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SHORT COMMUNICATION

Simulated image of electrospun nonwoven web of PVA and corresponding nanofiber diameter distribution

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Abstract−Fiber diameter is the most important characteristic in electrospun nonwoven webs. Understanding how it is influenced by the electrospinning parameters is essential to produce webs with desired characteristics. In this contribution, Direct Tracking method for measuring electrospun fiber diameter is described. To evaluate the accuracy of the technique, a simulation algorithm for generating webs with known characteristics was employed. To verify the applicability of the method on real samples, an electrospun polyvinyl alcohol (PVA) mat, as a representative of real webs, was used. Since the Direct Tracking method uses a binary image as its input, local thresholding was applied to segment the SEM micrograph of the electrospun web. The results indicate that the method could be used successfully for determining fiber diameter in electrospun nonwoven webs.

Key words: Electrospinning, Fiber Diameter, Image Analysis, Direct Tracking, μ-Randomness, Local Thresholding

INTRODUCTION

Nanofibers are ultra-fine fibers notable for their very small diameter, large surface area per unit mass and extremely small pore size. Electrospinning is a straightforward method for producing nanofibers. In this process, a high electric field is generated between a polymer fluid and a collection target. When the electric field strength dominates the surface tension of polymer solution, a charged jet is ejected. The jet is elongated during its passage to the target and this process is accompanied by the rapid evaporation of the solvent. The dried fibers accumulate on the surface of the collector, resulting in a nonwoven random fiber web with nanometer diameter scale [1-7].

It is well known that the distribution of structural characteristics such as fiber orientation [8-13], fiber diameter [14,15], pore size [16], uniformity [17] and other structural characteristics [15] are very important when determining the properties of nonwoven textiles. The properties of electrospun nonwoven webs are determined by the above-mentioned features as well. However, in this case, fiber diameter is more important than the others. Understanding how fiber diameter and its distribution are affected by the electrospinning parameters is essential to producing webs with the desired properties. Consequently, accurate and automated measurement of fiber diameter is useful and crucial. Recently, image analysis techniques have been used to evaluate nonwoven structures [8-17]. The objective of our research is to develop an image analysis based method to serve as a simple and efficient alternative for electrospun fiber diameter measurement.

METHODOLOGY

1. Measuring Electrospun Fiber Diameter

The typical way of measuring electrospun fiber diameter is through a manual method, which consists of the following steps: setting the scale, measuring the number of pixels between two edges of fiber perpendicular to the fiber axis, converting the number of pixels to nm using the scale and recording the result. Typically, 100 diameters are measured. Finally, a histogram of fiber diameter is generated.

However, this process is tedious and time-consuming especially for a large number of samples. Furthermore, it cannot be used as on-line method for quality control since an operator is needed for performing the measurements. Thus, developing an automated technique that eliminates the use of operator and has the capability of being employed as on-line quality control is of great importance.

To that end, we developed an image analysis based method called *Direct Tracking*. This method uses a binary image as its input. Fiber diameter is measured from two scans: first a horizontal and then a vertical scan, which is started from the mid point of the white pixels (representative of fiber) in horizontal scan. The number of white pixels is counted in each scan. The counting process stops when a black pixel (representative of background) is reached. If the black pixel is not found, the method fails. Having the number of pixels of a fiber scanned in either direction, the fiber diameter is determined through a simple geometrical relationship.

In electrospun nonwoven webs, fibers cross each other at intersection points and this brings about some untrue measurements in these areas. To circumvent this problem a process called *fiber identification* is employed. First, black regions are labeled and a couple of regions between which a fiber exists are selected. In the next step, the two selected regions are connected by performing a *dilation* operation with a large enough *structuring element*. In the following process, an *erosion* operation with the same structuring element is performed and the fiber is recognized. Then, in order to enhance the processing speed, the image is cropped to the size of selected regions. Afterwards, fiber diameter is measured according to the previously explained algorithm. This trend is continued until all of the fibers are analyzed. Finally, the data in pixels may be converted to nm and the histogram of fiber diameter distribution is plotted.

