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Processing and Tensile Properties of Twisted Core-Shell Yarns Fabricated by Double Nozzle Electrospinning Device

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Abstract: In this study, a double nozzle electrospinning device consisting of a take-up/twister unit applied to develop core-shell structured nanofibrous yarns with potential applications in the production of functional textile materials. The process was performed by introducing a pre-electrospun core yarn into the electrical field between two oppositely charged nozzles, where nanofibers cover it by a certain arrangement owning to the twisting procedure. Herein, the main goal was to investigate how the core and shell structures influenced by process parameters, can contribute to the ultimate mechanical properties of the electrospun core-shell yarn. Accordingly, the same solution of nylon 66/formic acid was used for electrospinning of both core and shell nanofibers. The response surface methodology (RSM) was applied to study the effect of twisting rate, take-up speed, and twist amount of core yarn (called pre-twist) on the morphological and tensile properties of electrospun core-shell yarns. SEM images confirmed that the nanofibers were assembled surround the core with a certain angle to the axis to form a twisted core-shell yarn. The take-up speed caused considerable effects on the yarn diameter by changing the number of fibers formed in the triangle zone of the electrospinning process. The diameter of the core-shell yarns decreased significantly by increasing the twist rate and take-up speed. Improvements in tensile stress were generally realized at low twist levels and higher take-up speeds. The contribution of the core on the mechanical behavior of core-shell yarn was also considered in detail. The electrospun core-shell yarns showed superior (~37 %) max stress when using a core yarn with low pre-twist amounts.

Keywords: Electrospinning, Core-shell yarn, Twist, Tensile properties, RSM

Introduction

Technical textiles for industrial purposes are a growing sector which supports a broad scope of other industries. Technical textile materials are most extensively used in composites, filtration, sensors, protective clothing, and biomedical fields, where the function is the primary criterion [1,2]. In all these applications, precise performance requirements must be met. Functional textiles, due to their intrinsic features, can afford special needs for industrial applications to provide today's life expectancy. Several approaches are presented to yield functional textiles. The fiber coating onto textile structures, particularly fabrics, filaments, and yarns, is a flexible method to provide diverse functionalities [3-5]. A core-shell yarn is a kind of functional textile structures composed of a detachable core embedded in the center surrounded by fibers, which termed as the shell [5-7]. Cotton-spandex yarn is a famous example of core-shell yarn in the textile industry, which is consisted of a spandex filament as core coated by cotton staple fibers [8-10].

Compared to conventional synthetic or natural fibers, the production of ultrafine fibers with micro to nanoscale diameters provides materials with specific features [11,12]. Aiming to benefit the outstanding characteristics of nanofibers, the electrospinning method presents an efficient procedure for the coating of nanofibers onto the different substrates. Several research works have been performed to provide functional textiles by producing nanofiber coated materials [8,12-15]. As recommended in the presented work, the electrospinning also can be applied to produce continuous core-shell structured yarns via twisting the electrospun fibers on nanofibrous or conventionally pre-spun yarns. Dabirian *et al.* [16] developed an electrospinning set-up equipped with positive/negative charged nozzles and a metallic disk collector to coat nanofibers onto the metal wire. Despite the growing research works on this area, it is not yet clearly realized how the core and shell structure contribute to the mechanical properties of the core-shell yarn. Moreover, further improvements in their geometrical structure and physical-mechanical performance require more attempts for effective fiber depositing, arranging, and assembling into a yarn. The physical and mechanical properties of electrospun twisted core-shell yarns may be affected from the individual fiber features (e.g., morphology, diameter, crystallinity, molecular orientation), yarn geometry (fiber deposition and arrangement, the twist angle of the fibers, interaction and friction between the fibers in the yarn structure), core yarn structure, core to sheath proportion, etc. [8,17-19]. The presented research work is mainly aimed to evaluate the core yarn/shell nanofiber structuring effects on the ultimate morphological and tensile properties of resultant electrospun core-shell yarns.

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In this study, we have modified a mechanism using an electrospinning set-up applied in our previous works to electrospin highly ordered nanofibrous twisted core-shell yarn. The manufacturing process mainly consists of feeding a pre-electrospun nanofibrous yarn to the modified electrospinning unit, where nanofibers cover it by a suitable arrangement. The twisting procedure during the electrospinning enables the integrity between two different layers. Using the presented mechanism, it is also possible to apply different kinds of polymers for electrospinning, twisting and wrapping on the surface of the pre-electrospun yarn, conventionally spun yarn, as well as carbon-based or metallic wires. Due to the versatile and straightforward characteristics of this process, it is feasible to develop new types of composite nanofibrous materials with certain microstructures, different morphologies, and advanced functions for a variety of applications. The art of altering the process parameters enables us to innovate novel products with unique functions. Manufacturing parameters were changed to study the effects of the process variables of twist rate, take-up speed, and twist level of the core (called pre-twist) yarn on the morphological structure and tensile properties of the resultant nanofibrous core-shell yarn. Using the response surface methodology (RSM), an empirical relationship between these process variables and response variables of max stress, and Young's modulus was obtained.

It is necessary to mention that a common idea in core-shell composite yarns is to take advantages of two components in the core and shell layers, and add fillers or encapsulate nanoparticles, drug, bioactive agents, or other nanomaterials for giving special properties, i.e., antibacterial, biological, mechanical, conductivity, etc. However, in this work, the same polymer solutions used for electrospinning of both core and shell nanofibers. Because, in the current work, we have performed a feasibility study on the production of continuous twisted yarn with a core-shell structure via the electrospinning method, and we have focused on the study of important process parameters that influenced the mechanical and morphological structure of the core-shell yarns, which is rarely investigated. It is clear that that the features of the core and shell components have considerable effects on the final properties of the produced yarn. For this reason, to escape the inherent characteristic effects of the raw materials, a similar nylon 66/acid formic solution was prepared for the electrospinning of both core yarn and sheath nanofibers.

