ELECTROSPUN NANOFIBER PROCESS CONTROL

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Received April 26, 2010

Fiber diameter is an important structural characteristic for electrospinning, due to its direct influence on the properties of the produced webs. In this paper, an image analysis-based method, called *direct tracking*, for measuring the electrospun fiber diameter, has been developed. The results obtained by direct tracking significantly excelled distance transformation, indicating that the method could be used for measuring electrospun fiber diameter.

Keywords: electrospinning, fiber diameter, image analysis, direct tracking, distance transformation

INTRODUCTION

In recent years, nanotechnology has become a topic of great interest to scientists and engineers, and is now established as a prioritized research area in many countries. Size reduction to the nanometer range creates new possibilities in terms of material properties, in particular with respect to the achievable surface, to volume ratios.

Electrospinning of nanofibers is a novel process for producing superfine fibers by forcing a solution through a spinnerette with an electric field. An emerging technology for manufacturing thin natural fibers is based on the electrospinning principle. In conventional fiber spinning, the mechanical force is applied to the end of a jet, whereas, in the electrospinning process, the electric body force acts on the element of the charged fluid. Electrospinning has emerged as a specialized processing technique for the formation of submicron fibers (typically between 100 nm and 1 μm in diameter), with high specific surface areas.

Due to their high specific surface area, high porosity and small pore size, the unique fibers have been suggested for wide range of applications. Electrospinning of nanofibers offers unique capabilities for producing novel natural nanofibers and fabrics with controllable pore structure.

About 4-9% of the cotton fiber is lost at textile mill, during the so-called opening and cleaning, which involves mechanically separated compressed clumps of fibers for the removal of trapped debris. Another 1% is lost during drawing and roving-pulling lengths of the fiber into longer and longer segments, which are then twisted together for strength. An average of 20% is lost during combing and yarn production. Typically, waste cotton is used in relatively low-value products, such as cotton balls, yarn and cotton batting. A new process for electrospinning waste cotton using a less harmful solvent has been developed.

Electrospinning is an economical and simple method used in the preparation of polymer fibers. Fibers prepared *via* this method typically have much smaller diameters than what is possible to attain by standard mechanical fiber-spinning technologies.[1] Electrospinning has gained much attention in the last few years as a cheap and straightforward method to produce nanofibers. Electrospinning differs from the traditional wet/dry fiber spinning in several ways, of which the most striking differences refer to the origin of the pulling force and to the final fiber diameters.

The mechanical pulling forces in the traditional industrial fiber spinning processes lead to fibers in the micrometer range, being contrasted in electrospinning by the electrical pulling forces that permit the production of nanofibers. Depending on the solution properties, the throughput of single-jet electrospinning systems is around 10 mL/min.

This low fluid throughput may limit the industrial use of electrospinning. A stable cone-jet mode, followed by the onset of the characteristic bending instability, which eventually leads to a considerable reduction in jet diameter, needs a low flow rate.[2] When the diameters of cellulose fiber materials are shrunk from micrometers (*e.g.* 10-100 mm) to submicrons or nanometers, several amazing characteristics appear, such as a very large surface area-to-volume ratio (for a nanofiber, this ratio can be up to 103 times higher than that of a microfiber), flexibility in surface functionalities, and superior mechanical performance (*e.g.* stiffness and tensile strength), compared to any other known form of the material.

These outstanding properties recommend polymer nanofibers as optimal candidates for many important applications,[3] including filter media, composite materials, biomedical applications (tissue engineering scaffolds, bandages, drug release systems), protective clothing for military, optoelectronic devices and semi-conductive materials, biosensors/chemosensors.[4] Another biomedical application of electrospun fibers, currently receiving much attention, refers to drug delivery devices. Researchers have monitored the release profile of several different drugs from a variety of biodegradable electrospun membranes. Also, electrospun fibers are used to create porous membranes for filtration devices. Due to the inter-connected network-type structure formed by the electrospun fibers, they exhibit good tensile properties, low air permeability and good aerosol protection abilities. Moreover, by controlling the fiber diameter, electrospun fibers can be produced over a wide range of porosities. Research has also been focused on the influence of the charging effects of electrospun non-woven mats on their filtration efficiency. The filtration properties slightly depend on the surface charge of the membrane, however the fiber diameter was found to have the strongest influence on aerosol penetration. Electrospun fibers are currently utilized for several other applications, as well. Conventional fiber spinning (like melt, dry and wet spinning) produces fibers with the diameter in the range of micrometer.

