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Electrospinning of continuous poly (L-lactide) yarns: Effect of twist on the morphology, thermal properties and mechanical behavior

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ABSTRACT

Electrospinning PLLA solutions from two oppositely charged nozzles gives a triangle of fibers, also called E-triangle, that assemble into yarns at the convergence point. The formed yarn at the E-triangle was taken up by a unit comprising a take up roller and coupled twister plate, which twist rate can be varied. At all twist rates, uniform and smooth fibers without any beads were formed. The apex angle of the deposited fibers at the E-triangle was larger at higher twist rates. By increasing the twist rate from 80 rpm to 320 rpm the orientation angle of fibers in the yarn changes from 18.8° to 41.5°. Increasing the twist rate revealed a higher polymer crystallinity likely due to the polymer orientation by the applied tension to the fibers. The ultimate strength and modulus of electrospun yarns were higher when prepared at higher twist rates. However, at the highest twist rates, the strength and modulus of electrospun yarns leveled off and even decreased slightly. The results revealed that the mechanical properties not only depend on the polymer crystallinity but also on the alignment of the fibers in the yarn and the angle at which they were deposited. These biodegradable materials are promising materials to be used in a wide range of applications where environmentally friendly products are required.

1. Introduction

Textiles are and remain an important part of the world manufacturing industry. Apart from traditional applications of textile materials in clothes and fabrics, they lately have been widely applied in composites, filtration devices, sensors, and in the biomedical field. More recently the production of synthetic fibers with nano- to micro sized diameters provides materials with distinct characteristics compared to conventional synthetic or natural fibers (Barani, 2014; Su et al., 2013; Zhou and Gong, 2008).

Electrospinning is an adaptive and simple method to produce micro or nanofibers. The formation of nanofibers through electrospinning process is based on the uniaxial stretching of the viscoelastic solution or melt by applying electrostatic forces (Abbasipour and Khajavi, 2013; Penchev et al., 2010; Shuakat and Lin, 2014; Su et al., 2013). Nanofibers with outstanding mechanical and physical characteristics have the potential to significantly improve textile technology and opens potential new applications (Ali et al., 2011a; Barani, 2014; Shuakat and Lin, 2014; Su et al., 2013).

In electrospinning of polymer solutions, due to the helical motion and instability of the jet, fibers are generally deposited randomly and form a nonwoven structure, which is acceptable in applications such as filtration, composite reinforcement and protective textiles. This method was also used for manufacturing non-wovens to be applied in the biomedical field as e.g. tissue engineering scaffolds, wound dressings and drug delivery materials (J.-X. He et al., 2013; Su et al., 2013; Wee-Eong et al., 2011).

For many of the textile based applications, like weaving and knitting, the electrospun fibers preferably are produced as continuous aligned fiber bundles or yarns (Ali et al., 2011b; J. He et al., 2013; Paneva et al., 2010; Wu and Qin, 2013; Zhou and Gong, 2008). Compared to non-woven mats the lateral interaction and friction between nanofibers in yarns assists in improving their mechanical properties. It can be expected that yarns composed of assembled nanofibers will find wider applications, not only in traditional textiles but also in reinforced materials and medical textiles (Abbasipour and Khajavi, 2013; Ali et al., 2011b; J.-X. He et al., 2013; Maleki et al., 2016, 2015, 2013; Semnani Rahbar et al., 2016; Shuakat and Lin, 2014; Smit et al., 2005; Su et al., 2013; Tian et al., 2013; Zhou et al., 2012).

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In recent years, various methods have been developed to prepare electrospun nanofiber yarns by manipulating the electric field or using mechanical collection devices (Abbasipour and Khajavi, 2013; Bazbouz and Stylios, 2008; Dabirian and Hosseini, 2009; Shuakat and Lin, 2014; Teo et al., 2007; Tian et al., 2013; Wang et al., 2008). In this work, to produce continuous twisted yarns, an electrospinning setup consisting of two oppositely charged nozzles was used. Using a take-up roller the fibrous yarns could be produced in a continuous way (Maleki et al., 2016, 2015, 2013; Semnani Rahbar et al., 2016). Combining the take-up roller with a twister unit in the production of yarns, twist is subjected to the fiber bundle. Because twist can increase lateral interactions and frictional forces between fibers within the yarn the mechanical properties may improve. Moreover, twist compresses the yarn and improves bundle uniformity (Abbasipour and Khajavi, 2013; J. He et al., 2013; Maleki et al., 2016, 2013; Shuakat and Lin, 2014).

