

# Synthesis of a commercial polymer, Nylon 6,6

Birudugadda Srivibhav  
22110050

IIT Gandhinagar  
birudugadda.srivibhav@iitgn.ac.in

Ankeshwar Ruthesha  
22110024

IIT Gandhinagar  
ankeshwar.ruthesha@iitgn.ac.in

Kirtankumar Patel  
22110185

IIT Gandhinagar  
kirtankumar.patel@iitgn.ac.in

Nikhil Kumar Lal  
22110166

IIT Gandhinagar  
nikhilkumar.lal@iitgn.ac.in

**Abstract**—This work presents the synthesis of nylon 6,6 through the polycondensation reaction between hexamethylenediamine and adipoyl chloride. Additionally, the functional groups present before and after the reaction are identified using FTIR spectroscopy.

## I. INTRODUCTION

Nylon 6,6 is a widely known synthetic polymer, recognized for its high strength, durability, and resistance to heat and wear. It was first discovered in the 1930s by Wallace Carothers and his team at the DuPont research laboratory. Since its discovery, many variations of nylon have been developed. Nylon 6,6 finds applications in numerous everyday products, including clothing, carpets, ropes, parachutes, automotive components, and industrial machinery. It is synthesized via the polycondensation reaction of hexamethylenediamine with adipoyl chloride, with sodium hydroxide added to the reaction mixture to facilitate amide bond formation.

## II. THEORY

### A. Polymers

Polymers are large macromolecules composed of repeating smaller units known as monomers, which bond together in a chain-like structure. Polymers can be either natural or synthetic and are present in a variety of materials, including those found in living organisms, minerals, and artificial products. Nylon 6,6 is an example of a synthetic polymer, consisting of two monomers: hexamethylenediamine and adipic acid, both containing six carbon atoms.

### B. Nylon 6,6

Nylon 6,6 is classified as a step-growth polymer, where each bond forms independently of others in a stepwise manner. It is also a condensation polymer, as it is produced by joining two molecules with the elimination of a smaller

molecule, such as water.

To synthesize nylon 6,6, a solution of adipoyl chloride in cyclohexane is layered over an aqueous solution of hexamethylenediamine. Due to the immiscibility of cyclohexane and water, a two-layer system is formed. Polymerization occurs at the interface between the two liquids, where adipoyl chloride, a diacid chloride, reacts with hexamethylenediamine, a diamine, to form nylon 6,6 through the formation of amide bonds. Hydrochloric acid (HCl) is released as a by-product during this polymerization. To facilitate amide formation, sodium hydroxide can be added to the hexamethylenediamine solution. The resulting nylon 6,6 polymer consists of alternating hexamethylene and adipoyl units, giving rise to a strong and flexible material.

As a synthetic polymer, nylon 6,6 is widely used in various industries due to its strength, durability, and resistance to abrasion and chemicals. It is commonly found in textiles such as clothing and carpets, as well as in industrial components like gears and bearings, and automotive parts. Its high mechanical properties and thermal stability contribute to its versatility, making it a valuable material for both consumer products and industrial applications.

### C. FTIR Spectroscopy

Fourier-transform infrared (FTIR) spectroscopy is a highly effective analytical method used to identify molecular structures by measuring a sample's absorption of infrared radiation. The resulting spectrum reflects the vibrational frequencies of the chemical bonds present in the sample. FTIR spectroscopy is extensively utilized for characterizing organic compounds, identifying functional groups, and analyzing complex mixtures. Its capability to offer detailed insights into molecular

interactions and chemical environments makes it a crucial tool in fields such as chemistry, materials science, and pharmaceuticals.

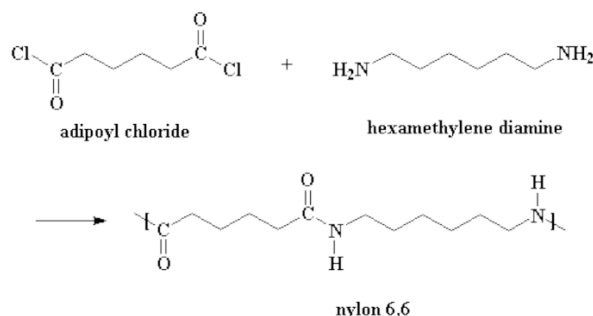


Fig. 1. Chemical reaction to synthesise Nylon 6,6 from adipoyl chloride and hexamethylenediamine