In recent years, *electrospinning* has gained much attention as a useful method to prepare fibers in the nanometer diameter range,[1-4] classified as *nanofibers*. The unique combination of high specific surface area,[5] extremely small pore size, flexibility and superior mechanical performance make nanofibers a preferred material for several applications. The proposed uses of nanofibers include tissue engineering,[6-8] drug delivery, wound dressing, protective clothing, filtration, reinforcement, electronic applications and space-based applications.[9-10]

The objective of this paper is to use image analysis for measuring the electrospun fiber diameter.

EXPERIMENTAL

Electrospun non-woven webs used as real webs in image analysis were obtained from electrospinning of PVA with an average molecular weight of 72000 g/mol, purchased from MERCK Company, at different processing parameters. The micrographs of the webs were obtained using a Philips (XL-30) Environmental Scanning Electron Microscope (SEM) under 10000X magnification, after being gold-coated.

RESULTS AND DISCUSSION

Two sets, each composed of five simulated images generated by a μ-randomness procedure, were used as samples with known characteristics, to demonstrate the validity of the techniques. The first set had a random orientation with increasing constant diameters; the second was also randomly oriented, but with a varying diameter, sampled from normal distributions with a mean of 15 pixels and standard deviations ranging from 2 to 10 pixels. Tables 1 and 2 present the structural features of these simulated images, shown in Figures 1 and 2.

The mean and standard deviation of nanofiber diameter for the first and second sets of simulated images obtained by different methods are shown in Tables 3 and 4, respectively. Figures 3 and 4 show the histograms of fiber diameter distribution for simulated images for the first and second set, respectively.

Table 1
Structural characteristics of the first set of images

Image no.	Angular range	Line density	Line thickness
C1	0-360	30	5
C2	0-360	30	10
C3	0-360	30	15
C4	0-360	30	20
C5	0-360	30	25

