



**6. ULUSLARARASI
MULTİDİSİPLİNER
ÇALIŞMALARI KONGRESİ**

**6TH INTERNATIONAL
MULTIDISCIPLINARY
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(26-27 Nisan 2019, Gaziantep, Türkiye)

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**ENGINEERING
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**CİLT 2
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Effect of Borax Decahydrate on Thermal Properties of Electrospun Nylon 6,6 Nanofibers

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Introduction

Recent progress in polymeric material engineering, especially in the processing of materials in nanoscale, has constituted promising opportunities for generating nanostructured materials having various functionalities. Nano or micro sized inclusions incorporated into polymers improve their multifunctional properties such as flame retardancy, UV protection, self-cleaning, antistatic, mechanical strength and antimicrobial activities. Nanofiber technology (fiber diameter less than 1 micrometer) is under development for the preparation of novel materials in nano-scale with multifunctional properties. Electrospinning is an elegant method for producing nanofibers with high porosity and high specific surface area (Beachley, 2009). The electrospun nanofibers with superior properties can be obtained controlling the diameter and distribution of the fibers (Huang, 2011). Attempts have been made to incorporate inclusions into fibers to develop their flammability and thermal properties (Agwuncha, 2017). Polymers, for example, polyurethane, polyvinyl alcohol, polyacrylonitrile and polyamide, and their composites had been fabricated to develop the thermal stabilities of industrial products. Nylon 6,6 is one of the best-known polymers used in textile or other industries due to its high mechanical, physical and thermal properties.

Nylon 6 and Nylon 6,6 are polyamides, which means they are molecules whose repeating units are linked by amide bonds. Some polyamides, such as silk, can be found naturally, but nylons are made in a lab. Nylon 6,6 is synthesized by polycondensation of hexamethylenediamine and adipic acid. Nylon 6,6 is flammable and also has dripping problem that limits its use in a wide range of applications.

The aim of the present work is to improve the thermal properties of Nylon 6,6 (Polyamide PA66) electrospun nanofibers. Nylon 6,6 nanofibers were fabricated using electrospinning method via incorporation of Borax decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) into the polymer matrix. There have been no studies using this combination to fabricate a nanofibrous polymer. The surface morphology and thermal properties of the nanofibers were characterized by Scanning Electron Microscopy (SEM) and Thermal Gravimetric Analysis (TGA), respectively. Additionally, FT-IR analysis was performed to evaluate the functional groups of the samples.

Experimental

Materials

Nylon 6,6 has been chosen because of its stability. Borax decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) was also used to improve the thermal property of Nylon 6,6. Borax decahydrate is the refined form of natural sodium borate. Composed of boric oxide (B_2O_3), sodium oxide, and water, this mild alkaline salt has excellent buffering and fluxing properties.

Preparation of Nylon 6,6-Borax nanofibers

Different solvents were used to make Nylon 6,6 spinnable with desired physical properties, then the optimization was evaluated to determine electro-spinning conditions. After number of trials, formic acid was selected to dissolve Nylon 6,6. In order to prepare Nylon-Borax solutions (0.8% w/w), firstly Borax was dissolved in formic acid solution with heating, then Nylon granules were added to the solution. Resulting mixture was stirred for 18 hours and subjected to electrospinning process.