### III. EXPERIMENTAL PROCEDURE

#### A. Apparatus and Materials Used

- **Adipoyl chloride:** A diacid chloride that reacts with diamines to form nylon via polycondensation. It is commonly used in the synthesis of nylon 6,6.
- **Hexamethylenediamine:** A diamine that reacts with adipoyl chloride to produce nylon 6,6. It is a colorless solid or liquid at room temperature.
- **NaOH solution:** Sodium hydroxide is used to neutralize the HCl produced during the polymerization reaction, facilitating the formation of amide bonds.
- **Cyclohexane:** A non-polar solvent used to dissolve adipoyl chloride. Cyclohexane is immiscible with water, which helps create a distinct phase boundary for polymerization.
- **Watch glass:** A shallow glass dish used to hold small amounts of liquid or for evaporation purposes. Here, it is used to contain the solutions during the nylon formation reaction.
- **Water:** Acts as a solvent in the preparation of hexamethylenediamine solutions and helps form an immiscible layer with cyclohexane for the interfacial polymerization.
- **Test-tube:** A cylindrical glass container used to mix and hold the reaction solutions during the polymerization process.
- **Heat gun:** A tool used to gently heat and dry the formed polymer after washing, removing residual moisture and solvents.
- **Forceps:** Small, tweezer-like tools used to handle and lift the delicate nylon polymer film formed at the interface of the liquid layers.
- **Droppers:** Small pipettes used to carefully add adipoyl chloride solution dropwise to the hexamethylenediamine solution, controlling the reaction rate.
- **Filter paper:** Used to blot and dry the polymer after it has been washed with water to remove impurities and by-products like NaCl.

- **Eppendorf tube:** A small, conical plastic container used for storing the synthesised nylon polymer after washing and drying.

#### B. Methodology

- 1) Begin by performing FTIR spectroscopy on the pure starting materials, adipoyl chloride and hexamethylenediamine, to obtain their respective spectra.
- 2) Dissolve adipoyl chloride in cyclohexane at a concentration of 1% v/v.
- 3) Prepare two solutions of hexamethylenediamine: one by dissolving it in a 0.5M NaOH solution (1% v/v) for polymerization in a basic medium, and another by dissolving it in water (1% v/v) for polymerization in a neutral medium.
- 4) Dispense 5 ml of each hexamethylenediamine solution into separate watch glasses or Petri dishes.
- 5) Using a dropper, carefully add the adipoyl chloride solution dropwise to the surface of both hexamethylenediamine solutions.
- 6) Allow the polymerization reaction to take place, leading to the formation of nylon 6,6 and the release of hydrogen chloride (HCl) as a by-product.
- 7) Use forceps to lift the polymer formed from the surface of the solution.
- 8) Drain any excess liquid from the polymer and transfer it to an Eppendorf tube.
- 9) Wash the polymer with water to remove impurities, dry it with filter paper, and gently heat it using a heat gun.
- 10) Finally, perform FTIR spectroscopy on the synthesized polymers and compare their spectra with those of the starting materials to evaluate the polymerization process.

### IV. CALIBRATIONS

The FTIR spectra obtained in this experiment display % Transmittance on the y-axis and wavenumber on the x-axis. In some cases, the y-axis represents absorbance, which can be converted to % Transmittance using the following formula:

$$\%T = \text{antilog}(2 - \text{absorbance})$$

To determine the coordinates of the peaks in the IR spectroscopy graphs, the Web Plot Digitizer software was used. Two known points along both the x and y axes were input to initialize the values for the graphs.

Additionally, in the spectrum obtained from the nylon synthesized in the NaOH solvent, the graph appears inverted. The values are centered around 100 % Transmittance, and several local maxima are observed at the same frequencies where local minima would be expected in the nylon 6,6 compound.

### V. RESULTS AND OBSERVATION

#### A. Results

- 1) The graphs below display the FTIR spectra of pure adipoyl chloride and hexamethylenediamine.

