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Prediction of nanofiber diameter for improvements in incorporation of multilayer electrospun nanofibers

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Abstract−Electrospun nanofiber web has many potential applications due to its large specific area, very small pore size and high porosity. However, the mechanical properties of nanofiber web are very poor for use in textile application. To remedy this defect, the laminating process could accomplish in order to protect nanofiber web versus mechanical stresses. In this paper, direct tracking method as an image analysis based technique for measuring electrospun nanofiber diameter has been presented. The usefulness of the method for electrospun nanofiber diameter measurement is discussed. Such automated measurement of nanofiber diameter can be used to obtain better laminated webs.

Key words: *Laminated nonwovens*, Electrospinning, Nanofibers, Fiber Diameter, Image Analysis

INTRODUCTION

In the electrospinning process, a high electric field is generated between a polymer solution held by its surface tension at the end of a syringe (or a capillary tube) and a collection target. In fabric lamination, producing an adhesive bond which guarantees no delaminating or failure in use requires lamination skills and information about adhesive types. It is relatively simple to create a strong bond; the challenge is to preserve the original properties of the fabric and to produce a flexible laminate with the required appearance, handle and durability. In other words, the application of adhesive should have minimum effect on the fabric flexibility and aesthetics during the lamination process; therefore, adhesive must be applied in a controlled manner. To achieve to this purpose, it is generally necessary that the least amount of a highly effective adhesive be applied and it penetrate to a certain extent of the fabric and cover the widest possible surface area. Too much adhesive and excessive penetration is likely to lead to fabric stiffening and it could result in thermal discomfort in the cloth, since the adhesive itself could form an impermeable barrier to perspiration.

The adhesives could be solvent/water-based or hot-melt. In first group, the adhesives are as solutions in solvent or water, and solidify by evaporating of the carrying liquid. In this group, solvent-based adhesives could ‘wet’ the surfaces to be joined better than water-based adhesives, and also could solidify faster. But unfortunately, they are environmentally unfriendly, usually flammable and more expensive. Of course it does not mean that the water-based adhesives are always preferred to laminating, since in practice, drying off water in terms of energy and time is expensive too. Beside, water-based adhesives are not resistant to water or moisture because of their hydrophilic nature. But in hot-melt adhesive group, the adhesives are as solids and melt under the action of heat. These types of adhesives are environmentally friendly, inexpensive, require less heat and energy, and so are now more preferred. They can be of several different chemical types, such as polyolefin (polyethylene, polypropylene), polyurethane, polyester, polyamide or blends of different polymers or copolymers in order to reach for a wide range of properties (including melting points, durability to washing and dry cleaning and heat resistance). Hot-melt lamination can be either continuous (hot calendars) or static (flat iron or Hoffman press) and is accomplished by two separate processes: first, a means of applying the actual adhesive; and second, bringing the two substrates together to form the actual bond under the action of heat and pressure. In this process, the heating is accomplished at temperatures above the softening or melting point of the adhesive. In addition, hot melt adhesives are available in several forms: as a web, as a continuous film, or in powder or granular form. The adhesive powders are available in most chemical types and also in particle sizes ranging from very small up to about 500 micrometers or so in diameter. Adhesives in film or web form are more expensive than the corresponding adhesive powders. The webs are discontinuous and produce laminates which are flexible, porous and breathable, whereas continuous film adhesives cause stiffening and produce laminates which

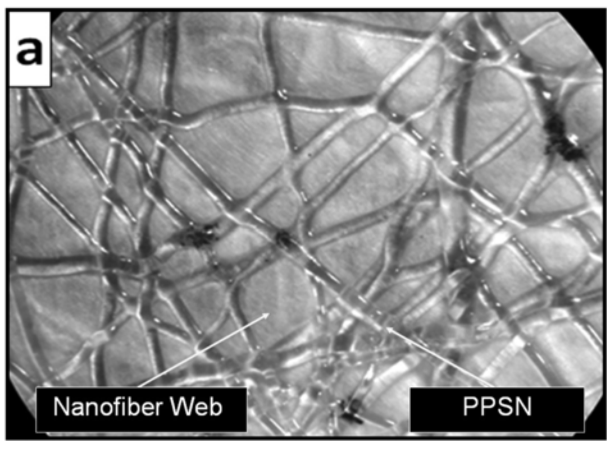

Fig. 1. The optical microscope images of multilayer nanofiber web (PPSN): polypropylene spun-bond nonwoven.

