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## Kompozit Nanolif ve Isı Uygulanmış Nanoliflerin Üretim ve Analizi

### Production and Analysis of Composite Nanofiber and Heat Applied Nanofiber

<sup>1</sup>Onur AYAZ, <sup>2</sup>Nuray UÇAR, <sup>3</sup>Elif BAHAR, <sup>4</sup>Mustafa OKSUZ, <sup>5</sup>Mehmet UCAR, <sup>3</sup>Aysen ONEN, <sup>2</sup>Ali DEMİR, <sup>6</sup>Youjiang WANG

<sup>1</sup>Istanbul Technical University, Nano Science and Nano Eng. Dept., Istanbul, Turkey

<sup>2</sup>Istanbul Technical University, Textile Eng. Dept., Istanbul, Turkey

<sup>3</sup>Istanbul Technical University, Polymer Science and Technology Dept., Istanbul, Turkey

<sup>4</sup>Marmara University, Material Education Dept. Istanbul, Turkey

<sup>5</sup>Kocaeli University, Mechanical Education Dept., Kocaeli, Turkey

<sup>6</sup>Georgia Insitute of Technology, School of Material Eng., Atlanta, USA

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<sup>1</sup>Onur Ayaz

<u> Araştırma Makalesi / Research Article</u>

## PRODUCTION AND ANALYSIS OF COMPOSITE NANOFIBER AND HEAT APPLIED NANOFIBER

TMMOB Tekstil Mühendisleri Odası

UCTEA Chamber of Textile Engineers Tekstil ve Mühendis Journal of Textiles and Engineer

> <sup>2</sup>Nuray Ucar\* <sup>3</sup>Elif Bahar <sup>4</sup>Mustafa Oksuz <sup>5</sup>Mehmet Ucar <sup>3</sup>Aysen Onen <sup>2</sup>Ali Demir <sup>6</sup>Youjiang Wang <sup>1</sup>Istanbul Technical University, Nano Science and Nano Eng. Dept., Istanbul, Turkey <sup>2</sup>Istanbul Technical University, Textile Eng. Dept., Istanbul, Turkey <sup>3</sup>Istanbul Technical University, Naterial Education Dept., Istanbul, Turkey <sup>4</sup>Marmara University, Material Education Dept., Kocaeli, Turkey <sup>5</sup>Kocaeli University, Mechanical Education Dept., Kocaeli, Turkey <sup>6</sup>Georgia Insitute of Technology, School of Material Eng., Atlanta, USA

**ABSTRACT:** In this study, two different applications related with nanofiber production have been studied. In one application, nanofiber was obtained from Maleic Anhydrite grafted Polypropylene (MAH PP) that it is not possible to produce nanofiber at the environmental temperature by solvent technique. The effect of the temperature of polymer solvent on nanofiber producibility has been investigated. It has been seen that it is not possible to produce nano fiber by solvent below 70°C. The bead on the nanofiber increases when solution temperature is around 100°C. Bead formation may be due to lower viscosity resulted from higher temperature. In the other application, elastomeric polymer (Polystyrene-block-poly(ethylene-ran-butylene)-block-polystyrene graft-maleic anhydride) (SEBS-g-MA) together with cellulose nanowhiskers (CNW) was used to produce composite nanofiber. The effect of feed rate and also the amount of water content on properties of composite nanofiber have been investigated. It has been seen that an increase of water content results to deformation of nanofiber morphology and decrease of feeding rate results to thinner fiber.

Key words: Nanofiber, Electrospinning, Polypropylene, Elastomeric Polymer, Cellulose Nano Whisker.

## KOMPOZİT NANOLİF VE ISI UYGULANMIŞ NANOLİFLERİN ÜRETİM VE ANALİZİ

**ÖZET:** Bu çalışmada, iki farklı nanolif uygulaması denenmiştir. Birinci uygulamada, çevresel sıcaklıkta solvent tekniğiyle nanolif elde edilemeyen Maleik Anhidrid graftlı Polipropilenden (MAH PP) nanolif elde edilmiştir. Çözelti sıcaklığının nanolif üretilebilirliğine etkisi incelenmiştir. Sonuçta 70 °C altında nanolif üretilemediği gözlenmiştir. Çözelti sıcaklığı 100 °C civarında iken bead yapısında artış görülmüştür. Yüksek sıcaklığın viskoziteyi düşürmesi bead oluşumunu arttırmıştır. Diğer uygulamada ise selüloz nanowhiskers (CNW) ile beraber elastomeric polimerden (Polystyrene-block-poly(ethylene-ran-butylene)-block-polystyrene graft-maleic anhydride) (SEBS-g-MA) kompozit nanolif üretilmiştir. Besleme hızı ve çözeltideki su miktarının lif yapısına olan etkisi incelenmiştir. Su miktarındaki artışın lif yapısında deformasyona neden olduğu, besleme hızındaki düşüşün ise daha ince lif yapısı oluşumuna katkıda bulunduğu gözlenmiştir.