Research on yarns mainly focused on the preparation, morphology, and structure of nanofiber yarns. The influence of twist rate on the mechanical properties has been less studied. In this work, the preparation and properties of twisted yarns from electrospun poly(L-lactide) (PLLA) fibers by a continuous process is presented. Poly(L-lactide) (PLLA) is a biodegradable and biocompatible material, which has attracted attention for biomedical applications (Toncheva et al., 2011). The effect of twist rate on the assembly process of fibers into a yarn and the resulting yarn mechanical and physical properties were described.

2. Experimental

2.1. Materials

Poly(L-lactide) (PLLA) with an inherent viscosity of 2.51 dL/g was purchased from Purac Biomaterials, The Netherlands. 2,2,2-Trifluoroethanol (TFE) was obtained from Merck and used without purification. To obtain homogeneous electrospinnable solutions, PLLA was dissolved in TFE at a concentration of 7 wt% and stirred gently at 40 °C for at least 24 h.

2.2. Preparation of electrospun yarns

As described in previous studies, to produce continuous twisted fiber yarns, an electrospinning setup consisting of two oppositely charged nozzles was used (Maleki et al., 2016, 2015, 2013; Semnani Rahbar et al., 2016) (Fig. 1). This setup consists of two digitally controlled syringe pumps (TOP-5300, Japan) and flat-tipped needles (22-gauge, ID 50.4 mm, OD 50.7 mm). Electrospinning was conducted at a constant feed rate of 0.3 mL/h. Two nozzles were horizontally positioned oppositely of each other at a distance of 30 cm. A DC high voltage-power supply was used to charge the needles at 13.5 kV but with opposite polarization. A grounded aluminum cylinder, 6 cm in diameter and 30 cm in length, was placed vertically at a distance of 2 cm from the center of the two nozzles. The take-up/twister unit consists of a take-up roller and a rotating plate for twisting the yarn was located at a distance of 30 cm from the syringe needles. The twister rotation speed can be varied up to 440 rpm. To produce the yarn, electrospinning was started from the two nozzles. At the convergence point, a triangle of fibers was formed, called the "electrospinning triangle (E-triangle)." The fibers were twisted by rotating the yarn around its axis and the yarn was taken up by the roller installed to the twister plate with a linear take up speed of 2.4 m/h. In order to investigate the effect of twist rate on the structural and mechanical properties of electrospun yarns, the rotational rate of the twister plate was changed to values of 80, 160, 240 and 320 rpm.

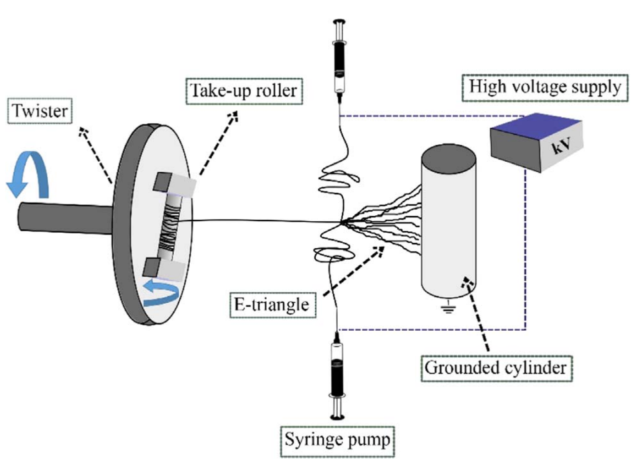

Fig. 1. Schematic illustration of the electrospinning setup to produce continuous twisted yarns.

2.3. Characterization

2.3.1. Morphology

A scanning electron microscope (SEM; XL 30, Philips) was used to investigate the effect of twist rate on the morphology of the electrospun fibers and yarns. Prior to analysis, samples were sputtered with a thin layer of gold and an accelerating voltage of 25 kV was applied. Based on the SEM images, the average diameter of fibers and yarns was determined using Digimizer.4.1.1.0 software.

