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Determination of Electrospun Fiber Diameter Distributions Using Image Analysis Processing

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Abstract: An image analysis processing method for the measurement of nanofiber diameter was developed. For the analysis, scanning electron microscopy (SEM) images of electrospun fiber were prepared and applied to the individual measurement of the fiber diameter by using the developed and the traditional manual methods. Both methods provided a similar fiber distribution. The fiber average diameters were similar but the variance of the new method was larger than that of the manual method. The average diameters from the two methods exhibited a linear relationship with a high coefficient. The developed method can be used as a practical tool to estimate the fiber diameter of the electrospun web.

Keywords: nanofiber, fiber diameter distribution, image analysis.

Introduction

Electrospinning is one of the most efficient processes for nanofiber production by charging high voltage to polymer solutions or melts. It is considered as the most potential techniques for industrialization due to its simple mechanism and various applicable polymers.

Previous researches in electrospinining have been focused on the effects of material properties and processing parameters on the spinability and the diameters of spun fibers using various polymers and solvents.[1-10] It was found that the fiber diameter was generally decreased to a certain limit as the applied voltage and TCD (Tip to Collector Distance) increased. However the fiber diameter was increased over a certain TCD level while higher voltage did not show significant effect on the fiber diameter. The effects can be explained in terms of the electrical field strength. Also the effects of material parameters were studied in terms of the concentration, viscosity and surface tension of polymer solution using various polymers and solutions. They found that the fiber diameter of the electrospun web was strongly depending on the polymer concentration, viscosity and surface tension.

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Although the researches provided fundamental understandings of the process, they have several drawbacks. One of the most significant drawbacks is that the fiber diameters and its distribution were measured from SEM images in most of researches. To obtain statistically reliable data, it is necessary to take large number of pictures on various places of the web and to measure the each fiber diameters. Although it is a painstaking and time-consuming job, the data are still unreliable due to small sampling areas compared with the total web size. The similar problem frequently happens in nonwovens. The morphological parameters such as fiber diameter, pore and fiber orientation show critical effect on the physical properties and its applications. To characterize and simulate the morphology, various image analyses were developed for each parameter.[11-15] Pourdeyhimi and his colleagues[15] suggested an image analysis for fiber diameter measurement using transmitted microscopic image from thin web. This process could not distinguish the individual fibers when the fibers were overlapped. It may overestimate the fiber diameter on the fiber overlapped areas.

In order to provide more precise characterization of the electrospun web morphology and structure, we developed a fiber diameter measuring process for nanofiber web using image analysis method from SEM image. The developed

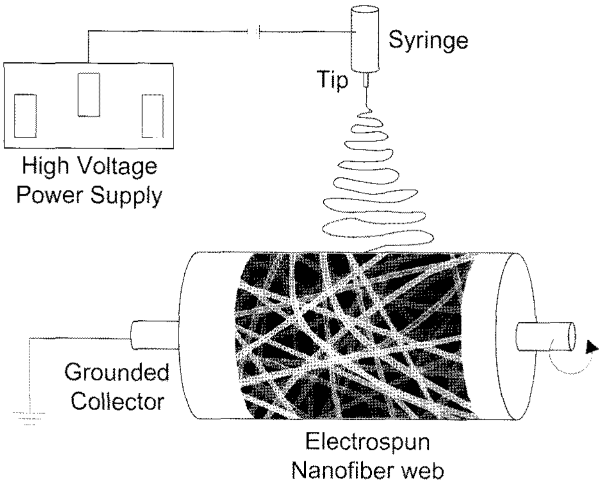


Figure 1. Schematic diagram for electrospinning apparatus.

Table I. Processing Parameters for Electrospinning

Variance		Level	
Concentration (wt%)		20	
Applied Pressure (MPa)	0.10	0.15	0.20
Spinning Time (hour)	1	2	3

method can estimate the fiber diameter from thick web by separating the desirable layer from the image. Also the method can distinguish the fiber boundaries when the fibers are overlapped or superposed each others. As a result we can estimate the fiber diameter precisely without overestimation. The validity of the method was tested by comparing the results from manual method and the developed method.

