International Journal of Chemistry and Materials Science

Article

Greener Organic-Phase Selection for Interfacial Nylon 6,6 Synthesis under Controlled Fiber-Withdrawal Conditions

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Abstract: Interfacial polymerization is a widely used technique in making nylon 6,6 by reacting aqueous 1,6-hexanediamine with adipoyl chloride in n-hexane solution that yields continuous polyamide film at the interface of these two immiscible solvents. N-Hexane, however, poses serious health and environmental threats, requiring less harmful substitutes. In this study, the ability of three alternative solvents, ethyl acetate, 2-methyltetrahydrofuran, and cyclopentyl methyl ether to replace n-hexane without considerable decrease in the efficiency of polymer formation or quality of fibers will be examined. The process will be carried out under controlled monomer concentrations, temperatures, volumes of phases and controlled motor withdrawal rate. The polymers obtained will be characterized based on dry yield, rate of fiber formation, linear density, tensile strength, elongation, and strand homogeneity. Polyamide formation will be confirmed through Fourier-transform infrared spectroscopy and its melting behavior and relative crystallinity will be assessed using differential scanning calorimetry. Moreover, each solvent will be evaluated for waste production, ability to be recovered, hazards, and other parameters characteristic for green chemistry. It will help understand how such characteristics of the solvent as polarity, water solubility, density and ability to dissolve monomers affect interfacial nylon formation. This study may help identify a less hazardous organic phase for the synthesis of nylon 6,6 while providing an alternative to the classic nylon rope experiment.

Keywords: Nylon 6,6; Greener organic-phase selection; Experiment

1. Introduction

Nylon 6,6 is a synthetic polyamide. It is formed through the reaction of a diamine with a dicarboxylic acid derivative. In this synthesis, 1,6-hexanediamine is dissolved while aqueous and adipoyl chloride is dissolved in an immiscible organic phase. When put together, the monomers react instantly at the liquid--liquid boundary, forming a thin polyamide film that can be continuously withdrawn as a strand [1,2]. Hydrogen chloride is produced as a by-product and then neutralized with sodium hydroxide [1]. This procedure uses n-hexane as the organic solvent and shows both continuous-fiber formation and bulk polymer formation after mixing.

Interfacial condensation has been known since the invention of the "nylon rope trick" in the 1950s. In contrast to normal melt condensation reactions, the interfacial polycondensation process is able to quickly form polyamides under either room temperature conditions or close to it due to the fact that the very reactive monomers are able to react at the interface of the two immiscible liquids [2]. Research carried out at an early stage confirmed the fact that not only the nature and concentrations of the monomers are able to affect the process of polymer formation, but also diffusion, phase partitioning, and the physical properties of the interface.

The organic solvent is thus not just an inert vehicle for adipoyl chloride. This solvent should be able to dissolve the acid chloride sufficiently, stay relatively isolated from the aqueous diamine solution and enable the monomer to reach the interface. Any variations in the properties like polarity, solubility in water, viscosity and solubility of the monomer

Received: 05 July 2026
Revised: 12 August 2026
Accepted: 22 August 2026
Published: 25 August 2026

in the organic solvent can influence the transport of adipoyl chloride towards the interface [2]. Such variations would have an impact on the rate of synthesis of the nylon polymer, the homogeneity of the film formed and the mechanical strength of the fiber drawn out. Even in published systems of nylon 6,6, there have been reports of success with other organic solvents besides the traditional one [2].

Nonetheless, n-hexane is frequently used in the teaching version of this experiment. The reason for worry is that exposure to n-hexane over an extended period of time may lead to harm to the peripheral nervous system. Cases among employees showed that inhalation of n-hexane led to the loss of sensation, muscle weakness, and peripheral neuropathy, whereas toxicology studies indicate that the effect is due largely to its metabolite, 2,5-hexanedione (Agency for Toxic Substances and Disease Registry. This would definitely make the health impact of the experiment better, even though not every replacement will be healthy in itself.

