

# Synthesis and characterization of nylon-6,6 polymer



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## Abstract

Synthesis and characterization of nylon-6, 6 polymer was performed in this experiment. Nylon-6, 6, was obtained from adipoyl chloride and 1, 6-hexanediamine monomers, dissolved in cyclohexane and water. Employing interfacial polymerization technique, thin film of nylon product was formed and collected. The final product were white peaces of small fragile strings with a total mass of 0. 0694 g and 4 cm in length. Infrared spectroscopy then characterized the final product and confirmed successeful synthesis of nylon-6, 6 polymer due to the appearance of N-H stretching, C= O stretching, and N-H bending from the secondary amide group and from the nylon polymer.

## Introduction

Polymers are macromolecules consisting of smaller subunits, called monomers. <sup>4</sup> There are two main types of synthetic polymers, according to the mechanism by which they are synthetically made: step- growth and chain-growth polymers. <sup>4</sup> One of the most popular synthetic polymers is nylon-6, 6 due to low cost and ease of synthesis. There is no human activity where plastics are not used on a daily basis. Nylon in particular is widely used, due to variety of properties such as: elasticity, flexibility, lightweight and strength. <sup>5</sup> Clothing, electronics, packaging, cars, airplanes, medical supplies, cookware, ropes - all examples of nylon fabrics. <sup>5</sup>

In this experiment nylon is made by step- growth polymerization and condensation reaction. The monomers involved in this polymerization reaction are adipoyl chloride and 1, 6-hexanediamine dissolved in

cyclohexane, respectively. The chloride from the acid and the hydrogen proton from the amine group form a hydrochloric acid. <sup>4</sup> The rest of the adipoyl chloride and hexanediamine will link together to form a larger polymer molecule. In each end of this polymer is a primary amine group in one end or an acid chloride group in the other end. <sup>4</sup> These groups can act in a similar way with other acid/amine groups from the adipoyl chloride and hexanediamine. If many steps of this process are repeated, nylon-6, 6 polymer will be formed. <sup>4</sup> Hydrochloric acid is generated as a byproduct upon nylon formation, therefore a strong base must be added to neutralize the acidity of HCl, and maintain the pH of the solution. In this experiment sodium hydroxide was used as base.

Infrared (IR) spectroscopy will be applied as method of characterization. It will provide information regarding functional groups present in a molecule because the location of the absorption peaks observed have been associated with specific types of chemical bonds. <sup>6</sup> It could be determine which functional groups from the starting monomers, are present in the nylon spectrum.

## Results

Table 1: Nylon-6, 6 Polymer

Mass of nylon-6, 6	0.0694 g
Length	4 cm

Appearance      White fragile  
strings

Table 2: IR spectrum of adipoyl chloride

Functional Group	Molecular Motion	Observed Wavenumber (cm <sup>-1</sup> )	Literature Value Range(cm <sup>-1</sup> )	Peak Intensity
Alkane	C-H Stretch	2952. 44	3000-2850	Weak
Acyl chloride	C= O Stretch	1779. 80	1820-1780	Weak
Alkane	C-H in plane bend	1404. 56	1480-1350	Medium
Acyl chloride	C-Cl Stretch	681. 43	800-600	Strong

Table 3: IR spectrum of 1, 6- hexanediamine

Functional Group	Molecular Motion	Observed Wavenumber (cm <sup>-1</sup> )	Literature Value Range(cm <sup>-1</sup> )	Peak Intensity
1° amine	N-H Stretch	3323. 78	3300-3250	Medium
Alkane	C-H Assym. Stretch	2921. 17	3000-2850	Strong
Alkane	C-H Sym.	2846. 91	3000-2850	Strong

## Stretch

1° amine	N-H Bend	1603. 91	1640-1530	Medium
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Table 4: IR spectrum of nylon-6, 6 polymer

Functional Group	Molecular Motion	Observed Wavenumber (cm <sup>-1</sup> )	Literature Value Range(cm <sup>-1</sup> )	Peak Intensity
2° amide	N-H Stretch	3299. 0	3300-3250	Strong
Alkane	C-H Assym. Stretch	2932. 0	3000-2850	Medium
Alkane	C-H Sym. Stretch	2859. 0	3000-2850	Medium
2° amide	C= O Stretch	1636. 0	1680-1640	Strong
2° amide	N-H Bend	1538. 0	1560-1530	Medium

## Discussion

In this experiment, sythesis of nylon-6, 6 was carried out. The final product had an appearance of white pieces of small fragile strings with a total mass of 0. 0694 g and 4 cm in length. A way of improving the yield could be: different molar ratios of water to solvent; changing the solvent. No theoretical yield could be calculated for this reaction because all the polymer chains will have variety of different lengths. <sup>1</sup>

The ability of nylon-6, 6 to undergo hydrogen bonding results in high molecular order and high degree of interchain interaction as shown below <sup>2</sup> (Figure 1). Each single chain of nylon fiber will interact with the adjacent one, forming a hydrogen bond between the nitrogen proton of the secondary amide and the lone pairs of the oxygen on the carbonyl, thus making the nylon polymers strong, difficult to separate and resistant to outside reaction.