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are not porous and permeable to both air and water vapor. This behavior is attributed to the impervious nature of adhesive film and its shrinkage under the action of heat.

Fig. 1 is an optical microscope image of a multilayer nanofiber web. Accurate and automated measurement of nanofiber diameter of laminated webs is useful and crucial, and therefore has been taken into consideration in this contribution. The objective of the current research would then be to develop an image analysis based method to serve as a simple, automated and efficient alternative for electrospun nanofiber diameter measurement with particular application in laminated nanofiber web.

2. Methodology

The algorithm for determining fiber diameter uses a binary input image and creates its skeleton and distance transformed image (distance map). The skeleton acts as a guide for tracking the distance transformed image, and fiber diameters are measured from the intensities of the distance map at all points along the skeleton. Fig. 2 shows a simple simulated image that consists of five fibers with diameters of 10, 13, 16, 19 and 21 pixels, together with its skeleton and distance map including the histogram of fiber diameter obtained by this method.

In this paper, we developed *direct tracking* method for measur-

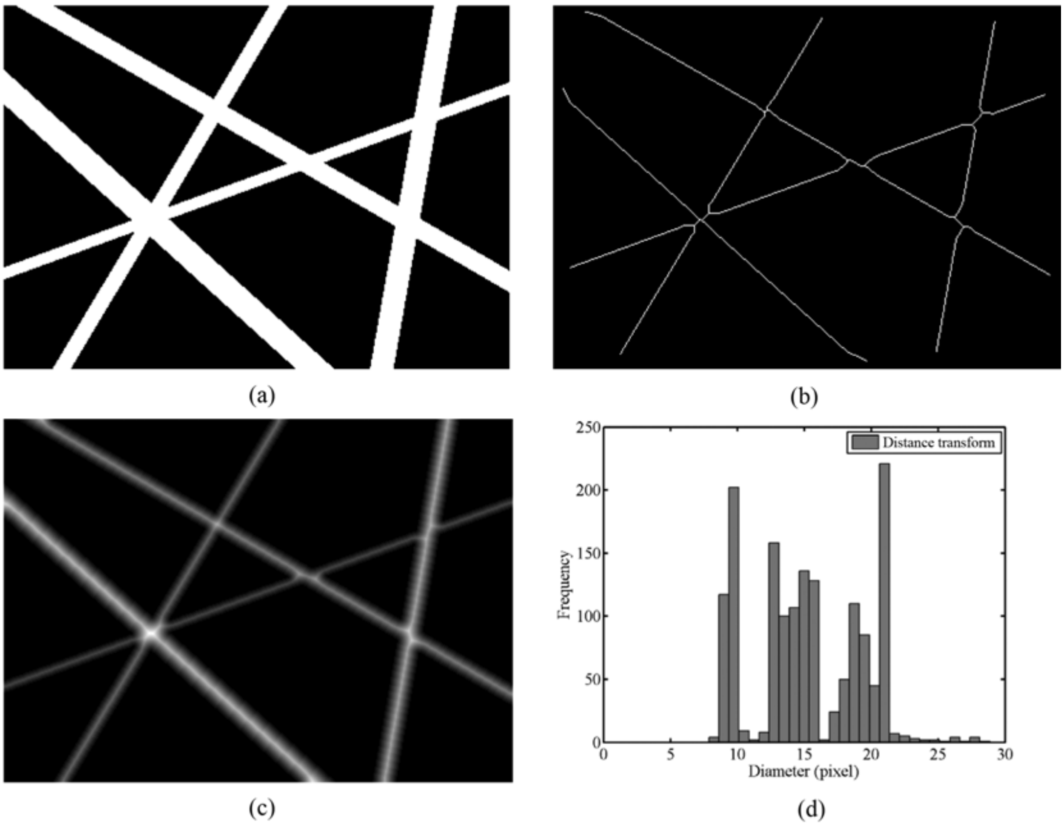

Fig. 2. (a) A simple simulated image, (b) Skeleton of (a), (c) Distance map of (a) after pruning, (d) Histogram of fiber diameter distribution obtained by distance transform method.

