

# [Synthesis and characterization of nylon-6,6 polymer](https://assignbuster.com/synthesis-and-characterization-of-nylon-66-polymer/)

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. 4 There are two main types of synthetic polymers, according to the mechanism by which they are synthetically made: step- growth and chain-growth polymers. 4 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. 5 Clothing, electronics, packaging, cars, airplanes, medical supplies, cookware, ropes – all examples of nylon fabrics. 5

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. 4 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. 4 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. 4 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. 6 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 -1 ) | Literature Value Range(cm -1 ) | Peak Intensity | Peak Shape |
| Alkane | C-H Stretch | 2952. 44 | 3000-2850 | Weak | Broad |
| Acyl chloride | C= O Stretch | 1779. 80 | 1820-1780 | Weak | Sharp |
| Alkane | C-H in plane bend | 1404. 56 | 1480-1350 | Medium | Sharp |
| Acyl chloride | C-Cl Stretch | 681. 43 | 800-600 | Strong | Sharp |

Table 3: IR spectrum of 1, 6- hexanediamine

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Functional Group | Molecular Motion | Observed Wavenumber (cm -1 ) | Literature Value Range(cm -1 ) | Peak Intensity | Peak Shape |
| 1° amine | N-H Stretch | 3323. 78 | 3300-3250 | Medium | Sharp |
| Alkane | C-H Assym. Stretch | 2921. 17 | 3000-2850 | Strong | Sharp |
| Alkane | C-H Sym. Stretch | 2846. 91 | 3000-2850 | Strong | Sharp |
| 1° amine | N-H Bend | 1603. 91 | 1640-1530 | Medium | Sharp |

Table 4: IR spectrum of nylon-6, 6 polymer

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

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 theorytical yield could be calculated for this reaction because all the polymer chains will have  variety of different lengths. 1

The ability of nylon-6, 6 to undergo hydrogen bonding results in high molecular order and high degree of interchain interaction as shown below 2 (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. 2

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 cm -1 . This value was within the range 3000-2850 cm -1 for the secondary alkane functional groups.  The weak and sharp C= O stretch appeared at 1779. 80 cm -1 thus correlating with the literature value 1820-1780 cm -1 . A C-H bend for alkanes should appear at 1480-1350 cm -1 , which did appear at 1404. 56 cm -1 as a medium and sharp peak. A C-Cl stretch was observed at 681. 43 cm -1 , within the expected range of 800-600 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 to 3250 cm -1 , which corresponds to the experimental value of 3323. 78 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 cm -1 correlates well with the literature value range of 1640-1530 cm -1 . There is C-H asymmetric stretch observed at 2921. 17 cm -1 and it corresponds to the literature value range of 3000-2850 cm -1 , caused by the alkane. It exhibited strong intensity. The C-H symmetric stretch falls in the appropriate range of literature values 3000-2850 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 cm -1 and it’s caused by the secondary amine. It correlates well with the literature values of 3300-3250 cm -1 and with the N-H stretch of 1, 6-hexadiamine at 3323. 78 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 cm -1 . It falls within the expected literature values for this peak 1300-1100 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 3 . The alkane C-H assymetric and symmetric stretches evident at 2932 cm -1 and 2832 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 cm -1 with medium intensity and sharp peak, is caused by the secondary amide. It is in the accepted literature data of 1560-1530 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 cm -1 . The other major difference is the N-H stretch observed at 3323. 78 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 experiments 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.