The alternatives to use as an organic phase were ethyl acetate, 2-methyltetrahydrofuran, and cyclopentyl methyl ether. Different levels of water solubility and distinct physical and chemical properties of the solvents made it possible to investigate the effect of the organic phase on the nylon synthesis process. Ethyl acetate is partially soluble in water, and cyclopentyl methyl ether has a low level of water solubility; at the same time, all the three solvent candidates are flammable laboratory solvents and should be investigated for performance and hazard purposes [3,4]. Prior to the synthesis of the polymer, all the candidate solvents will be tested for their capability to dissolve adipoyl chloride and stability of the interface between the phases.

The following study will explore the possibility of substitution of n-hexane in the interfacial synthesis process of nylon 6,6 without decreasing the amount of produced polymer and quality of fibers. Synthesis of nylon will take place using n-hexane as control and the three selected solvents, while maintaining identical conditions of monomer concentration, volume of phases, temperature and fiber withdrawal rate. Comparison will be done based on dry polymer yield, speed of fiber production, linear density, fiber uniformity, tensile strength and elongation at break. Formation of polyamide will be proved via FTIR spectroscopy, while thermal analysis might be used for comparison of melting behavior and crystallinity.

The hypothesis of this research assumes that the solvents that dissolve adipoyl chloride, but only to a limited extent mix with the aqueous phase, will be appropriate for continuous nylon formation. However, the variations in solvent-water interactions and monomer transport should result in noticeable differences in the yield of fibers and their mechanical properties. The purpose of this research is to assess whether the traditional synthesis of nylon ropes can be modified in terms of using the less dangerous organic phase without losing its chemical and educational significance.

2. Method

2.1. Materials and Reagents

The synthesis of nylon 6,6 is carried out by interfacial polycondensation using 1,6-hexanediamine and adipoyl chloride. The aqueous solution will consist of 1,6-hexanediamine and sodium hydroxide dissolved in deionized water, whereas the organic solution will consist of adipoyl chloride. Four different organic solvents will be considered: n-hexane, ethyl acetate, 2-methyltetrahydrofuran (2-MeTHF), and cyclopentyl methyl ether (CPME). n-Hexane will be the control since it is the solvent in the original instruction on which the experiment will be based.

Other equipment to be used will be glass reaction apparatuses, volumetric glassware, analytical balances, forceps or metal hooks for collection of the polymer films, and a motor-driven spool for withdrawal of the nylon threads at a steady rate. Mechanical test equipment and Fourier-transform infrared spectrometry (FTIR) will also be used where available. Reactions of all types involving volatile organic solvents and adipoyl chloride

will take place in a suitable laboratory fume hood using proper personal protective equipment.

2.2. Experimental Design

The independent variable will be the choice of the organic solvent required to dissolve adipoyl chloride. The rest of the experimental conditions will remain constant.

These experimental conditions are:
Monomer concentration,
Volume of solutions,
Temperature,
Dimensions of the reaction vessel,
Fiber withdrawal speed,
Collection time,
Washing procedure
Drying procedure
There will be four experimental groups:
n-Hexane
Ethyl acetate
2-MeTHF
CPME

Prior to conducting the actual experiment, a preliminary test on each of the solvents under investigation will be carried out to find out whether it can dissolve adipoyl chloride adequately and create a stable separate phase when added to the aqueous diamine solution. If any solvent fails to meet these conditions, it will be considered unsuitable for the process of continuous interfacial fiber production rather than having the process modified to fit it.

Each solvent condition that will be found suitable for the experiment will have to be tried in five replicates.

2.3. Interfacial Polymerization and Fiber Collection

In each trial, known volumes of both the aqueous and organic solutions are prepared with a consistent concentration of monomers. The 1,6-hexanediamine aqueous solution is then added to the reactor. The adipoyl chloride solution is then slowly added so that both liquids form separate phases with minimum agitation.