Image Acquisition. Nanofiber webs were prepared using 20 wt% PAN/DMF solution via electrospinning process at 28 kV as shown in Figure 1. Table I summarized the electrospinning processing condition.

The nanofiber web images for the image process were obtained using SEM (Hitachi, S-4700).

Image Analysis Processing. Figure 2 shows a flow chart of the image analysis process developed in the study. The analysis was carried out in two phases. In the first phase, the boundaries of each fiber on the image were detected. The boundary can easily be detected from binary images, which were obtained from the thresholded SEM images. However, the fiber boundaries at fiber-to-fiber cross-over area are difficult to be identified from the binary image. To separate the each fiber boundary at the fiber-to-fiber cross-over areas, Canny Edge detecting method[16-19] was applied.

In the second phase, the fiber center line was defined using skeletonization.[16-19] The distance from the center to the boundary was calculated using distance transform.[17-19] The detail of each process is explained in the following sections.

Fiber Boundary Detection. Figure 3 shows the images transformed by the image analysis.

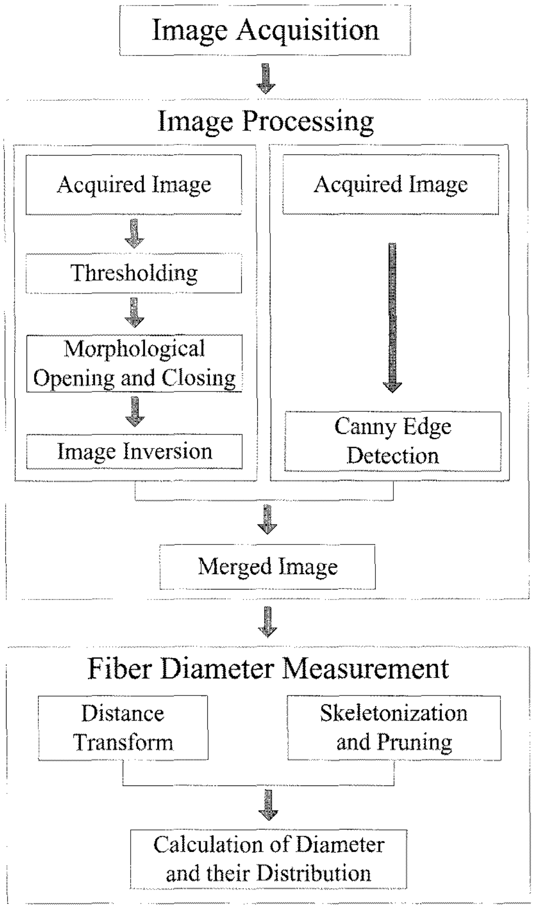


Figure 2. Flowchart of the image process.

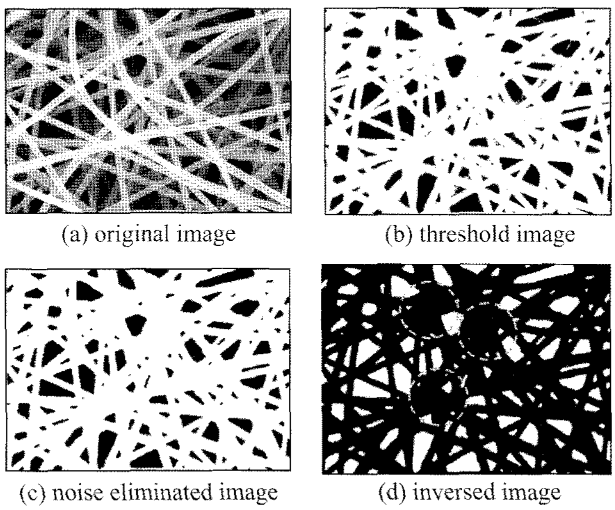


Figure 3. Processed images by the image analysis.