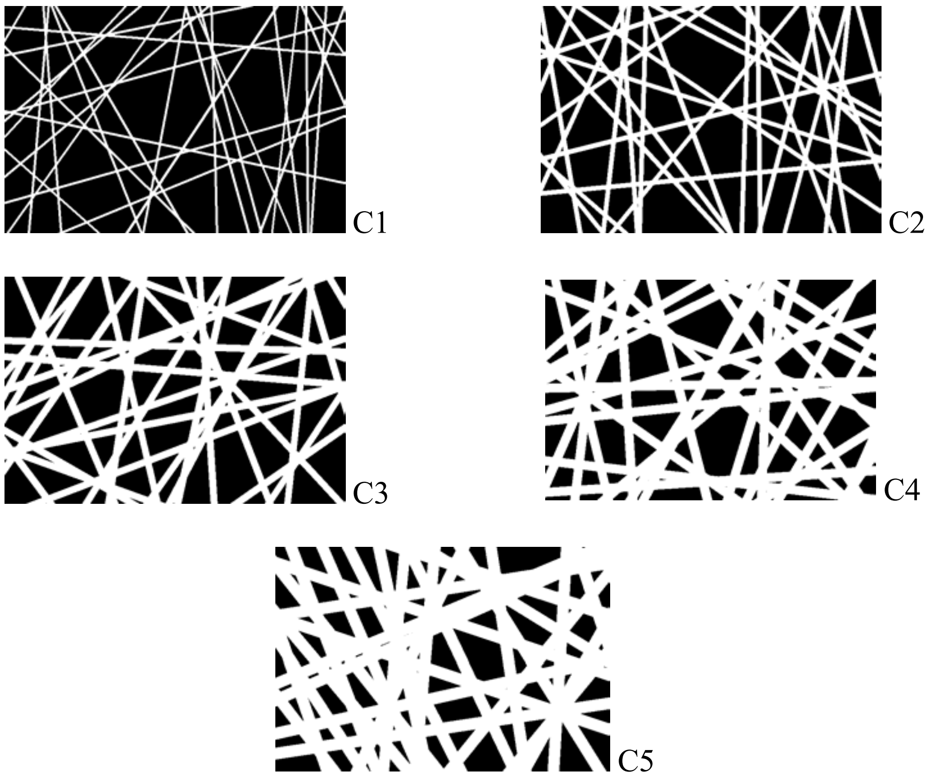
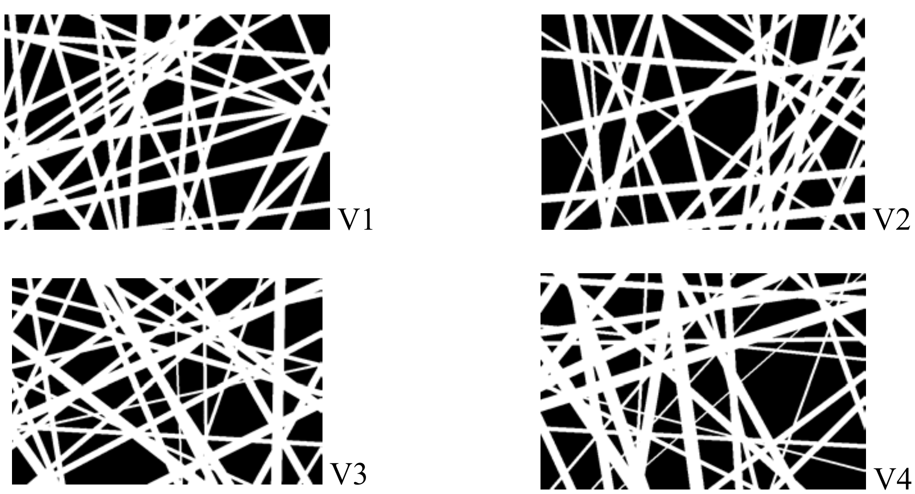


Figure 1: Simulated images with constant diameter



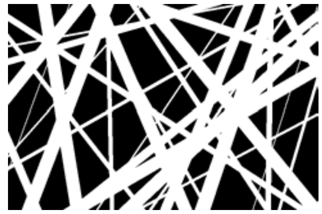

V5

Figure 2: Simulated images with varying diameter

Table 2
Structural characteristics of the second set of images

Image no.	Angular range	Line density	Line thickness	
			M	Std
V1	0-360	30	15	2
V2	0-360	30	15	4
V3	0-360	30	15	6
V4	0-360	30	15	8
V5	0-360	30	15	10

Table 3
Mean and standard deviation for series 1

		C1	C2	C3	C4	C5
Simulation	M	5	10	15	20	25
	Std	0	0	0	0	0
Distance transformation	M	5.486	10.450	16.573	23.016	30.063
	Std	1.089	2.300	5.137	6.913	10.205
Direct tracking	M	5.625	11.313	17.589	22.864	29.469
	Std	1.113	2.370	4.492	5.655	7.241

Table 4
Mean and standard deviation for series 2

		V1	V2	V3	V4	V5
Simulation	M	15.247	15.350	15.243	15.367	16.628
	Std	1.998	4.466	5.766	8.129	9.799
Distance transformation	M	16.517	16.593	17.135	17.865	19.394
	Std	5.350	6.165	7.597	9.553	11.961
Direct tracking	M	16.075	15.803	16.252	16.770	18.756
	Std	2.606	5.007	6.129	9.319	10.251

In the first set, for simulated images with a line thickness of 5 and 10 pixels, the distance transformation presents a closer mean and standard deviation of fiber diameter from the fiber diameter of the simulated picture. For a line thickness of 15, the standard deviation of the diameter obtained by the direct tracking method is closer to the data artificially obtained by simulation.

