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Towards an Automatic Classification of SEM Images of Nanomaterials Via a Deep Learning Approach

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Abstract. Nanofibrous materials produced by electrospinning process, may exhibit characteristic localized defects and anomalies (i.e. beads, speck of dust) that make the nanostructure a network of nonhomogeneous nanofibers, unsuitable for an industrial production at large scale of the nanoproducts. Therefore, monitoring and controlling the quality of nanomaterials production has become increasingly important and intelligent anomalies detection systems have been emerging. In this study, we propose an innovative framework based on machine (deep) learning for automatic anomaly detection. Specifically, a deep Convolutional Neural Network (CNN) is proposed to automatically classify Scanning Electron Microscope (SEM) images of homogeneous (HNF) and nonhomogeneous nanofibers (NHNF), interpreted as two different categories. The proposed approach has been validated on experimental SEM images acquired through SEM images analyzer on Polyvinylacetate (PVAc) nanofibers produced by electrospinning process. Experimental results showed that the designed deep CNN achieved accuracy rate up to 80% and average precision, recall, F_score of, 78.5%, 79%, 78.5% respectively. These promising results encourage the use of this effective technique in industrial production.

Keywords: SEM images, Nanomaterials, Deep Learning, Convolutional Neural Network

1 Introduction

Nanofibrous materials are ultra-fine fibers with diameters lower than 10^2 nm, typically produced by an effectiveness and efficient technique called *electrospinning* (or electrostatic spinning) [1]. In recent years, the applications of such nanostructures have attracted a great deal of interest in fields as biomedicine

[2], electronics [3], drug delivery and tissue engineering [4]. However, the production of nanofibers (NF) is still difficult to monitor as several parameters (i.e. applied voltage, polymeric concentration, temperature etc) may cause structural defects during the electrospinning process [5]. The most common defects are *beads*, namely, micro or nano polymeric particles obtained mainly when the concentration of the solution is very low. These anomalies reduce the large surface area per unit volume, which is a typical advantage of electrospinning over competing techniques of production, and influence time and cost of production. The most efficient approach of monitoring the electrospun material is acquiring Scanning Electron Microscope (SEM) images from the nanofibrous sample and analyzing its structure. As the expert visual inspection is not the most effective method to identify defects, also for practical reasons, there is an increasing interest in developing automatic anomalies detection systems (ADS) through the analysis of SEM images. However, a few works are reported in literature. Carrera et al. [6] used the so called *novelty detection* algorithm also known as one-class classification to address the issue of anomaly detection within SEM images. The dictionary of only normal (anomaly-free) images patches previously developed by Boracchi et al. [7] was employed. This dictionary was used during the test to identify defects in a patch-wise modality. Experimental results showed that the proposed method was able to outperform state-of-art algorithms (i.e. STSIM, Coding) and provided good performance in detecting small defects. Recently, advanced machine learning techniques known as *deep learning* (DL) have been also employed in this context [8]. It has been proved that DL algorithms achieve human-level performances in several real-world applications (i.e. speech recognition [9], biomedicine [10],[11], cybersecurity [12], nondestructive testing and evaluation [13]), so DL based systems for anomalies detection in SEM images have been emerging. Specifically, Carrera et al. [14] , proposed a DL method based on Convolutional Neural Network (CNN) for automatic detection and localization of defects. They used the ResNet-18 network pre-trained on scene and object images defined by the ILSVRC 2015 competition and built a dictionary of features vectors extracted from normal patches. Abnormal patches were evaluated through similarity between a testing patch and an anomaly-free patch of the dictionary. The performances were measured in terms of ROC curve and coverage factor. Experimental results show that the proposed framework outperformed the state-of-the-art of about 5%. However, similarly to their previous works, the authors developed the DL system through a one-class classifier. In this paper an Automatic Classification of SEM images of Nanomaterials Via a Deep Learning Approach is proposed. Specifically, a deep Convolutional Neural Network is developed to detect SEM images of homogeneous nanofibers (HNF, anomalies-free) and SEM images of nonhomogeneous nanofibers (NHNF, with defects). A dataset of 160 SEM images (85 NHNF and 75 HNF) of Polyvinylacetate (PVAc) nanofibers were manually collected after electrospinning process at the *Materials for Environmental and Energy Sustainability Laboratory* of the University Mediterranea of Reggio Calabria (Italy). Experimental results (Table 2) showed that the 2-way deep CNN classifier achieved accuracy up to 80%.

