

## Fabrication and Characterization of Electrospun Wool Nanoparticles/Nylon 6 Composite Nanofiber Yarn

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**Abstract**-The present study attempts to combine the properties of natural and synthetic fibers by using nanotechnology. Considering the potential of nylon nanofiber structures in some applications such as controlled drug release, moisture micro-sensors and antibacterial filters, the moisture absorption of the nanofibers could be improved via using biocompatible natural fibers such as wool. In this investigation, it was achieved to produce yarns of composite nylon 6 nanofibers, incorporating wool nanoparticles (WNPs), via electrospinning. WNPs were added to the electrospinning solution of nylon 6 in various concentrations. Scanning electron microscopy was employed to characterize the morphology of composite nanofibers. The existence of WNPs in the nanofibers was confirmed by transmission electron microscopy. Fourier transform infrared spectroscopy (FTIR) spectra showed that nanofibrous composite formation did not influence the chemical bonds of both the wool nanoparticles and nylon 6, and no new steady bonds were formed. It was found that an increase in the WNPs concentration increased the diameter of composite nanofibers from  $152\pm 16$  nm (pure nanofibers) to  $266\pm 51$  nm (7 wt% nanoparticles). The assessment of moisture regain of composite nanofiber yarns showed that the moisture absorption of nylon 6 nanofibers improved by introducing the WNPs as hydrophilic components. The moisture regain of the

composite nanofibers yarn containing 7 wt% nanoparticles was found to be higher than that of the pure nanofibers yarn by about 117.1%. On the other hand, tensile strength and elongation-at-break of composite nanofiber yarns initially decreased and then increased with the increase in WNPs concentration.

**Keywords:** nylon 6, electrospinning, nanofibers, wool nanoparticles, composite nanofibers yarn

### I. INTRODUCTION

Natural fibers have played an important role in fibrous materials since ancient times. They are still widely used to develop modern textiles, due to their unique properties such as high moisture absorption and enhanced comfort. Wool is a biodegradable and biocompatible natural fiber that has caught much attention for being used in textiles. The reason lies in its important properties, e.g., its high moisture recovery, excellent flexibility, good insulation capacities, low heat conductivity, and decent affinity for dyes [1]. Unsuitable fibers for spinning in the textile process wool fibers or poor quality raw wool could be the sources of some short fibers called wastes [2]. Developing a new method for reusing these fibers has large marketing potentials due to their excellent intrinsic properties [3-5]. Since protein powder can keep the original properties of the materials without destroying their microstructure, it has been widely applied in modern industries and some hi-tech related fields due to some special properties [6-8]. Recently, there have been several studies relevant to methods of making and using wool particles. One review [9] focused on the method of making fine particles from protein fibers and how to apply protein fiber particles.

To improve some properties of synthetic polymers, like dyeability, elastic recovery, warmth retention, water vapor

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permeability, and moisture retention, the wool powder was produced [10-16]. Wool particles also appear as fillers in composites to boost both reinforcement and mechanical properties [17]. To recycle the discarded protein resources, most researchers have developed keratin films. These films could be used as packaging materials and replace the daily-used synthetic packaging films which are very difficult to degrade inside nature [18,19]. Donato *et al.* (2019) [20] described keratin-based biomaterials. Furthermore, researchers [21] studied the uses of keratins extracted from feathers, hair, wool, and horn. Wool-keratin-based composites [1,22-25] such as composite nanofiber structures [26-29] can be utilized in tissue engineering, wound bandages, biomedical and drug-delivery systems, and antimicrobial filters. A high-efficiency method to extract keratin from wool was developed by Shen *et al.* (2019). Afterward, an Ag-doped keratin/PA6 composite nanofiber membrane was prepared that had enhanced filtration, excellent water-vapor transmission rate, and antibacterial performance [29].

Recent years have witnessed much attention directed at the use of electrospun composite nanofibers with various nanoparticles such as silver (Ag) [30], TiO<sub>2</sub> [31], silica nanospheres (KCC-1) [32], and electrospun composite nanofibers like gelatin/nylon 6 [33-35] and polyamide 6/CNT [36]. A fact that is mainly due to their activities.

Electrospinning has already emerged as an ideal route for producing aligned nanofibers composites [37-40]. The technique involves applying a certain high voltage to a polymer solution in a syringe. In this way, the polymer solution overcomes the surface tension, ejecting a fine jet of liquid from the needle's tip, which then deposits on the collector as nanofiber [41-45]. As a 3D nanofiber structure, electrospinning nanofiber yarn including a good twist, orientation, large surface area, and secondary processability has a unique consumption in functional textiles, flexible and wearable electronic devices, and carbon nanofiber yarn formation [46-49]. Up to now, various methods like coupled-electrodes [50], liquid bath [51], funnel twisting [52], rotating airflow [53], suction wind [54], etc., have been examined for preparing the nanofiber yarns. Compared to these technologies, the new one is more stable and effective for rotating the winding units [55] in the coupled-electrodes method to convert nanofibers into continuous twisted yarns. In terms of yarn structure, nanofiber yarns can be divided into pure nanofiber and composite nanofiber yarns. The latter is considered to be a new research direction to improve some yarn properties.

A novel mechanism to spin continuous twisted composite nanofiber yarns has been reported by Bazbous and Stylios (2008) [56]. The resultant nanofiber has several

important characteristics, e.g., a very large surface area to volume ratio ( $10^3$  times that of a micro-fiber) and flexibility in the surface functionalities. One study has investigated the production of continuous nylon 6/single-wall nanotubes (SWNTs) composite nanofiber yarns via the electrospinning method with a liquid bath as a collector. The mechanical and electrical properties and electric resistance-tensile strain sensitivity of the composite nanofiber yarns have been studied [51]. Wang *et al.* (2021) presented a simplified dry electrospinning system for the self-assembling fabrication of composite nanofiber yarn. The polymer in their study was a mixture of cellulose nanocrystals (CNC), polyvinyl acrylate (PVA), and poly(3,4-ethylene dioxythiophene) polystyrene sulfonate (PEDOT: PSS). The novelty lay in the self-triggered thread formation during electrospinning is enabled by electrostatic forces between conductive nanofibers made from polymer composite [57].

