

This experiment involves the addition of adipoyl chloride to hexamethylenediamine in either water (neutral conditions) or NaOH (basic conditions) causing the polymerization to occur at the liquid-liquid interface. The resulting nylon 6, 6 polymer is collected using forceps and rolled around a dropper and then later washed and dried using an air gun. Then FTIR spectroscopy is performed on the reactants and the final polymer product to confirm the synthesis of nylon 6, 6.

II. Materials and Methods

A. Materials Required

1) Hexamethylenediamine
2) Adipoyl Chloride
3) 0.5M NaOH Solution
4) Cyclohexane
5) 3 Test Tubes
6) 2 Petri Dishes
7) 2 Eppendorf Tubes
8) 5 Droppers
9) Heat Gun
10) Forceps
11) Filter Papers
12) Safety Glasses
13) Gloves

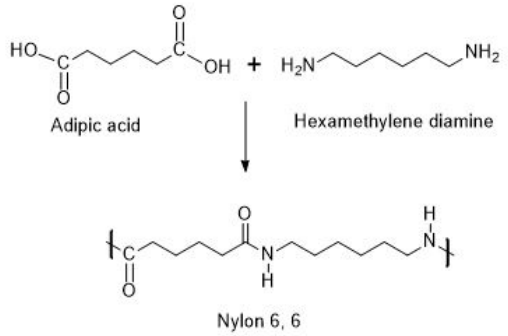


Fig. 1: Synthesis of Nylon 6, 6

B. Methods

1) Dissolve adipoyl chloride in cyclohexane at a 1% v/v concentration, mixing well to achieve a homogeneous solution.
2) Add hexamethylenediamine to a 0.5 M NaOH solution at a concentration of 1% v/v.
3) Mix hexamethylenediamine with water in another container at the same 1% v/v concentration.
4) Take two dry, clean petri dishes and add 5 ml of the prepared hexamethylenediamine solutions on each.
5) Slowly add 2 ml adipoyl chloride solution drop by drop to the surface of both solutions from steps 3 and 4.
6) Let the polymerization reaction occur. Nylon 6,6 will be produced at the interface of the two solutions, with hydrogen chloride (HCl) as a byproduct.
7) Carefully pick up the polymer at the interface using forceps from the solvent and wrap it around a dropper.
8) Allow any excess solution to drain off from the polymer.
9) Wash the polymer well with water to eliminate any residual solvent or reactants with gentle agitation. After

washing, dry the Nylon 6,6 with filter paper and heat it gently with a heat gun.

10) Then, place the polymers in an Eppendorf tube or other container and label them (neutral medium and basic medium).
11) Measure the FTIR spectra of the polymer when completely dry and compare it with the FTIR spectra of the starting materials.

III. Results

A. FTIR spectroscopy on the reactants

On performing FTIR spectroscopy of adipoyl chloride using a spectrometer, the spectrum labeled Fig. 2 was obtained.

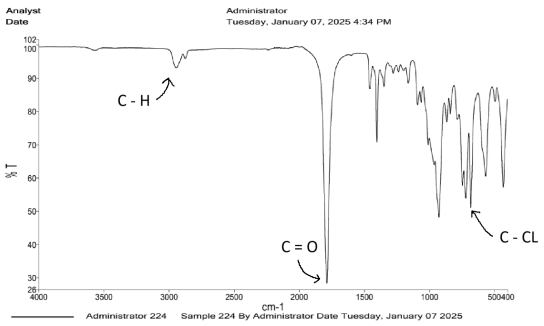


Fig. 2: FTIR spectroscopy of adipoyl chloride

The peaks and their corresponding confirmed functional groups in adipoyl chloride's FTIR spectra are listed below in Table 1.

TABLE I: Wave numbers absorbed by bonds in adipoyl chloride

Bond	Wave Number (cm^{-1})
C - H	2800-3000
C = O	1700-1800
C - Cl	600 - 800

On performing FTIR spectroscopy of hexamethylenediamine using a spectrometer, the spectrum labeled Fig. 3 was obtained.

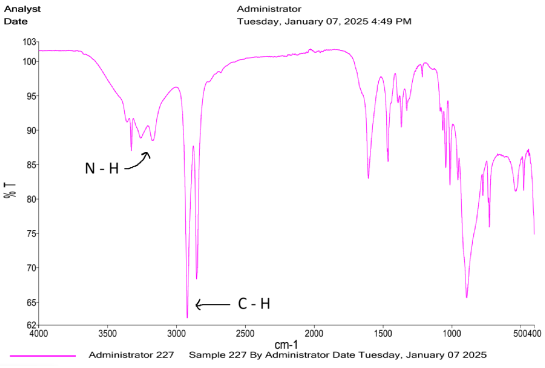


Fig. 3: FTIR spectroscopy of hexamethylenediamine

The peaks and their corresponding confirmed functional groups in hexamethylenediamine's FTIR spectra are listed below in Table 2.