2.3.2. Differential scanning calorimetry (DSC)

The thermal properties of the PLLA electrospun yarns were determined by differential scanning calorimetry using a TA Instrument DSC SW 9.01 calorimeter. Samples were heated from 25 to 250 °C at a scanning rate of 10 °C/min under a nitrogen atmosphere. From the DSC thermograms the glass transition temperature (T_g), melting temperature (T_m) and cold crystallization temperature (T_{cc}) were determined. The degree of crystallinity (% χ) of the PLLA was calculated from the melting enthalpy and cold crystallization enthalpy according Eq. (1):

$$\chi_c(\%) = \frac{\Delta H_m - \Delta H_{cc}}{\Delta H_m^0} \times 100 \tag{1}$$

where, ΔH_m is the melting enthalpy, ΔH_{cc} is the cold crystallization enthalpy and $\Delta H°_m$ is the melting enthalpy of 100% crystalline PLLA (93.7 J/g) (Asran et al., 2010; Maleki et al., 2013).

2.3.3. Mechanical properties

Mechanical properties of the electrospun yarns were measured using a tensile tester (Instron Elima EMT-3050). The twisted fibrous yarns were cut at random into pieces with a length of 50 cm and then weighted to obtain the linear mass density (Tex) of the yarns. The mechanical data were reported in cN per Tex (cN/Tex). The applied gauge length and cross head speed were 20 mm and 10 mm/min, respectively.

3. Results and discussion

3.1. Effect of twist rate on the geometry of the E-triangle

Using an electrospinning set up with two oppositely charged nozzles (Fig. 1), a triangle of electrospun fibers forms in the area between a neutral cylindrical surface and convergence point. At this so-called "E-triangle" the oppositely charged fibers assemble into a yarn that can be drawn to a take-up roller with twister unit. Experimental observations showed that by varying the twist rate, the geometry of the E-triangle changes. The geometry affects the tension on the fibers and thus the structure of the produced yarn.

Characteristic images of the formed E-triangle during electrospin-

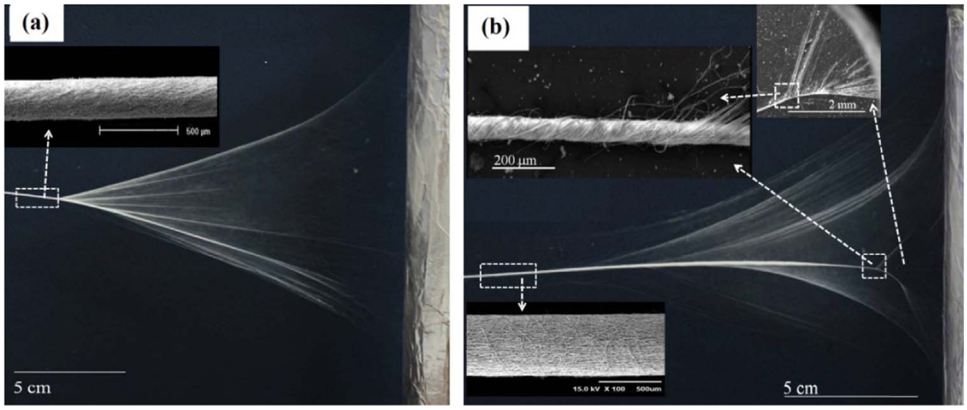

Fig. 2. Images of the E-triangle formed in the electrospinning of a PLLA/TFE solution and fiber assembly into yarns. Twist rates were 80 rpm (a) and 320 rpm (b).

Table 1
Geometry of the E-triangle at different twist rates.

Twist rate (rpm)	Height (cm)	Base (cm)	α (°)
80	19.6 ± 1.0	15.2 ± 0.6	18.3 ± 1.6
160	12.4 ± 0.6	11.9 ± 0.5	23.4 ± 2.6
240	10.7 ± 0.8	5.7 ± 0.5	33.1 ± 1.2
320	8.5 ± 0.3	3.7 ± 0.6	50.8 ± 2.4

ning of a PLLA-trifluoroethanol solution at twist rates of 80 and 320 rpm are presented in Fig. 2. The height and base of the E-triangle and angle (α) at the convergence point axis were determined using Digimizer.4.1.1.0 software (Table 1).

The formed E-triangle was approximately symmetric at all twist rates. In addition, when keeping the electrospinning process parameters constant, an increase in the twist rate places the fiber convergence point closer to the neutral cylinder. At the convergence point the twist angle α increased with twist rate while the height and the base of the triangle decreased (Table 1). The considerable reduction of E-triangle dimensions at twist rates of 240 and 320 rpm leads to a limited space for fiber deposition at the convergence point and thus for yarn formation. As a result, at high twist rates and due to the high amount of produced fibers, electrospun “out of control” fibers are depositing and wrapping on the already formed yarn (Fig. 2(b)). This phenomenon leads to an increase in yarn diameter at twist rates of 240 and 320 rpm.