Experimental

Materials

Nylon 66 granules were supplied from Alyaf Co., Iran. Formic acid (98 %) was purchased from CHEMLAB-ANALYTICAL, Belgium.

Preparation of Polymer Solution

To produce electrospinning solution, a weighted amount of nylon 66 granules was dissolved in formic acid to reach 15 wt% concentration. The mixture was stirred at room temperature for at least 4 h to obtain a homogeneous solution. The viscosity and conductivity of the prepared solutions were determined using a MCR 301 Physical Rheometer (Anton Paar, USA), and an Orion 160 conductivity meter (USA), respectively. The 15 wt% nylon 66 solution in formic acid showed a viscosity of 0.2487 Pa·s and conductivity of 4.19 mS·cm^{-1}.

Electrospinning

Using the presented mechanism, the electrospinning of core-shell yarn was performed in two main steps, as follows:

(1) Electrospinning of the polymer solution via the ordinary process to produce nanofibrous yarn, which used as core yarn for the next step.

(2) Electrospinning of continuous twisted core-shell yarn using the modified set-up.

A schematic illustration of the electrospinning set-up used for producing nanofibrous core-shell yarns is presented in Figure 1. As explained in our previous works [19-21], for electrospinning of continuous twisted yarns, a double nozzle apparatus was utilized. The nozzles equipped with needles (22-gauge, ID=0.4 mm, OD=0.7 mm) placed against each other at a distance of 30 cm. Two syringe pumps were employed to feed the polymer solutions at a constant flow rate of 0.3 m*l* h^{-1}. The needles were oppositely charged using a DC high voltage power supply with an applied voltage of 13 kV. A conductive cylinder (6 cm diameter×30 cm length) was grounded at a distance of 2 cm from the center of the two needles. The electrospinning was started between the grounded cylinder and a convergence point in needles' center, and consequently, a triangle of fibers (called E-

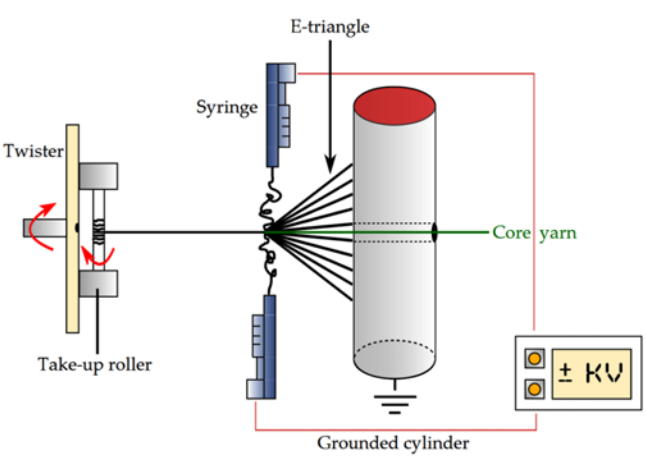

Figure 1. Schematic illustration of the electrospinning set-up used for the production of core-shell yarns.

triangle) was formed in this zone. The bundle formed at convergence point was twisted by rotating around its axis, and simultaneously, the twisted yarn was pulled by a take-up roller installed to the twister unit. The distance of the needles' center to the take-up/twister unit was set at 30 cm.

To produce a yarn with a core-shell structure, as shown in Figure 1, a hole was created in the middle of the grounded cylinder for feeding the core yarn. The fibers at the convergence point were collected on the fed core, which was then collected by the take-up roller. Simultaneously, the electrospun fibers were twisted by rotating the yarn around its axis and wrapped to around the core yarn. Using this set-up, the yarn fabrication process was continuous, and the electrospun nanofibers were arranged with an angle to the yarn axis (named twist angle) within the yarn structure. To evaluate the impact of take-up speed on the structural and tensile properties of electrospun yarns, the linear take-up speed was altered using a stepper motor. According to the required yarn mechanical performance, it is possible to give the desired level of the twist during this process. In this study, the twist effect was evaluated for both the single and core-shell yarns and, the twist rate was changed in two steps. Firstly, to realize the contribution of core in the mechanical behavior of core-shell yarn, the core was electrospun via the ordinary electrospinning process with three different twist amounts (called pre-twist) ranging from 1000 TPM (turns per meter) to 7000 TPM. Further, these diverse pre-electrospun yarns fed as core to produce core-shell yarn, and during the modified electrospinning process, the rotational speed of the twisting unit also changed in the range of 40 to 280 rpm to produce core-shell yarns with TPM of 1000 to 7000.

Table 1. Experimental ranges for process variables

Variable	Values of the levels	
	Low	High
Pre-twist amount of core yarn (TPM)*	1000	7000
Twist (TPM)	1000	7000
Take-up speed (m/min)	0.02	0.06

*To produce the core yarn, the linear take-up speed was adjusted at a constant value of 0.04 m/min.

Experimental Design

In the current work, a central composite design (CCD) was selected to analyze the influence of electrospinning processing parameters on the tensile properties of nanofibrous core-shell yarns using the response surface methodology (RSM) (Design Expert 8.0.7.0, Stat-Ease). In the preliminary tests, process conditions for the ordinary electrospinning of single yarn was determined by varying the concentration, flow rate, voltage, and distances. At the obtained effective ranges, the electrospinning of the uniform and bead-free fibers and yarn performed in a stable and continuous process, and without yarn rupture. Further, a set of preliminary experiments were carried out by changing the twist rate, take-up speed, and feeding rate of the pre-spun core yarn to determine the efficient experimental boundaries for continuous production of core-shell yarn without breakage of core yarn. The experimental ranges of process variables are presented in Table 1.