Figure 3(a) shows an original SEM image used for the analysis. By thresholding process,[18-20] the image was transformed into binary image, which is black and white (Figure 3(b)). The SEM image gives clear binary image when the threshold value lies between 0.2 and 0.25. We kept the threshold value as 0.21 in this study. The smaller value generally results in overestimation of the fiber thickness. The image after thresholding still contained undesirable noise and images out of focus, which can be the source of significant error in the analysis. To remove the noise and the images out of focus, morphological opening and closing was carried out repeatedly and the resulted image was shown in Figure 3(c). For further analysis, the image was inversed as illustrated in Figure 3(d). As a result, black pixels stand for fiber and white pixels stand for pore.

Fiber Individualization. When fibers were located in the superposed areas such as the circled parts in Figure 3(d), it is impossible to separate the superposed fibers into individual fibers without any treatment. Without defining the each fiber boundaries, the fiber diameters in those areas can be overestimated. The fiber boundaries in those areas were distinguished using Canny edge detection,[17-20] which is effective for gradient boundary. From the original image, the local gradient and edge direction were computed at each direction using Sobel method. The gradient of a 2-dimensional function, $f(x, y)$, is defined as the vector.

$$\nabla f = \begin{bmatrix} G_x \\ G_y \end{bmatrix} = \begin{bmatrix} \frac{\partial f}{\partial x} \\ \frac{\partial f}{\partial y} \end{bmatrix} \tag{1}$$

The magnitude of this vector is

$$\nabla f = mag(\nabla f) = [G_x^2 + G_y^2]^{1/2} \approx |G_x| + |G_y| \tag{2}$$

An edge point is defined as a point of which strength has local maximum in the direction of the gradient. The edge direction is calculated by

$$\alpha(x,y) = \tan^{-1}\left(\frac{G_y}{G_x}\right) \tag{3}$$

Figure 4 shows the results of Canny edge detection using the image in Figure 3(a). It was overlaid on Figure 3(d) for

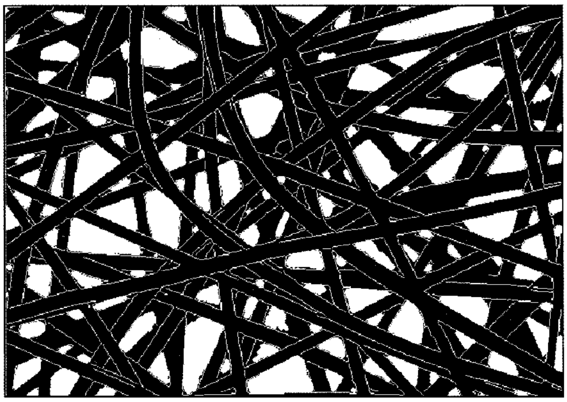


Figure 4. Canny edge detection results superposed on the inversed image.

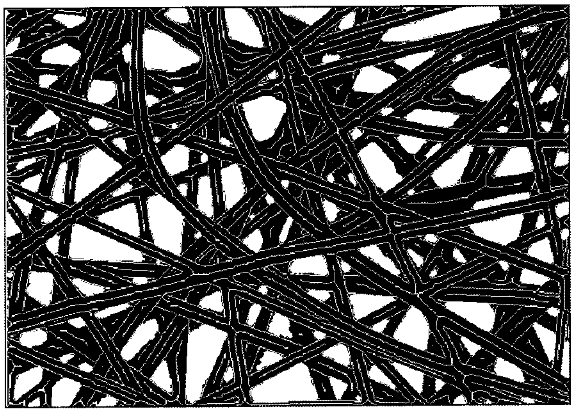


Figure 5. Skeletonized and pruned image.

better understanding.