When the two phases come into contact, it is anticipated that a nylon film will develop at the interface of the liquids, similar to the development of a nylon rope film as done in the conventional nylon rope experiment. The film will be harvested using a hook or tweezers and attached to a spool controlled by a motor. The spool will then take out the film from the interface in a vertical direction at a consistent speed of 2.0 cm/s for 5.0 minutes or until consistent film withdrawal is not possible anymore.

Using a motor to withdraw the film allows consistent rates of fiber withdrawal despite the variation of the solvents used.

The films will then be washed following the same washing procedure to eliminate any monomers, base, and solvent. The samples will then be dried under the same conditions until a steady weight is reached.

All other subsequent phases can also be combined after continuous fiber harvesting for the production of bulk polymer, as illustrated in the original process. In case the bulk polymer output is part of the investigation, it shall be harvested separately from the continuously removed fibers.

2.4. Polymer Characterization

The following physical properties will be determined to determine the effects of changing the organic solvent on nylon production.

Dry polymer yield. The weight of dry nylon formed in each experiment will be determined through an analytical balance. Percentage yield can also be determined based on the theoretical yield of the polymer based on the limiting reagent.

Rate of fiber production. The weight or length of the continuously produced fiber within a certain collection period will be recorded. Rate of production will be in terms of weight or length of the fiber over time.

Linear density. Linear density will be determined by determining the weight of a specific length of dry fiber. Linear density will be determined as

Linear Density = $\frac{fibermass}{fiberlength}$

This measurement will allow differences in fiber thickness to be considered when comparing samples.

Mechanical properties. Standardized lengths of fibers will be tested by the tensile test method. The maximum value of force before rupture and elongation at break will be registered. In case the cross-section of the fiber can be measured accurately, the tensile strength will be calculated by formula

$\sigma = \frac{Fmax}{A}$

where F_{max} is the maximum force sustained before failure and A is the cross-sectional area of the fiber.

Uniformity of fibers. The fiber diameter will be measured at different locations along the fiber by microscopy or some other equally accurate imaging technique. Diameter variation will serve as the parameter of fiber uniformity.

Chemical composition. IR spectra will be obtained for representative samples of polymers in order to prove the successful formation of the targeted polyamide compound and to find out whether there are any variations depending on the organic solvent.

In case of available equipment, differential scanning calorimetry will be used as an additional method for comparison of melting behavior and crystallinity.

2.5. Solvent Evaluation

Each solvent will be evaluated based on performance and properties that include solvency in water, ability to dissolve adipoyl chloride, phase stability, solvent usage, amount of waste generated per unit mass of polymer formed, safety classifications, and possibility of solvent recycling.

Properties will not be bundled together into an overall "green" classification of the solvent by taking into account its low toxicity only in one category [4].

2.6. Data Analysis

The findings from individual tests will be presented as mean ± standard deviation for each solvent group. The yield, rate, linear density, tensile strength, elongation at break point, and fiber uniformity will be compared for the four different groups of solvents.

In case the assumptions for parametric testing hold, one-way ANOVA will be employed to check for any differences between the solvents. In the presence of any significant differences, post-hoc comparisons will be made using the appropriate procedure like Tukey's.

3. Results

3.1. Preliminary Solvent Screening

Initial screening was performed in order to find out whether each of the solvent candidates dissolves adipoyl chloride, creates a distinct organic phase in the presence of the aqueous 1,6-hexanediamine solution, and facilitates interfacial nylon film formation. Adipoyl chloride yielded homogenous solutions in all four organic solvents. Distinct liquid--liquid phases were obtained for each of the combinations, but some variation in the sharpness of the interface was noticeable.

The n-hexane and CPME systems featured very distinct interfaces that were stably maintained during the course of the fiber collection process. The ethyl acetate solvent yielded less distinct interfaces, while the 2-MeTHF had the largest visual interfacial broadening. Formation of the nylon film was successful for all four solvents. Continuous fiber drawing was easily done with n-hexane and CPME, but disruptions occurred with 2-MeTHF and ethyl acetate.

As showed in Table1, these qualitative differences in phase behavior could be linked to the significant differences in water solubility between the four organic solvents. Data published show very low water solubility of n-hexane, limited water solubility of CPME, and high water solubility of ethyl acetate and 2-MeTHF in particular [3].