3.2. Effect of twist rate on fiber morphology in the E-triangle zone

SEM images of collected electrospun fibers from the E-triangle zone and their corresponding diameter distribution are presented in Fig. 3. From these images the diameter distribution of the fibers was measured using Digimizer software and the average values of 100 measurements are reported in Table 2. At all twist rates, uniform and smooth fibers were formed without any beads. The results revealed that the twist rate had a significant effect on the average diameter of the fibers in the E-triangle zone. By increasing the twist rate from 80 to 320 rpm, the average fiber diameter decreased from 810 nm to 530 nm. This decrease is mainly due to the high tension on the fibers at the high twist rate applied. Moreover, at a high twist rate of 320 rpm fibers with a narrow diameter distribution were obtained.

3.3. Effect of twist rate on morphology of electrospun yarns

SEM images of electrospun PLLA yarns prepared at different twist raters are presented in Fig. 4. Higher magnifications show that the fibers are oriented and arranged in an angle to the yarn axis called twist angle. The results showed that by increasing the twist rate from 80 rpm to 320 rpm the twist angle increased from 18.8° to 41.5° (Table 3). Compared to fibers in the E-triangle zone the fiber diameter in the yarn is somewhat smaller caused by the rolling and twisting (Table 3).

Despite the tension exposed to the fibers upon electrospinning and subsequent twisting and rolling, the yarn diameter increased by increasing the twist rate. As mentioned above, by increasing the twist rate, the dimensions of the E-triangle zone become smaller, and part of the electrospun fibers wrap the formed yarns. This phenomenon leads to form bulky yarns at high twist rates. Actually, the electrospun yarns produced at high twist rates of 240 and 320 rpm can be considered to be composed of an inner layer with deposited fibers assembled on it. The difference between these two layers is depicted in Fig. 4c and d. For example, in Fig. 4d, image (2) is related to the inner layer of the yarn which is formed from twisting the fibers in the E-triangle zone. In this part of the yarn, the fibers are densely packed with high orientation. Image (1) shows the surface layer of the yarn which is formed by wrapping of out of control fibers. The fibers in this outer layer have a lower orientation and are less densely packed.

3.4. Effect of twist rate on fiber crystallinity

The crystallinity of the PLLA electrospun yarns was determined for it will largely influence the mechanical properties and degradation rate (Richard-Lacroix and Pellerin, 2013). Using Differential Scanning Calorimetric (DSC) the thermal properties of electrospun PLLA yarns prepared at different twist rates were determined (Fig. 5).

All thermograms of electrospun yarns revealed similar trends. A glass transition is followed by an endothermic peak at around 64–65 °C. A similar transition was observed previously and was ascribed to enthalpy relaxation and release of remaining stress resulting from the rapid formation of the fibers during the electrospinning process (Branciforti et al., 2009; Monticelli et al., 2013). Cold crystallization around 80–86 °C was observed for all electrospun yarns. The observed cold crystallization indicates that the fast solvent evaporation during fiber formation in the electrospinning process results in a partial alignment of polymer chains thereby hampering PLLA crystallization (Monticelli et al., 2013; Tan and Lim, 2006; Zhou et al., 2006). A melt transition is observed at 178–180 °C. Due to increasing tension at higher twist rates molecular chains likely arrange longitudinally to the yarn axis and the crystallinity, as calculated by Eq. (1), increases at higher twist rates (Table 4).

3.5. Effect of twist rate on the mechanical properties of the electrospun yarns

The electrospinning process combined with the take up roller and twist unit produces filament type fibers assembled into yarns. As indicated above the orientation of polymer molecules in the fibers likely affects the crystallinity. Moreover, fiber orientation in the yarn