Nylon is one of the most widely used commercial polymer fibers with a wide range of applications [58]. In particular, nylon 6 is a synthetic and biocompatible polymer with good mechanical strength, excellent spinnability, and stability [59]. Various techniques have been used to synthesize nylon 6 fibers, including melt spinning, wet spinning, dry spinning, and electrospinning. The synthesized fibers are composed of various quantities of the two common crystalline forms of nylon 6, the thermodynamically stable  $\alpha$  form and the less stable  $\gamma$  form. The obtained crystalline form depends on the collecting speed, thermal treatment, and thermo-mechanical history [60]. Due to the capabilities of nylon 6, in the present study, it was used for the synthesis of composite nanofibers via electrospinning. While composite nanofibers are mainly produced by silver (Ag) [30], TiO<sub>2</sub> [31], silica nanospheres [32], gelatin [33-35], carbon nanotubes (CNTs) [36], there is no reported work on nanofibrous structures such as composite nanofiber yarn with nanoparticles of natural fibers such as wool so far. Since the wool fiber is mainly composed of fibrous protein and keratin, as a biodegradable and biocompatible natural fiber, in the previous studies, the extraction of protein powder and keratin of these fibers and their application has been considered. Although keratins have a high potential for several applications because of inherent biological activity and biocompatibility, their usage is rather limited due to their poor mechanical properties.

In this work, electrospun composite nanofiber yarns containing wool nanoparticles (WNPs) with enhanced moisture absorption ability were investigated. Firstly, WNPs, which were produced by mechanical processes, were dispersed in the solution of electrospinning. Secondly, in the process of electrospinning, WNP/nylon 6 composite

nanofiber yarn was produced by the coupled-electrode method and characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier transform infrared (FTIR) spectroscopy, and particle size analyzer (PSA). Some mechanical, physical, and morphological properties of composite nanofiber yarn were tested and reported.

## II. EXPERIMENTAL

### A. Materials

In this study, nylon 6 ( $M_w$  35,000) was purchased from Aliaf Co. (Iran) and formic acid (FA,  $\text{CH}_2\text{O}_2$ , 99–100%,  $46.03 \text{ g}\cdot\text{mol}^{-1}$ ) was supplied from Merck (Germany). Baluchi wool fiber (22.5 den, Iran) was used to prepare the wool nanoparticles.

### B. Preparation of Wool Nanoparticles

Wool nanoparticles were prepared as recycled material through a physical method. The research pulverized wool fibers into a nanoscale particle step by step which are described briefly as follows:

The wool fibers were first washed and cleaned according to the method described in ASTM D 584 [61], then to get dried. After that, the wool fibers were cut into pieces, 1–2 cm long, and then were crushed into small pieces by a mechanical rotating crusher, resulting in a particle length of around  $300 \mu\text{m}$ . The first wool particles were then pulverized with a planetary ball mill (PM 100, © Retsch GmbH, Innovative FFCS Technology, Germany) with one grinding station for grinding jars with a nominal volume of 12 mL to 500 mL. This ball milling machine was charged with 500 g stainless steel balls (20 mm diameter) as grinding media. The speed of ball milling was set at 300 rpm. In this step, the wool particles were pulverized for seven hours discontinuously. After this pulverization step, the majority of the wool particles were smaller than  $2 \mu\text{m}$  in optical microscopy trials. Then, the second pulverized wool particles were sieved to remove large and uncrushed particles. After crushing and sieving, the obtained particles were re-pulverized into nanoscale wool by implementing the previous steps again. After the milling process reiteration, the particles' size was smaller than 200 nm in diameter (see III.A).

### C. Polymer Solution Preparation

The electrospinning solution with 12 wt% concentration was prepared by dissolving the nylon 6 polymer in formic acid, which was stirred for 24 h at an ambient temperature. To produce the composite nanofibers, at first, different masses of WNPs (3, 5, and 7% in respect to the polymer weight) were weighted and added to the formic acid. Then, nylon 6

granules were added to this mixture and stirred for 24 h until the polymer was uniformly dissolved in the solvent. Before the electrospinning process, the solution, comprised of WNPs and nylon 6 in formic acid, got sonicated for 10 min, using an Ultrasonic Homogenizer model UP200H (Hielscher), operating at 24 kHz. So WNPs were dispersed to disrupt the possible agglomeration in any solution. These mixtures were kept in an airtight bottle to prevent formic acid from evaporating. Thus, the final solutions contained various concentrations of WNPs from 0 wt% to 7 wt% in 12 wt% nylon 6 solutions. These concentrations were determined because these conditions were able to produce bead-free nanofibers in stable conditions.

### D. Electrospinning Setup and Composite Nanofiber Yarn Production

Two needles with opposite voltages were used simultaneously to spin the nanofiber yarn. This enables nanofibers with opposite charges to pull in, stick together and form a yarn after drafting towards the collecting surface. Then, the neutral yarn can be easily collected. It is an advantage compared to the conventional technique wherein one electrode is used as the collector (grounded or connected to a negative voltage). Fig. 1 illustrates the schematic of the setup for electrospinning. Flat-tipped and stainless steel needles (0.6 mm OD) were installed in opposite directions at a distance of about 18 cm. Polymer solutions were pumped to needles by two infusion pumps each including one syringe. Typical operating regimes had feeding rates of 0.24 mL/h, with the two needles' applied voltages adjusted to 11 kV. While voltages were applied to the needles with polymer solution droplets at the tip, the polymer jets were ejected simultaneously. Then the opposite jets met each other which results in the formation of a cluster of fibers. The fibers, emerging from the two

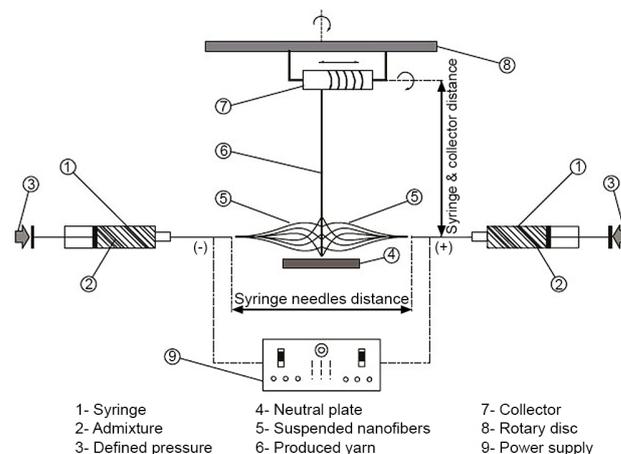


Fig. 1. Schematic of electrospinning set up for collection and twisting of continuous nanofibers yarn.

needles are combined into a yarn body which was wound on a cylindrical collector rotating with a speed of 1.6 (cm. min<sup>-1</sup>). So, the twist value (twists per meter, TPM) for the nanofiber yarns was about 3000 TPM.

