A novel method for measuring the porosity and pore size of nanowebs

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Method Article

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Nano-CT as a novel method for porosity measurements of nanowebs

Abstract

Nanofiber layers have privileged properties such as fine fiber diameter, small pores, suitable relationship between pores, special surface area and high porosity. These properties make nanofibers applicable for a variety of applications, such as high-performance filters and tissue engineering scaffolds. Different applications of electrospinning layers require different porosities. Therefore, it is very important to measure the porosity of nanofiber layers to express their application. To date, many methods have been expressed for this purpose, but all methods have disadvantages. Therefore, presenting the best method is a challenge that will be addressed in this project.

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1. Introduction

In recent years, multifunctional properties have been expressed for nanofibers obtained from polymers, metal composites and metal oxides. Additionally, surface modification nanofibers are easy to produce, economical and comfortable. This is due to the specific properties of nanofibers, which include high tensile strength, high specific surface area (surface area per unit mass) and porosity[1, 2]. Nanofibers have a high length-to-diameter ratio [3]. This makes nanofiber properties important for high-performance filters, absorbent textiles, medical textiles, drug release and many other applications[1,4].

Electrospinning is a continuous method for the continuous production of nanofibers with diameters below micrometers to nanometers. This process can produce layers with a high specific surface area, high porosity and good mechanical properties[5].

Electrospinning is a method widely used in the preparation of scaffolds. Various patterns of electrolyzed nanofibers are used to prepare layers that have medical applications, from artificial skin to endocrine organs and from the nervous system to cardiovascular applications[6].

Electrospinning layers have different porosities according to their application. In some applications, porosity is needed less than usual, and in some cases, it requires more porosity than usual. Therefore, there are different methods to achieve sufficient porosity.

In this project, a suitable solution for measuring porosity will be presented.

1.1. Electrospinning

Electrospinning is the only method of preparing large-scale nanofibers. The reason is comfortable control, high speed, minimum solution consumption, control of diameter and pores and makeup of fibers, process comfort, inexpensive, simple and reproducible fiber production process and technical advances [7]. This process can use a variety of polymers to obtain polymer fibers in the submicron range, which is difficult to achieve by conventional spinning methods [8]. Many parameters, including environmental and soluble parameters and processes, affect electrospinning. Environmental parameters include relative temperature and humidity. Soluble parameters include concentration, conductivity, molecular weight and viscosity. Process parameters also include feeding rate, voltage and needle head distance to the collector. With increasing temperature, the diameter of fibers decreases, and with increasing humidity, the diameter increases[7, 6]. The diameter of the electrospun fibers ranged from 10 nm to 100 μm. In most cases, polymer solutions are used for spinning electricity, but in some cases, polymeric melts with higher direct current voltages can also be used to produce fibers with diameters less than micrometers[6].

1.2. Porosity

Porosity is the amount of air, gas or vacuum in a solid material, often as the percentage of nonsolid portion volume on the total volume (total solid and nonsolid volume) of a unit of matter[14]. The porosity of the ratio of fluid volume or empty space in the filter environment is based on the total volume of the filter and therefore has no unit, and its value can be from zero to one. Porosity is one of the most important parameters in the design and operation of filters. The porosity and sufficient surface area of the nanofiber layer have made nanofiber coatings the most important candidates for high-performance filters. [12] Porosity, pore size distribution and membrane bending make it easy to pass steam through the membrane and collect steam as a filter outlet[9]. Previous studies have shown that the thickness and porosity of the nanofiber layer can be controlled by changing the sedimentation rate of fibers. The total porosity in the electrospinning layers is related to the pore space in the layer[12].

Nonwoven materials have a pore structure, and this feature is very important in their application. There can be three types of vents in matter. Package vents are not accessible. Restricts the blind pores inside the material and does not allow fluid to pass through. Open pores are outwards and allow fluid to pass. Open pores are useful for many

applications of nonwoven textiles. Their main characteristics are the highest stenification of the pore diameter, the largest pore diameter, the distribution of pores, the surface area, the permutation of gas and the perimeter of the liquid.

2. Discussion

In this chapter, the best method of measuring porosity and summarizing the previous parts are discussed.

Among the porosity measurement methods that were discussed in the previous season, all had disadvantages. Methods based on sample liquidizers had more difficulty in making the sample, which caused changes in structure and errors in measurement. In addition, non-wetting liquids can also rupture due to the need for pressure, which can change the structure of the layer and can even rupture it. The density method is also in error due to the need to measure dimensions, and the scale is necessary with appropriate accuracy. Of course, SEM images can be used accurately to measure dimensions, but in any case it is some a problem to measure and in addition to the thickness of the layers is not uniform and because the dimensions are too large, then the error increases, on the other hand, many methods are incapable of measuring the pore size of the pores, i.e., much smaller than the pores of nanofiber layers. It is suitable for the pores of the fork itself. SEM images are two-dimensional and the layers are stacked together and still question how to consider pores, the best method is nano-CT scan, but this device is not in many places and its advantages are measuring pore size by machine and operator, the method gives a completely intuitive accurate method of layer structure and is very precise. Nanofiber imaging with micro-CT scanners is not possible due to the thickness limit of at least 200 μm, and it is good for microscale images, not nanoscale images.