Within the ranges given for the pre-twist amount of core yarn, twist levels, and take-up speed, a series of 15 experiments were performed, and the results were statistically analyzed by the RSM. Max stress, and Young's modulus of electrospun core-shell yarns were considered as the response variables.

Table 2. RSM experimental design and measured results for max stress, Young's modulus, and diameter of electrospun core-shell yarns

RUN	Pre-twist amount of core yarn (TPM)	Twist level (TPM)	Take-up speed (m/min)	Max stress (cN/tex)	Young's modulus (cN/tex)	Average diameter of yarn (μm)
1	4000	7000	0.04	5.97	233.03	86.23
2	7000	1000	0.06	6.29	302.21	133.67
3	4000	4000	0.04	6.58	210.75	88.07
4	7000	7000	0.02	5.87	167.72	172.42
5	4000	4000	0.04	6.58	210.75	86.24
6	4000	4000	0.04	6.58	206.46	87.07
7	7000	4000	0.04	4.20	180.53	126.38
8	1000	7000	0.06	4.23	162.96	119.93
9	1000	4000	0.04	6.61	250.67	128.51
10	1000	1000	0.02	5.45	265.88	138.53
11	4000	4000	0.04	6.92	217.96	86.88
12	4000	4000	0.04	6.52	208.95	87.34
13	4000	4000	0.02	5.15	165.17	211.43
14	4000	1000	0.04	8.05	258.55	96.73
15	4000	4000	0.06	6.46	248.82	86.20

The experimental conditions and the mechanical properties of electrospun core-shell yarns are shown in Table 2.

The statistical significance of the model determined based on the p-value with a 95 % confidence level. The results were investigated entirely through analysis of variance (ANOVA). The regression correlation coefficient (R^2) and adjusted R^2 (R^2_{adj}) were considered to estimate the quality of the fit model with the observed response.

Characterization

The morphology of the electrospun yarns was studied by a scanning electron microscope (SEM; VEGA\\TESCAN-LMU) at an accelerating voltage of 15 kV. Before analysis, samples were coated with a thin layer of gold. Fiber and yarn diameters were measured from SEM images using Digimizer 4.1.1.0 software. Mechanical properties of the electrospun yarns were evaluated using a tensile tester (Kardotech Co. 2012 v3.0.1.7_i, Iran). The electrospun samples were randomly cut into pieces with a length of 50 cm and weighted to obtain the linear mass density (Tex) of the yarns. The max stress and Young's modulus were recorded in cN per tex (cN/tex). At least 20 pieces of every yarn sample were tested, and the average values were reported. In line with the previous research works, the applied gauge length and crosshead speed set to 100 mm and 50 mm min^{-1}, respectively.

Results and Discussion

As reported on our previous works, highly oriented nanofibrous deposits in the form of twisted yarns could be produced via a continuous electrospinning process. By applying a double-nozzle electrospinning set-up polarized with opposite charges, a triangle of electrospun fibers connected to the grounded cylinder created (Figure 1, E-triangle zone). The formed yarn at the vertex point of the triangle was twisted by rotation of the twister plate and simultaneously, pulled toward the take-up roller [19,21]. In this work, by modifying this set-up, core-shell yarns were prepared by feeding the pre-electrospun core yarn through the hole embedded in the middle of the grounded cylinder. The nanofibers at the convergence point were collected with the core yarn and twisted to different levels, and then pulled toward the take-up roller. To optimize the properties of yarns for special applications, the influence of processing parameters on the physical and mechanical performance of such nanofibrous structures was studied. Usually, changing the process variables would require many individual tests. Statistical approaches such as RSM can be employed to use a sequence of designed experiments and obtain an optimal response by estimating the different variables and their interactions simultaneously. Within the ranges are given for twisting level, take-up speed, and pre-twist amount of core yarn, a set of 15 experiments were performed, and the results were statistically analyzed. The detailed information of experimental design and measured results for the diameter, max stress, and Young's modulus of electrospun core-shell yarns are reported on Table 2. The analysis of variance results of the determined model for each response are summarized in Table 3. Values of p>F less than 0.05 approves that the applied model was significant at the 95 % confidence level. The lack-of-fit test states the variation of the data from the fitted model. A significant lack of fit ($p<0.05$) indicates that the model does not fit. As presented in Table 3, for both responses, the lack-of-fit was not statistically significant and the probability of lack of fit values were higher than 0.05 that approved the adaptation of the models. The values of R^2 and R^2_{adj} approached 1, which also confirm that the proposed models for both responses fit well to the experimental data. The detailed analysis of the models via ANOVA results are reported in Tables S1 and S2. The model equations in terms of coded factors obtained from the analysis of the experimental data are presented in Table S3.

Table 3. Analysis of variance results for the response parameters

Response	F value	p > F	Lack of fit	R^2	R^2_{adj}
Max stress	37.87	0.0005	0.1170	0.9855	0.9595
Young's modulus	61.02	< 0.0001	0.1672	0.9879	0.9717

Morphology

Using the presented electrospinning mechanism, the number of nanofibers formed in the E-triangle zone, twist rate, and take-up speed are practical factors to control the fibers and yarn diameter and yarn count. Also, the experimental considerations have been approved that the geometry of the E-triangle varies by the twisting rate and affects the tension on the fibers, and thus the structure and physical-mechanical properties of the produced yarn [19,22].

Figure 2 shows a series of SEM images captured from single and core-shell yarns electrospun at different twist rates. The morphological study demonstrated that uniform yarns composed of smooth and bead-free fibers were formed. It can be seen from the images with higher magnification, most of the nanofibers are oriented and arranged along a specified direction in an angle to the yarn axis. The visual comparison of SEM images indicated that the twist angle increased by twist rate (see Figures 2(a) and 2(b)).