#### E. Structural and Morphological Characterization

The surface morphologies of scattered nanofibers and nanofiber yarns were observed by scanning electron microscope (SEM, XL30, Philips, Netherlands) operating at 17 kV. The samples were sputter-coated with gold-palladium (SCD005, BAL-TEDC) to achieve a thin layer of the conductive layer for SEM samples. The average diameter of nanofibers was measured from at least 100 points nanofiber samples by Image-J software (National Institute of Health) [54].

A transmission electron microscopy (TEM–Philips CM120 with an accelerating voltage of 120 kV) was utilized to qualitatively determine the size distribution of WNP in the composite nanofiber on Cu grids for samples of randomly-collected fibers.

Particle size analysis (PSA), carried out with Nanophax-Sympatec GmbH 2008, Sympatec's Photon Cross-Correlation Spectroscopy (PCCS), is a novel technique that allows the simultaneous measurement of particle size and stability in the range of about 1 nm to a few μm in opaque suspensions and emulsions. This experimental system is operated with a HeNe laser light (632.8 nm) and a detector positioned at a scattering angle of 90°. The measurement time was 60 s and the measurement temperature was 25 °C. Each sample was tested at least three times. Raw data were evaluated by the non-negative least squares (NNLS) model which did not imply any particle shape or distribution.

Fourier transform infrared spectroscopy (FTIR) (BOMEN-MB-series 100, Hartman and Braun, Canada) was employed to study any possible reaction or chemical structural changes as a result of the milling process and blending of WNP with nylon 6 in formic acid solvent to produce the electrospinning solution. FTIR spectra were recorded from 30 scans in an ambient temperature and the absorbant mode.

To measure the tensile mechanical properties of the produced yarns, the universal testing machine (Zwick tensile tester, 1446-60, Ulm, Germany) was used including a load cell of 50 N and extension rate of 15 mm.min<sup>-1</sup>. The nanofiber yarn sample for this test was fixed vertically in a paper frame at a gage of 25 mm clamp distance. The yarn fineness as tex was measured in each group vibroscope method to normalize the tensile results. The mechanical properties of composite nanofiber yarns were measured considering two various states, before

and after standardization (pre-standard and post-standard conditions). Initially, the results were obtained before the exposure to standard conditions and then after the exposure for 24 h under the standard conditions (65±2% relative humidity and 21±1 °C) in which the samples absorbed the moisture. The tensile mechanical test was repeated for 25 different samples for each studied group.

The moisture regain is a property to evaluate the moisture absorption capacity of a yarn in which the ratio between the mass of the moisture absorbed into the yarn and the dry mass of the yarn is called its moisture regain. The moisture regain of the composite nanofiber yarns was determined by oven drying the yarns at 95 °C for 1 h. The yarns were then allowed to absorb moisture under the standard testing conditions (21±1 °C and 65±2% relative humidity) for 24 h. The difference between the mass of the sample after conditioning and the oven-dried mass was used to calculate the moisture regain of the yarn which was calculated as Eq. (1):

$$\text{Moisture regain (\%)} = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

Where,  $W_1$  and  $W_2$  represent the dry weight and conditioned weight of the samples, respectively. Moisture regain in 5 samples was measured for each studied group.

Data values obtained in the experiments were analyzed statistically based on a one-way analysis of variance (ANOVA) at a level of 95% confidence interval for the mean, employing SPSS software. The results were reported as a mean±SD. Differences in the properties of the nanofiber yarns were determined by Fisher's least significant difference (LSD) mean discrimination test.

### III. RESULTS AND DISCUSSION

#### A. Characterization of Wool Nanoparticles

Fig. 2 shows the particle size distribution of ball-milled wool fibers covering the size range of 60 nm to 200 nm. 95% of the powder particles are between 80 and 180 nm

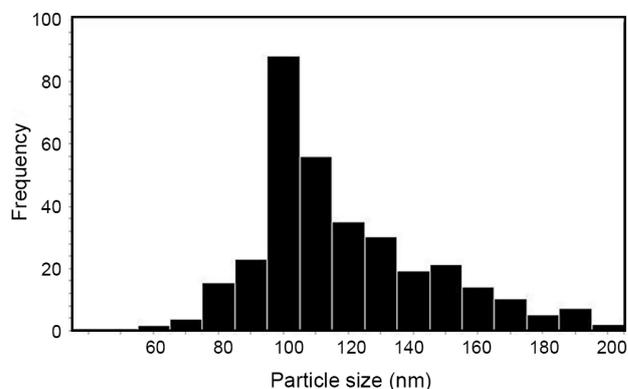
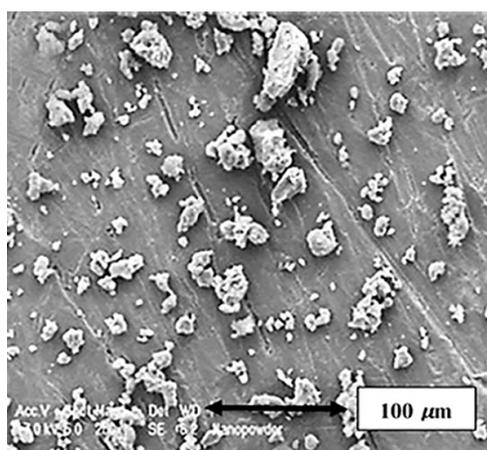
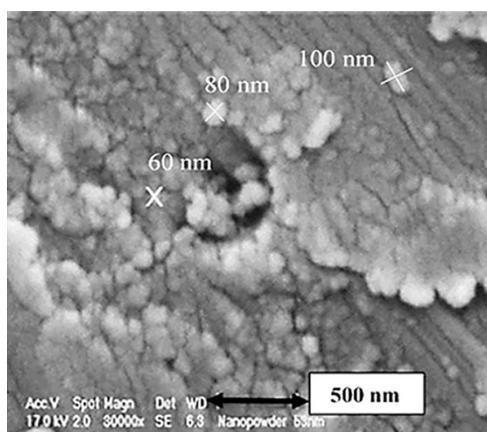