The rest of this paper is organized as follows. Section 2 describes the proposed methodology, including the electrospinning process, the experimental setup and the proposed deep CNN classifier. Section 3 discusses the results achieved. Section 4 concludes this paper.

2 Methodology

The flowchart of the procedure is illustrated in Figure 1. Firstly, the polymeric solution of PVAc dissolved in EtOH solvent is prepared and used to produce PVAc nanofibers by electrospinning process (Figure 1 (a)). The morphology of the electrospun nanofibers is examined by a scanning electron microscope, and SEM images sized 1024 x 1024 of homogeneous and not-homogeneous nanofibers are manually collected. Afterwards, given the jth SEM image, a sub-region (sized 128 x 128) is selected (Figure 1 (b)). This operation has been necessary due to the limitations of the available processor. Finally, a deep learning classifier based on CNN is employed to identify HNF and NHNF images (Figure 1 (c)).

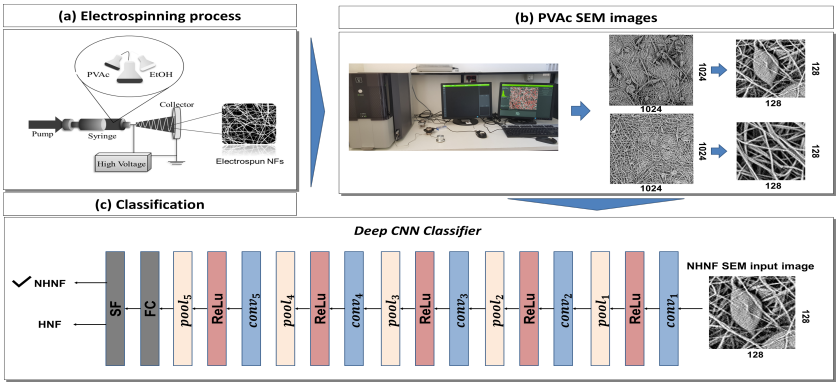

Fig. 1. Flowchart of the method proposed.

2.1 Electrospinning Process

The electrospinning apparatus is schematized in Figure 2. It includes three basic components: a high voltage supply, an extruder and a metallic collector screen. The polymeric solution is initially placed into a glass syringe and pushed through the metallic needle by the injection pump, which allows controlling the flow-rate. A high voltage is applied between the needle (anode) and the collector (cathode), which are electrostatically charged to a different electric potential. As the electric field increases, the formed droplet loses surface tension and takes the form of a cone, referred to as Taylor cone. When the electrostatic force exceeds the surface tension, the polymeric jet is stretched within the high electric field; meanwhile the solvent evaporates and is deposited on the collector in the form of nanofibers.

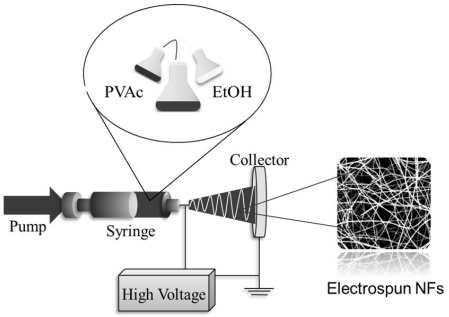

Fig. 2. Electrospinning setup.

The viscosity has a great influence on the jet and diameter of nanofibers. Specifically, when the concentration of the solution is very low, micro or nano polymeric particles are obtained and the electrospray phenomenon is observed [15]. However, applied voltage, tip-collector distance and flow-rate have also remarkable effects on the fibers [16].

Material In this study, Polyvinylacetate (PVAc; average molecular weight (Mw): 170,000) and Ethanol (EtOH) were used as polymer and solvent, respectively. The spinnable solution was prepared by dissolving PVAc in EtOH and stirring until a clear solution was obtained. The spinning process was carried out at 20±1 C temperature and 40% relative air humidity, using a CH-01 Electro spinner 2.0 (Linari Engineering s.r.l.) and a 20 mL syringe, equipped with a 40 mm long 0.8 mm gauge stainless steel needle. All reactants were supplied by Sigma-Aldrich. The effects of concentration (v_1), applied voltage (v_2), flow rate (v_3) and tip-collector distance (v_4) were evaluated to study the nanofibers production process. The Phenom Pro-X scanning electron microscope (SEM), equipped with an energy-dispersive x-ray (EDX) spectrometer, examined the morphology of the electrospun fibers. Finally, the average diameter, the distribution of the nanofibers and the detection of beads were obtained by using Fibermetric software (a SEM images analyzer).