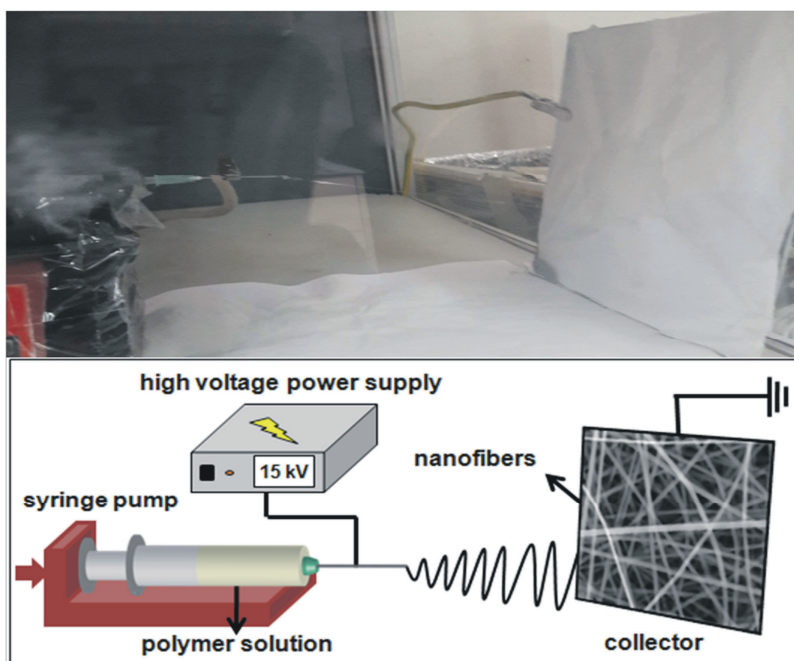


Figure 1. Experimental set up for electro-spinning technique

The surface morphologies and thermal properties of fabricated nanofibers were characterized by Scanning Electron Microscopy (SEM) and Thermal Gravimetric Analysis (TGA), respectively. FT-IR analysis was performed to evaluate the functional groups of the samples.

Results and Discussion

Scanning Electron Microscopy (SEM)

In order to evaluate the effect of Borax on the diameter of nanofibers, SEM analysis was performed. As seen from the Figure 2, nanofibers were successfully obtained in the presence of Borax. It can be concluded that uniform Nylon fibers without beads could be prepared by electrospinning upon controlling the process parameters.

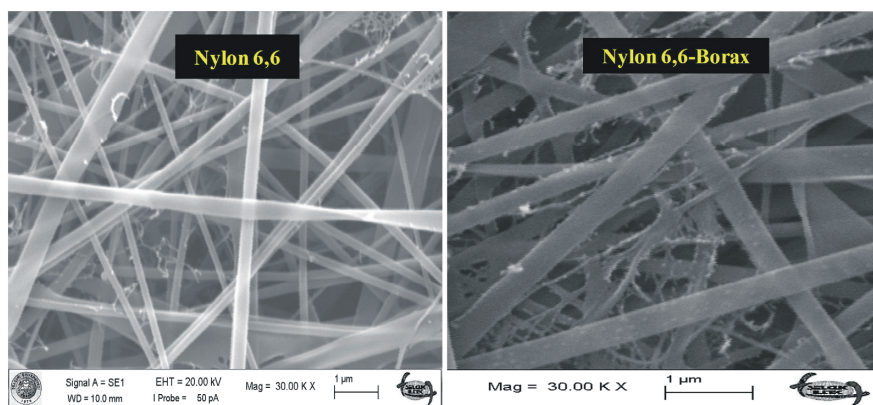


Figure 2. SEM micrographs of pristine Nylon 6,6 and Nylon 6,6-Borax nanofibers.

Effect of polymer solution feeding flow rate

In this study, different feeding rates were applied to investigate the effect of the feeding rate on diameter of fibers. Fibers beadless were successfully collected through the plate for all three feeding rates. SEM images of Nylon 6,6-Borax nanofibers produced by applying various feeding rates are presented in Figure 3. They show a difference in the diameter of the nanofibers. Even though the solution could be successfully spun into nanofibers, the resultant nanofibers created with the feeding rate of 0.05 mL/h, were usually not uniform because of its electrical current. The larger diameter nanofibers were spun at a lower feeding rate (e.g., 0.05 mL/h), and smaller and uniform nanofibers were observed in the samples spun at a higher feeding rate (e.g., 0.3 mL/h). Generally, the electrical current increases with the feeding rate increasing in polymer solutions. This phenomena occurs depending on the polymer type used to obtain nanofibers.