However, in this case, the distance transformation measured average diameter more accurately. For the simulated webs with a line thickness higher than 15 in the first set, the direct tracking method gave a better estimation of the mean and standard deviation of fiber diameter. This is due to the fact that, as the lines get thicker, branching during skeletonization (or thinning) is much more possible, the branches remaining even after pruning. Although these branches are small, their orientation is typically normal to the fiber axis, thus widening the distribution obtained by the distance transformation method. For fibers with small diameters, however, these branches are lower in number, and more accurate measurements are obtained by distance transformation.

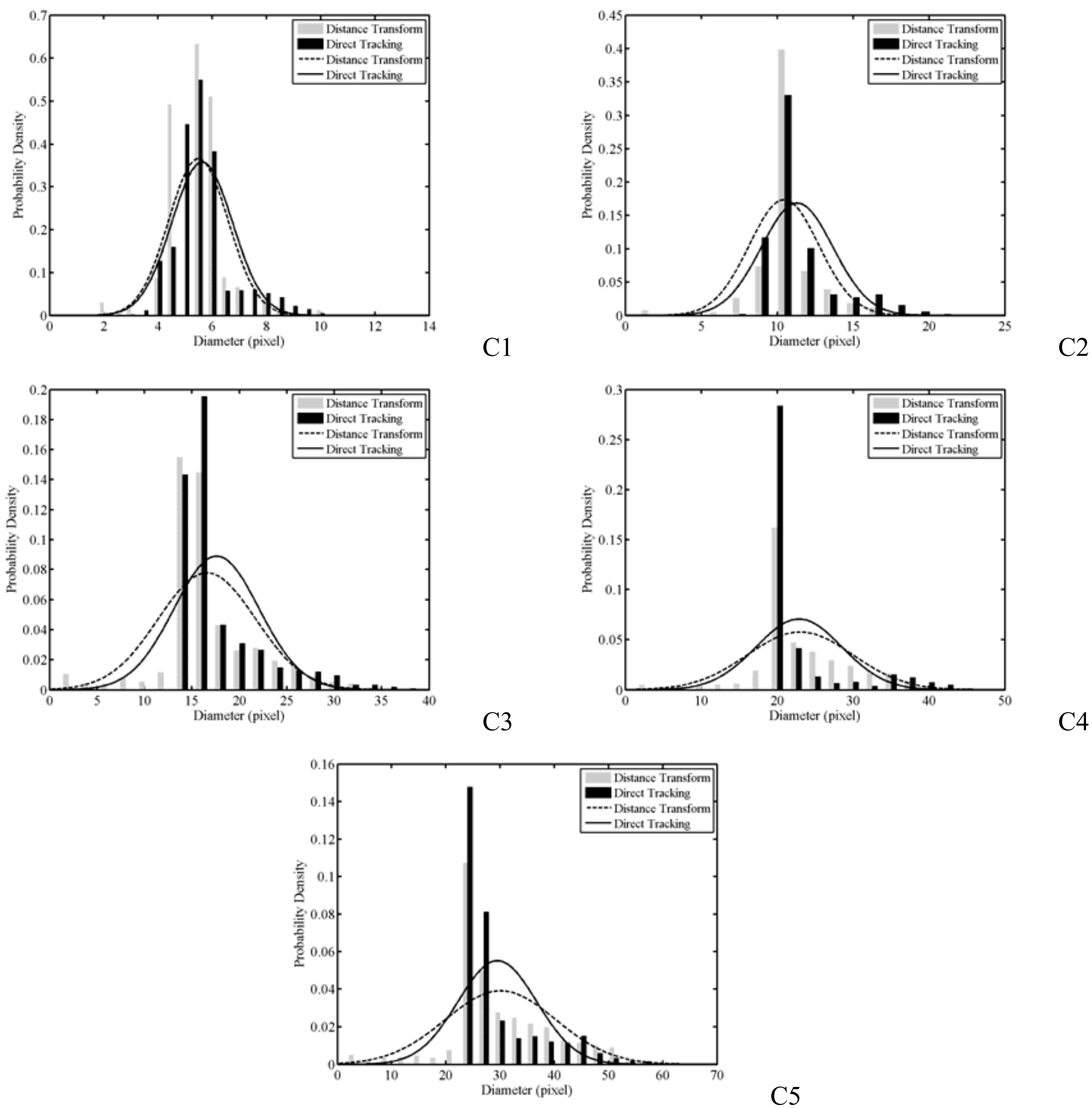


Figure 3: Histograms for simulated images with constant diameter

Furthermore, in the case of the distance transformation method, the value of the object center in the distance map is related to fiber diameter only for a single fiber. At intersections, where two or more fibers cross each other, it is associated to more than one fiber, being no longer related to fiber diameter. Both the distance-transformed image and the skeleton are broken at intersections. The problem becomes more serious as fibers get thicker in the points where more fibers cross each other. Hence, the method fails to measure fiber diameter at intersections, causing over-estimation of fiber diameter. Since, in the direct tracking method, the image is divided into parts where single fibers exist, and the effect of the intersections, which causes inaccurate measurement of fiber diameter, is eliminated. Therefore, fiber diameter will be better estimated.

In the second set, regardless of the line thickness used in simulation, for all simulated webs, direct tracking resulted in a better measurement of the mean and standard deviation of fiber diameter. Note that mean and standard deviation of the diameter for the simulated images with varied diameter are slightly different from those set as simulation parameters.

