Experiment 3
Synthesis of commercial polymer, Nylon 6,6

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Contents

I. Introduction

Nylon, a widely used polymer, exists in two primary forms: Nylon 6,6 and Nylon 6. Developed in the 1930s, both variants share striking similarities. Nylon 6,6, initially synthesized in the USA by Wallace Carothers at DuPont, serves as a pivotal material in textile and plastic manufacturing. Its production involves combining Hexamethylenediamine with either Adipoyl chloride or Adipic acid, resulting in the formation of high molecular weight Nylon 6,6 by polycondensation.

To synthesize Nylon 6,6 in the laboratory, specific procedures are followed. Hexamethylenediamine is mixed with water under neutral conditions or with a solution of NaOH under basic conditions. Subsequently, the material undergoes drying to eliminate excess solvent. Following this, FTIR spectroscopy is performed to understand Nylon 6,6 synthesis and identification of constituent functional groups.

Safety Precautions:

i Avoid direct contact with any of the chemicals provided.
ii Wear gloves and safety glasses throughout the experiment.
iii Handle all laboratory equipment with care to prevent accidents.
iv Use the heat gun carefully to avoid burning the Nylon.

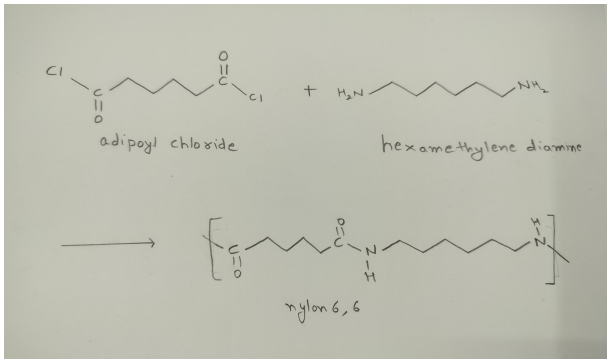


Fig. 1: Formation of Nylon 6,6

II. Experiment Details

A. Apparatus

1) Test tubes
2) Heat gun
3) Petri dishes
4) Forceps
5) Dropper
6) Gloves
7) Protective goggles
8) Eppendorf tube

B. Materials

1) Adipoyl chloride
2) Hexamethylenediamine
3) 0.5 M NaOH
4) Cyclohexane

C. Procedure

i Dissolve Adipoyl chloride in Cyclohexane. Use a concentration of 1% v/v, ensuring thorough mixing to obtain a homogeneous solution.
ii Add Hexamethylenediamine to a 0.5 M NaOH solution. Again, use a concentration of 1% v/v.
iii In a separate container, add Hexamethylenediamine to water. Maintain the same concentration of 1% v/v.
iv Take two watch glasses or petri dishes and add 5 ml of the prepared Hexamethylenediamine solutions to both. Ensure that the surfaces are clean and dry before proceeding.

v Carefully add the Adipoyl chloride solution drop by drop onto the surfaces of both solutions obtained in steps iii and iv.

vi Once the Adipoyl chloride solution is added, allow the polymerization reaction to occur. Nylon 6,6 will form at the interface of the two solutions, while hydrogen chloride (HCl) will be released as a byproduct.

vii Using forceps, carefully lift the polymer formed at the interface of the two solutions from the solvent.

viii Allow any excess solution to drain from the polymer.

ix Rinse the resulting polymer with water to remove any residual solvent or reactants. Use gentle agitation to ensure thorough washing. After washing, dry the polymer with some tissue paper to remove excess water, and gently heat it with a heat gun. Then it can be collected in an Eppendorf tube or similar container.

x Once the polymer is completely dry, measure FTIR spectra and compare them with the IR spectra of starting materials.

III. Results

We draw patterns between the obtained FTIR spectra and reference data [1] and get the following results for the bond and the corresponding wave number.

