

STRUCTURES AND PROPERTIES OF ELECTROSPUN PAN NANOFIBERS COLLECTED FROM TWO DIFFERENT METHODS INCLUDING WATER FLOW AND ROTATIONAL DEVICE

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Introduction

The electrospun PAN copolymer nanofibers with high degree of alignment and high-drawing were new material to produce continuous high-strength nano-scale carbon fibers^[1]. The preparation of ordered nanofibers became an important topic in electrospinning^[2]. Following techniques are some possible means which have been attempted to align electrospun nanofibers: A thin wheel with sharp edge^[3], an auxiliary electrode/electrical field^[4], parallel plate electrodes^[5], static water bath to receive^[6], a cylinder collector with high rotating speed^[7], conductive template method^[8], magnetic spinning method^[2] and so on. These methods can get unidirectional nanofibers arranged in a certain degree, but there were some flaws are more or less. Besides the static water bath collector other methods can't collect fibers for a long time, and the nanofibers can't be used as long fibers. Although the static water bath collector was possible to collect long-fibers, and not subject to time constraints, it collected nanofibers only in a low speed of 0.05m/s, so the nanofibers collected from the static water bath had a low degree in unidirectional arrangement.

As the water is good conductor of electricity, and nanofibers need further removal of residual solvents in water in the follow-up processes, we combined with the existing wet spinning technology and successfully designed a new way to get one-way arranged precursor continuous fibers from a continuous flow water bath collector. In this paper, structures and properties of electrospun PAN nanofibers collected from two different methods including water flow and rotational device were studied, and the results were compared to those acquired from the conventional PAN microfibers (SAF 3K provided by the Courtaulds, Ltd in the UK).

Experimental

The PAN precursor fibers used in this article were the Special Acrylic Fibers (SAF 3K) provided by the Courtaulds, Ltd in the UK, the copolymer Composition is AN 92.8%, IA1.2%, MA6.0%. N, N-dimethylformamide (DMF).

The SAF 3K fibers were dissolved in DMF to prepare a 16 wt. % solution. Subsequently, removed gel particles. A specially designed spinneret was utilized for conducting electrospinning. The spinneret consisted of a high-density polypropylene tube and a stainless steel hemispherical head

which had an orifice with the diameter of 1mm at the center. The electrospinning setup also consisted of a high voltage power and a laboratory-built roller with a diameter of 10 inches. During electrospinning, a positive high voltage of 25kV was applied through a metal rod to the spin dope held inside the spinneret. Electrospun nanofibers were collected on the water first, then they were collected onto the roller. The rotational speed of the roller during electrospinning was set at 0.15m/s. In this manner, the process was extremely stable; and the electrospinning jet could run continuously without breaking for many hours. The obtained nanofibers were thus a single nanofiber loosely aligned along the rolling direction.

In the article we also use the roller with the same rotating speed of 0.15m/s to collect electrospun nanofibers. The codes and descriptions of the samples prepared and studied in this research are shown in Table 1.

Table 1. Codes and descriptions of the samples prepared and studied in this research

Sample	Samples' Experimental conditions	Diameter
W-0	the nanofibers collection method of water flow	632nm
R-0	the nanofibers collection method of rotational device	756nm
C	Courtaulds 3K precursor fibers	7 μ m

A Zeiss Supra 40VP field-emission SEM was employed to examine the fiber morphologies. Prior to SEM examinations, the specimens were sputter-coated with gold to avoid charge accumulations. XRD experiments were performed using a Rigaku D/MAX-2500UBZ+PC X-ray diffractometer. The X-ray tube operating at 40 kV and 50 mA with the CuK α radiation (wavelength $\lambda=0.154$ nm) was used. The XRD profiles were recorded with the 2 θ angles from 5 $^{\circ}$ to 40 $^{\circ}$ at the scanning speed of 5 $^{\circ}$ /min. Shrinkage of the fibers in Boiling water: The fiber's boiling water shrinkage is refers to GB 6505-86 "the synthetic fiber filament and the distortion silk boiling water shrinkage experimental technique".