Anahtar kelimeler: Nanolif, Electrospinning, Polipropilen, Elastomerik Polimer, Selüloz Nano Whisker.

**\*Sorumlu Yazar/Corresponding Author:** ucarnu@itu.edu.tr **DOI:** 10.7216/130075992012198502 www.tekstilvemuhendis.org.tr

### **1. INTRODUCTION**

Electrospinning is a well recognized and effective technique which yields a fiber which diameter is in the range from a few nanometers to a few micrometers. With their small diameter and extremely high surface area to volume ratio electrospun nanofibers can find many application areas such as filtration, tissue engineering, wound dressing, etc. [1, 5].

However, in the literature there is very few studies related with nanofiber produced from Polypropylene. Most of studies related with polypropylene nanofiber were done by melt technique instead of solvent technique, since there is many of limitation to produce polypropylene nanofiber by solvent technique [2]. In this study, for the first time, Maleic Anhydrite grafted Polypropylene has been studied for the production nanofiber by solvent technique and effect of the polymer solution's (MAH-PP) temperature on the nanofiber producibility has been examined. Several properties of polypropylene such as solubility, adhesion was impoved because of Maleic anhydrite grafting. For example, Liu *et.al.* [3] in their studies indicates that, as a result of comparision between chlorinated polypropylene and chlorinated polypropylene grafted maleic (CPP) anhydride (MAH-CPP); miscibility of PP increases with the increase of the MAH grafted content. Also studies of Funasaka et.al. [4] and Qui, et.al. [8] indicate that, grafting of maleic anhydride is effctive for the improvement of adhesive strength.

As known, there are also many of studies related with composite nanofiber reinforced with nano filler such as carbon nano tube, nano clay, etc. However, there are very limited studies done for composite nanofiber filled with cellulose nano whisker (CNW) which is very promising nano filler for improvement of breaking strength and thermal strength [6, 7]. In this study, composite nanofiber filled with CNW has also been produced, i.e., Maleic anhydrite grafted elastomeric polymer (SEBS-g-MA) is polymer matrix while CNW is nanofiller. The effect of water content and feeding rate of polymer solution (SEBSg-MA) on morphology of nanofiber has been examined. Thus; contribution to limited literatures related with composite nanofiber filled with CNW has been provided.

#### 2. MATERIALS AND METHODS

#### 2.1. Materials

Maleic Anhydrite grafted Polypropylene (MAH PP-Epolen E 43-Wax, Westlake Chemical Corp. USA). Elastomeric polymer (Polystyrene-block-poly (ethyleneran-butylene)-bloc-polystyrene graft-maleic anhydride from Sigma Aldrich). Cyclohexane (Merck), Dimethylformaid (DMF, Merck) and Tetrahydrofuran (THF, Merck). Microcrystalline Cellulose (MCC) Avicel type GP1030 (from FMC Biopolymer)

#### 2.2. Preparation of solutions

In this study, two different applications related with nanofiber production have been studied. In one application, nanofiber was obtained from Maleic Anhydrite grafted Polypropylene (MAH PP- Epolen E 43-Wax, Westlake Chemical Corp. USA). As a solvent Cyclohexane (Merck) was used to solve MAH PP. Dimethylformaid (DMF, Merck) and Tetrahydrofuran (THF, Merck) were used to improve the electrospinnability. The percentage of Cyclohexane, DMF and THF is 70, 20, 10 % respectively. The concentration of MAH-PP in solution is 10 %.

In the other application, elastomeric polymer (Polystyrene-block-poly (ethyleneran-butylene)-blocpolystyrene graft-maleic anhydride from Sigma Aldrich-SEBS-g-MA) together with cellulose nanowhiskers (CNW) was used to produce composite nanofiber. The concentration of SEBS-g-MA in solution is 10 %. The percentage of Cyclohexane, DMF and THF is 70, 20, 10 % respectively. The rate of CNW to SEBS-g-MA (in weight) is 50%. Microcrystalline Cellulose (MCC) (Avicel type GP1030 from FMC Biopolymer) was used to produce cellulose nanowhiskers (CNW)