2. Simulation

Since the proposed method is a new technique, its accuracy needs

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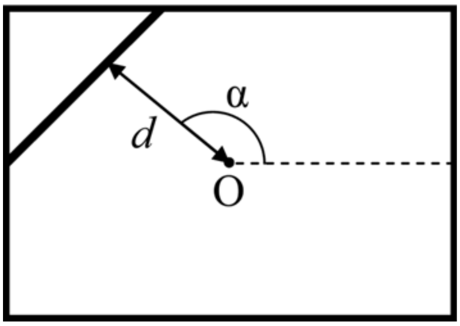

Fig. 1. μ-Randomness procedure.

to be evaluated by using samples with known characteristics. To that end, a simulation algorithm for generating nonwoven webs with known characteristics has been employed. The use of simulation is not a new idea. Algorithms for simulating nonwoven mats of continuous and discontinuous fibers have been proposed by Abdel-Ghani et al. [18] and Pourdeyhimi et al. [8]. The most important component of simulation is the way in which lines or curves are generated.

The aim is to produce an unbiased sample that is spatially homogeneous. Lately, it was discovered by Pourdeyhimi et al. [8] that the best way to simulate nonwovens of continuous fibers is through *μ-Randomness* procedure in which idealized straight lines are used for the simulation. It is assumed that the lines are so long that they intersect the boundaries. This process is illustrated in Fig. 1. Under this scheme, a line with a specified thickness is drawn perpendicular to α direction at distance d (limited to the diagonal of the image) away from a fixed reference point O [8,18]. Several variables are allowed to be controlled during the simulation: line density, angular density, distance from the reference point and line thickness. These variables can be sampled from given distributions or held constant.

3. Segmentation

As it was mentioned before, the Direct Tracking algorithm for measuring fiber diameter needs binary images as its input. Therefore, images first have to be converted to black and white. This is done by segmentation (known also as thresholding). The simplest thresholding technique is to segment the image by using a single constant threshold. This approach is called *global thresholding*. First, a threshold is selected, then pixels with a value more than or equal to the threshold belong to the objects (here fibers) and the remaining pixels belong to the background. Typically, the threshold is chosen by trial and error. The main problem here is that global thresholding can fail when the background illumination is uneven. For handling such a situation, *local thresholding* could be applied. In this approach, the original image is divided to subimages and a different threshold is used to segment each one. To automatically select the appropriate threshold *Otsu's method*, which minimizes the intraclass variance of the black and white pixels could be employed. In advance of the segmentation, an *intensity adjustment* operation and a two dimensional *median filter* are applied in order to enhance the contrast of the image and remove noise [19-21].

EXPERIMENTAL

The electrospun nonwoven web used in this study was obtained from the electrospinning of PVA with average molecular weight of 72,000 g/mol (purchased from MERCK Company) at a concentration of 12% (w/w), applied voltage of 15 KV, flow rate of 0.2 ml/h and tip to collector distance of 10 cm. The micrograph of the web, which was used as a real web in image analysis, was obtained by using a Philips (XL-30) environmental Scanning Electron Microscope (SEM) under magnification of 10,000× after being gold coated.

RESULTS AND DISCUSSION

Two simulated images were generated using μ-randomness procedure. The first had random orientation with constant diameter of 20 pixels; the second was also randomly oriented but with varying diameter sampled from a normal distribution with a mean of 15 and standard deviation of 8 pixels. In both cases, the line density was set to 30 and angular density was sampled from a random distribution in the range of 0-360°. In addition to simulated images, there was a real web in this report obtained from electrospinning of PVA to test the applicability of the method for real samples. The SEM micrograph of the real web was thresholded for diameter measurement. The fiber diameter distribution was determined for each image by using the Direct Tracking method.

We employed the proposed method here for measuring fiber diameter of an electrospun PVA mat. However, it may be applied on electrospun webs obtained from other polymers as well since the electrospun webs of different polymers are quite analogous (*i.e.* all of them consist of fibers which are randomly oriented and the only features which may differ are the diameter of these fibers and the way they are arranged).

A summary of the mean and standard deviation of fiber diameter for the simulated images and the real web image utilizing the Direct Tracking method is shown in Table 1.

Fig. 2 and Fig. 3 show the simulated images and their fiber diameter distributions. A micrograph of a real web together with its diameter distribution (in terms of nm) is given in Fig. 4. The curved line over the histogram in each case corresponds to its fitted *normal distribution*.