From the SEM images, the diameter values of yarns and fibers in the yarn structure were determined using Digimizer software. The measured diameter for each yarn sample electrospun according to the RSM designed conditions are described in Table 2. Depending on the twist rate, and take-up speed, the diameter of electrospun core-shell yarns varied from 86 μm to 211 μm (Table 2). The average values of

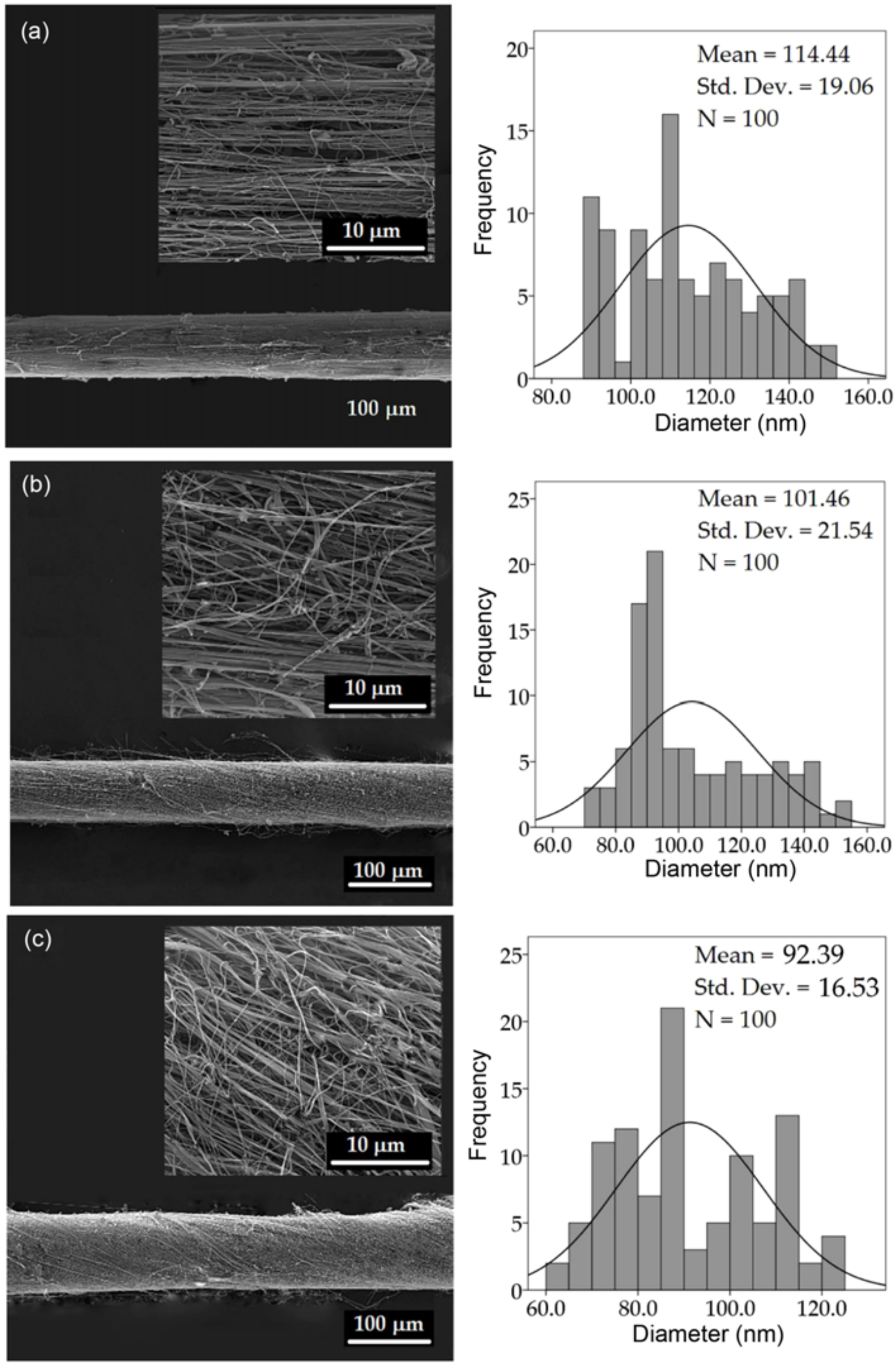

Figure 2. Examples of SEM images of the electrospun yarns prepared at a constant take-up speed of 0.04 m/min and their corresponding diameter distribution of nanofibers; (a) single yarn with twist amount of 1000 TPM, (b) single yarn with twist amount of 4000, and (c) core-shell yarn with twist amount of 4000 TPM, having a core with pre-twist amount of 1000 TPM.

diameter measurements at a constant take-up speed of 0.04 m/min and different twist levels are summarized in Table 4. The results indicated that the twist rate had a significant influence on the average diameter of the electrospun fibers and yarns. By increasing the twist rate, the average diameter of the fibers in the yarn structure decreased. During electrospinning process to produce core yarn at a constant feed rate of 0.04 m/min, by increasing the twist level from 1000 TPM to 7000 TPM, the average diameter of yarns slightly decreased from 90 μm to 79 μm

Table 4. Average diameter of electrospun yarns and fibers in the yarn structure at a take-up speed of 0.04 m/min