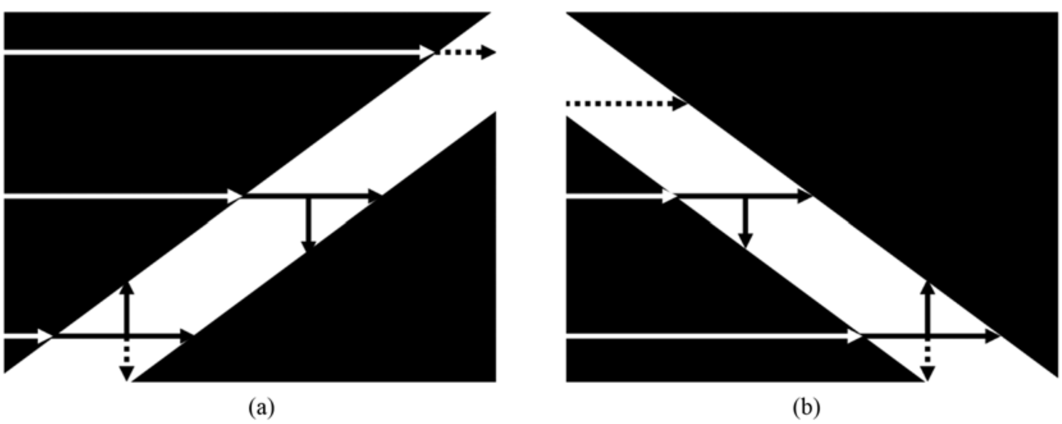

Fig. 3. Fiber diameter measurement based on two scans in direct tracking method.

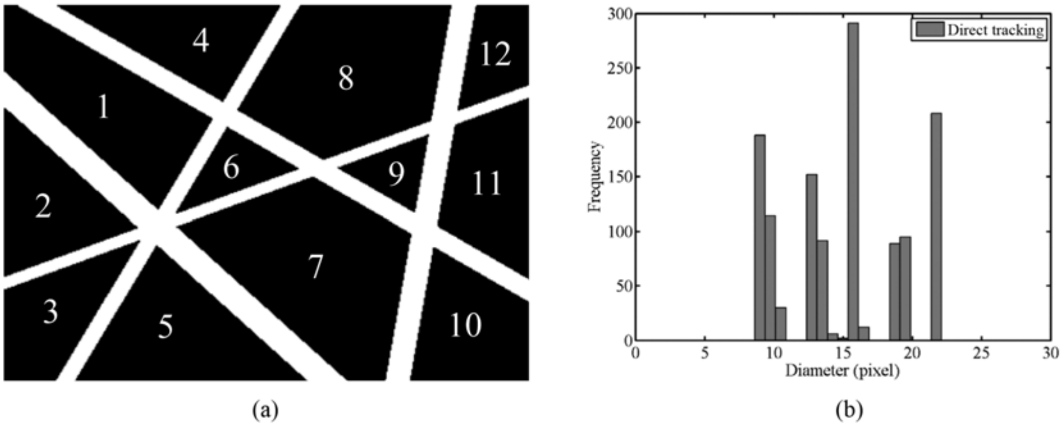

Fig. 4. (a) The labeled simulated image, (b) Histogram of fiber diameter distribution obtained by direct tracking method.

ing electrospun nanofiber diameter. This method, which also uses a binary image as the input, determines fiber diameter based on information acquired from two scans: first a horizontal and then a vertical scan. In the horizontal scan, the algorithm searches for the first white pixel (representative of fibers) adjacent to a black (representative of background). Pixels are then counted until reaching the first black. Afterwards, the second scan is started from the midpoint of the horizontal scan and pixels are counted until the first vertical black pixel is encountered. The direction will change if the black pixel is not found (Fig. 3). Having the number of horizontal and vertical scans, the number of pixels in the perpendicular direction, which is the fiber diameter in terms of pixels, can be measured through a simple geometrical relationship.

In electrospun webs, nanofibers cross each other at intersection points, and this brings about the possibility for some untrue measurements of fiber diameter in these regions. 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. Fig. 4 depicts the labeled simulated image and the histogram of fiber diameter obtained by direct tracking method.