It was also reported by Zhuo et al. that increasing the feeding flow rate would decrease the fiber diameter, and the surface charge density would decrease as the flow rate increases. A different morphology could be achieved by control of the feeding rate at a given electric field because a certain minimum value of the solution volume suspended at the end of the spinneret should be maintained to form an equilibrium Taylor cone on the morphology of the nanofibers (Zhuo, 2008).

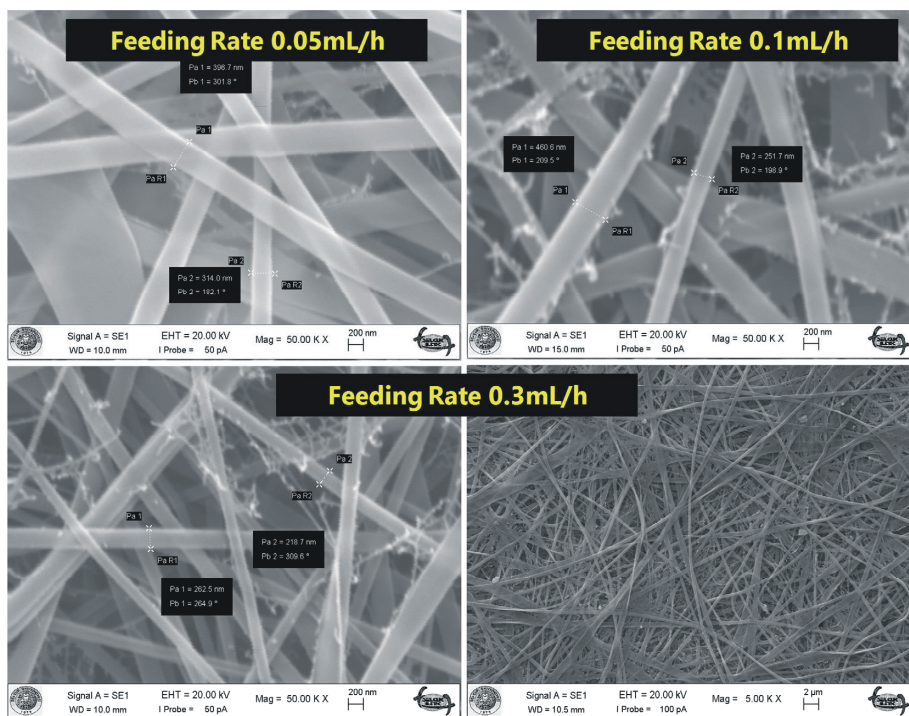


Figure 3. SEM micrographs of Nylon 6,6-Borax nanofibers produced by applying various feeding rates.

Fourier Transform Infrared Spectroscopy

FT-IR spectra of Nylon 6,6 and Borax loaded Nylon 6,6 nanofibers. Nylon 6,6 exhibited the characteristic bands in region of $3200\text{--}3300\text{ cm}^{-1}$ (deformation of O-H groups), at 2940 ve 2860 cm^{-1} (C-H stretching and O-H groups), 1640 cm^{-1} (C=O deformation), 1420 cm^{-1} (C-O-H deformation), 1256 cm^{-1} (stretching of C-O groups).

The characteristic peaks of the sample ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) are described as follows: the band at 1652 cm^{-1} and the broad band at 3405 cm^{-1} can be assigned to the HOH bending and OH stretching modes respectively, which shows that compound contain water molecules.

If the spectrum is closely investigated, some peaks of Nylon 6,6 fiber have diminished because of the Borax decahydrate.