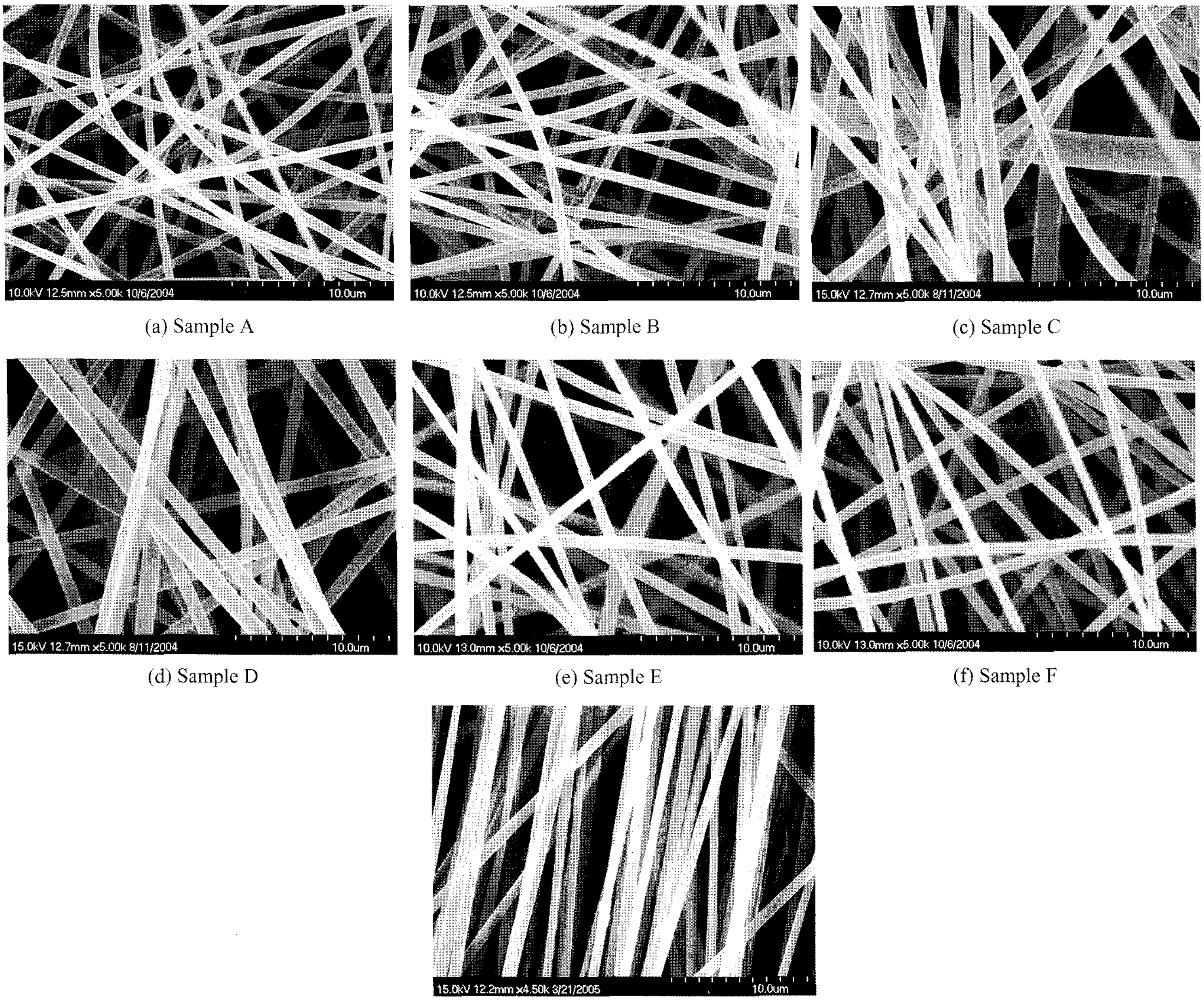
Distance Transform. The fiber diameter can be estimated from the distance from the fiber center line to the fiber boundary. The fiber center line was defined using skeletonization process. Although we obtained a single center line in the middle of the fiber by the process, the skeletonized line is split into two lines as a shape of 'Y' at the both ends of the fiber. The 'Y' shaped line was converted into a single line by pruning process.[18-20] The skeletonized and pruned image was superposed on the binary image as shown in Figure 5.