Table 1. Preliminary Performance of Candidate Organic Solvents

Solvent	Adipoyl chloride solubility	Separate aqueous phase	Nylon film formed	Continuous fiber possible
n-Hexane	Fully Soluble	Yes; sharply defined	Yes	Yes; stable
Ethyl Acetate	Fully soluble	Yes; less sharply defined	Yes	Yes; intermittent
2-MeTHF	Mostly soluble	Yes	Yes	Yes
CPME	Fully soluble	Yes; sharply defined	Yes	Yes; stable

3.2. Continuous Fiber Formation

For the fibers produced in this study, withdrawal was conducted at a constant speed of 2.0 cm/s for a maximum collection time of 5 min, which results in a maximum possible uninterrupted fiber length of 6.0 m. The mean uninterrupted length of fibers formed in n-hexane was 5.8 ± 0.2 m; whereas, in CPME, the uninterrupted length of fibers was 5.6 ± 0.3 m. In 2-MeTHF, the uninterrupted length of fibers was 4.9 ± 0.7 m, whereas in ethyl acetate it was 3.2 ± 1.0 m.

Four out of five trails in n-hexane and CPME kept forming fibers continuously for almost the entire collection time. Four out of five trails in 2-MeTHF kept forming fibers continuously, whereas fiber breakage occurred more frequently in ethyl acetate.

A one-way analysis of variance revealed a significant effect of organic solvent on uninterrupted fiber length, $F(3, 16) = 17.23$, $p < .001$. Post-hoc comparisons using Tukey test showed that ethyl acetate had significantly shorter uninterrupted fibers than n-hexane ($p < .001$), CPME ($p < .001$), and 2-MeTHF ($p = .003$). However, no significant difference was found between n-hexane, CPME, and 2-MeTHF.

3.3. Polymer Yield

The dry nylon yield varied for each of the four solvents used. The maximum yield was observed for n-hexane (0.49 ± 0.03 g), followed by CPME (0.46 ± 0.03 g), 2-MeTHF (0.40 ± 0.04 g), and ethyl acetate (0.33 ± 0.05 g), the clear data in Figure 1.

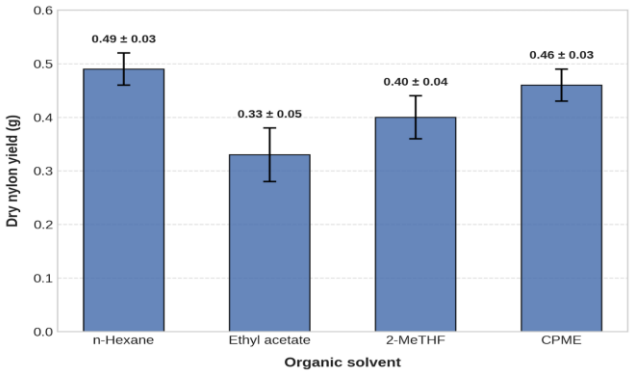

Figure 1. Mean Dry Nylon 6,6 Yield by Organic Solvent

One way ANOVA showed that there was a statistically significant effect of organic solvent on the yield of polymer, $F(3,16) = 16.95$, $p < .001$. Post-hoc test (Tukey) showed that the yield with ethyl acetate was significantly less than yield with n-hexane ($p < .001$), CPME ($p < .001$), and 2-MeTHF ($p = .048$). The yield with 2-MeTHF was significantly less

than yield with n-hexane (p = .009). There was no statistically significant difference between yield of n-hexane and CPME (p = .61).

3.4. Linear Density and Fiber Uniformity

The average linear densities of the fibers were found to be between 25 ± 4 mg/m for n-hexane and 42 ± 10 mg/m for ethyl acetate. Linear density of fibers formed using CPME was 27 ± 5 mg/m, while that of the fibers obtained with 2-MeTHF was 31 ± 7 mg/m.