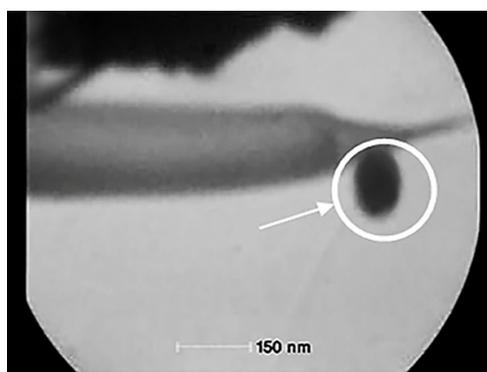
Fig. 2. Particle size distribution.



(a)



(b)



(c)

Fig. 3. SEM images of wool powder with scale bars: (a) 100 μm, (b) 500 nm, and (c) TEM image of a wool nanoparticle.

in diameter and the average particle size is 100 nm. The SEM images of typical wool powder depict these findings in comparison with the TEM images of wool nanoparticle-embedded nylon 6 nanofiber in Fig. 3.

### B. FTIR Analysis

A comparison of FTIR absorbance was used to investigate the possibility of changes in the chemical structure of wool due to the milling process. Fig. 4 is the FTIR analysis results

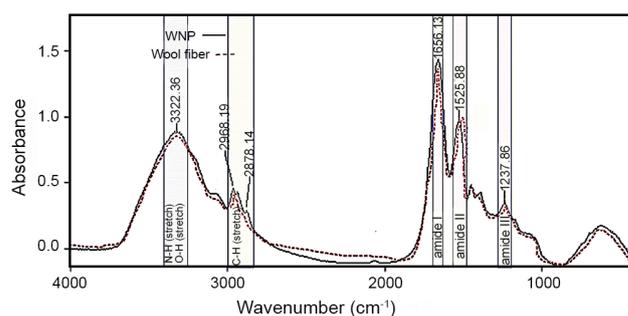


Fig. 4. FTIR spectra of wool nanoparticles (WNP) and wool fiber.

of wool fiber before milling and as nanoparticles. The two curves exhibit similar absorption bands around 3322 cm<sup>-1</sup> (the combined effect of N-H and O-H stretching vibrations), 2968 cm<sup>-1</sup> (C-H asymmetric stretching vibrations), 2870 cm<sup>-1</sup> (C-H symmetric stretching vibrations), 1656 cm<sup>-1</sup> (amide I), 1525 cm<sup>-1</sup> (amide II), and 1237 cm<sup>-1</sup> (amide III). These bands are similar to the FTIR spectra related to the protein fibers that are in good agreement with the other related works [62-64]. Fig. 4 also indicates that the chemical structures of wool fiber and WNP are much similar and no new functional groups or chemical bonds were produced in the WNP after the milling process.

FTIR analysis was also performed to investigate any possible reaction or chemical structural changes as a result

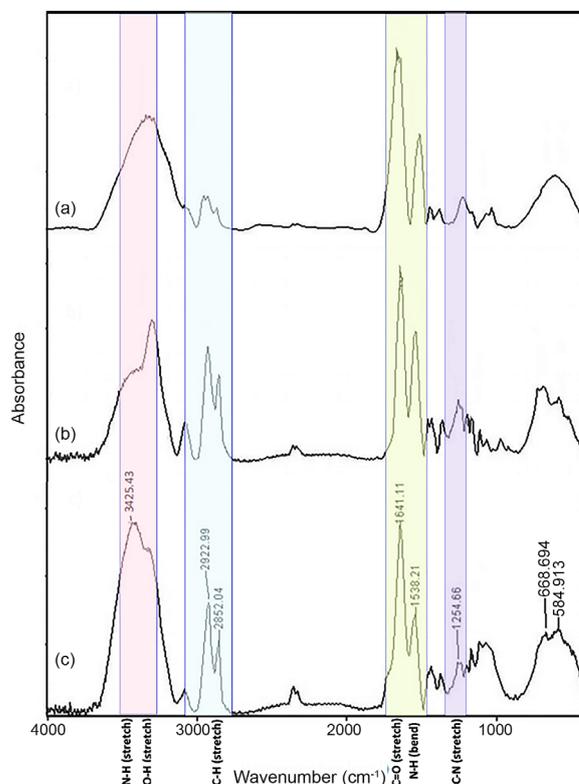


Fig. 5. FTIR spectra of: (a) wool nanoparticles, (b) nylon 6 nanofibers, and (c) WNP/nylon 6 composite nanofibers.

of the blending wool nanoparticles with the nylon 6 solution. Fig. 5 presents the FTIR spectra of nylon 6 composite nanofiber containing 7 wt% WNPs in comparison with nylon 6 nanofibers and wool nanoparticles. There were several absorption bonds in the curves. The bonds at 3425 and 3309  $\text{cm}^{-1}$  are due to the stretching vibration of OH groups in nylon 6 and wool nanoparticles and NH group in  $-\text{NHCO}$ , respectively, whereas the bonds at 2922 and 2852  $\text{cm}^{-1}$  are attributed to the stretch vibration of C-H groups in nylon 6 chain and amino acids of protein polymer. The spectra of the composite nanofiber yarn exhibit the absorbing bonds around 1641 (amide I, C=O stretching), 1538 (amide II, secondary N-H bending), and 1254  $\text{cm}^{-1}$  (amide III, C-N stretching), indicating the characteristic vibration bonds of nylon 6 and protein of wool. Other absorbing bonds around 1428, 1370, 1171, 1074, 668, and 584  $\text{cm}^{-1}$  were also found in the composite nanofiber structures. Therefore, the FTIR spectra of the WNPs/nylon 6 composite nanofiber show the characteristic absorbing bonds of both wool nanoparticles and nylon 6. Likewise, it is deduced that the nanofibrous composite formation does not influence the chemical bonds of both the wool nanoparticles and nylon 6, and no new steady bonds are

formed by a simple physical mixture.