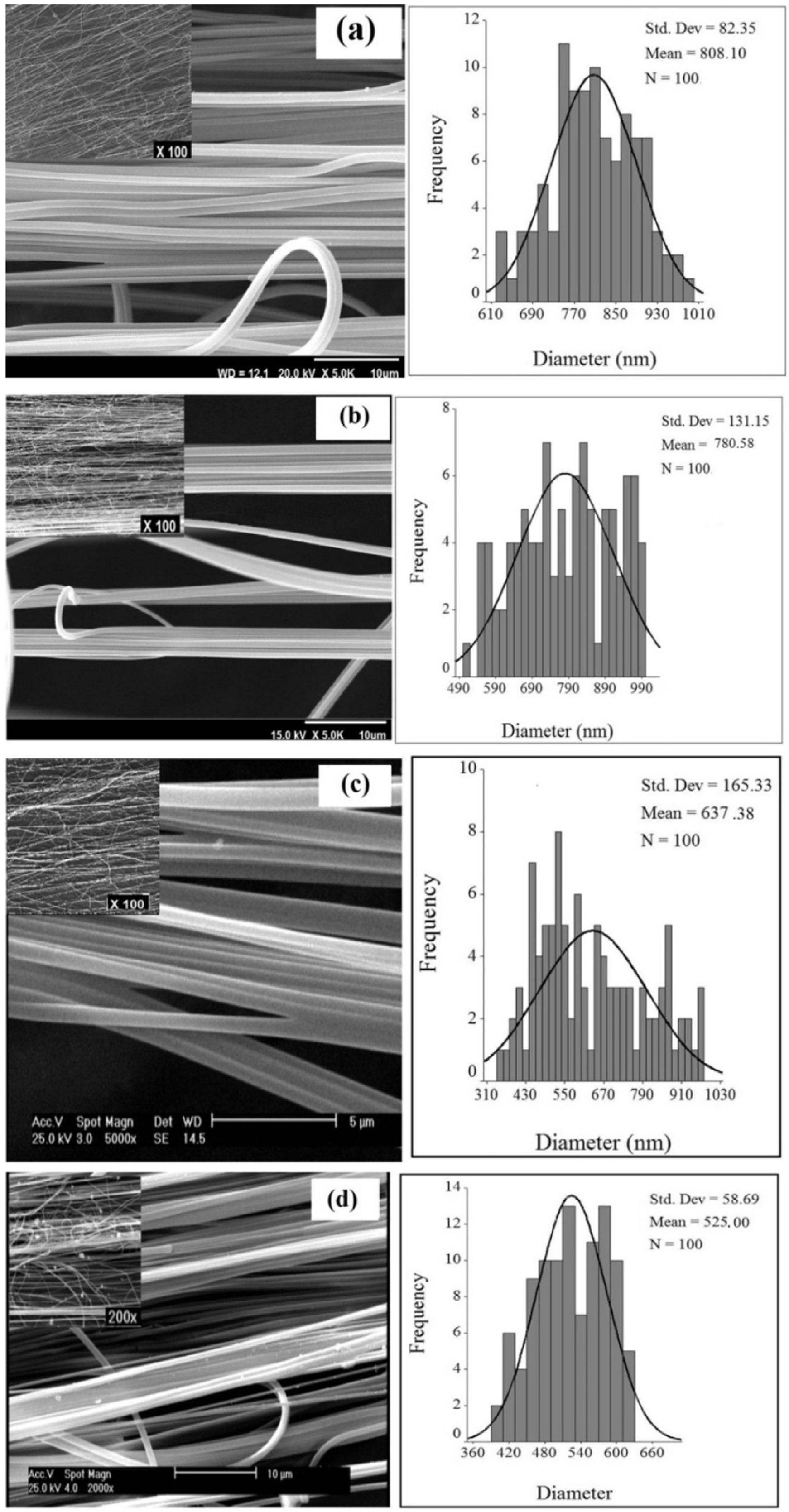

Fig. 3. SEM images and corresponding diameter distributions of electrospun PLLA fibers in the E-triangle zone at different twist rates: (a) 80 rpm, (b) 160 rpm, (c) 240 rpm, and (d) 320 rpm.

will affect the interaction between the fibers and consequently influence the mechanical behavior of the yarns. (Baji et al., 2010; Maleki et al., 2013; Yang et al., 2011; Zhou et al., 2012).

The effect of twist rate on the mechanical behavior of the electrospun yarns samples were compared to yarns prepared at a low twist rate of 20 rpm and samples prepared without twist (0 rpm). The

Table 2
Mean diameter of electrospun fibers in the E-triangle zone.