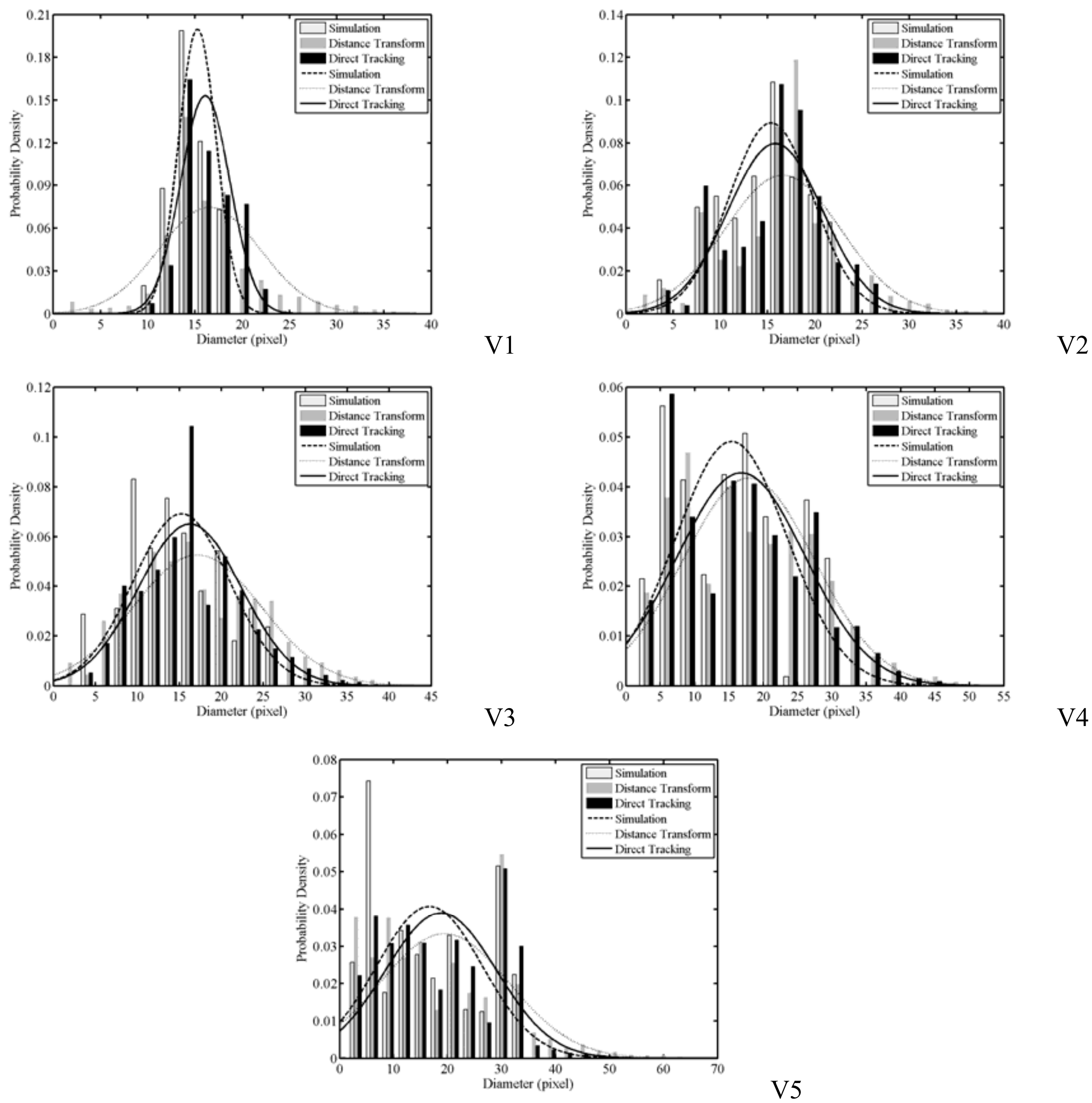


Figure 4: Histograms for simulated images with varying diameter

There are several reasons for the deviation of the computed results when using direct tracking and the real collected results. The differences observed can be attributed to the failure of the technique in correctly distinguishing between the multiple fibers joined together and a single fiber. Also, a 1-pixel error occurs in the selection of the mid point pixel (as a starting point for the second scan), when the number of pixels in the first scan is even. Furthermore, fiber segments should have minimum lengths, for permitting to measure the diameter. For dense webs or dense regions in a web, the fiber identification process creates some artifacts, other than the fibers, which result in untrue measurements. Further advancements in this field could include the improvement of the fiber identification process and the circumvention of the other problems mentioned.