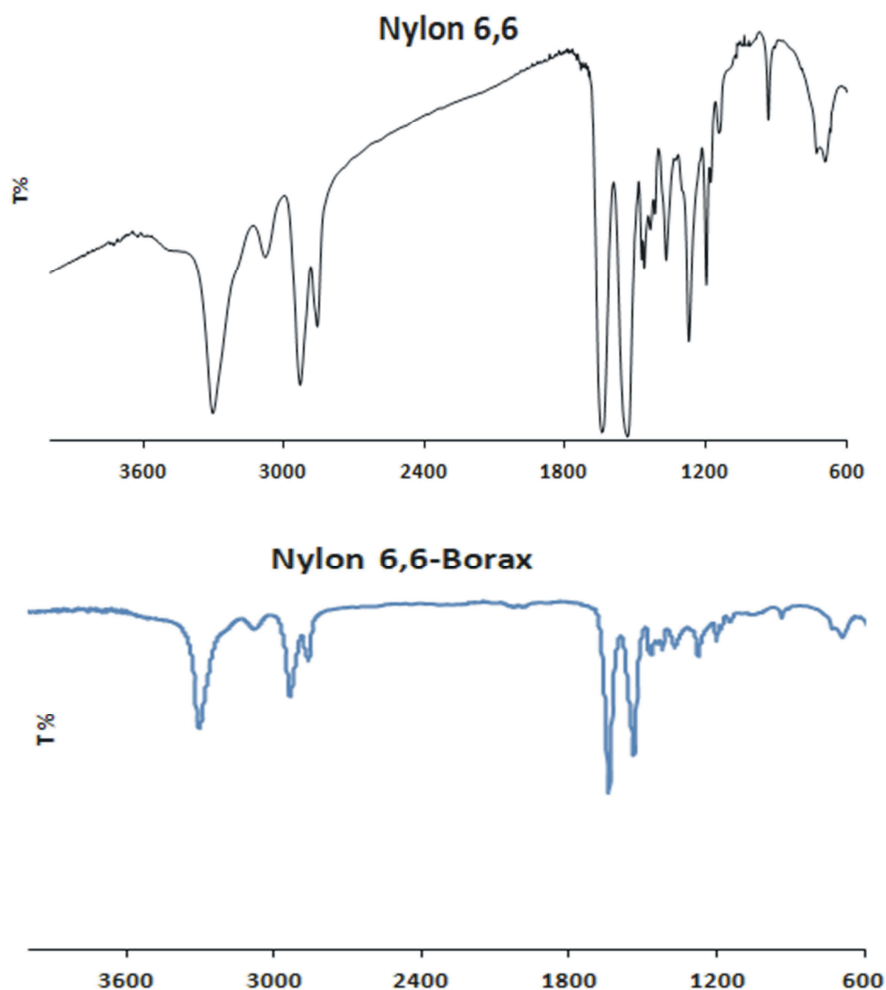


Figure 4. FT-IR spectra of Nylon 6,6 and Nylon 6,6-Borax nanofibers

Thermal Gravimetric Analysis

Figure 5 presents the thermogravimetry curves of Nylon 6,6 and Nylon 6,6-borax nanofibers. The thermal stability was examined in nitrogen atmosphere to assess the degradation behavior of the nylon nanofibers in the presence of borax. All the samples were heated from 25 to 700 °C with a heating rate of 10°C/min. Detailed data, including various temperature, temperature for the maximum decomposition rate and char residues at 700 °C are shown in Table 2.

Although, the inclusion of Borax with very low concentration, positively influenced the thermostability and reduced the mass loss rate of Nylon 6,6.

The Borax on Nylon could promote smoldering and the formation of char. Char is an important indicator of the reduced flammability of the materials. The formed char could serve as a thermal barrier to protect the nylon based materials from further degradation. Furthermore, a rapid boost up in char yield was observed with the loading of borax which

was due to generation of thermally stable inorganic boron-containing products. It was assumed that the boron-bearing moiety in the char layers exhibited excellent thermo-oxidative stability at higher temperature, and thus stabilized the residues and increased the amount of char yield.