TABLE II: Wave numbers absorbed by bonds in hexamethylenediamine

Bond	Wave Number (cm^{-1})
N - H	3200 - 3300
C - H	2800 - 3000

B. FTIR spectroscopy on the resulting polymers

On performing FTIR spectroscopy of nylon 6, 6 formed in basic medium using a spectrometer, the spectrum labeled Fig. 4 was obtained.

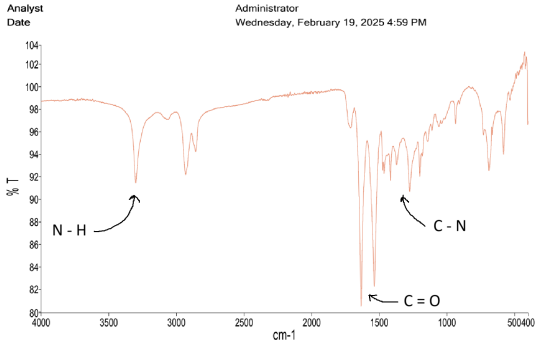


Fig. 4: FTIR spectroscopy of nylon 6, 6 (basic medium)

The peaks and their corresponding confirmed functional groups in (basic medium) nylon's FTIR spectra are listed below in Table 3.

TABLE III: Wave numbers absorbed by bonds in nylon 6, 6 (basic medium)

Bond	Wave Number (cm^{-1})
N - H	3200 - 3300
C = O	1700 - 1800
C - N	1200 - 1300

The rate of reaction was faster and a clear structure of nylon-6,6 polymer because HCl, the side product kept being removed upon neutralization with the NaOH present in the reaction solution to form salt and water. This does not disturbed the flow of reaction by causing a delay or decreasing speed of reaction. Also, no harm was caused to the structure of nylon-6, 6 .

On performing FTIR spectroscopy of nylon 6, 6 formed in neutral medium using a spectrometer, the spectrum labeled Fig. 5 was obtained.

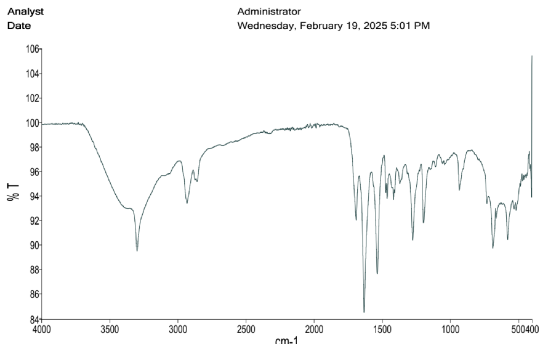


Fig. 5: FTIR spectroscopy of nylon 6, 6 (neutral medium)

Broad N-H peaks and unclear or missing C-N peaks indicated weaker bond formation, highlighting the impact of unneutralized HCl.

The rate of reaction was slow and a faded structure of nylon-6, 6 polymer appeared because of the production of HCl, the side product, was not removed up by any base and hence either made the reaction backward into the reactant form according to Le-Chateiler's principle or went on to react with the -NH group in nylon-6, 6 for protonation. This made the nylon-6,6 polymer bit unstable and the amount produced was also less.

IV. Conclusion

In this experiment, we synthesized nylon 6, 6, a polymer that is widely used in the plastics and textiles industry. The procedure involved making an adipoyl chloride solution in cyclohexane and hexamethylenediamine solution in basic and neutral mediums and then adding the adipoyl chloride solution to the hexamethylenediamine solutions leading to poly-condensation, forming a white rubbery nylon 6, 6 film which was collected, washed, dried and heated.

We also did FTIR spectra analysis of the reactants and the polymers formed using a spectroscope to verify that the target polymer was actually made. We identified the reactants and products by noticing the peaks in the spectra and relating them to the functional groups that absorb that energy. This experiment showed how spectroscopy which might have seemed like a abstract concept earlier can be used to help us understand the reactions around us and learn more about the world.

V. Author Contributions

1) Akshit Chhabra, 24110026
   - Performed the experiment and assisted in obtaining the nylon film and washing, drying and heating it to prepare it for the FTIR spectra analysis.
   - Completed the 'Abstract' and 'Author Contributions' sections and compiled, formatted the report on LaTeX.
2) Andhale Advait Bapurao, 24110039
   - Performed the experiment and assisted in measuring and mixing of the chemical solutions.
   - Completed the 'Conclusion' section.
3) Rayan Talukder, 24110294
   - Performed the experiment and assisted in obtaining the nylon film and washing, drying and heating it to prepare it for the FTIR spectra analysis.
   - Completed the 'Results' section.
4) Rhythem Soni, 24110296
   - Performed the experiment and assisted in measuring and mixing of the chemical solutions.
   - Completed the 'Introduction' section and assisted in the 'Materials and Methods' section.
5) Roshia Shweta, 24110304
   - Performed the experiment and assisted in preparing the chemical solutions.
   - Assisted in the 'Materials and Methods' section.

References

[1] BS 192 Chemistry Lab Manual