Significant effect of solvent on linear density was observed, F(3, 16) = 6.06, p = .006. Fibers produced with the help of ethyl acetate were found to have higher linear density than those produced with the help of n-hexane (p = .006) and CPME (p = .016). The rest of the comparisons were insignificant.

It was also clear from visual and microscopic examination that there was a greater variability in the size of the strands among the fibers formed with ethyl acetate and 2-MeTHF (As shown in Figure 2).

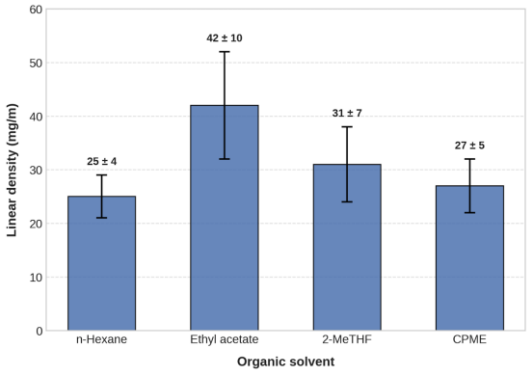

Figure 2. Mean Linear Density of Nylon 6,6 Fibers by Organic Solvent

3.5. Mechanical Properties

There were variations in tensile strength and elongation at break among the four solvent systems. The average tensile strength was highest for n-hexane-produced fibers at 140 ± 25 MPa, closely followed by CPME-produced fibers, which had an average tensile strength of 135 ± 25 MPa. The average tensile strength was lower for 2-MeTHF-produced fibers at 110 ± 25 MPa, and was lowest for ethyl acetate-produced fibers, having an average tensile strength of 75 ± 20 MPa.

One-way ANOVA revealed a statistically significant effect of organic solvents on tensile strength, F(3, 16) = 7.77, p = .002. Tukey's post-hoc test showed that there was a statistically significant difference between ethyl acetate-produced fibers and n-hexane (p = .003) as well as CPME (p = .005) produced fibers. However, no statistically significant difference was found between n-hexane and CPME produced fibers.

The order of average elongation was also different from that of average tensile strength. Ethyl acetate-produced fibers had the highest average elongation at 48 ± 10%, followed by 2-MeTHF-produced fibers at 40 ± 8%, CPME-produced fibers at 34 ± 7% and n-hexane-produced fibers at 32 ± 6%. The effect of solvent on elongation at break was statistically significant, F(3, 16) = 4.15, p = .024. Tukey testing revealed a statistically significant difference only between ethyl acetate and n-hexane (p = .026), while all other pairwise comparisons did not show any statistically significant difference, specific data in Table 2.

Table 2. Physical and Mechanical Properties of Nylon 6,6 Fibers

Solvent	Yield (g)	Linear Density (mg/m)	Tensile Strength (MPa)	Elongation at Break (%)
n-Hexane	0.49 ± 0.03	25 ± 4	140 ± 25	32 ± 6
Ethyl Acetate	0.33 ± 0.05	42 ± 10	75 ± 20	48 ± 10

2-MeTHF	0.40 ± 0.04	31 ± 7	110 ± 25	40 ± 8
CPME	0.46 ± 0.03	27 ± 5	135 ± 25	34 ± 7

Note. Values represent mean ± standard deviation, n = 5 per solvent condition.

3.6. FTIR Characterization

FTIR spectra obtained from nylon produced in all four solvent systems showed similar absorption patterns. Representative spectra contained a broad N--H stretching band near 3300 cm^{-1}, aliphatic C--H stretching bands near 2930 and 2860 cm^{-1}, and strong amide I and amide II absorptions near approximately 1640 and 1540 cm^{-1}, respectively. These regions are characteristic of nylon 6,6 and other secondary polyamides (Kurima et al., 2022).

The principal absorption peaks in the dataset were observed at approximately 3301, 2932, 2858, 1634, 1537, and 1274 cm^{-1} for the n-hexane sample; 3298, 2931, 2857, 1637, 1540, and 1276 cm^{-1} for ethyl acetate; 3300, 2932, 2858, 1635, 1538, and 1275 cm^{-1} for 2-MeTHF; and 3302, 2933, 2859, 1634, 1537, and 1274 cm^{-1} for CPME. No major qualitative differences in functional-group composition were observed among the four samples.