Twist rate (rpm)	Diameter of fibers in the E-triangle zone	
	Average (nm)	S.D.
80	810	80
160	780	130
240	640	170
320	530	60

mechanical properties of PLLA electrospun yarns are presented in Table 5 and typical strength-elongation curves are shown in Fig. 6.

By twisting the aligned fiber bundles at a rate of 80 rpm the strength at break of electrospun yarns increased compared to untwisted yarns. However, a further increase in twist up to rate of 320 rpm, the strength at break leveled off and even becomes somewhat lower (Hearle et al., 1959; Kilby, 1964). Compared to untwisted aligned fiber bundles, the higher strength upon twisting can be related to the increased cohesion and frictional forces between fibers. Actually, twisting allows the distribution of tension between the fibers and prevents local stress concentrations (Hearle et al., 1959; Kilby, 1964; Rao and Farris, 2000).

As twisting causes the fibers deposited at an angle to the yarn axis the direction of an applied force is an important factor. It is known that a force applied in a direction perpendicular to the aligned fibers, the mechanical properties appear much lower (Mauck et al., 2009; Ye et al., 2004). Because the angle at which fibers were deposited increases with increasing twist rates the yarn strength will reduce (Sugimoto et al., 2013). Taken together, twisting prevents fiber slippage by increasing friction between the fibers and thus improves the tensile properties. However, due to an increase in the angle of fiber deposition to yarn, a decrease in tensile strength will be observed (Naik and Madhavan, 2000; Zhou et al., 2012).

Similar to the tensile strength of the yarns the modulus increased by twisting but levels off and decreased at high twist rates. Electrospun

Table 3
Average values of the twist angle and diameters of electrospun PLLA yarns and fibers in the yarn.

Twist (rpm)	Twist angle (°)	Yarn diameter (μm)	Diameter of fibers in the yarn (nm)
80	18.8 ± 1.7	358 ± 73	789 ± 114
160	23.1 ± 2.6	381 ± 64	763 ± 119
240	31.7 ± 4.6	435 ± 73	595 ± 129
320	41.5 ± 4.7	470 ± 79	481 ± 48

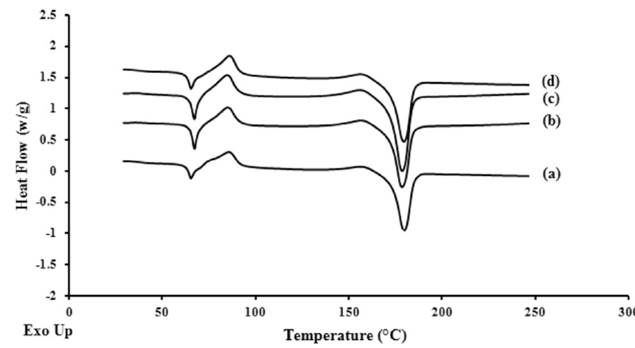

Fig. 5. DSC thermograms of electrospun yarns prepared at different twist rates of (a) 80 rpm, (b) 160 rpm, (c) 240 rpm, and (d) 320 rpm.

Table 4
Thermal properties of electrospun PLLA yarns prepared at different twist rates.

Sample		T_g (°C)	T_{cc} (°C)	T_m (°C)	ΔH_m (J/g)	ΔH_{cc} (J/g)	χ %
Electrospun yarns:	80	64.1	85.5	179.3	57.0	19.9	39.6
Twist rate (rpm)	160	64.8	84.9	178.1	58.0	17.1	43.7
	240	64.6	84.6	178.2	69.9	21,0	52.2
	320	64.2	85.8	179.0	71.3	16.4	58.6