Results and Discussion

In order To confirm the unidirectional arranged degree of the water bath collected electrospun nanofibers further, fibers' shrinkage in boiling water was tested. When a bunch of fibers composed of many single fibers deformed along length direction, the agglutination network limited the deformation of the fibers in the boiling water. The results were shown in Table 2:

Table 2. Shrinkage of W-0, R-0 and C in the boiling water

Samples	W-0	R-0	C
shrinkage%	7.88	1.48	7.04

As shown in Table 2, the shrinkage of W-0 in the boiling water was the largest, and R-0's was the lowest. Among the three kinds of fibers, W-0 and Courtaulds fibers had the similar boiling water shrinkage, indicating that the water bath collected electrospun nanofibers had the same degree of

unidirectional arrangement along the axial direction with the Courtaulds fibers. R-0's shrinkage in boiling water was only 1.48%, and was far from Courtaulds fibers. It also further clarified the roller electrospun nanofibers distributed as reticular fiber membrane, the distribution of fibers were disorder-free chapters, and there were many cross-link points between the monofilament within the fiber bundles.

The SEM pictures of the W-0 and R-0 were shown in Figure 1. The two ways of electrospinning collection had the same condition (density was 16%wt, spinning jet and the collecting device's distance was 13cm, voltage was 25KV, rotational speed was 0.15m/s), basipetal in turn was enlarged 10000 times, 5000 times and 1000 times. It showed that the water bath collected electrospun nanofibers' unidirectional arrangement degree is higher than the roller collected nanofibers under the same condition. Figure 2 was the SEM pictures of the nanofibers collection method of high-speed rotational device (rotational speed is 800m/min), the enlargement of which respectively are 10000 and 20000 times. The fibers in the picture still were the netted distribution. Therefore, the water bath collected electrospun nanofibers' unidirectional arrangement degree is higher than the roller collected nanofibers under the high rotational speed.

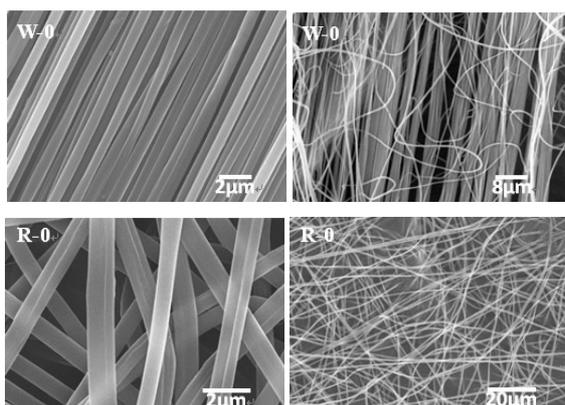


Fig. 1 SEM images showing the representative morphologies of W-0 and R-0

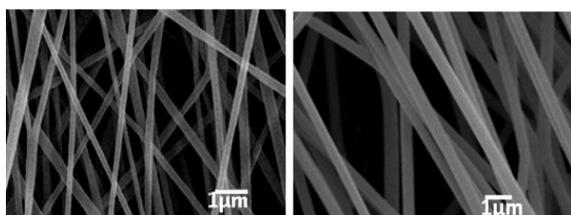


Fig. 2 SEM images showing the representative morphologies of the nanofibers collection method of high-speed rotational device

In order to study the differences of the fibers' macromolecular crystal structure and orientation between the two kinds of nanofibers we did the XRD test. The test's results were shown in Table 3:

Table 3- Orientation degree and Crystallinity of the different ways collected fiber

Samples	Crystallinity /%	Orientation degree /%
W-0	42.83	55.0
R-0	5.72	51.1

As shown in Table 3, the orientation degree of W-0 was 55% that was higher than 51%. The reason why carbon fiber has excellent tensile strength was that the carbon network's high level of orientation along the fiber axis. This required the original fibers had a high degree of orientation and crystallization. Thus, on the basis of studying on the effects of one-way collection, it was needed to further study on the primary fibers' stretching, and to study the stretching's influence on the fibers' structure and properties.

Conclusions

Shrinkage of the fibers in the boiling water and SEM results were shown that the nanofibers collection method of water flow was superior to the nanofibers collection method of rotational device.

The crystallinity of the nanofibers collection method of water flow was 42.83% that was higher than the nanofibers collection method of rotational device (5.72%). the nanofibers collection method of water flow had a higher orientation degree. The orientation degree of the nanofibers collection method of water flow were 55.0%, and the orientation degree of the nanofibers collection method of rotational device was 51.1%.

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