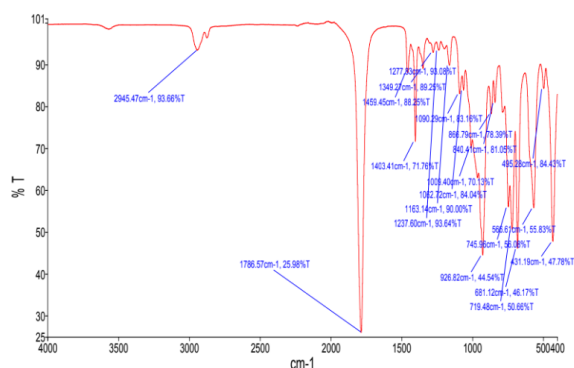


Fig. 2. FTIR spectra of Adipoyl chloride

- The peak at  $2945.47\text{ cm}^{-1}$  is due to the stretching of the alkane bond between carbon and hydrogen (C-H).
- The peak at  $1786.57\text{ cm}^{-1}$  is due to the presence of acid chloride functional group.
- The peaks in the range  $1450\text{--}1300\text{ cm}^{-1}$  are due to the bending of alkane bond.
- The peaks in the range  $785\text{--}540\text{ cm}^{-1}$  are due to the C-Cl bond.
- From this FTIR spectra, we can observe all the functional groups present in the adipoyl chloride.

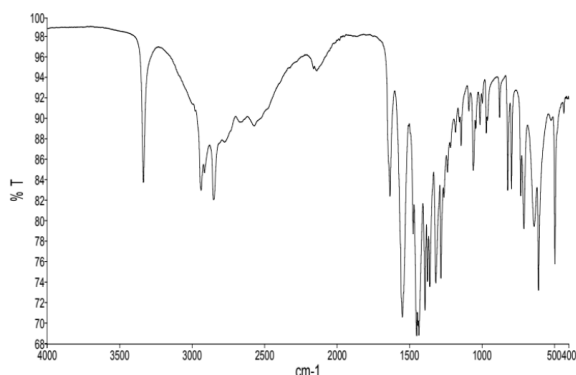


Fig. 3. FTIR spectra of Hexamethylene diamine

- The peak at  $3350\text{--}3370\text{ cm}^{-1}$  is due to the stretching of the N-H bond in primary amines.
- The peaks at  $2800\text{--}3000\text{ cm}^{-1}$  are due to the stretching of the alkane bond between carbon and hydrogen (C-H).
- The peaks at  $1500\text{--}1700\text{ cm}^{-1}$  are due to the bending of the N-H bond in primary amines.
- The peak around  $1450\text{ cm}^{-1}$  is due to the bending of the alkane bond between carbon and hydrogen (C-H).
- The peak around  $1100\text{ cm}^{-1}$  is due to the C-N bond.
- From this FTIR spectra, we can observe all the functional groups present in the hexamethylene diamine.

- The graph below shows the FTIR spectrum of nylon 6,6 when synthesized with hexamethylenediamine dissolved in water solvent.

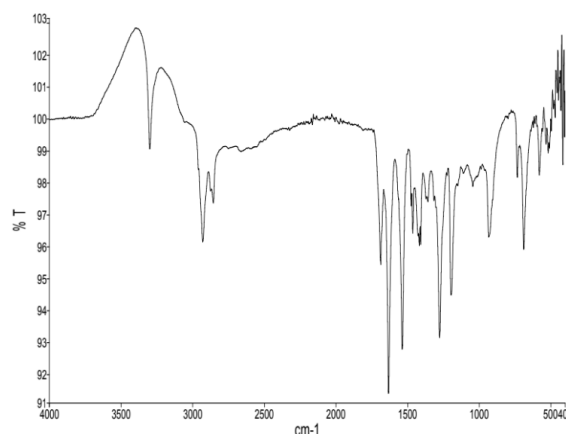


Fig. 4. FTIR spectra of Nylon 6,6 in water solvent

- The peak around  $3300\text{ cm}^{-1}$  is due to the stretching of the N-H bond.
- The peak around  $3000\text{ cm}^{-1}$  is due to the stretching of the alkane bond between carbon and hydrogen (C-H).
- The peak around  $1650\text{ cm}^{-1}$  is due to the amide functional group.
- The peak around  $1550\text{ cm}^{-1}$  is due to the bending of the N-H bond.
- The peaks around  $1200\text{--}1300\text{ cm}^{-1}$  are due to the C-N bonds.
- The peak around  $700\text{ cm}^{-1}$  is due to the remaining reactant (C-Cl bond in adipoyl chloride)
- From this FTIR spectra, we can observe all the functional groups present in Nylon-6,6 synthesised in water solvent.