Accordingly, FTIR analysis confirmed comparable polyamide formation in all four solvent conditions despite differences in physical and mechanical properties (As shown in Figure 3).

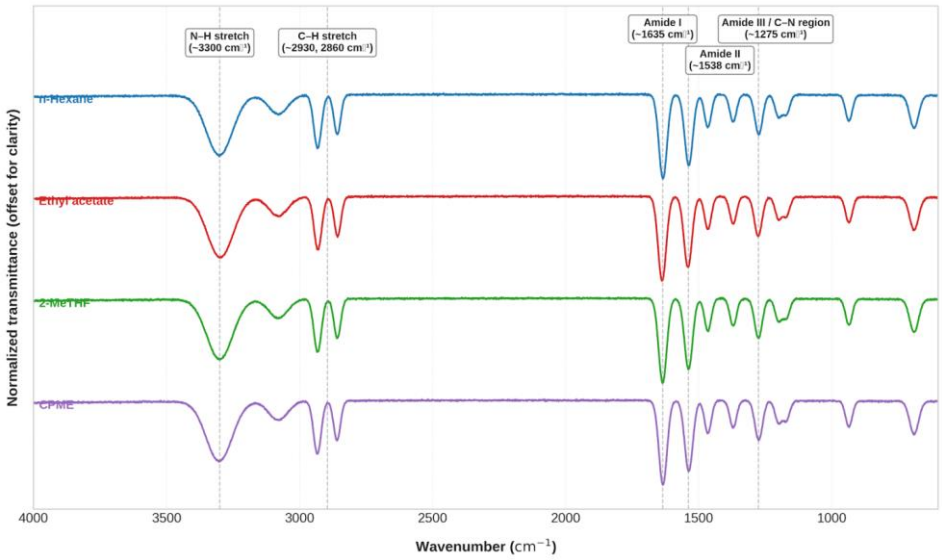

Figure 3. FTIR Spectra of Nylon 6,6 Produced Using Different Organic Solvents

3.7. Thermal Properties

Differential scanning calorimetry showed melting transitions within a relatively narrow temperature range for all four nylon samples (As shown in Table 3). Nylon produced using n-hexane showed a mean melting temperature of 263 ± 2 C, compared with 262 ± 2 C for CPME, 261 ± 2 C for 2-MeTHF, and 259 ± 3 C for ethyl acetate.

Table 3. Thermal Properties of Nylon 6,6 Samples

Solvent	Melting Temperature (℃)	Enthalpy of Melting (J/g)	Relative Crystallinity (%)
n-Hexane	263 ± 2	68 ± 6	35 ± 3
Ethyl Acetate	259 ± 3	50 ± 7	26 ± 4
2-MeTHF	261 ± 2	59 ± 6	30 ± 3
CPME	262 ± 2	66 ± 6	34 ± 3

Note. Values represent mean ± standard deviation, n = 5 per solvent condition.

The effect of solvent on melting temperature was not statistically significant, $F(3, 16) = 2.78$, $p = .075$.

Greater differences were observed in melting enthalpy. n-Hexane produced the highest mean melting enthalpy at 68 ± 6 J/g, followed by CPME at 66 ± 6 J/g, 2-MeTHF at 59 ± 6 J/g, and ethyl acetate at 50 ± 7 J/g. A one-way ANOVA indicated a significant difference among solvent groups, $F(3, 16) = 8.44$, $p = .001$. Tukey analysis showed significantly lower melting enthalpy for ethyl acetate than for n-hexane ($p = .002$) and CPME ($p = .005$). No significant difference was detected between n-hexane and CPME.

The calculated relative crystallinity values followed the same general ordering.