Sample	Twist level (TPM)		Yarn count	Average diameter±S.D.	
	Core yarn	Core-shell yarn	(tex)	Yarn (μm)	Fibers in the yarn structure (nm)
Single yarn	1000	-	1.71±0.22	89.94±4.26	114.44±19.06
	4000	-	1.28±0.14	82.06±4.13	101.46±21.54
	7000	-	1.60±0.19	78.64±2.11	84.85±13.78
Core-Shell yarn	1000	4000	3.06±0.43	128.51±2.03	92.39±16.53
	4000	1000	2.93±0.27	96.73±3.55	110.85±21.33
		4000	3.06±0.21	88.07±5.40	97.67±18.44
		7000	3.16±0.37	86.23±2.53	78.84±13.66
	7000	4000	3.78±0.66	126.38±3.58	86.05±16.58

(~12 %). This decrease may be due to the more tension applied to the fibers during the twisting procedure and the denser structure of the yarn at the higher twist rates [19,22,23]. Likewise, compared to the samples produced with a twist amount of 1000 TPM (114 nm), the fiber diameter in the yarn structure reduced 25 % compared to the twist of 7000 TPM (85 nm) (Table 4). A similar decrease in the average diameter of fibers and yarn at higher twist rates was observed in the electrospun yarns with the core-shell structure. For example, as presented in Table 4, at constant take-up speed of 0.04 m/min and 4000 TPM pre-twist amount of core yarn, the increasing of twist rate caused a decrease of about 12 % in yarn diameter. Also, the average diameter of nanofibers in the yarn body with a twist amount of 1000 TPM was 111 nm, and when the twist rate increased, the values reached 79 nm at a twist level of 7000 TPM and showed about 29 % decrease.

In the presented process, it is found that the take-up speed was also an important process parameter affecting the morphological structure of the produced yarns. Figure 3 shows the effect of take-up speed on the diameter of the electrospun core-shell yarns. SEM images of the core (pre-twist of 4000 TPM)-shell yarns prepared at a constant twist level of 4000 TPM also are exhibited in Figure 3. The images clearly show that the take-up speed caused considerable effects on the yarn diameter by changing the number of fibers formed in the E-triangle zone. Yarns collected at a take-up speed of 0.06 m/min had a diameter of 88.07±5.40 μm. At lower take-up speed, the accumulated fibers in the E-triangle zone and then in the yarn structure increased. Thereby, the diameter of the core-shell yarns showed a significant increase of 139 % at a take-up speed of 0.02 m/min. Following that, the influence of take-up speed on the linear mass density (tex) of yarns was also studied. The data analysis revealed that at a constant value of twist rate, the linear mass density of yarns significantly changed with take-up speed. By increasing the take-up speed, due to the less fibers deposited in E-triangle zone, the weight mass per length of yarn decreased. For this reason, the linear density of the core-shell yarns showed a decrease around 45 % from 4.72 tex at a take-up speed of 0.02 m/min to 2.62 tex at a higher take-up speed of 0.06 m/min.

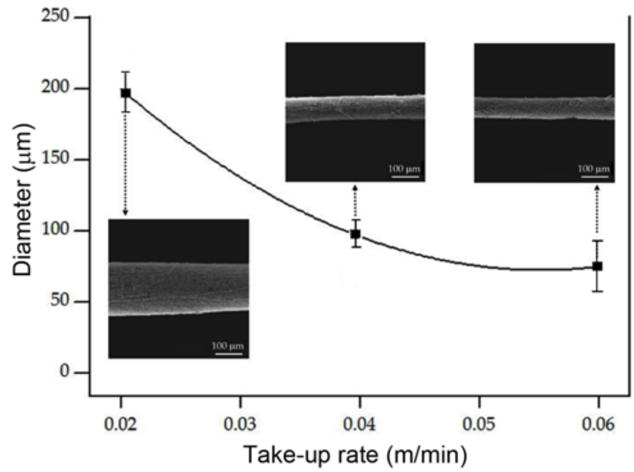

Figure 3. Effect of take-up speed on the diameter of electrospun core (pre-twist of 4000 TPM)-shell yarns, at a constant twist level of 4000 TPM.

Tensile Properties

Characterization of the mechanical properties of nanofibrous core-shell yarns is the main issue that plays an important role in perceiving the potential of them in the end-use application. Fiber arrangement in the yarn structure influences the interaction between the fibers and consequently alters the mechanical behavior of the yarns. Moreover, stretching and drawing applied during the electrospinning and twisting process may lead to higher molecular chain orientation and further arrangement of them [19,24,25]. Accordingly, mechanical features of samples produced with the modified electrospinning method were investigated to determine the effects of the process variables of twist rate, take-up speed, and pre-twist level of the core yarn on the mechanical performance of the electrospun core-shell yarns.

The ANOVA results revealed that the p-values for independent process variables of twist rate, take-up speed,

Table 5. Tensile properties of single electrospun yarns prepared at different twist levels

Yarn twist (TPM)	Max stress (cN/tex)	Young's modulus (cN/tex)
1000	6.46±0.66	324.14±33.23
4000	8.53±1.75	397.95±35.74
7000	7.18±1.06	356.55±32.62

and pre-twist amount of core yarn, were smaller than 0.05 ($p<0.05$, the corresponding confidence level is 95 %) and thus had significant effects on the max stress, and Young's modulus of electrospun core-shell yarns (see Table 3 and Tables S1 and S2). Among that, the twist was an important factor which had different effects on the mechanical behavior of yarns (see Table S4). The twisting procedure may improve the mechanical properties by enhancing lateral interactions and frictional forces between fibers within the yarn structure and hindering fiber slippage. Besides, twist condenses the yarn and improves its structure uniformity. Diversely, at higher twist rates, due to increasing the angle of fiber deposition to the yarn axis, a decline in tensile strength may be observed [22,25-27].