#### 2.3. Preparation of CNW

Acid hydrolysis has been applied to the microcrystalline cellulose (MCC, from Biopolymer) to obtain cellulose nanowhiskers [9]. Obtained CNW was mixed and dispersed in solved polymer solution by sonification method (Bandelin Sonoplus ultrasonic homogenizator)

#### 2.4. Electrospinning

Nanofiber production has been done with a conventional electrospinning unit showed in Figure 1. The syringe pump used to feed solution (Sino mdt, sn-50c6/c6t). High power supply (Matsusada) was connected to steel needle (gauge 0.8 x 38 mm). An aluminum plate was used as a collector. The electropsinning process performed at room temperature. Feeding rate was 2ml/h for MAHPP and 1ml/h, 0.5ml/h for SEBS-g-MA. Applied voltage was 20kV for MAHPP and 20kV for SEBS-g-MA, the distance between needle and collector was 15 cm.

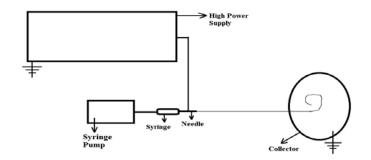


Figure 1. Schematic illustration of electrospinning unit

In this study, Scanning Electron Microscopy (SEM, JEOL, Model JSM-5910LV) and optical microscopy with 1000 times magnification (Olympus BX51) have been used to examine the morpholgy of Maleic Anhydrite grafted Polypropylene nanofiber and composite nano fiber.

#### **3. RESULTS AND DISCUSSION**

As known from literatures, there is a very limited study related with polypropylene nanofiber because of limitation of producibility of polypropylene nanofiber (especially by solvent technique). However, in this study, nanofiber from Maleic Anhydrite grafted Polypropylene (MAH-PP) could be produced. It has been seen that while it is not possible to produce MAH-PP nanofiber in the environmental temperature condition, it is possible to produce MAH-PP nanofiber at the temperature between 75°C and 100°C. The bead on the nanofiber increases when solution temperature is around 100°C. Bead formation may be due to lower viscosity resulted from higher temperature (Figure 2 and Figure 3).

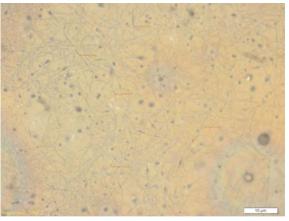


Figure 2. Nanofibers produced at 75°C (x1000)

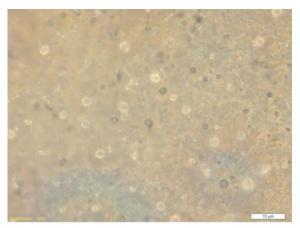


Figure3. Nanofibers produced at 100°C (x000)

At the other application, it could be possible to produce composite nanofiber which is consisted of elastomeric polymer (Polystyrene-block-poly (ethyleneran-butylene) -bloc-polystyrene graft-maleic anhydride) as polymer matrix and cellulose nanowhiskers (CNW) as nanofiller. The effect of feed rate and also the amount of water content on properties of composite nanofiber have been investigated. It has been seen that an increase of water content on the solution results to deformation of nanofiber morphology (Figure 4) and decrease of feeding rate from 1 ml/h to 0.5 ml/h results to thinner fiber. Viscosity was increased from 48cP to the 290cP, when CNW with water was added into polymer solution. From Figure 4 and Figure 5 the positive effect of absence of water on morphology of electrospun could be observed. Presence of water prevents the dispersion of CNW and causes aggregation and also evaporation of solvents is slower because of water.

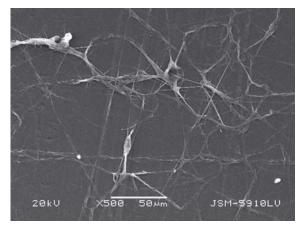


Figure 4. SEM image of SEBS-g-MA+CNW electrospun with the 20kV, 1ml/h, 15cm electrospinning parameters.

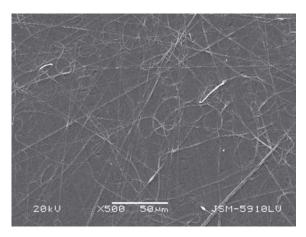


Figure 5. SEM image of SEBS-g-MA+CNW electrospun nanofiber without water inclusion.

#### 4. CONCLUSIONS

From this study, it has been seen that it is possible to get nanofiber from Maleic anhydrite grafted polypropylene, if polymer solution is heated. The water content affects negatively the morphology of composite nanofiber. In the future; it will be tried to produce composite nanofiber by use of heated polypropylene solution and the effect of parameters such as temperature, amount of filler, setting, etc on the morphology of composite polypropylene nanofiber will be searched.

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