From Table 1 it is apparently observed that mean and standard deviation obtained from Direct Tracking method are quite close to the simulation values. It must be considered that the true mean and standard deviation of diameter in simulation of sample with varying line thickness are slightly different from those of used as simulation variables (true values are given in Table 1). The results of Direct Tracking for the real web are in agreement with the manual method. It is noteworthy that, in the manual method, since the number of measurements is limited (to mostly 100) due to the time-con-

Table 1. Mean and standard deviation for the simulated images and the real web

		Mean (pixel)	Std (pixel)
Constant diameter	Simulation	20	0
	Direct tracking	20.809	1.497
Varying diameter	Simulation	15.367	8.129
	Direct tracking	16.770	9.319
Real web	Manual	24.358	3.193
	Direct tracking	27.195	4.123

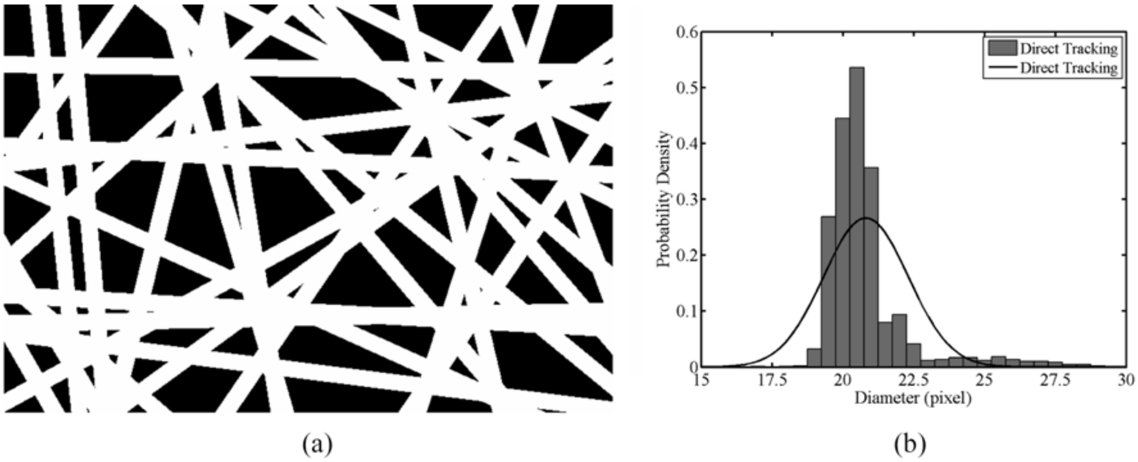

Fig. 2. (a) Simulated image with constant diameter, (b) Corresponding fiber diameter distribution.

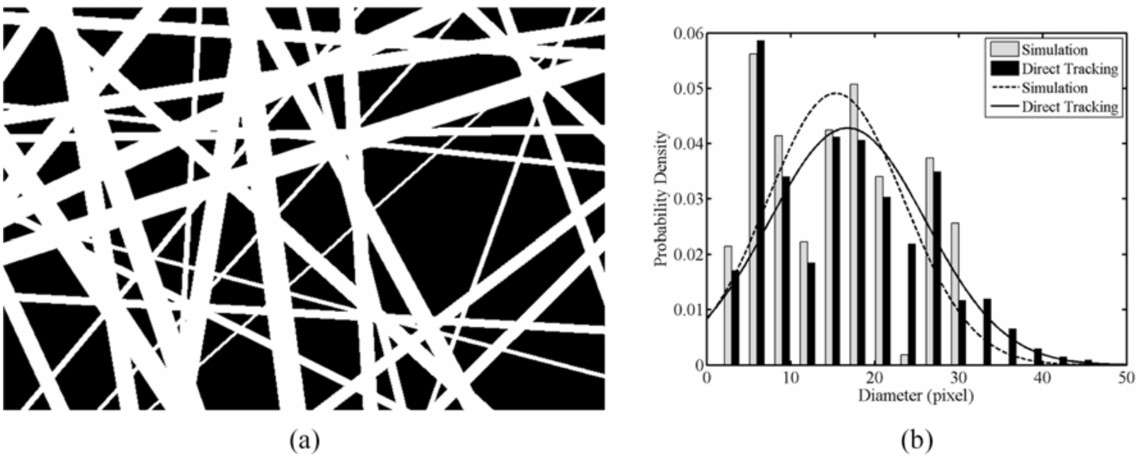

Fig. 3. (a) Simulated image with varying diameter, (b) Corresponding fiber diameter distribution.