Now, reliable evaluation of the accuracy of the developed methods requires samples with known characteristics. Since it is neither possible to obtain real electrospun webs with specific characteristics through the experiment nor is there a method which measures fiber diameters precisely with which to compare the results, the method will not be well evaluated using just real webs. To that end, a simulation algorithm has been employed for generating samples with known characteristics. In this case, it is assumed that the lines are infinitely long so that in the image plane, they intersect the boundaries. Under this scheme, which is shown in Fig. 5, a line with a specified thickness is defined by the perpendicular distance d away from a fixed reference point O located in the center of the image and the angular position of the perpendicular α. Distance d is limited to the diagonal of the image. Several variables are allowed to be controlled during simulation: line thickness, line density, angular density and distance from the reference point. These variables can be sampled from given distributions or held constant.

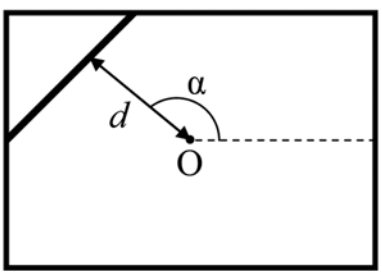

Fig. 5. μ-Randomness procedure.

Distance transform and direct tracking algorithms for measuring fiber diameter both require a binary image as their input. Hence, the micrographs of electrospun webs first have to be converted to black and white. This is carried out by the *thresholding* process (known also as *segmentation*), which produces a binary image from a grayscale (intensity) image. This is a critical step because the segmentation significantly affects the result. Prior to the segmentation, an *intensity adjustment* operation and a two dimensional *median* filter are often applied in order to enhance the contrast of the image and remove noise.

In the simplest thresholding technique, called *global thresholding*, the image is segmented by using a single constant threshold. One simple way to choose a threshold is by trial and error. Each pixel is then labeled as object or background, depending on whether its gray level is greater or less than the value of threshold, respectively.

The main problem of global thresholding is its possible failure in the presence of non-uniform illumination or local gray level unevenness. An alternative to this problem is to use *local thresholding* instead. In this approach, the original image is divided into subimages and different thresholds are used for segmentation. As shown in Fig. 6, global thresholding resulted in some broken fiber segments. This problem was solved by using local thresholding.

EXPERIMENTAL

Electrospun nanofiber webs used as real webs in image analysis were prepared by electrospinning aqueous solutions of PVA with average molecular weight of 72,000 g/mol (MERCK) at different processing parameters. The micrographs of the webs were obtained with a Philips (XL-30) environmental scanning electron microscope (SEM) under magnification of 10,000× after gold sputter coating.

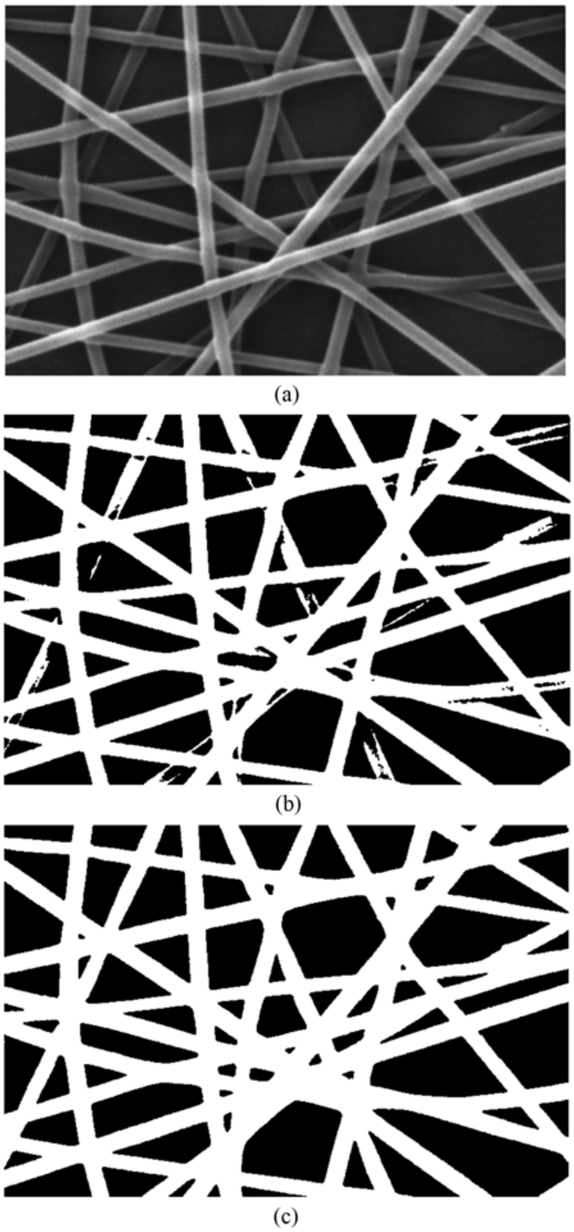

Fig. 6. (a) A typical electrospun web, (b) Global thresholding, (c) Local thresholding.