### C. Morphological Characterizations

Fig. 6 presents the diameter distributions for electrospun nanofibers containing various concentrations of WNPs. The results show that an increase in the concentration of WNPs led to an increase in average composite nanofibers' diameter. The nanofibers' diameter increased from  $152 \pm 16$  nm (the pure nylon 6 nanofibers) to  $266 \pm 51$  nm when composite nanofiber yarn was produced with 7 wt% nanoparticles. Comparing nylon 6 nanofibers, adding 7 wt% WNPs to the electrospinning solution caused an increase of 75.4% in nanofiber diameter that these changes were significant ( $p$ -value $<0.05$ ).

The effects of WNPs concentration on the solution viscosity and diameter of the resulting nanofibers are shown in Table I. According to this table, increasing nanoparticle content significantly ( $p$ -value $<0.05$ ) increased solution viscosity and nanofibers' diameter. Similar results have been reported in studies [26,65], in which by increasing the nanoparticle concentration in composite nanofiber compared to that of pure nanofiber, solution viscosity, and consequently, nanofibers' diameter increased.

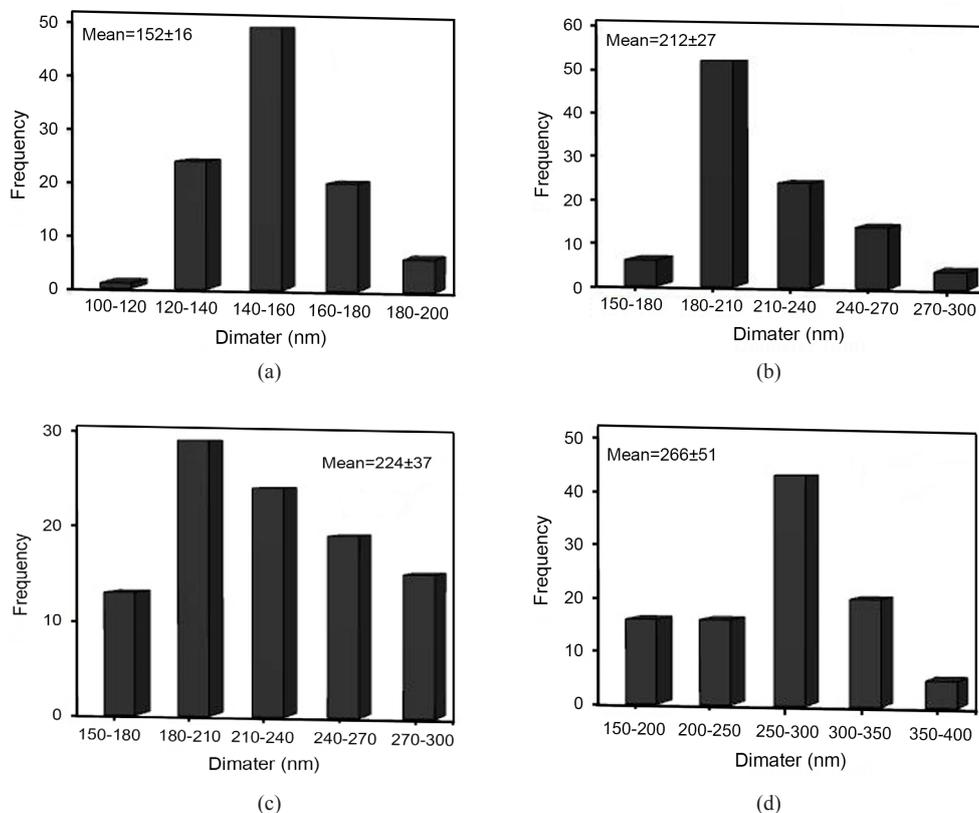


Fig. 6. Frequency distribution diagrams of electrospun nanofibers containing various concentrations of WNPs in 12 wt% nylon 6 solution: (a) 0%, (b) 3%, (c) 5%, and (d) 7%.

TABLE I  
SOLUTION VISCOSITY AND NANOFIBER DIAMETER WITH DIFFERENT WNPS CONTENTS

Sample	Viscosity (Pa×s)	Nanofibers' diameter (nm)
12% nylon 6	0.34±0.03	152±16
3% WNP/nylon 6	0.43±0.01	212±27
5% WNP/nylon 6	0.55±0.04	224±37
7% WNP/nylon 6	0.63±0.02	266±51

SEM images (Fig. 7) show some morphological features of the yarns and nanofibers in the structure of yarns. These images show the composite nanofibers are uniform

and bead-free. The surface morphology of related yarns containing various concentrations of WNPs from 0 wt% to 7 wt% in 12 wt% nylon 6 solution is also partially

