

– Table of Contents

– CONTENTS

I	Introduction	1
II	Apparatus	1
III	Materials	1
IV	Safety Measures	1
V	Procedure	1
VI	Observations and Results	1
VI-A	IR Spectrum of Hexamethylenediamine	1
VI-B	IR Spectrum of Adipoyl Chloride	2
VI-C	IR Spectrum of Nylon 6,6 in H_2O	2
VI-D	IR Spectrum of Nylon 6,6 in NaOH	2
VII	Conclusion	3
VIII	References	3
IX	Author Contributions	3

Preparation of Nylon 6,6

In both neutral and basic medium

Faayza Vora: 23110109, Goraksh Bendale: 23110118, Hriday Pandya: 23110136, Dishant Tanmay: 23110100, Haravath Saroja: 23110127

I. INTRODUCTION

One of the most commonly used polymers around us is Nylon. There are two types of Nylon: Nylon 6 and Nylon 6,6. Both types of nylon have similar properties and were invented in the late 1930s. In this experiment, we synthesised Nylon 6,6, a polyamide-based polymer. It is a step-growth polymer usually produced by polycondensation of Hexamethylenediamine, Adipoyl chloride, or Adipic acid. It is commonly used in the textile and plastic industries.

The following experiment was conducted in two conditions, i.e. under a basic medium (NaOH) and a neutral medium (H_2O). The resultant product's excess solvent was removed using a heating gun. The FTIR spectrum was obtained for the products, which helped us analyze the functional groups present in the product. The experiment under both conditions gave the same product; however, the yield was different. The reaction is given as:

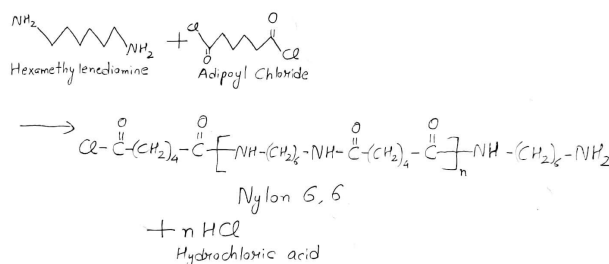


Fig. 1: Formation of Nylon 6,6

II. APPARATUS

- 1) Test-tubes
- 2) Droppers
- 3) Petri-dishes
- 4) Pasteur pipettes
- 5) Forceps

III. MATERIALS

- 1) Adipoyl chloride
- 2) Hexamethylenediamine
- 3) Sodium Hydroxide(NaOH)
- 4) Cyclohexane
- 5) Water

IV. SAFETY MEASURES

- 1) Wear the safety eyeglasses and gloves throughout the lab
- 2) Avoid contact with any chemicals in the lab
- 3) Handle the heating gun with care
- 4) Do not use the same droppers for all chemicals

V. PROCEDURE

- 1) Begin by measuring the FTIR spectra of the pure starting materials: Adipoyl chloride and Hexamethylenediamine.
- 2) Dissolve 1% v/v of Adipoyl chloride in Cyclohexane.
- 3) Prepare a 0.5M NaOH solution and add 1% v/v of Hexamethylenediamine for polymerization under certain conditions.
- 4) Alternatively, for polymerization under neutral conditions, add 1% v/v of Hexamethylenediamine to water.
- 5) Fill two Petri dishes or watch glasses with 5 ml of Hexamethylenediamine solutions.
- 6) Drop by drop, add the Adipoyl chloride solution onto the surfaces of the Hexamethylenediamine solutions.
- 7) Allow the reaction to proceed, resulting in the polymerization of Nylon 6,6 and the formation of HCl as a byproduct.
- 8) Using forceps and dropper, carefully remove and roll the formed polymer from the solvent over dropper.
- 9) Drain any excess solution and collect the polymer in an Eppendorf tube.
- 10) Wash the resultant polymer with water and gently heat it with a heat gun, ensuring it is first dried with tissue paper.
- 11) Determine the weight of the resulting polymer.
- 12) Finally, compare the FTIR spectra of the starting materials with those of the resulting Nylon 6,6 polymers.
- 13) Wash the used test tubes and Petri dishes.