Experimental Setup and Dataset Description Sixteen experiments (ξ_i, i=1,2,..,16) were carried out by changing one parameter at time in the following well-defined range of working: 10-25 wt.% concentration; 10-17.5 kV applied voltage; 100-300 μL/min flow rate; 10-15 cm TCD. Table 1 reports the details of the experiments. However, since the purpose of this study was the development of a classification system based on SEM images, the average diameter was not taken into account and it is not reported in Table 1.
Given the ith material sample under analysis, 10 significant and representative areas were arbitrarily selected and evaluated through the SEM images analyzer,

Table 1. Electrospinning setup of the 16 experiments.

ξ	**Concentration** (v_1) [$\%wt$]	**Applied Voltage** (v_2) [kV]	**Flow Rate** (v_3) [μL/min]	**TCD** (v_4) [cm]
ξ_1	10	15	10	100
ξ_2	15	10	10	100
ξ_3	15	13,5	10	100
ξ_4	15	15	10	100
ξ_5	15	15	10	200
ξ_6	15	15	10	300
ξ_7	15	15	12,5	100
ξ_8	15	15	13,5	100
ξ_9	15	15	15	100
ξ_{10}	20	10	10	100
ξ_{11}	20	11,5	10	100
ξ_{12}	20	13,5	10	100
ξ_{13}	20	15	10	100
ξ_{14}	20	16	10	100
ξ_{15}	20	17,5	10	100
ξ_{16}	25	15	10	100

in order to augment the dataset. A grand total of 160 SEM images sized 1024 x 1024 was collected. Each image was examined by an expert operator, and labeled as SEM image of homogeneous nanofibers (HNF) or nonhomogeneous nanofibers (NHNF). Specifically, 75 images were classified as HNF and 85 as NHNF. Figure 3 shows representative SEM images of NHNF and HNF achieved during the experiments. As can be observed, lower concentrations cause low viscosity of the solution, instability of the polymeric jet and consequently beads structures (Figure 3 a). With increasing TCD, the electrospun solution is affected by a less intense electric field and causes a mixed morphology of fibers and beads (Figure 3 b). Higher applied voltages produce smaller dimensions of beads as they are elongated and stretched by the higher electric field. Therefore, high voltages, together with high concentrations, produce homogeneous networks of nanofibers (Figure 3 c).
However, due to the computational limit of the available processor, we did not process the whole SEM sized 1024 x 1024. Given the jth SEM image belonging to NHNF or HNF class, it was firstly split into 64 sub-images sized 128 x 128, then one representative sub-image was selected and used as input of the proposed Convolutional Neural Network. It is worth noting that there is no downsampling of the original SEM image to prevent distortion of the size of nanofibers.

2.2 Convolutional Neural Network

Convolutional Neural Networks (CNNs) are deep learning architectures able to learn discriminating features directly from raw input data through a deep hierarchical organization. A typical CNN consists of subsequent modules of *convolution*, *activation* and *pooling* layers. The convolutional layer performs the convo-

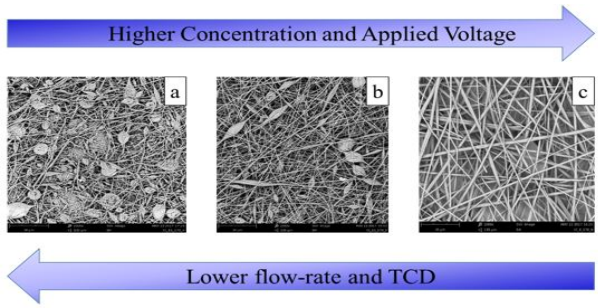

Fig. 3. Effect of the parameters variation on the morphology. (a-b) SEM images of not-homogeneous nanofibers (NHNF) due to the presence of beads. (c) SEM image of homogeneous nanofibers (HNF).

