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Investigation of Image Analysis in the Characterization of Electrospun Polycaprolactone/Graphene Oxide/Fe_3O_4 Nanocomposites

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In this study, one of the most promising methods of tailoring a composite scaffold material in nanosized diameters, electrospinning method were used to fabricate polycaprolactone (PCL)/ graphene oxide (GO)/iron(II,III) oxide (Fe_3O_4) nanocomposite fibers as biocompatible scaffolds for biomedical applications. The prepared nanofibers were characterized using the scanning electron microscopy. Grayscale scanning electron microscopy images of composite nanofibers were analyzed by a new image analysis procedure for determination of fibers diameter, diameter distribution, orientation of nanofibers and the compactness of electrospun nanofibers. The developed image analysis procedure consists of three phases, preprocessing phase, morphological operation phase and feature extraction phase. In first phase, preprocessing methods are applied to enhance scanning electron microscopy images. After preprocessing phase, grayscale images are converted into binary images. In second phase, morphological operations such as erosion, dilation are applied on binary regions. Then, region of interests are detected on binary images. In the feature extraction phase, morphological features are extracted and calculated from region of interests.

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PACS/topics: SEM images, image processing, graphene, electrospinning method

1. Introduction

Nanocomposites are kind of materials that may be multi-dimensional. At least one dimension of nanocomposite materials is at the nanoscale. There are various nanocomposite applications such as increasing the speed of the healing process for broken bones, producing structural components with a high strength-to-weight ratio, making lightweight sensors [1]. Electrospinning of polymers and polymeric nanocomposites is a useful method for processing and producing nanocomposite materials for biomedical applications such as scaffolds for tissue engineering [2].

Electrospinning is a feasible method to produce continuous fibers with a diameter from 10 nm to few micrometers [3]. This technique allows obtaining nanofiber structures with a large surface area to volume ratio, flexibility and high porosity [4, 5]. These features are important for biocompatibility by providing the familiar environment for cells to recognize, which results with better attachment and increased cell proliferation [6].

Polycaprolactone (PCL) is a thermoplastic polymer which is very popular in this field due to its desirable features such as being flexible, thermally stable, easy to process [7]. Graphene is the lightest material known with 1 square meter coming in at around 0.77 mg, the strongest compound discovered that is approximately 100–300 times stronger than steel and also the best conductor of electricity known [8].

Graphene oxide (GO) is one of the most important derivatives of graphene because of advantageous physical and biological features [9]. Iron oxides (Fe_3O_4) are chemically stable and superconductive nanoparticles which also has low cost.

Image processing is a method to perform some operations on an digital image, in order to get an enhanced image or to extract some useful information and features from the image. Morphologic image processing is a collection of non-linear operations related to the shape or morphology of features in an image. Image processing has a wide application area in biomatarials and biomedical engineering [10–14].

In this paper, character analysis of the bioactive PCL/GO/Fe_3O_4 nanocomposites is performed using image processing techniques. The developed method is accurate and faster than other methods for the character analysis process.

2. Developed image analysis procedure

The block diagram of the developed character analysis procedure is presented in Fig. 1. Nanofibers are de-

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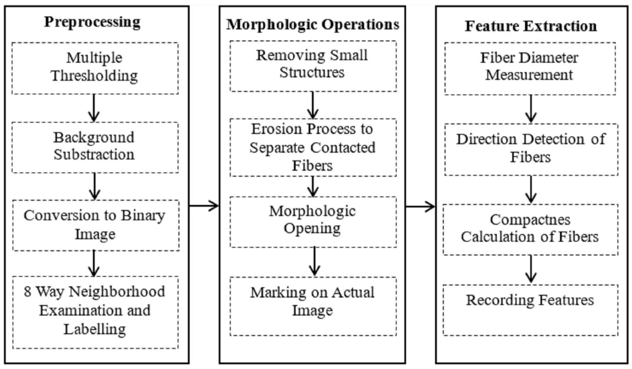

Fig. 1. Block diagram of the developed procedure.

tected and identified and labelled in preprocessing stage. Other structures are eliminated and removed from scanning electron microscopy (SEM) images in morphologic operation stage. In the feature extraction stage, diameters of labelled nanofibers are measured and their directions are detected. Also compactness of the nanofiber are calculated and all features are recorded in this stage.