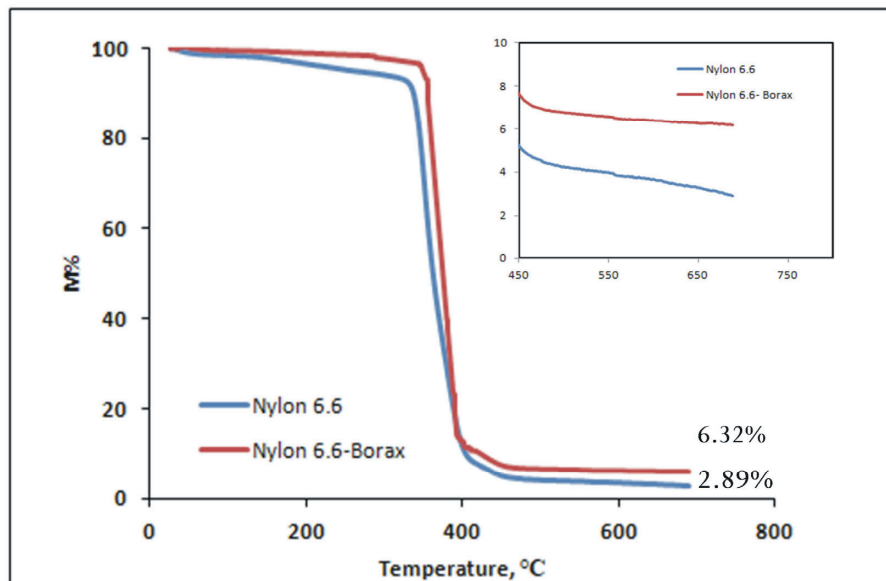


Figure 5. Thermal gravimetric analysis of Nylon 6,6, and Nylon 6,6-Borax

Furthermore, van Krevelen described an empirical equation $LOI=17.5+0.4(CY)$ relating char yield (CY) and the limiting oxygen index (LOI), a numerical index defined as the minimum volume percent of oxygen. The LOI value is considered to be one of the most important and economic and practical methods to examine the flammability of material. In this study, LOI was calculated from van Krevelen equation. It can be seen that an increase in the value of LOI is achieved by the incorporation of 0.8 wt% Borax. Even though the loading amount of Borax was very low in total composition, it can be claimed that it is very effective on thermal resistance of Nylon 6,6.

Table 1. Thermogravimetric data of Nylon 6,6 and borax loaded Nylon 6,6.

	200°C	250°C	300°C	450°C	700°C	LOI
Nylon 6,6	96.1%	95.4%	94.12%	5.2%	2.89%	18.65
Nylon 6,6-Borax	99.5%	98.8%	97.80%	8.4%	6.32%	20.02

Conclusion

The results showed that compared to pristine Nylon 6,6 nanofibers, enhanced thermal properties were obtained for Nylon 6,6-Borax with the loading of %0.8 borax into the polymer matrix. Therefore, an enhanced thermal behavior, and an appropriate physical

property lead to use borax loaded Nylon 6,6 nanofibers for the fabrication of Nylon based products. In addition, the results suggest that the combination of Borax decahydrate and other fire retardant additives can be applied in polymer designs in further studies to effectively improve the stability of Nylon 6,6 polymer.

References

Agwuncha, S. C., Ibrahim, I. D., & Sadiku, E. R. (2017). Improving the thermal and flame resistance properties of polyolefins. In *Polyolefin Fibres* (pp. 421-448). Woodhead Publishing.

Beachley, V., & Wen, X. (2009). Effect of electrospinning parameters on the nanofiber diameter and length. *Materials Science and Engineering: C*, 29(3), 663-668.

Huang, L., Bui, N. N., Manickam, S. S., & McCutcheon, J. R. (2011). Controlling electrospun nanofiber morphology and mechanical properties using humidity. *Journal of polymer science part B: Polymer physics*, 49(24), 1734-1744.

Zhuo, H., Hu, J., Chen, S., & Yeung, L. (2008). Preparation of polyurethane nanofibers by electrospinning. *Journal of Applied Polymer Science*, 109(1), 406-411.