lution operation between a set of F_j learnable filters (or kernels) and the input map $I_i \in R^{hxw}$. The result is the so called *features map* $O_j = \sum I_i * F_j + B_j$ where B_j is the bias term and * indicates the convolution operator. Each filter convolves with a local area of I_i and then scans the whole plane with a stride s, sharing the same values of weights. The O_j features map is sized o_1 x o_2 where $o_1 = \frac{h-f_1+2p}{s} + 1$, $o_2 = \frac{w-f_2+2p}{s} + 1$ and p is the zero padding parameter used to control the output size by padding the input edges with zeros. The convolutional layer is typically followed by the "*Rectified Linear Unit*" (ReLu, $f(z) = max(0, z)$) activation function, as it aids the system in generalization and improves learning time [17]. The pooling layer reduces the input features map through an average (*average pooling*) or maximum (*max pooling*) operation. In this study, the max pooling operation is used. The F_j filter scans the input features map with stride $\tilde{s}$ producing a sub-sampled representation of O_j sized $\tilde{o}_1$ x $\tilde{o}_2$ where $\tilde{o}_1 = \frac{y_1-\tilde{f}_1}{\tilde{s}} + 1$ and $\tilde{o}_2 = \frac{o_2-\tilde{f}_2}{\tilde{s}} + 1$. Finally, the learned features are the input of a standard multi-layer neural network (MLP) for classification task.

Architecture Proposed and Learning Setup Figure 1 (c) shows the schematic of the proposed deep CNN . It contains 5 convolutional (*conv*) layers, 5 max pooling (*pool*), 1 fully connected layer (*FC*) with 40 hidden neurons and 1 softmax (*SF*) output layer for binary classification task (NHNF - HNF). All convolutional layers have filters size of 3 x 3, stride s=1 and padding parameter p=1; whereas, all max pooling layers have filters size of 2 x 2 and stride s=2. The rectified linear unit is employed as activation function and batch normalization is applied to the hidden layers to avoid covariate shift phenomenon [18]. The details of architecture configuration are reported in Table 2. Learning set up was based on the practical recommendations of [19], [20]. The layers are initialized from a Gaussian distribution with zero mean and standard deviation of 10^{-2}. The stochastic gradient descent (SGD) algorithm with momentum of 0.9, weight decay of 10^{-4}, learning rate of 0.001 and mini-batch size of 32 shuffled SEM im-

ages is used to train the deep neural network. The network was implemented using MATLAB R2017b (The MathWorks, Inc., Natick, MA, USA) and trained for 300 iterations on a single CPU of HP xw4600 workstation with 12 GB RAM.

Table 2. Layers configuration of the deep CNN proposed.

	conv1	**pool1**	**conv2**	**pool2**	**conv3**	**pool3**	**conv4**	**pool4**	**conv5**	**pool5**	**FC1**	**FC2**
Input (I)	128 x 128	128 x 128	64 x 64	64 x 64	32 x 32	32 x 32	16 x 16	16 x 16	8 x 8	4 x 4	2048 x 1	40 x 1
Number of filters (F)	16	16	32	32	64	64	96	96	128	128	-	-
Size of filters (f1 x f2)	3 x 3	2 x 2	3 x 3	2 x 2	3 x 3	2 x 2	3 x 3	2 x 2	3 x 3	2 x 2	-	-
Stride (s)	1	2	1	2	1	2	1	2	1	2	-	-
Padding (p)	1	-	1	-	1	-	1	-	1	-	-	-
Output (O)	128 x 128	64 x 64	64 x 64	32 x 32	32 x 32	16 x 16	16 x 16	8 x 8	8 x 8	4 x 4	40 x 1	2 x 1

3 Experimental results

The dataset included 160 SEM images (75 HNF and 85 NHNF) sized 128 x 128 (Subsection 2.1, *Experimental setup and Dataset Description*). A subset of dataset was used to train the deep CNN (70% training dataset) and the remaining 30% was used to test the trained model. The performance of the proposed deep CNN was evaluated using standard metrics: precision, recall, F-score and accuracy:

$$Precision = \frac{TP}{TP + FP} \tag{1}$$

$$Recall = \frac{TP}{TP + FN} \tag{2}$$

$$F_score = 2 * \frac{Precision * Recall}{Precision + Recall} \tag{3}$$

$$Accuracy = \frac{TP + TN}{TP + TN + FP + FN} \tag{4}$$

where true positives (TP) represent the number of SEM images correctly identified as images of nonhomogeneous nanofibers; true negatives (TN) represent the number of SEM images correctly identified as images of homogenous nanofibers (anomalies-free); false positives (FP) are the number of normal images erroneously identified as images of nanofibers nonhomogeneous (with anomalies); false negatives (FN) are the number of anomalies SEM images missclassified as anomalies-free. The best CNN configuration was chosen by evaluating the effects on the performance of changing the number of hidden layers. Table 2 reports the outcome of the experiments on test set. Experimental results showed that the CNN_1 architecture with only one module of convolution ($conv_1$), ReLU ($relu_1$) and pooling ($pool_1$) layer produced the lowest performance (50% accuracy) and was not capable of discriminating SEM images anomalies-free and with defects;