3. Experiments

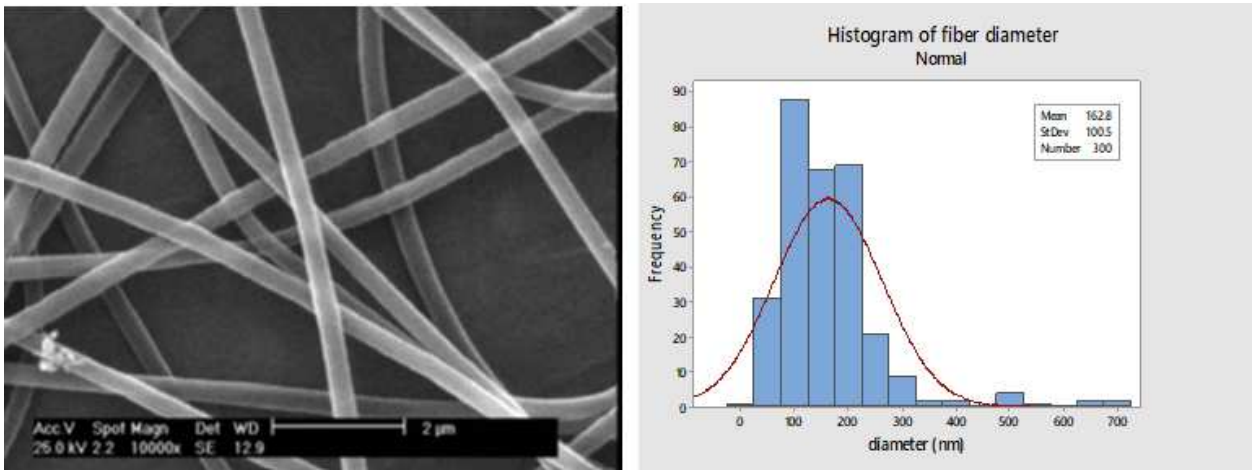
3.1. Materials

PA6 pellets with a melt flow index of 26.3 g/10min with textile grade prepared from Parsilon Khorramabad company (Iran). Formic acid and n-butanol with analytical grade prepared from Merck Company. 1 nozzle horizontal electrospinning (digital pump from DAWHA company with MS-2211 model, drum collector, voltage supply with voltage range of 1-20kV) used to prepare (polyamide 6) PA6 nanoweb. The electrospinning condition was 15wt%, 15cm, 0.5ml/h and 20kV. Electrospinning continued for 4h.

3.2. SEM

SEM images of the nanofibers prepared from Seron Technology (South Korea, AIS2100 model) microscope. Nanober's diameter is measured by ImageJ software. The average of 100 nanofiber diameters used (figure 1).

Figure 1. The SEM images of the nanofiber web.



3.3. Nano-computed tomography (Nano-CT)

The electrospun fibers were placed inside a 1 mm Kapton tube (DuPont, Shanghai, China). All specimens were scanned by a nano-CT (SkyScan 2211 Multiscale X-ray Nano-CT System, Bruker micro-CT, Kontich, Belgium) with a 20–190 kV tungsten X-ray source and a dual detection system: an 11- megapixel cooled 4,032 × 2,670-pixel CCD-camera and a 3-megapixel 1,920 × 1,536 pixel CMOS flat panel. The specimens were scanned at 40 kV, 300 µA and 1000 ms over 360^{o} with a rotation step of 0.290°, leading to a final voxel size of 400 nm. The scan duration for samples was about one hours. Nano-CT projections were reconstructed using the system-provided software. NRecon (version 1.7.4.6) with smoothing kernel 2, ring artifact correction 9, and beam hardening correction of 20%. The 3D image sets were visualized with CTAn (Bruker micro-CT, Kontich, Belgium, version 1.18.4.0).

4. Results

As the above nano-CT images could give the real pore size and porosity of the nanowebs, this is the best way to measure the porosity and pore size of nanowebs, without any of the discussed problems of the previous methods.