3. Preprocessing stage

The preprocessing stage consists of four phases. In the first phase, a multiple thresholding method with two threshold values, a minimum and a maximum threshold value, was applied to SEM images to detect nanofiber candidate structures. The pixel values between the threshold values constitute the structures will be examined to decide nanofiber or not. Other pixel values will be equal to zero. After thresholding, background of the SEM image is subtracted to clarify foreground structures. Then, the subtracted SEM image converted into a binary image so that morphological image processing operations can be performed. A binary image consists only 1 and 0 pixel values. In last phase of preprocessing stage, binary structures are labelled using a neigborhood examination of pixel values with 1. This process is useful for recording the features of structures.

4. Morphologic image processing operations stage

In morphologic operations stage is the second stage of the developed method. In first phase of this stage, small structures which are non fibers are removed by morphologic erosion operation using a disk shape structure element with diameter size five pixels. Then, another erosion operation is carried out to separate the contacting fibers using a line structure element. After separation of contacted fibers, morphologic opening operation for different angles is performed to specify fibers for each angle. In last phase of the morphologic operations stage, pixel coordinates of detected fibers are recorded and marked on actual image.

5. Feature extraction stage

Feature extraction is the last stage of the developed system. In this stage, detected fibers on previous stage are examined. First, their diameters are measured and recorded. Then, number of fibers according to their direction is recorded. Compactness ratio of fibers are calculated. Formula of the compactness ratio is

$$C = 4\pi A/p^2,$$

where A is area and p is equivalent perimeter of the shape.

6. Experimental study

Experimental studies were performed to measure performance of the developed procedure. In order to perform experiments, SEM images of the polycaprolactone/graphene oxide/Fe_3O_4 nanocomposites are used. Samples for experiments were prepared using varying concentrations (0.02, 0.04, 0.06, 0.08, and 0.1 wt%) of GO. Experiment results indicates the developed procedure measures fiber diameters accurately. The procedure also detects directions of fibers and groups them according to their directions. The developed procedure performs these processes faster than other methods. Execution time of whole processes is less than 60 s. In Fig. 2a, a part of an original SEM image of the GO rate of 0.02% is shown. A binary image of after preprocessing stage is presented in (b). Skeleton of the nanofibers for morphologic operations is shown in (c). Detected nanofibers for direction of 135° is presented in (d). In Fig. 3, a chart of fiber groups according to diameter size is presented.

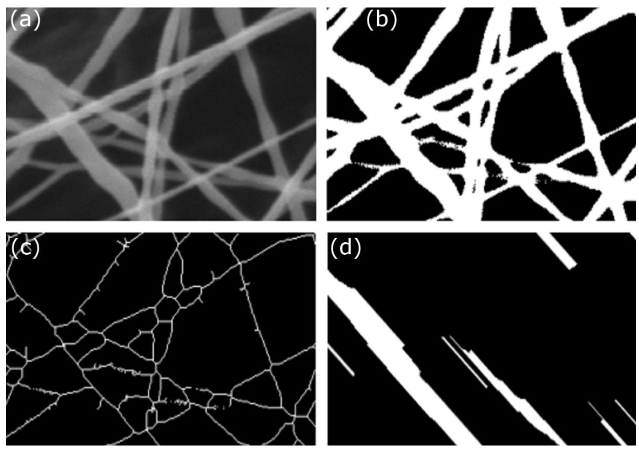

Fig. 2. (a) Original SEM, (b) thresholded binary image, (c) skeleton of the nanofibers, (d) parts of detected nanofibers of 135° angle.

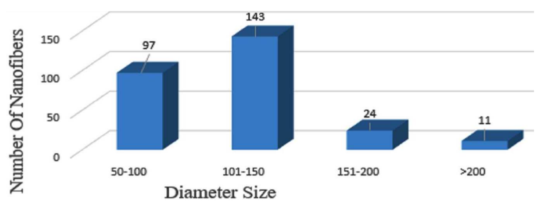

Fig. 3. Diameter size groups (nanometers).

7. Conclusion

In this study, character analysis of electrospun polycaprolactone/graphene oxide/Fe_3O_4 nanocomposites for biomedical applications is realized using image processing techniques. The developed image processing procedure measures diameters of fibers accurately and faster than classical methods on SEM images. Also the developed procedure detects orientation of fibers and calculates their compactness. Thus, the developed method facilitates the biomaterial characterization process. These advantages provided by the developed method will enable more accurate and rapid evaluation of experimental studies on nanocomposite biomaterial production.

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