The distance between the center line and the boundary can be calculated by direct method. However, for efficient computation the distance matrix was applied in the study. Based on the direct calculation, the distance from a certain pixel to every neighboring pixel can be obtained, which allows a distance matrix. According to the distance definition, several distance transforms are available, such as Euclidean, City block, Chess board, Quasi-Euclidean.[17-20] In the study, we used Euclidean transform matrix to calculate the distance between neighboring boundaries obtained from Canny edge detection algorithm.

Comparison of Manual Analysis to Image Analysis. In order to test the reliability of the developed method, the fiber diameter distributions of several electrospun web images (Figure 6) determined by manual and the developed methods were compared.

The averages and variances of fiber diameters for each image obtained by both methods were summarized in Table II.

The average diameters from both methods gave similar values for every case. It revealed that the new method can determine the average fiber diameter in correct. However they showed small difference in their variance. It can be explained in terms of sample number used for population variation estimation. While small number of sample was used in manual analysis, the developed process used at least 5000 measurements for the variation estimation. That means the developed method as well as the manual measurement can equally be good estimating tools for fiber average diam-



(a) Sample A (b) Sample B (c) Sample C

(d) Sample D (e) Sample E (f) Sample F

(g) Sample G

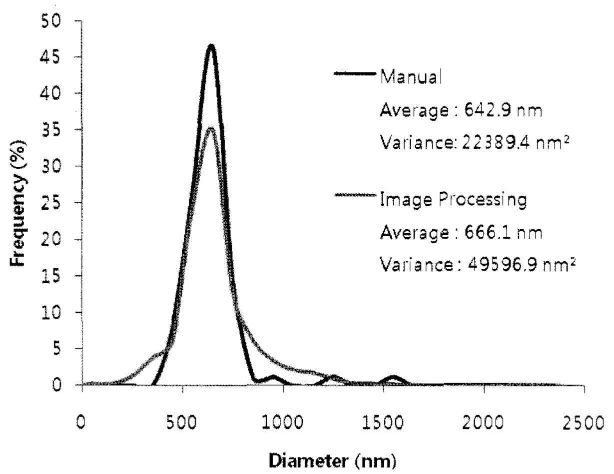
Figure 6. SEM image of electrospun fiberweb for the comparison.

Table II. Comparison of Results from the Two Methods for Samples

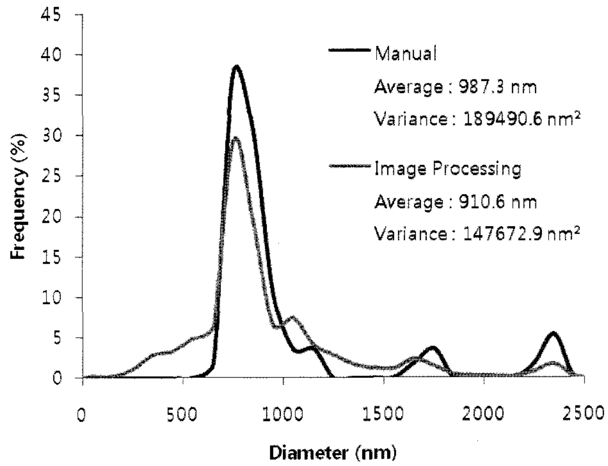
Sample	Manual Measurement		The Image Processing Method	
	Mean (nm)	Variance (nm)	Mean (nm)	Variance (nm)
A	571.5	13417.6	598.4	16974.9
B	642.9	22389.4	666.1	49596.9
C	987.3	189498.6	910.64	147672.9
D	906.9	43264.6	870.2	81618.7
E	739.7	41122.4	737.6	54933.9
F	735.2	6597.2	728.7	33717.1
G	670.9	27148.0	628.9	50002.2

eter. However, due to large sampling number, the developed method is supposed to be a better estimating tool for population variation.