VI. OBSERVATIONS AND RESULTS

A. IR Spectrum of Hexamethylenediamine

- In the region of $3300-3500\text{ cm}^{-1}$, the nitrogen and hydrogen atoms come closer, which leads to the contraction and relaxation of Amine (N-H) bonds. Therefore, the peaks observed in this region are due to the stretching vibrations of Amine (N-H) bonds. In the region of $1600-1650\text{ cm}^{-1}$, the peaks are due to the bending vibrations of the Amine (N-H) bonds.
- In the region of $2800-2950\text{ cm}^{-1}$, the peaks are due to the vibrations in the Alkyl (C-H) bonds. These vibrations are weaker than the Amine (N-H) bond stretching vibrations, thus giving them a lower peak. In the $1400-1490\text{ cm}^{-1}$ region, the peaks are due to the bending vibrations of Alkyl (C-H) bonds.
- In the region of $1200-1400\text{ cm}^{-1}$, the peaks are due to the stretching vibrations of C-N bonds.
- In the region of $1050-1300\text{ cm}^{-1}$, the peaks are due to the stretching vibrations of C-C bonds.

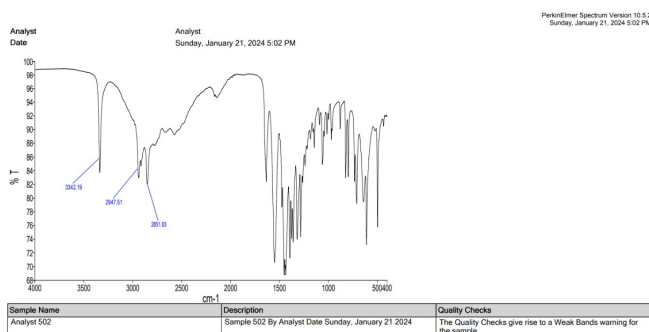


Fig. 2: IR Spectrum of Hexamethylenediamine

B. IR Spectrum of Adipoyl Chloride

- In the region of $1680\text{--}1800\text{ cm}^{-1}$, the peaks are due to the stretching vibrations of Carbonyl ($\text{C}=\text{O}$) bonds.
- In the region of $3100\text{--}2900\text{ cm}^{-1}$ the peaks are due to stretching vibrations of Alkyl (C-H) bonds.
- The main reason for this difference in peaks is the dipole moments of the bonds. Carbonyl ($\text{C}=\text{O}$) bonds have a higher dipole moment than Alkyl (C-H) bonds.
- In the region of $1000\text{--}1500\text{ cm}^{-1}$, the peaks are due to the bending vibrations of Alkyl (C-H) bonds.
- In the region of $550\text{--}750\text{ cm}^{-1}$, the peaks are due to the stretching vibrations of C-Cl bonds. Since chlorine is heavier than hydrogen, C-Cl bonds have lower stretching frequencies responsible for lower peaks.

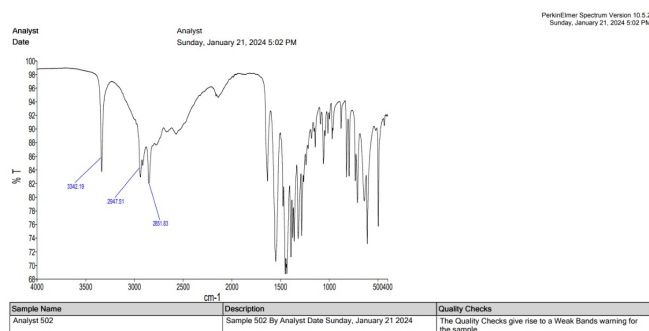
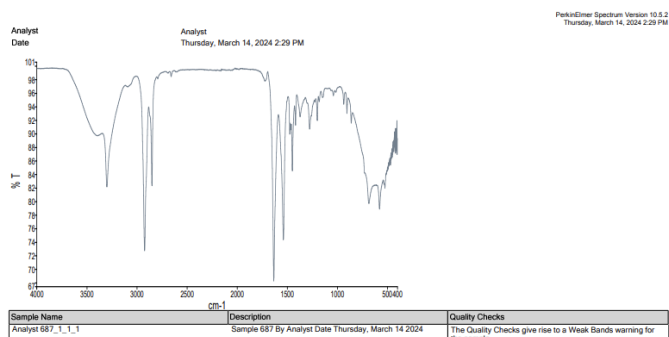