whereas, the best classification result was reached with CNN_5 achieving accuracy rate up to 72%. All the networks (CNN_1 - CNN_5) included a softmax output layer for binary classification. However, it was observed that the accuracy performance improved of 8% by adding a fully connected layer with 40 hidden neurons and ReLu activation function. As can be observed in Table 4, the $CNN_5 + FC + SF$ architecture achieved accuracy rate up to 80% and average precision, recall and F_score of, 78.5%, 79%, 78.5% respectively. To our best knowledge, this is the first work on classification among SEM images of homogeneous (anomaly-free) and nonhomogeneous (with anomaly) PVAc nanofibers (produced by electrospinning process) by using DL techniques. However, it woth mentioning that, recently, Napoletano et al. [14] proposed a per-pixel one-class classification based on CNN but for detection and localization of defects within SEM images. Specifically, the proposed patch-wise based method identifies defects through visual similarity between the test-patch under analysis and a reference dictionary of normal subregions.

Table 3. Accuracy performance of deep CNN with different numbers of hidden layers.

CNN architecture	**Accuracy** [%]
CNN_1 [$conv_1$+$relu_1$+$pool_1$]+SF	50
CNN_2 [CNN_1+$conv_2$+$relu_2$+$pool_2$] + SF	66
CNN_3 [CNN_2+$conv_3$+$relu_3$+$pool_3$] + SF	68
CNN_4 [CNN_3+$conv_4$+$relu_4$+$pool_4$] + SF	70
CNN_5 [CNN_4+$conv_5$+$relu_5$+$pool_5$] + SF	72
CNN_5^* [CNN_4+$conv_5$+$relu_5$+$pool_5$] + FC +SF	80

Table 4. Precision, Recall, F_score of the deep CNN_5^* classifier.

SEM image	**Precision** [%]	**Recall** [%]	**F_score** [%]
NHNF	84	82	83
HNF	73	76	74
Average	78.5	79	78.5

4 Conclusions

In this paper, we proposed a deep learning based system to detect anomalies in Scanning Electron Microscope (SEM) images of nanofibrous materials produced by electrospinning process. Specifically, a deep convolutional neural network was developed to classify SEM images of homogeneous (HNF) and nonhomogeneous nanofibers (NHNF). The polyvinylacetate (PVAc, Mw 170,000) dissolved in ethanol solvent was electrospun at the *Materials for Environmental and Energy Sustainability Laboratory* of the University Mediterranea of Reggio

Calabria (Italy) and 16 experiments were carried out under different experimental conditions. A total of 160 images of PVAc nanofibers was extracted from the scanning electron microscope according the procedure described in Section 2 (*Experimental setup and Dataset Description*). A deep CNN was employed to learn the most relevant features from the raw SEM input data and classify PVAc HNF and NHNF images. Figure 4 shows examples of the features learned by $conv_1$ and $conv_3$ layer on a NHNF and HNF SEM image sized 128 x 128. Experimental results showed that the proposed deep CNN was able to correctly discriminate HNF and NHNF with accuracy up to 80%. However, it is to be noted that this study supposes to be a preliminary work for a more accurate and versatile system. Future works will address the limit of SEM image processing sized 1024 x 1024 (and above). Moreover, a larger number of experiments on PVAc polymer will be performed through electrospinning process. In addition, in the future, we intend to integrate the work here presented with the methodology presented in [14] for detection and localization of defects in SEM images.

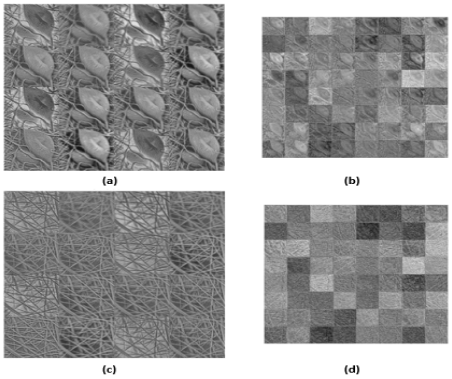

Fig. 4. Features maps learned by $conv_1$ and $conv_3$ on a SEM image input of homogeneous (HNF) and nonhomogeneous nanofibers (NHNF). (a-c) 16 feature maps sized 128 x 128 learned by $conv_1$ of NHNF and HNF, respectively. (b-d) 64 feature maps sized 32 x 32 learned by $conv_3$ of NHNF and HNF, respectively.

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