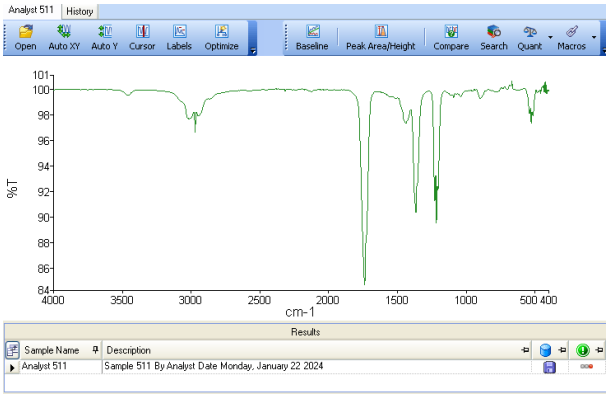


Fig. 2: FTIR spectrum of Adipoyl chloride

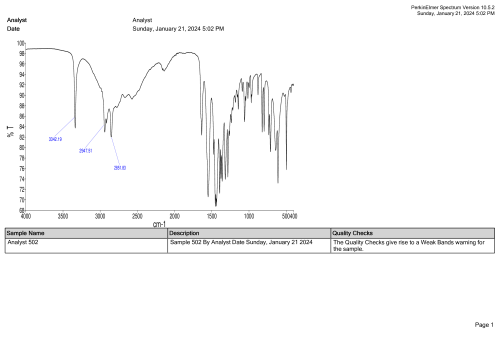


Fig. 3: FTIR spectrum of Hexamethylenediamine

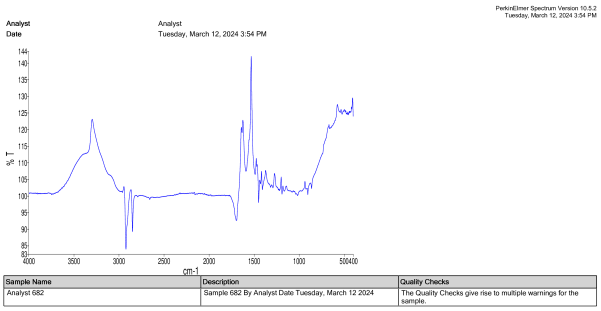


Fig. 4: FTIR spectrum of Nylon 6,6 synthesized under basic conditions (NaOH)

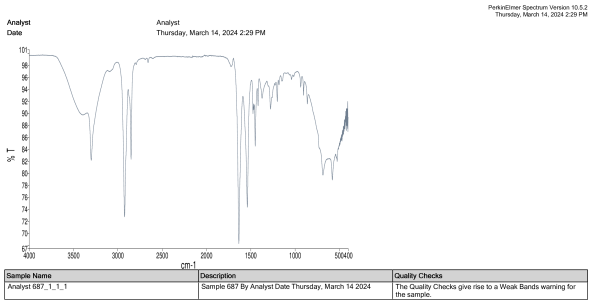


Fig. 5: FTIR spectrum of Nylon 6,6 synthesized under neutral conditions (H_2O)

A. Adipoyl chloride

Bond	Wave Number	Remarks
C-H	3000-2840	-
C=O	1680	Secondary Amide
C=O	1800-1770	Acid Halide
C-Cl	1150-1080	-

The FTIR analysis shows dips corresponding to the C-H, C=O (Acid Halide), and C-Cl ranges.

B. Hexamethylenediamine

Bond	Wave Number	Remarks
C-N	1250-1020	-
N-H	3500-3300	Primary Amine
N-H	3350-3310	Secondary Amine

The FTIR analysis shows dips corresponding to the C-H and N-H (Primary Amine) ranges.

C. Nylon 6,6

We observed that the Nylon 6,6 obtained from the water and NaOH solutions had identical patterns. We see dips corresponding to N-H (Secondary Amine), C-H, and C=O (Secondary Amide) ranges.

IV. Conclusion

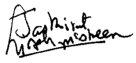
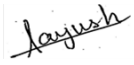
We successfully synthesized Nylon 6,6 from Adipoyl chloride and Hexamethylenediamine under both basic and neutral conditions, which was confirmed by the FTIR spectra of

original compounds as well as the formed product. Both the Nylon 6,6 formed under basic and neutral conditions gave about the same FTIR spectra.

V. References

[1] Libretexts, "Infrared spectroscopy absorption table," Chemistry LibreTexts, https://chem.libretexts.org/Ancillary_Materials/Reference/Reference_Tables/Spectroscopic_Reference_Tables/Infrared_Spectroscopy_Absorption_Table (accessed Mar. 17, 2024).

VI. Author Contribution

Name	**Contribution**	**Signature**
Jaskirat Singh Maskeen (23110146)	Document structure, Results, and Conclusion, Wrapping Nylon 6,6 on forceps	
Nishchay Bhutoria (23110222)	Introduction, Wrapping Nylon 6,6 on forceps.	
Aayush Bundel (23110005)	Experimental details, Dropping Adipoyl chloride using dropper	
Kavya Lavti (23110164)	Experimental details, Gathering the chemicals	
Kanhiyalal (23110155)	Equation, Gathering the chemicals