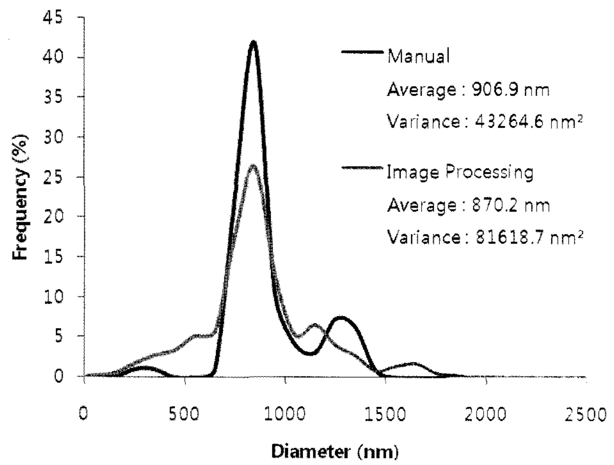
For close comparison, the fiber diameter distribution of samples B, C, D and G were measured using the two methods as shown in Figure 7. Both the manual and the devel-



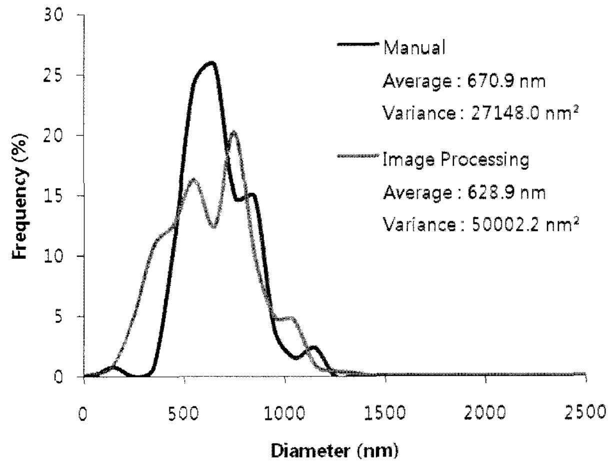
(a) Comparison of results from the two methods for Sample B



(b) Comparison of results from the two methods for Sample C



(c) Comparison of results from the two methods for Sample D



(d) Comparison of results from the two methods for Sample G

Figure 7. Comparison of results from the two methods.

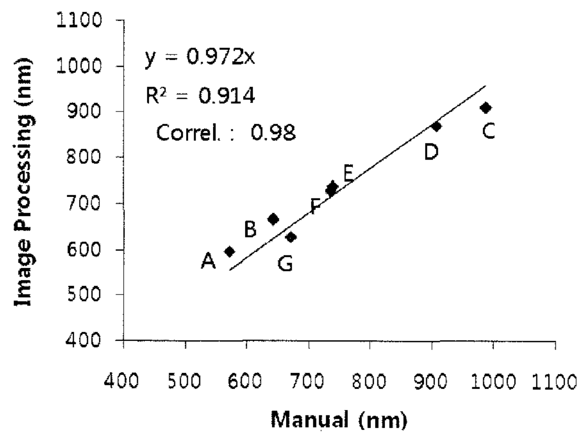


Figure 8. The linear regression result between the fiber average diameters obtained by the two methods.

oped methods showed similar fiber diameter distributions for sample B, C, D and G. The modes of each distribution were found in similar values and the shapes of the distribution were almost same. The measured diameter by the developed method showed broader distribution than those by the manual method. However, the two methods gave significant difference in the fiber diameter distributions for sample G. It can be explained in terms of fiber orientation. The fibers in sample G were highly oriented in a certain direction. Due to the orientation, the fiber boundaries were not clearly distinguished by Canny edge detection.

Figure 8 shows the linear regression result between the fiber average diameters obtained by the two methods. In regression analysis, the intersection with y-axis was controlled as 0. The result shows significant linear relationship with high coefficient of determination ($R^2 = 0.914$). The slope is almost 1 (slope = 0.972), which means that the two methods gives the same values.

Conclusions

The developed method using image analysis technique successfully estimate the fiber diameters produced by electrospinning. The developed method and the manual method did not showed significant difference in the fiber diameter distributions. Both the methods provide similar fiber average diameters, while the variances from the image analysis were 50-200% larger than those from the manual method. The regression analysis confirms that two methods give the same values. It reveals that the developed method can be used as a practical tool to estimate the fiber diameter of electrospun web.

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