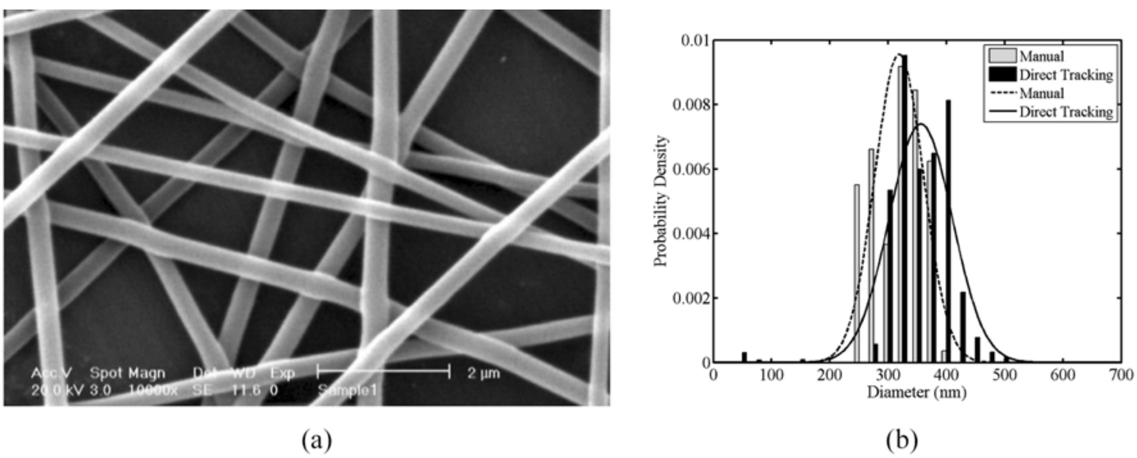

Fig. 4. (a) Micrograph of a real electrospun web, (b) Corresponding fiber diameter distribution.

suming nature of the process and measurement lines are not exactly drawn perpendicular to the fiber axis, the resulting distribution could not be the true distribution of fiber diameter of the sample. Nevertheless, due to lack of standard technique, the manual method is often employed as the typical way for measuring electrospun fiber diameter. Thus, in order to test the validity of any other techniques, the results of other methods should be compared with the manual method. In Direct Tracking, over 2000 diameters are measured exactly perpendicular to the fiber axis. The differences observed can be attributed to the failure of the technique to correctly distinguish between multiple fibers being 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 must be of minimum lengths so that the diameter may be measured. For dense webs or dense regions in a web, the fiber identification process creates some artifacts other than fibers, which results in untrue measurements. Further advancements in this field could involve improving the fiber identification process and trying to circumvent the other mentioned problems.

CONCLUSION

Fiber diameter is one of the important structural characteristics which influences the properties of electrospun nonwoven webs. Electrospun fiber diameter is often measured by manual method, which is an operator-based technique with low number of measurements that cannot be used as an automated method for quality control. Hence, developing an automated technique for measuring fiber diameter is highly demanded. In this paper, Direct Tracking as an alternative has been presented. In order to examine the accuracy of the method, samples with known characteristics were produced by using μ-Randomness procedure. Mean and standard deviation of fiber diameter obtained from Direct Tracking were quite close to simulation values. The method was also applied to measure fiber diameter of a real web produced from electrospinning of PVA. However, it could be used for measuring fiber diameter of any other electrospun webs; the choice of PVA was quite arbitrary and mostly due to ease of web formation of PVA. SEM micrograph of the web was converted to black and white by using local thresholding. Otsu's method was used to automatically determine the appropriate threshold. Mean and standard deviation of fiber diameter obtained from Direct Tracking for the real web were acceptably close to the manual method. The results indicate that the attempt to develop a high speed, computer automated algorithm for measuring electrospun nanofiber diameter has been successful.

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