Fig. 3: IR Spectrum of Adipoyl Chloride

C. IR Spectrum of Nylon 6,6 in H_2O

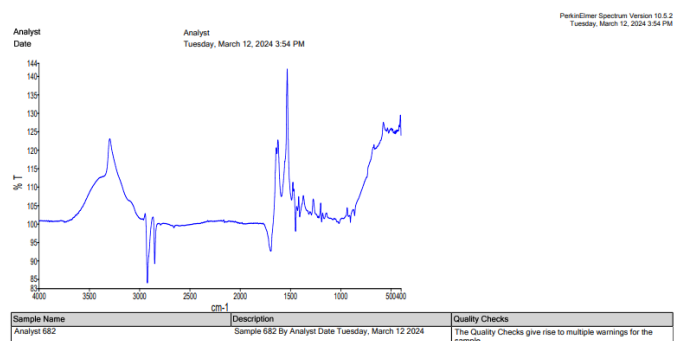
- In the region of $1610\text{--}1700\text{ cm}^{-1}$, the peaks are due to the stretching vibrations of Carbonyl ($\text{C}=\text{O}$) bonds, and the peaks in the region of $1470\text{--}1580\text{ cm}^{-1}$ are due to the stretching of Alkyl (C-H) and Amine (N-H) bonds within the amide bonds of Nylon 6,6 showing the presence of Hydrogen bonding in the polymer.
- In the region of $1710\text{--}1770\text{ cm}^{-1}$, the peaks are due to the bending vibrations of Carbonyl ($\text{C}=\text{O}$) bonds, and the peaks in the region of $1550\text{--}1400$ are due to the bending vibrations of the Amine (N-H) bonds within the amide groups.

- In the $2750\text{--}3000\text{ cm}^{-1}$ region, the peaks are due to the stretching vibrations of Alkyl (C-H) bonds in the polymer chain.
- In the region of $3200\text{--}3600\text{ cm}^{-1}$, the peaks are due to the stretching vibrations of hydroxyl bonds (O-H) bonds of H_2O , and the peaks in the region of $1600\text{--}1700\text{ cm}^{-1}$ are due to the bending vibrations of hydroxyl bonds (O-H) bonds of H_2O .

Fig. 4: IR Spectrum of Nylon 6,6 in H_2O

D. IR Spectrum of Nylon 6,6 in NaOH

- In the region of $1600\text{--}1700\text{ cm}^{-1}$, the peaks are due to the amide linkages showing the reaction between the amide group and the base and the stretching vibrations of Carbonyl ($\text{C}=\text{O}$) bonds within the amide group.
- In the $1500\text{--}1580\text{ cm}^{-1}$ region, the peaks are due to the deformation vibrations of the Amine (N-H) bonds coupled with C-N bonds caused by the reaction between the amide group and the base.
- In the region of $3200\text{--}3600\text{ cm}^{-1}$, the peaks are due to the hydrogen bonding between the base, O-H and the Amine (N-H) bonds.
- In the $2800\text{--}3000\text{ cm}^{-1}$ region, the peaks are due to the stretching vibrations of Alkyl (C-H) bonds in the polymer chain.

Fig. 5: IR Spectrum of Nylon 6,6 in NaOH

By comparing the reaction of Hexamethylenediamine and Adipoyl Chloride in H_2O and in NaOH , we observe that when Hexamethylenediamine and Adipoyl Chloride react to form Nylon 6,6 with HCl as a byproduct. In the case of a neutral medium, i.e. H_2O , it reacts with HCl , leading to

the formation of H^+ ions, which react with the N-H bonds of Hexamethylenediamine and tend the reaction to move in the backward direction, decreasing the yield of Nylon 6,6. However, in the case of a basic medium, i.e. NaOH, the HCl reacts with NaOH to form NaCl and H_2O , reducing the concentration of products, which pushes the reaction in the forward direction, increasing the yield of Nylon 6,6

VII. CONCLUSION

- In conclusion, the experiment demonstrated the successful synthesis of Nylon 6,6 from Adipoyl chloride and Hexamethylenediamine under basic and neutral conditions.
- The comparison of FTIR spectra confirmed the formation of Nylon 6,6, indicating the presence of characteristic functional groups corresponding to the polymer structure.
- The experiment helped us understand the polymerisation process and highlighted the impact of reaction conditions on the yield of the polymer.

VIII. REFERENCES

https://personal.utdallas.edu/~scortes/ochem/OChem_Lab1/recit_notes/ir_presentation.pdf

IX. AUTHOR CONTRIBUTIONS


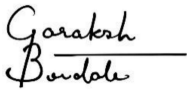


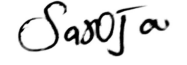
Name	Roll number	Contribution	Signature
Faayza Vora	23110109	Introduction, Materials and apparatus, Safety measures, Compilation, Collection of the product	
Goraksh Bendale	23110118	Compilation, Editing, Handling of the chemicals in lab, Collection of the product	
Dishant Tanmay	23110100	Procedure, Collection of the product in the lab	
Hriday Pandya	23110136	Observations, Results, Handling the chemicals in the lab, Collection of the product	
Haravath Saroja	23110127	Conclusion, Collection of the product in the lab	

TABLE I: Author's Contribution