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The signals for the IR spectrum of adipoyl chloride, are presented in Table 2. The spectrum shows the characteristic weak and narrow alkane peak, C-H stretch at  $2952.44\text{ cm}^{-1}$ . This value was within the range  $3000\text{-}2850\text{ cm}^{-1}$  for the secondary alkane functional groups. The weak and sharp C=O stretch appeared at  $1779.80\text{ cm}^{-1}$  thus correlating with the literature value  $1820\text{-}1780\text{ cm}^{-1}$ . A C-H bend for alkanes should appear at  $1480\text{-}1350\text{ cm}^{-1}$ , which did appear at  $1404.56\text{ cm}^{-1}$  as a medium and sharp peak. A C-Cl stretch was observed at  $681.43\text{ cm}^{-1}$ , within the expected range of  $800\text{-}600\text{ cm}^{-1}$ . This peak appeared weak to strong and sharp.

The IR spectrum for 1, 6-hexanediamine exhibits the following stretches: N-H stretch; N-H bend; alkane C-H asymmetric stretch; and an alkane C-H symmetric stretch. The primary amine is causing the N-H stretch. The literature value for this stretch is from  $3300\text{ to }3250\text{ cm}^{-1}$ , which corresponds to the experimental value of  $3323.78\text{ cm}^{-1}$ . The peak intensity is medium and the shape is sharp. There is another sharp peak with medium intensity caused by the primary amine as well, which appear to be a N-H

bend. The experimental value of  $1603.91\text{ cm}^{-1}$  correlates well with the literature value range of  $1640\text{-}1530\text{ cm}^{-1}$ . There is C-H asymmetric stretch observed at  $2921.17\text{ cm}^{-1}$  and it corresponds to the literature value range of  $3000\text{-}2850\text{ cm}^{-1}$ , caused by the alkane. It exhibited strong intensity. The C-H symmetric stretch falls in the appropriate range of literature values  $3000\text{-}2850\text{ cm}^{-1}$  and it is caused by the alkane as well (See table 3).

Analysis of IR spectrum of nylon-6, 6 polymer showed successful synthesis. There were five distinct peaks associated with this compound include the following: N-H stretch; C= O stretch; C-H symmetric stretch; C-H antisymmetric stretch; and N-H bend. The N-H stretch had an observed peak value of  $3300.81\text{ cm}^{-1}$  and it's caused by the secondary amine. It correlates well with the literature values of  $3300\text{-}3250\text{ cm}^{-1}$  and with the N-H stretch of 1, 6-hexadiazine at  $3323.78\text{ cm}^{-1}$ . The two peaks have the same shapes, just slightly different intensities. Nylon-6, 6 has strong intensity whereas the 1, 6- hexanediamine, medium one. The C= O stretch had an observed peak value of  $1634.04\text{ cm}^{-1}$ . It falls within the expected literature values for this peak  $1300\text{-}1100\text{ cm}^{-1}$ . It appears at lower wavelength, compared to the adipoyl chloride. The proximity of the carbonyl to the nitrogen, lowers the energy of that stretch, due to interchain molecular interactions<sup>3</sup>. The alkane C-H asymmetric and symmetric stretches evident at  $2932\text{ cm}^{-1}$  and  $2832\text{ cm}^{-1}$  are nearly identical with the C-H stretches of 1, 6- hexanediamine (See Tables 2 and 3). Differing only in their intensities- medium for the nylon and strong for the 1, 6- hexanediamine. All of the observed values fall within

their literature range. The N-H bend at  $1538\text{ cm}^{-1}$  with medium intensity and sharp peak, is caused by the secondary amide. It is in the accepted literature data of  $1560\text{-}1530\text{ cm}^{-1}$ .

The major peak not present in the nylon-6, 6 spectrum and evident in the starting material spectra for adipoyl chloride is the C-Cl stretch. The acyl chloride stretch is missing because there is no chlorine in the nylon composition, thus explaining why there are no observed stretches below  $1000\text{ cm}^{-1}$ . The other major difference is the N-H stretch observed at  $3323.78\text{ cm}^{-1}$  in the hexanediamine spectrum, which appears at lower wavelength and becoming one peak for a secondary amide in the nylon spectrum.

## Conclusion

This experiment demonstrated successful synthesis of nylon-6, 6 polymer. It was accomplished using infrared spectroscopy technique. The IR analysis revealed the appearance of N-H stretching, C=O stretching and N-H bending from a secondary amide group and from the nylon polymer. It showed that there is no C-Cl stretch explaining the absence of chlorine in the nylon polymer as well as the N-H observed on the hexanediamine spectrum, appears on a lower wavelength as a secondary amide in the nylon spectrum. The final product had an appearance of white pieces of small fragile strings with a total mass of 0.0694 g and 4 cm.

However, this experiment allows for multiple errors; such as improper pipetting technique; improper washing technique; incorrect rate of taking out the nylon product from the reaction beaker. A chemical error could be not



adding enough sodium hydroxide to neutralize the acidity of HCl, not allowing the reaction to be pushed towards the product.