More detailed information about the effect of core structure on the mechanical properties of core-shell yarn can be achieved by comparing the features of the core-shell yarn and the pre-electrospun single yarn that used as the core in the further process. The mechanical characteristics of single electrospun yarns are presented in Table 5. Twisting the electrospun fibers up to a level of 4000 TPM, caused an increase up to 32 % in max stress and up to 23 % in Young's modulus. The higher stress can be described by the enhanced cohesion and frictional forces between fibers upon twisting. However, by further increase of the twist up to 7000 TPM, a small decrease observed for both the max stress and Young's modulus (Table 5).

Statistical results (Tables S1 and S2) showed that the effect of core yarn pre-twist was significantly meaningful on the mechanical properties of the core-shell yarn. Figures 4 and 5 demonstrate the max stress and Young's modulus of the core-shell yarns collected at constant take-up speed of 0.04 m/min, with three different core yarns. Tensile test results revealed that the max stress of core-shell yarns decreased as core yarn pre-twist increased. For example, at a constant twist level of 4000 TPM, electrospun core-shell yarns showed superior max stress of 6.61 cN/tex and 6.58 cN/tex when using a core yarn with low pre-twist amounts of 1000 TPM and 4000 TPM, respectively. While around the 37 % decrease in core-shell yarn max stress was evident in the presence of core yarn with a pre-twist amount of 7000 TPM (4.2 cN/tex) (Figure 4). During the process to electrospin core-shell yarn, the secondary twist that enters to the core yarn with a low pre-twist amount may increase the entanglement and cohesion between fibers in both core and shell layers of the yarn. Besides, the secondary twisting allows the distribution of tension between the fibers of two layers. Also, this twist compresses as-spun fibers (core fibers) to form a yarn in a monolith structure and improves bundle uniformity. For these reasons, electrospun core-shell yarns showed superior (~37 %) max stress when using a core yarn with low pre-twist amounts. During the process for producing core-shell yarn, the sheath fibers may slip along the core yarn when applying core yarn with the high pre-twist amount. In addition, as reported in Table 5, yarn with the pre-twist amount of 7000 TPM showed a lower value of max stress (6.18 cN/tex) and this property might then be transferred to the core-shell yarn. This can be another reason for low strength of the core-shell yarn having a core yarn

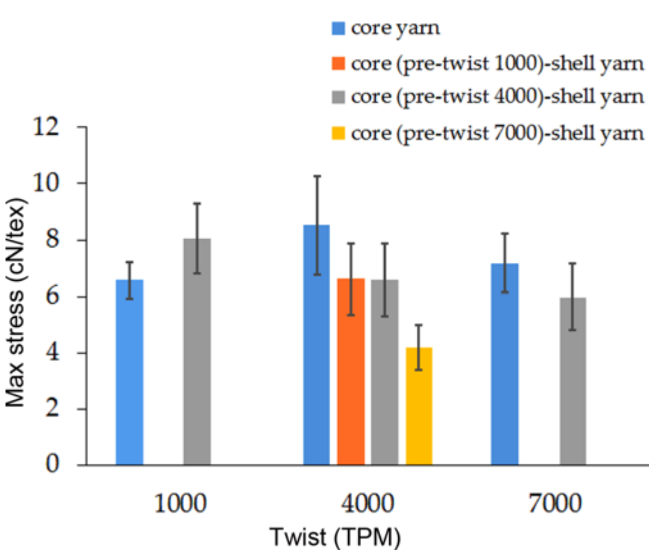

Figure 4. Effect of twist amount on the max stress of electrospun yarns at the take-up speed of 0.04 m/min.

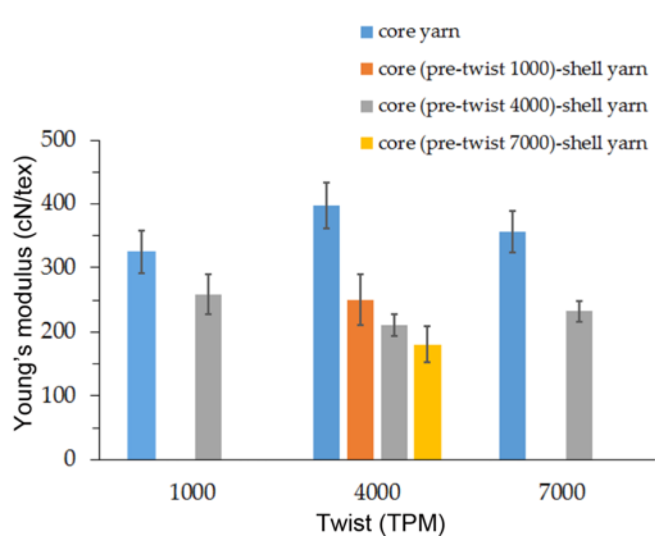

Figure 5. Effect of twist amount on the Young's modulus of electrospun yarns at the take-up speed of 0.04 m/min.

with the highest pre-twist amount of 7000 TPM. Consequently, the core-shell yarn with the highest max stress could be achieved on the twist level of 1000 TPM and made of a core yarn with the pre-twist amount of 4000 TPM.

Likewise, regarding Young's modulus, a decreasing trend was observed versus the pre-twisting rate of the core yarn. At the constant twist amount of the 4000 TPM and take-up speed of 0.04 m/min, the increasing of core yarn pre-twist from 1000 TPM to 7000 TPM resulted in a significant decrease (250.67±39.20 cN/tex; -28 %) in Young's modulus of the core-shell yarns.

Tensile properties of electrospun core-shell yarns for the set of the selected twist levels (1000 TPM, 4000 TPM, and 7000 TPM), and take-up speeds (0.02 m/min, 0.04 m/min, and 0.06 m/min) were considered. The interactive effects of twist level and take-up speed found to have the most significant contribution to explain the mechanical behavior of electrospun core-shell yarns (Table S4). Figure 6(a, b) shows the effects of the twist level and take-up speed on the max stress of the electrospun core-shell yarns when using a core yarn with the constant pre-twist amount of 4000 TPM.