The applicability of the techniques was also tested using five real webs obtained from electrospinning of PVA. SEM micrographs of the webs (Fig. 5) were first thresholded for diameter measurement. Fiber diameter distributions were determined for each image by distance transformation and direct tracking methods, the results being compared to those obtained by the manual method. Table 5 shows the results for real webs in terms of pixel and *nm*. The histograms for real webs are given in Figure 6. For the real webs, the mean and standard deviation of fiber diameter for direct tracking were closer to those obtained by the manual method, which agrees with the trends observed for the simulated images.

Table 5
Mean and standard deviation for real webs

			R1	R2	R3	R4	R5
Manual	M	pixel	24.358	24.633	18.583	18.827	17.437
		nm	318.67	322.27	243.11	246.31	228.12
	Std	pixel	3.193	3.179	2.163	1.984	2.230
		nm	41.77	41.59	28.30	25.96	29.18
Distance transformation	M	pixel	27.250	27.870	20.028	23.079	20.345
		nm	356.49	364.61	262.01	301.94	266.17
	Std	pixel	8.125	7.462	4.906	7.005	6.207
		nm	106.30	97.62	64.18	91.64	81.21
Direct tracking	M	pixel	27.195	27.606	20.638	21.913	20.145
		nm	355.78	361.15	269.99	286.68	263.55
	Std	pixel	4.123	5.409	4.148	4.214	3.800
		nm	53.94	70.77	54.27	55.14	49.72