References

[1]	Thenmozhi, S., Dharmaraj, N., Kadirvelu, K. and Kim, H.Y., 2017. Electrospun nanofibers: New generation materials for advanced applications. *Materials Science and Engineering: B*, *217*, pp.36-48.
[2]	Ciocoiu, M. and Maamir, S. eds., 2016. *Nanostructured polymer blends and composites in textiles*. CRC Press.
[3]	Ramakrishna, S., Fujihara, K., Teo, W.E., Yong, T., Ma, Z. and Ramaseshan, R., 2006. Electrospun nanofibers: solving global issues. *Materials today*, *9*(3), pp.40-50.
[4]	Krifa, M. and Yuan, W., 2016. Morphology and pore size distribution of electrospun and centrifugal forcespun nylon 6 nanofiber membranes. *Textile Research Journal*, *86*(12), pp.1294-1306.
[5]	Yu, Y., Ma, R., Yan, S. and Fang, J., 2018. Preparation of multi-layer nylon-6 nanofibrous membranes by electrospinning and hot pressing methods for dye filtration. *RSC advances*, *8*(22), pp.12173-12178.
[6]	Tan, G.Z. and Zhou, Y., 2020. Electrospinning of biomimetic fibrous scaffolds for tissue engineering: a review. *International Journal of Polymeric Materials and Polymeric Biomaterials*, *69*(15), pp.947-960.
[7]	Ray, S.S., Chen, S.S., Nguyen, N.C. and Nguyen, H.T., 2019. Electrospinning: A versatile fabrication technique for nanofibrous membranes for use in desalination. In *Nanoscale Materials in Water Purification* (pp. 247-273). Elsevier.
[8]	Tornello, P.R.C., Caracciolo, P.C., Cuadrado, T.R. and Abraham, G.A., 2014. Structural characterization of electrospun micro/nanofibrous scaffolds by liquid extrusion porosimetry: a comparison with other techniques. *Materials Science and Engineering: C*, *41*, pp.335-342.
[9]	Cipitria, A., Skelton, A., Dargaville, T.R., Dalton, P.D. and Hutmacher, D.W., 2011. Design, fabrication and characterization of PCL electrospun scaffolds—a review. *Journal of Materials Chemistry*, *21*(26), pp.9419-9453.
[10]	Xue, J., Wu, T., Dai, Y. and Xia, Y., 2019. Electrospinning and electrospun nanofibers: Methods, materials, and applications. *Chemical reviews*, *119*(8), pp.5298-5415.
[11]	Frey, M.W. and Li, L., 2007. Electrospinning and porosity measurements of nylon-6/poly (ethylene oxide) blended nonwovens. *Journal of Engineered Fibers and Fabrics*, *2*(1), p.155892500700200103.

[12]	Sreedhara, S.S. and Tata, N.R., 2013. A novel method for measurement of porosity in nanofiber mat using pycnometer in filtration. *Journal of Engineered Fibers and Fabrics*, *8*(4), p.155892501300800408.
[13]	Pham, Q.P., Sharma, U. and Mikos, A.G., 2006. Electrospun poly (ε-caprolactone) microfiber and multilayer nanofiber/microfiber scaffolds: characterization of scaffolds and measurement of cellular infiltration. *Biomacromolecules*, *7*(10), pp.2796-2805.
[14]	Ramakrishna, S., Fujihara, K. and Teo, W., 2005. LTC. An introduction to eletrospinning and nanofibers.
[15]	Jena, A. and Gupta, K., 2003. Liquid extrusion techniques for pore structure evaluation of nonwovens. *International Nonwovens Journal*, (3), pp.1558925003os-1200313.
[16]	Guarino, V., Causa, F., Taddei, P., Di Foggia, M., Ciapetti, G., Martini, D., Fagnano, C., Baldini, N. and Ambrosio, L., 2008. Polylactic acid fibre-reinforced polycaprolactone scaffolds for bone tissue engineering. *Biomaterials*, *29*(27), pp.3662-3670.
[17]	Ghasemi‐Mobarakeh, L., Semnani, D. and Morshed, M., 2007. A novel method for porosity measurement of various surface layers of nanofibers mat using image analysis for tissue engineering applications. *Journal of applied polymer science*, *106*(4), pp.2536-2542.
[18]	Karageorgiou, V. and Kaplan, D., 2005. Porosity of 3D biomaterial scaffolds and osteogenesis. *Biomaterials*, *26*(27), pp.5474-5491.
[19]	Mayer, R.P. and Stowe, R.A., 1965. Mercury porosimetry—breakthrough pressure for penetration between packed spheres. *Journal of colloid Science*, *20*(8), pp.893-911.
[20]	Shi, G., Cai, Q., Wang, C., Lu, N., Wang, S. and Bei, J., 2002. Fabrication and biocompatibility of cell scaffolds of poly (l‐lactic acid) and poly (l‐lactic‐co‐glycolic acid). *Polymers for advanced technologies*, *13*(3‐4), pp.227-232.
[21]	Lu, X., Bertei, A., Finegan, D.P., Tan, C., Daemi, S.R., Weaving, J.S., O'Regan, K.B., Heenan, T.M., Hinds, G., Kendrick, E. and Brett, D.J., 2020. 3D microstructure design of lithium-ion battery electrodes assisted by X-ray nano-computed tomography and modelling. *Nature communications*, *11*(1), pp.1-13.
[22]	Yu, Y., Ma, R., Yan, S. and Fang, J., 2018. Preparation of multi-layer nylon-6 nanofibrous membranes by electrospinning and hot pressing methods for dye filtration. *RSC advances*, *8*(22), pp.12173-12178.