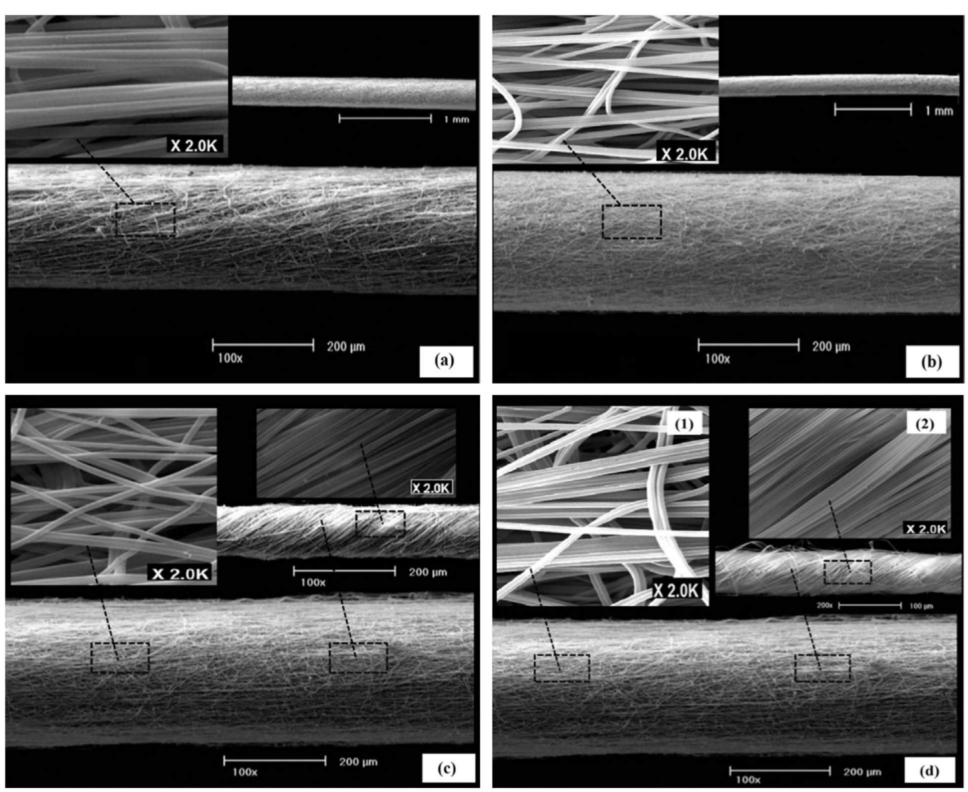

Fig. 4. SEM images of the electrospun PLLA yarns prepared at different twist rates; (a) 80 rpm, (b) 160 rpm, (c) 240 rpm, and (d) 320 rpm.

Table 5
Mechanical properties of PLLA electrospun yarns prepared at different twist rates.

Yarn twist (rpm)	Tensile Strength (cN/Tex)	Young's Modulus (cN/Tex)	Elongation at break (%)
0	1.98	15.75	121
20	2.59	18.28	153
80	4.10	32.58	187
160	3.98	29.37	203
240	3.47	24.09	238
320	3.18	23.14	267

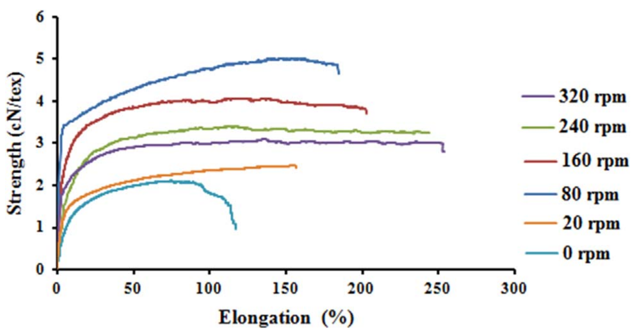

Fig. 6. Strength-elongation curves of electrospun yarns prepared at different twist rates.

yarns have a structure similar to conventional filament yarns and their mechanical properties can be assumed to follow Hooke's law. In a study performed by Yan and coworkers, a simulation based on the Hearle equation (Eq. (2)) was performed on electrospun poly(acrylonitrile) yarns prepared at different twist rates and revealed a good agreement obtained with this theory (Hearle et al., 1959; Yan et al., 2011).

$$E_y = E_f \cos^2 \alpha \tag{2}$$

In this equation, α is the twist angle and E_f and E_y are the fiber modulus and yarn modulus, respectively.

Applying twist to the aligned fibrous bundle, the cohesion between fibers and the lateral forces increases. Moreover, at higher twist rates, the angle of the fiber arrangement to the yarn axis increases. According to Eq. (1), the modulus reduces. Generally, there is a critical value in the twist rate where the modulus is maximal and similar results were observed in other studies (Fennessey Sian et al., 2006; Rao and Farris, 2000; Sugimoto et al., 2013; Yan et al., 2011; Zhou et al., 2012).