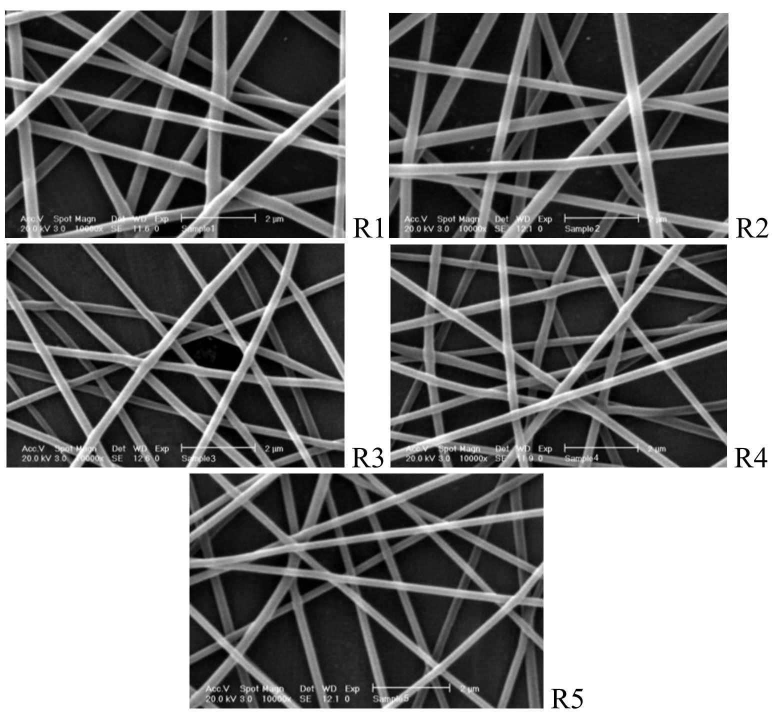


Figure 5: Micrographs of electrospun webs

Besides the previously mentioned reasons, the small discrepancies between the results obtained may be also attributed to the different number of measurements employed in each technique. Distance transformation and direct tracking measure over 1000 diameters. By the manual methods, however, the number of measurements is limited to mostly 100, as due to the time-consuming nature of the procedure.

CONCLUSIONS

The general applicability of the method using real webs was also demonstrated with five real electrospun non-woven webs obtained by electrospinning of PVA. Since the methods needed binary images as input, the images had first to be segmented. A local thresholding method was employed together with Otsu's method, for automatically computing the appropriate threshold.

The results obtained for real webs confirm the trends suggested by the simulated images. The mean and standard deviation values obtained by direct tracking were significantly closer to those of the manual method, compared to those obtained by distance transformation, suggesting that direct tracking could generally perform better; however, in the webs with very low fiber diameter, distance transformation may produce more accurate results. The results show that the use of image analysis for determining fiber diameter in electrospun non-woven webs has been successful.

REFERENCES

[1] S-H. Tan and R. Inai, *Polymer*, **46**, 6128 (2005).
[2] J. M. Deitzel, *Polymer*, **42**, 8163 (2001).
[3] S. Zarkoob, *Polymer*, **45**, 3973 (2004).
[4] S. Atheron, *Polymer*, **45**, 2017 (2004).
[5] M. Ziabari, V. Mottaghitalab, S. T. McGovern and A. K. Haghi, *Chim. Phys. Lett.*, **25**, 3071 (2008).
[6] M. Ziabari, V. Mottaghitalab, S. T. McGovern and A. K. Haghi, *Nanoscale Research Letter*, **2**, 297 (2007).
[7] M. Ziabari, V. Mottaghitalab and A. K. Haghi, *Korean J. Chem. Eng.*, **25**, 919 (2008).
[8] M. Ziabari, V. Mottaghitalab and A. K. Haghi, *Korean J. Chem. Eng.*, **25**, 923 (2008).
[9] M. Ziabari, V. Mottaghitalab and A. K. Haghi, *Korean J. Chem. Eng.*, **25**, 905 (2008).
[10] A. K. Haghi and M. Akbari, *Physica Status Solidi*, **204**, 1830 (2007).