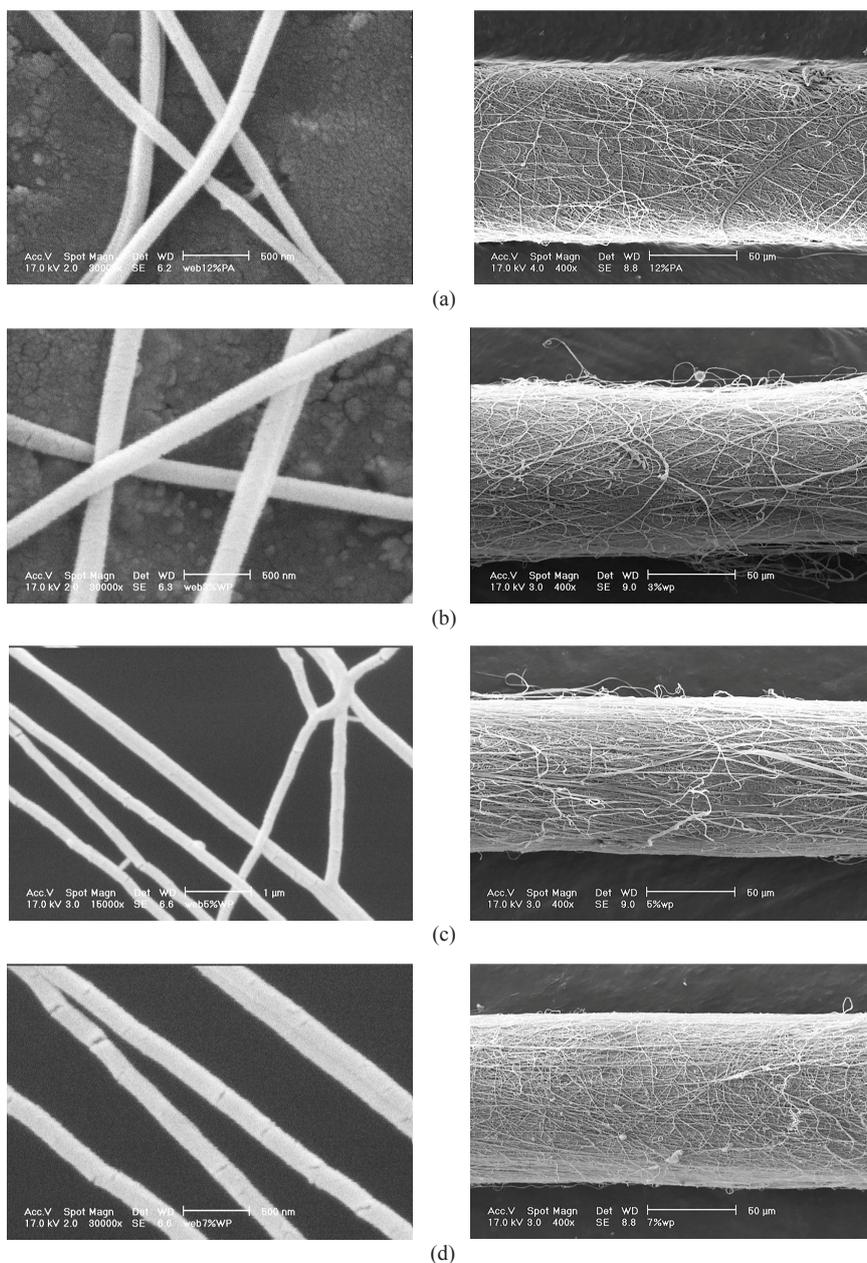
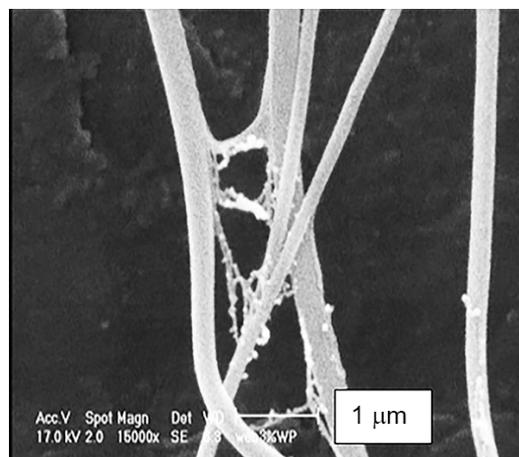


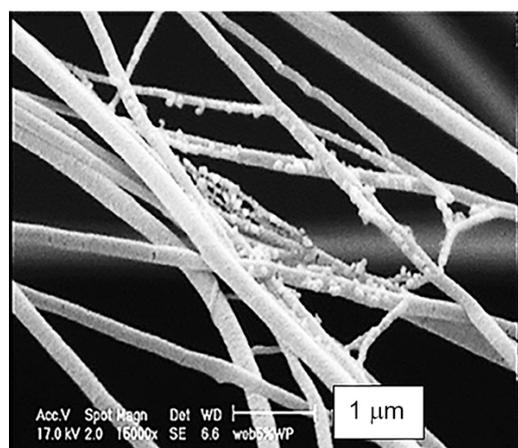
Fig. 7. SEM images of WNPs/nylon 6 composite nanofibers and yarns containing: (a) 0 wt%, (b) 3 wt%, (c) 5 wt%, and (d) 7 wt% wool nanoparticles. Left images show nanofibers in the structure of yarn and the right ones show the nanofiber yarns.

demonstrated in this figure.

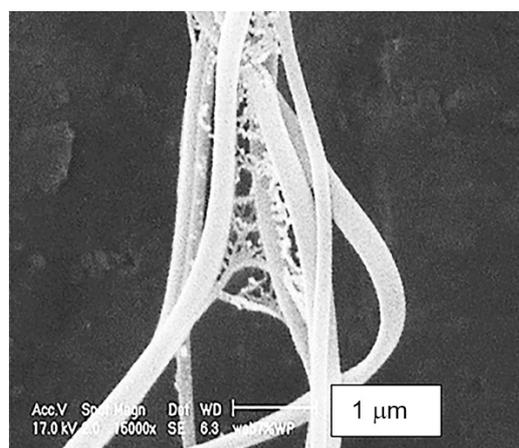
Dispersing the WNPs inside the nylon 6 nanofibers body and placing them along the axis of the fibers was a challenging issue, which was achieved through utilizing



(a)



(b)



(c)

Fig. 8. SEM images of external surfaces of the nanofibers produced from WNP/nylon 6 containing various concentrations of WNPs: (a) 3 wt%, (b) 5 wt%, and (c) 7 wt%.

the electrospinning process. Poor dispersion may occur due to the tendency of WNPs for agglomeration after migration in the solution flow. Also, the build-up of internal stresses could have resulted from the agglomeration of WNPs at higher concentrations, where a larger portion of WNPs is used. To avoid the agglomeration of nanoparticles in this work, the solution was electrospun immediately after sonication, leading in turn to the formation of a stable jet via electrospinning. This means that WNPs were distributed within the nanofibrous matrix without any agglomerations, and the process was well designed to achieve the surface tension and viscosity of polymer composite solution after the WNP component increased in the solution. By using WNPs in the nylon 6 nanofibers matrix, the properties of natural and synthetic fiber had the chance to be combined.

It can also be seen in Fig. 7 that with the addition of nanoparticles up to 7 wt%, some cracks on the surface of nanofibers were created. It is probably due to different hygroscopy of the protein (WNPs) and nylon polymer. Also, the absorption of moisture by wool nanoparticles can cause swelling, and on the other hand, nylon loses its moisture much faster than wool, which these conditions can cause cracks.