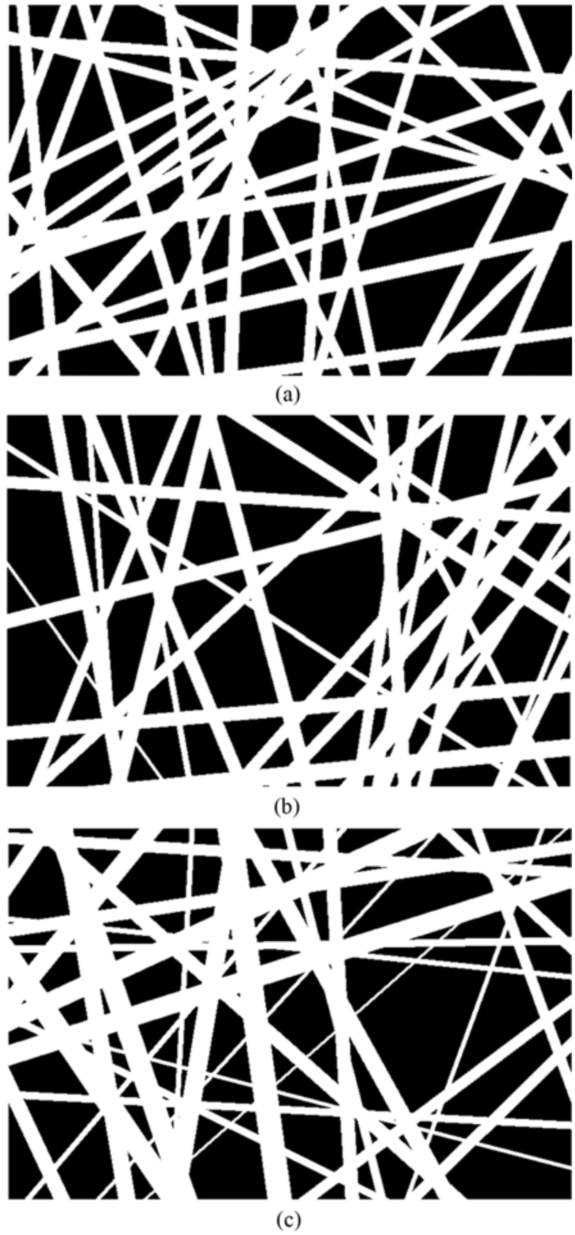

Fig. 7. Simulated images generated using μ-randomness procedure.

RESULTS AND DISCUSSION

Three simulated images generated by μ-randomness procedure were used as samples with known characteristics to demonstrate the validity of the techniques. They were each produced by 30 randomly oriented lines with varied diameters sampled from normal distributions with mean of 15 pixels and standard deviation of 2, 4

Table 1. Structural characteristics of the simulated images generated using μ-randomness procedure

No.	Angular range	Line density	Line thickness	
			M	Std
1	0-360	30	15	2
2	0-360	30	15	4
3	0-360	30	15	8

Table 2. Mean and standard deviation of fiber diameters for the simulated images

		No. 1	No. 2	No. 3
Simulation	M	15.247	15.350	15.367
	Std	1.998	4.466	8.129
Distance transform	M	16.517	16.593	17.865
	Std	5.350	6.165	9.553
Direct tracking	M	16.075	15.803	16.770
	Std	2.606	5.007	9.319