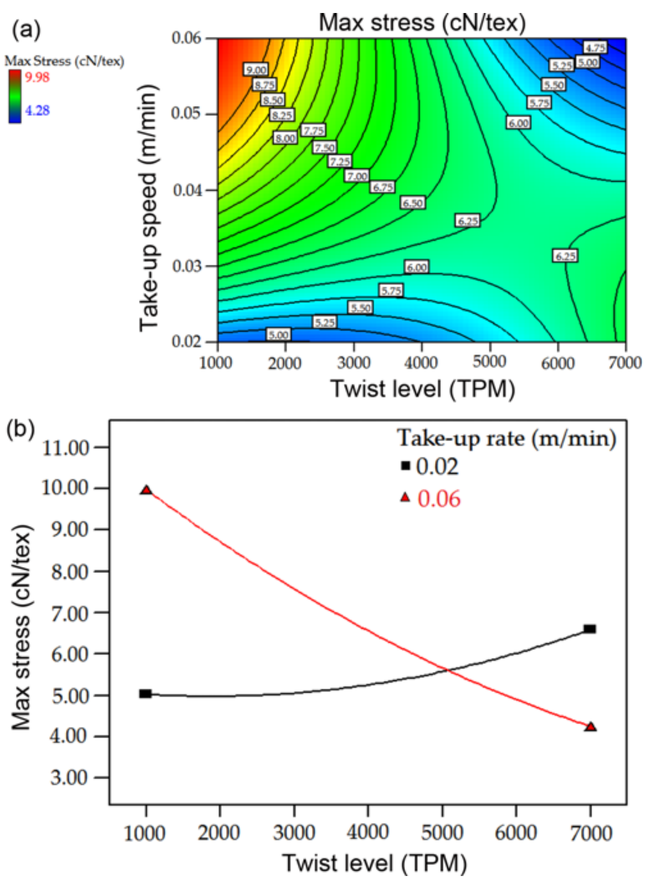

Figure 6. Contour plot (a) and interaction plot (b) for max stress of electrospun core-shell yarns versus independent process variables of twist level and take-up speed when using core yarn with the pre-twist amount of 4000 TPM.

It is obvious in Figure 6, the max stress showed different behavior on the settings of both twist level and take-up speed. The trend lines reveal that lower twist level and high take-up speed are the optimum conditions to gain maximum stress values. Within the twist range up to 5000 TPM, the samples collected at a take-up speed of 0.06 m/min, exhibited higher max stress (Figure 6(a)). Due to a greater drawing of the fibers and increasing tension at higher take-up speeds, molecular chains likely arrange longitudinally to the yarn axis, resulting in the yarn with higher stress values. However, at high twist amounts of 7000 TPM and the take-up speed of 0.06 m/min, a decrease in max stress was observed. These findings confirmed in Figure 6(b). At the take-up speed of 0.02 m/min, the max stress of core-shell yarns enhanced by increasing the twisting rate. This increase was more evident at the twist levels higher than 5000 TPM. At low take-up speed, the accumulated fibers in the E-triangle increase, and sheath fibers with more layers would be formed, leading to better cohesion. These layers, because of their spiral shape caused by twisting, generate radial pressure on the core yarn and thereby restrict slippage. Moreover, at low take-up speed, there is more time for fibers to adhere and form sheath layers and place them into the yarn structure upon the twisting. Thus, this is expected that by increasing the twist rate at a constant take-up speed of 0.02 m/min, the max stress of the core-shell yarns increases.

Similar to the max stress, the changes in Young's modulus of electrospun core-shell yarns also considerably depend on the settings of the twist level and take-up speed. Figure 7 shows the variation of Young's modulus versus twist and take-up speed when employing a core yarn with the pre-twist amount of 4000 TPM. It is clear that the maximum values of Young's modulus achieved at higher take-up speeds and lower twist levels. But, in contrast to the max stress data shown in Figure 6, the lowest Young's modulus values of the core-shell yarns did not appear in the contour

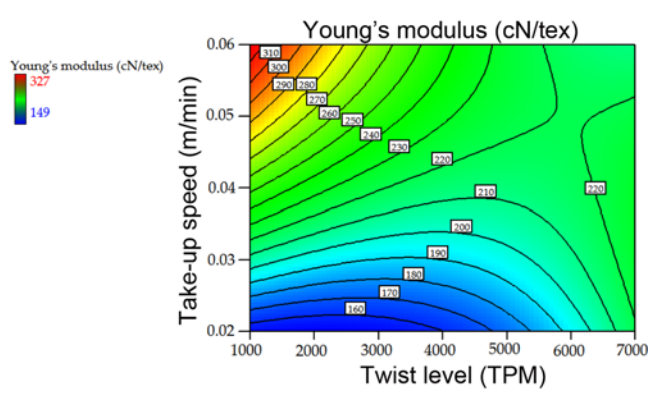

Figure 7. Contour plot for Young's modulus of electrospun core-shell yarns versus independent process variables of twist level and take-up speed when using core yarn with the pre-twist amount of 4000 TPM.