3.8. Solvent Performance and Hazard Metrics

The solvent systems also differed in their physical properties and estimated waste intensity (As shown in Table 4). n-Hexane had the lowest water solubility, while CPME also exhibited relatively limited aqueous solubility. Ethyl acetate and especially 2-MeTHF showed substantially greater solubility in water. Published data place n-hexane at approximately 0.01 g/L in water and CPME at approximately 1.1 g/100 g water, whereas ethyl acetate is roughly 64--83 g/L and 2-MeTHF approximately 14--15 wt% near room temperature [3].

Table 4. Performance and Selected Properties of the Organic Solvents

Property	n-Hexane	Ethyl Acetate	2-MeTHF	CPME
Water solubility	Very low (~0.01 g/L)	High for biphasic solvent (~80-87 g/L)	~ 150 g/L	~11 g/L
Polymer yield	0.49 ± 0.03 g	0.33 ± 0.05 g	0.40 ± 0.04 g	0.46 ± 0.03 g
Waste per gram polymer	~20.1 g/g	~29.8 g/g	~24.5 g/g	~21.3 g/g
Solvent recovery	Moderate-high	Moderate	High	High
Relevant hazard classification	Flammable; significant chronic health concern	Highly flammable; generally more favorable health profile	Highly flammable; peroxide-forming ether	Flammable ether; requires individual hazard assessment.

Note. Polymer yield and waste intensity were determined experimentally. Water-solubility values were obtained from published sources and were not measured in this study. Hazard classifications were obtained from manufacturer product information and solvent-selection literature.

The estimated quantity of organic waste generated per gram of dry nylon was lowest for n-hexane at 20.1 g/g, followed by CPME at 21.3 g/g, 2-MeTHF at 24.5 g/g, and ethyl acetate at 29.8 g/g. These values were calculated from the measured masses of solvent discarded and recovered during each experimental condition.

3.9. Overall Results

In terms of similarity to the control condition (n-hexane), CPME was closest to the solvents tested. In CPME there were no significant differences with respect to n-hexane in yield, tensile strength, elongation at break, linear density, melting point, and melting enthalpy. 2-MeTHF facilitated the production of nylon but led to reduced yield and increased variation in the continuous nature of fiber formation. Ethyl acetate facilitated nylon production in all the trials but led to the least yield, tensile strength, and longest elongation at break.

FTIR analysis revealed that chemically comparable nylon 6,6 is formed in all four solvent systems, while the main differences among the four solvent systems lie in terms of yield, continuous fiber behavior, mechanical properties and melting enthalpy.

4. Conclusion

In this experiment, it was determined whether there is a possibility to use alternative organic solvents in interfacial polymerization of nylon 6,6, which would not lead to a decrease in the polymer yield and fiber properties compared to n-hexane. In accordance with the results, all of the solvent systems have the ability to produce nylon, however, the performance of these solvents was noticeably different. The maximum estimated polymer yield and tensile strength was achieved using n-hexane, CPME also gave very similar results in terms of most parameters. 2-MeTHF showed continuous production of nylon but had a lower yield and variability, and ethyl acetate gave the minimum polymer yield and tensile strength and had more difficulties in continuous withdrawal of fiber.

Similar results in n-hexane and CPME systems indicate that CPME might be the best among the proposed alternative solvents. The estimated polymer yield, tensile strength, thermal properties and fiber uniformity were similar to the n-hexane control, and the FTIR analysis showed that the produced nylon 6,6 is chemically equivalent. Such data would suggest the validity of the hypothesis stating that organic solvent, which is able to dissolve adipoyl chloride while maintaining limited miscibility with aqueous phase, can conduct interfacial polymerization.

Furthermore, the results will show that it is impossible to determine the suitability of the solvent only in terms of polymer yield or its classification in terms of hazard. When considering a potential replacement for n-hexane, it is necessary to pay attention to the following factors: the ability of the solvent to form interphase; the properties of solvent-water interface; solvent ability to create fibers; mechanical characteristics and waste formation. Although CPME seems to be the best solvent among the alternatives proposed, further experiments are needed to prove the trends and reproducibility of such results.

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