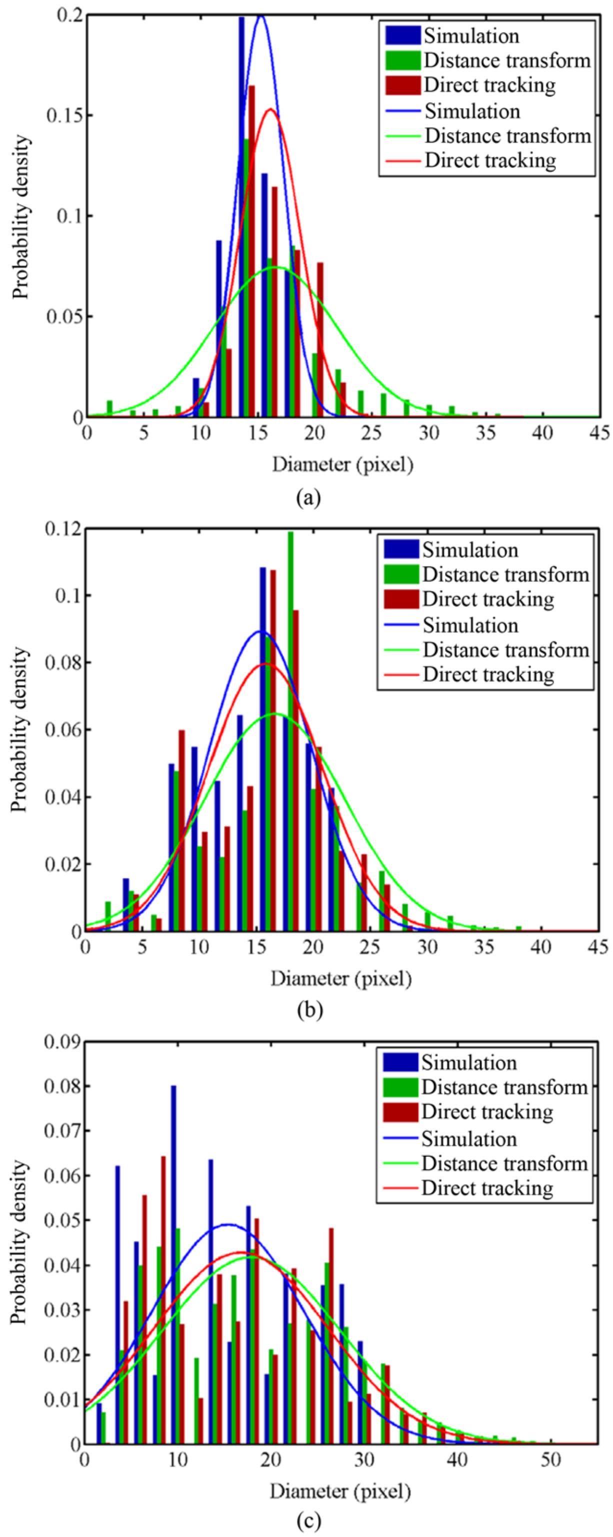

Fig. 8. Histograms of fiber diameter distribution for the simulated images.

and 8 pixels, respectively. Table 1 summarizes the structural features of these simulated images which are shown in Fig. 7.

Mean and standard deviation of fiber diameters for the simulated images obtained by direct tracking as well as distance transform are listed in Table 2. Fig. 8 shows histograms of fiber diameter distribution for the simulated images obtained by the two methods. To make a true comparison, the original distribution of fiber diameter in each simulated image is also included. The line over each histogram is related to the fitted normal distribution to the corresponding fiber diameters.

Table 2 and Fig. 8 clearly demonstrate that for all simulated webs the direct tracking method resulted in mean and standard deviation of fiber diameters which are closer to those of the corresponding simulated image (the true ones). Distance transform method is far from making reliable and accurate measurements. This may be due to some branches remaining in the skeleton even after pruning. The thicker the line, the higher the possibility of branching during skeletonization (or thinning). Although these branches are small, their orientation is typically normal to the fiber axis, thus causing a widening of the distribution obtained by distance transform method.

CONCLUSION

Fiber diameter is one of the most important structural characteristics in electrospun nanofiber webs. Electrospun nanofiber diameter is often measured by manual method, a labor intensive, time consuming, operator-based technique which only utilizes a low number of measurements and is thereby inefficient for automated systems, e.g., online quality control. In this study, an automated technique called direct tracking for measuring electrospun nanofiber diameter has been developed that is fast and has the capacity for automation, enabling improved quality control of large-scale electrospinning operations.

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