Fig. 8 shows the SEM images of parts of the nanofibers in the yarn structure where the nanofibers were finer and the wool nanoparticles tended to migrate to the outer surface of the nanofibers. It can be said that the availability of nanoparticles on the surface of finer nanofibers was increased. This result was observed for finer composite nanofibers containing 3, 5, and 7 wt% of wool nanoparticles. TEM images were taken to confirm the embedding of the nanoparticles in the nanofibers. Fig. 9 shows the TEM images of electrospun nylon 6 composite nanofibers containing 7 wt% WNPs. These images confirm the distribution and existence of WNPs in the nylon 6 composite nanofibers. As a result, TEM images can indicate the stability of the nanoparticles in the electrospinning solution and the good compatibility of wool nanoparticles with formic acid and nylon polymer.

#### D. Tensile Mechanical Properties

The mechanical properties of the nanofiber yarns in terms of the tensile strength (TS) and elongation-at-break (strain%) were investigated and compared with each other in both pre-standard and post-standard conditions (Fig. 10). The results showed that there was a statistically significant difference in the mechanical properties of nylon 6 nanofiber yarn and composite nanofiber yarns. It was observed that the tensile strength and elongation of nanofiber yarns first decreased and then increased with the increase in WNPs concentration. Under pre-standard

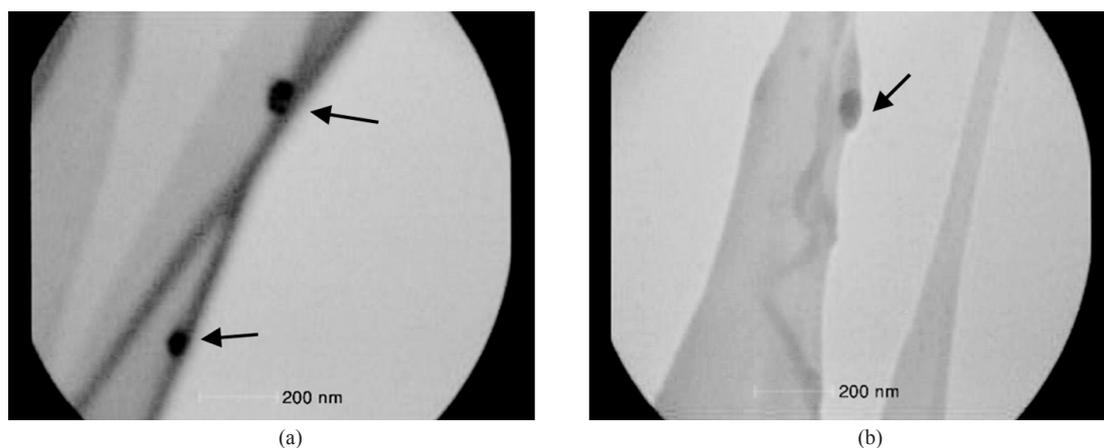


Fig. 9. TEM images of nylon 6 nanofibers containing 7 wt% WNPs in 12 wt% nylon 6 solution (arrows show the wool nanoparticles).

conditions, nylon 6 nanofiber yarn exhibited an average TS of  $8.82 \text{ cN.tex}^{-1}$ , which decreased significantly ( $p\text{-value} < 0.05$ ) upon blending with 3 wt% wool nanoparticles, up to  $5.95 \text{ cN.tex}^{-1}$ . In previous researches, similar results are reported regarding the effect of protein content on the tensile properties of nanofibers membranes. Cho *et al.* [66] indicated that increasing the content of soy protein isolate (SPI) caused a sharp reduction of both the breaking strength and elongation of the SPI/poly(vinyl alcohol) hybrid nanofibers. This result was consistent with the fact that SPI protein, due to its globular structure, could not produce strong fibers. Furthermore, Zhu *et al.* [26] reported that the incorporation of wool keratin decreased the mechanical properties of wool keratin/PCL nanofiber membranes, which might be due to the hard protein nature of the regenerated keratin [26]. In the present study, composite nanofiber yarns had ended up with less mechanical strength and elongation than the nylon nanofiber yarn. This is probably due to the distribution of WNPs and the creation of stress concentration sites in the vicinity of the crack

tips, which affects the mechanical properties of nanofibers. Also, the addition of wool nanoparticles might destroy the continuous and uniform internal structure of pure nylon 6 nanofibers yarn. These results are similar to those reported for the tensile properties of nylon 6/CeO<sub>2</sub> composite nanofibers [28] in which the addition of CeO<sub>2</sub> to nylon 6 nanofibers resulted in the reduction of tensile strength and easy fracture of fibers due to the stress concentration point in nylon 6 fibers.

Besides, in the composite yarns, the obtained results showed that with the addition of nanoparticles, tensile strength, and elongation of the composites increased, particularly at high nanoparticle content (7 wt%), which was approximately close to the strength of nylon 6 nanofiber yarn. This can be attributed to the compatibility of nylon 6 and WNPs in yarn structure or due to the good dispersion of WNPs as rigid-filler in solution, which improved the mechanical properties. A similar result was reported by Li *et al.* [27] in which PCL/keratin composite nanofibers layers showed improved tensile strength when keratin was

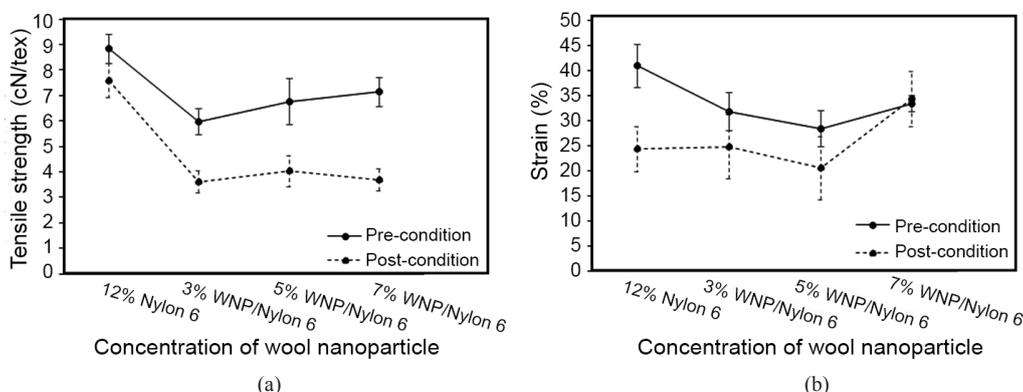


Fig. 10. Mechanical properties of the produced nanofibers containing various concentrations of WNPs in 12 wt% nylon 6 solution under pre-standard and post-standard conditions.

added. This result was described by the compatibility of keratin and PCL in a limited range or by the rigid-filler role of hard keratin [27]. In another study, tensile strength values of feather keratin/poly(vinyl alcohol) composite nanofibers enhanced with an increase in protein content. It is explained that the increased tensile strength was possibly related to the favorable compatibility and strong interactions between feather keratin and poly(vinyl alcohol) [67].