Twisting of electrospun PLLA fibers in processing of a yarn revealed an increase in the elongation at break. According to Hearle and coworkers, as a part of their studies on the mechanical properties of filament yarns, an applied twist imposes strain on every single filament (ε_f) according Eq. (3).

$$\varepsilon_f = \varepsilon_y \cos^2 \alpha \tag{3}$$

In this equation, ε_y is the yarn strain and α is the twist angle (Hearle et al., 1959). In the case of untwisted yarns or yarns prepared at low twist rates, the friction between the fibers is low and each fiber will rupture when gained its elongation at break. However, twisting causes friction between the fibers increasing the elongation at break. Moreover, by increasing the twist rate, the twist angle of fibers to the yarn axis increases, and according to Eq. (3), under the same tension, the elongation at break of yarn will be larger. Both of these factors can increase the elongation at break of yarns by increasing the twist rate (Ali et al., 2011b; Fennessey et al., 2006; He et al., 2014; Rao and Farris, 2000; Treloar and Riding, 1963).

The failure of electrospun PLLA yarns was determined using scanning electron microscopy and typical images of the rupture point of a yarn prepared at twist rate of 240 rpm are presented in Fig. 7. The images clearly show buckling, plastic deformation, and formation of multiple necks.

4. Conclusion

The E-triangle of fibers formed in electrospinning PLLA solutions from two oppositely charged nozzles has a large influence on the assembly of fibers and consequent properties of twisted yarns. It was

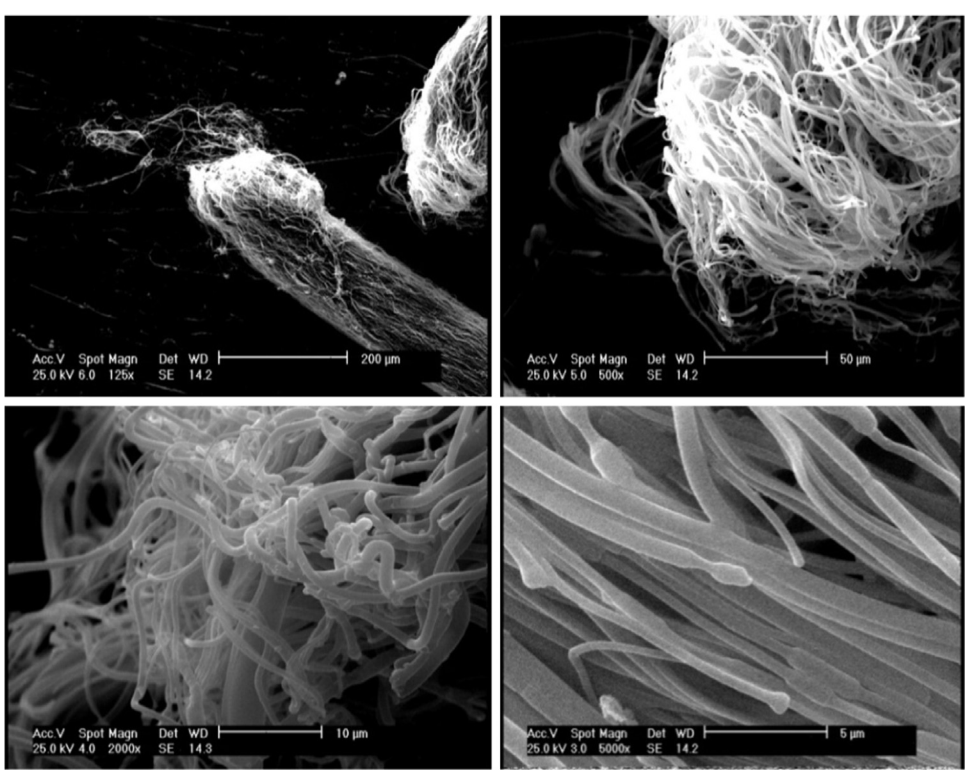

Fig. 7. SEM images of the end failure of electrospun PLLA yarns prepared at a twist rate of 240 rpm at different magnifications.

shown that by changing the twist rate the average fiber diameter decreased as the twist rate increased. At higher twist rates, despite the compression of the fibers in the inner structure of the yarn, the yarn diameter increased due to the formation of an outer layer of out of control depositing fibers. Increasing the twist rate gave an increased tension on the fibers, resulting in a crystallinity. The mechanical properties of the yarns showed that the strength at break and modulus of electrospun yarns increased up to a twist rate of 160 rpm and leveled off and even decreased slightly at the higher twist rates. Increasing the twist amount caused an increase in the elongation at break of electrospun yarns.

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