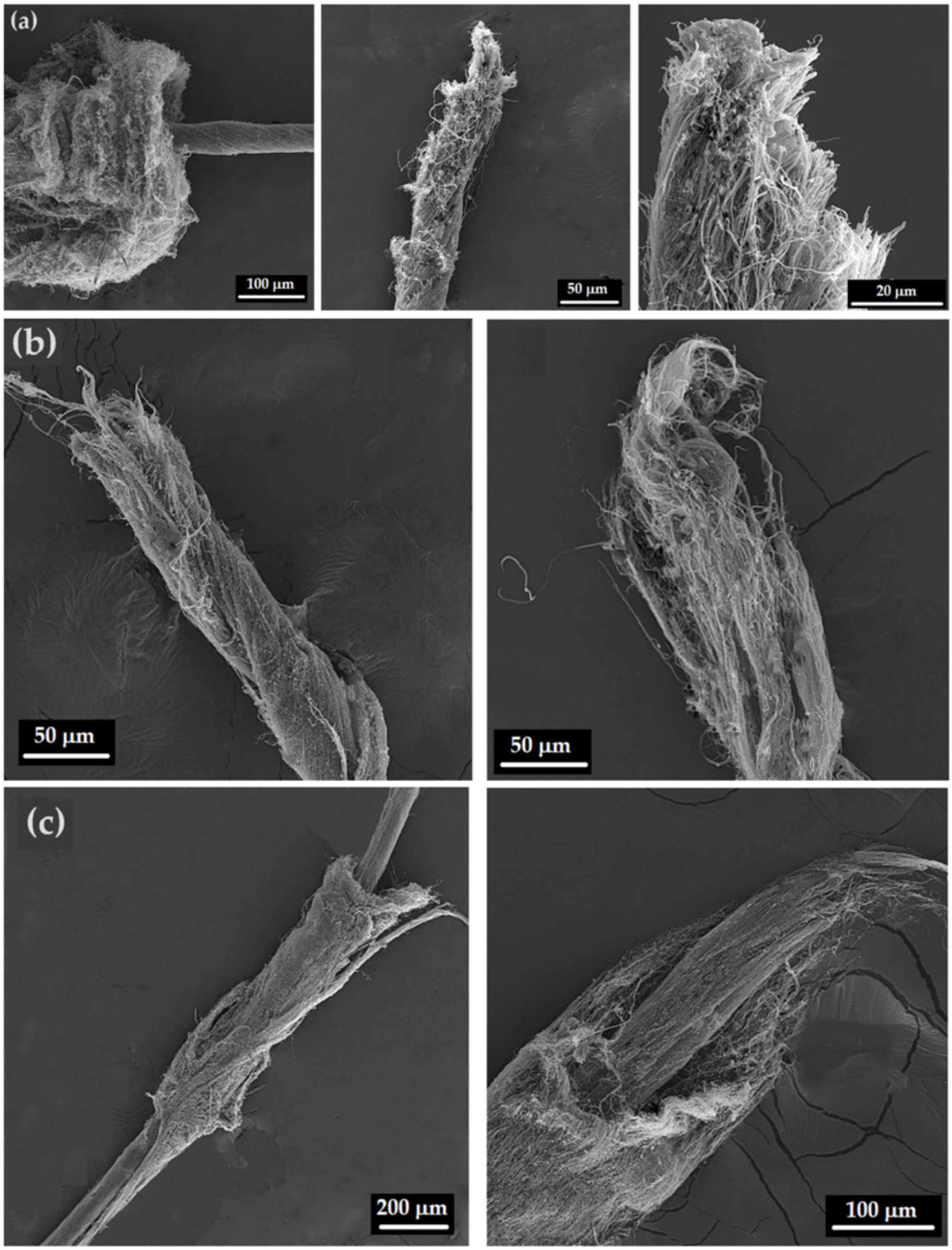

Figure 8. SEM images of the end failure of electrospun core-shell yarns at different processing conditions; (a) twist amount of 4000 TPM, and core yarn with the pre-twist amount of 4000 TPM, (b) twist amount of 7000 TPM, and core yarn with the pre-twist amount of 1000 TPM, and (c) twist amount of 1000 TPM, and core yarn with the pre-twist amount of 7000 TPM. In all samples, linear take-up speed was set to 0.06 m/min.

lines of the highest twist levels at the higher take-up speeds.

The failure of electrospun core-shell yarns was studied using scanning electron microscopy, and the example images of the rupture point of electrospun core-shell yarns prepared at different conditions are presented in Figure 8. The samples for SEM test are prepared from two steps of partial (shell failure) and total (both core and shell failure) rupture during tensile testing. The presented images confirm the core-shell structure of the electrospun yarns. The images also clearly show buckling, plastic deformation, and the formation of multiple necks.

Conclusion

The electrospinning technique was employed to manufacture continuous core-shell structured yarns via twisting the nanofibers on a pre-electrospun yarn. The process variables of twist, take-up speed, and pre-twist amount of core yarn were considered to evaluate the contribution of core and shell structure on the tensile properties of the core-shell yarn. The response surface methodology (RSM) was used to investigate process parameters systematically. The morphological study from the SEM images demonstrated that depending on the twist rate, and take-up speed, uniform yarns with diameters between 86 and 211 μm composed of smooth and bead-free fibers were formed. By increasing the twist rate, the mean diameter of the fibers in the yarn structure decreased due to higher tension on the fibers during the twisting procedure. Subsequently, the diameter of the corresponding yarns also reduced. The take-up speed caused a significant decrease in the yarn diameter by decreasing the number of the fibers accumulated in the E-triangle zone. Tensile test results revealed that the max stress and Young's modulus of core-shell yarns decreased as core yarn pre-twist increased. At the constant twist of 4000 TPM and 0.04 m/min take-up speed, the increasing of core yarn pre-twist from 1000 to 7000 TPM resulted in a significant decrease of around 37 % and 28 % in max stress and Young's modulus of the core-shell yarns, respectively. The interactive effects of twist and take-up speed found to have the most considerable contribution to the mechanical behavior of electrospun core-shell yarns. Lower twist level and high take-up speed were the optimal conditions to gain maximum strength and Young's modulus values. The presented mechanism is a facile and flexible process to develop new types of composite nanofibrous materials with specified structures, distinct morphologies, and novel functions for a variety of applications.

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