On the other hand, studies have shown that the diameter of the nanofibers is an important factor affecting the tensile properties of nanofiber yarns and bundles [42,45,68-69]. As the concentration of solution increases, the larger diameters for nanofibers are obtained in the yarns [45,68] and nanofiber membranes [42], which in turn increase the tensile properties. In the present study, according to Fig. 6, the mean diameter of WNP/nylon 6 composite nanofiber increased with the increasing concentration of wool nanoparticles. It can be said that the structure of the composite yarn is affected by the diameter of the electrospun nanofibers, so the mechanical properties of the yarn are also affected and improved by increasing the diameter of the nanofibers.

An analysis of variance (ANOVA) at a level of 95% confidence interval was performed to examine the significance of differences in both pre-standard and post-standard conditions through the SPSS software program. Observing the curves (Fig. 10), it becomes evident that yarn samples were weakened more and more under post-standard conditions, especially the samples of composite yarns that contain more nanoparticles. The WNP/nylon 6 composite nanofiber yarns showed lower tensile strength significantly ( $p$ -value $<0.05$ ) after absorbing moisture than the nylon 6 nanofiber yarn, which was like the results obtained in pre-standard conditions. As a result of these conditions, WNP containing nanofibers got the chance of moisture absorption and swelling, thereof. Consequently, some sites were created for stress concentration which in turn decreased the tensile strength of the composite nanofiber yarns. However, the statistical analysis showed no significant difference between the average strength of composite nanofiber yarn in these conditions ( $p$ -value $>0.05$ ). Moreover, elongation values of nylon 6 nanofiber yarn and composite yarns containing 3 and 5 wt% wool nanoparticles did not differ significantly ( $p$ -value $>0.05$ ). It was also observed that with the addition of nanoparticles up to 7 wt%, elongation of the composite nanofiber yarn, significantly ( $p$ -value $<0.05$ ) increased after absorbing the moisture. It can be stated that with the increase in the wool nanoparticles in this sample, moisture absorption and swelling increased, so the bonds between the molecular chains were weakened and the movement of

TABLE II  
MOISTURE ABSORPTION OF WNPS/NYLON 6 NANOFIBERS AT  
20 °C AND 65% R.H.

Sample	Moisture regain (%)
12% nylon 6	5.73
3% WNP/nylon 6	8.56
5% WNP/nylon 6	10.23
7% WNP/nylon 6	12.44

chains became easier, thus, the yarn elongation increased.

#### E. Moisture Regain

In previous researches, the suitable effect of protein particles on the moisture absorption of blended synthetic polymer films and fibers has been reported. Huang *et al.* [70] reported that the polypropylene/superfine down powder blend films showed higher moisture regain than the pure polypropylene film. Superfine wool powder blended synthetic polymer films also exhibited increased water permeability and moisture regain with the decreased mechanical properties due to an increase in the particle/polymer ratio [71]. Incorporating wool powder into the synthetic polymers could also improve the moisture absorption of viscose fiber [72]. This study demonstrates that moisture absorption of WNP/nylon 6 composite nanofibers was greater than that of nylon 6 nanofibers. The moisture regains (Table II) of the nanofibers of nylon 6 soared from 5.73% of the pure nylon 6 nanofibers yarn to 12.44% of composite nanofibers yarn with 7 wt% nanoparticles component. Amino groups of wool protein fibers made it possible for them to absorb water through hydrogen bonding [73].

Also, this characteristic seems to be the result of thicker fibers having pores with bigger sizes between fibers in the yarn [29,74], which causes the moisture absorption to increase. In the present study, the pore size of WNPs/nylon 6 composite nanofibers yarns with thicker nanofibers was larger than that of pure nylon 6 composite nanofibers yarns, and these pores permitted the water molecules to be absorbed between the structure of composite yarn more easily [75].

#### IV. CONCLUSION

Despite many studies on composite nanofiber yarns, there is no reported work about the production of nanofiber composite yarn with nanopowder of natural fibers such as wool so far. The present study aimed at improving the hygroscopic properties of nylon 6 nanofibers by introducing wool nanoparticles (WNPs) into nylon 6 nanofibers and spinning composite nanofiber yarns with various contents of WNPs. Consequently, some physical,

mechanical, and morphological properties of composite nanofibers and yarns were evaluated. The fourier transform infrared (FTIR) results showed that no probable chemical structural change occurred in wool fiber during milling. Also, the FTIR spectra of the WNPs/nylon 6 composite nanofiber yarn showed the characteristic absorbing bonds of both wool nanoparticles and nylon 6. Similarly, the nanofibrous composite formation did not influence chemical bonds of either the WNP or nylon 6, with no new steady bonds formed by a simple physical mixture. The study indicated that nanotechnology has the quite potential to use the wool nanoparticles in the bulk of nanofibers to enhance moisture absorption. The solutions containing 12 wt% nylon 6 polymer and (0-7 wt%) WNP in formic acid were successfully electrospun via optimizing electrospinning parameters, followed by conversion to composite nanofiber yarn after twisting. An increase in the concentration of nanoparticles had increased composite nanofibers' diameter due to the increase in the solution viscosity. The incorporation of wool nanoparticles with the nylon 6 nanofibers was confirmed by TEM images. Assessment of moisture regain of composite yarns revealed the effect of WNPs content, i.e., moisture absorption of nylon 6 nanofibers improved by introducing the WNPs as a hydrophilic sector. Under pre-standard conditions, tensile strength and elongation-at-break of composite nanofiber yarns initially decreased significantly and increased with the increase in WNPs concentration thereafter. ANOVA results showed there was no significant difference between composite nanofiber yarns under post-standard conditions in tensile strength, and the difference was only in elongation of the composite nanofiber yarn. This study suggests developing a new area in protein and polymer-blended nanofibrous materials such as composite nanofiber web and composite nanofiber yarn.

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