- The following graph displays the infrared spectrum of Nylon 6,6 compound upon synthesis using hexamethylenediamine dissolved in 0.5 M NaOH solvent.

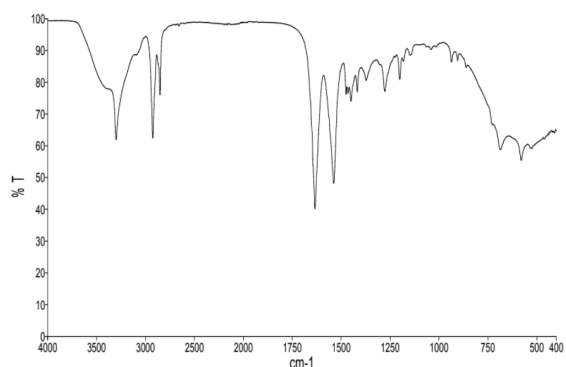


Fig. 5. FTIR spectra of nylon 6,6 in NaOH solvent

- The peak around  $3300\text{ cm}^{-1}$  is due to the stretching of the N-H bond.

- b) The peak around  $2900\text{ cm}^{-1}$  is due to the stretching of the alkane bond between carbon and hydrogen (C-H).
- c) The peak around  $1650\text{ cm}^{-1}$  is due to the amide functional group.
- d) The peak around  $1500 - 1550\text{ cm}^{-1}$  is due to the bending of the N-H bond.
- e) The peaks around  $1200 - 1300\text{ cm}^{-1}$  are due to the C-N bonds.
- f) The peaks around  $600 - 750\text{ cm}^{-1}$  is due to the remaining reactant (C-Cl bond in adipoyl chloride)
- g) From this FTIR spectra, we can observe all the functional groups present in Nylon-6,6 synthesised in NaOH solvent.

## VI. DISCUSSION

The shifting of the peak due to the carbonyl bond, from  $1786.57\text{ cm}^{-1}$  in adipoyl chloride to around  $1650\text{ cm}^{-1}$  in nylon 6,6 prepared under the presence of  $\text{H}_2\text{O}$  and NaOH solvents, indicates that an amide bond has been formed in lieu of an acid chloride. This transformation is a key marker of the successful synthesis of nylon 6,6, as the carbonyl stretching vibration in amides occurs at a lower frequency compared to the acid chloride due to the difference in electron distribution around the carbonyl carbon.

The formation of the amide bond is further supported by the appearance of a peak around  $3300\text{ cm}^{-1}$  corresponding to N-H stretching, a characteristic of amides. The presence of C-N stretching vibrations around  $1200\text{--}1300\text{ cm}^{-1}$  also confirms the creation of the polyamide structure. Additionally, the disappearance of peaks around  $600\text{--}750\text{ cm}^{-1}$ , which are attributed to the C-Cl stretching of adipoyl chloride, indicates the complete conversion of the starting material into the polymer.

The different solvents used in the synthesis,  $\text{H}_2\text{O}$  and NaOH, affected the rate and nature of the polymerization. In the basic NaOH environment, the polymerization was more efficient as the hydroxide ions neutralized the HCl by-product, shifting the reaction equilibrium and promoting the formation of the polyamide. On the other hand, the neutral medium with water alone may have resulted in slower reaction rates and incomplete conversion due to the accumulation of HCl, which could hinder further polymerization.

Overall, the FTIR spectra provide strong evidence of successful polymerization, and the analysis of the peak shifts corroborates the formation of nylon 6,6 from the starting materials.

## VII. CONCLUSION

In this experiment, we successfully synthesized nylon 6,6 from adipoyl chloride and hexamethylenediamine. We examined how these compounds react in different solvents and observed the formation of the polymer, along with

the release of HCl gas as a by-product. Our observations underscored the importance of controlling reaction timing: sufficient reaction time is crucial for the formation of the desired polymer, whereas prolonged exposure may lead to overreaction and the formation of unintended by-products.

Using infrared spectroscopy, we were able to distinguish the functional groups present before and after the reaction. The FTIR results confirmed the complete conversion of the starting materials into nylon 6,6, as indicated by the disappearance of peaks characteristic of adipoyl chloride and the appearance of peaks corresponding to amide bonds in the nylon polymer. This analytical method provided clear evidence of successful polymerization and the chemical transformations that occurred during the reaction.

## VIII. ACKNOWLEDGMENT

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