Fabrication and Characterization of Rotary Forcespun Styrofoam Fibers

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Abstract. Styrofoam fibers have been fabricated using a rotary forcespinning route. The fibers were produced by utilizing the centrifugal force derived from the high-speed motor. Styrofoam was dissolved in d-limonene at the concentration of 10, 15, 20, 25, and 30 wt\%, then the solution was subjected to the rotary forcespinning at a rotational speed of 12,000 rpm. The effect of concentration on fibers’ morphology was studied. SEM images showed that the fibers produced had a straight and smooth surface with no beads. The diameter of the fibers ranged from 600 nm to 3 μm. The solution concentration parameter is important in fiber formation. An increase in the concentration of the solution increased the diameter of the fibers. The analysis of FTIR spectra suggested that FTIR peaks of styrofoam fibers had the same peaks like those in the styrofoam bulk and there was no peak of d-limonene detected in the samples, which indicates the solvent has evaporated. XRD analysis showed that the styrofoam fibers had an amorphous structure. The use of rotary forcespinning to produce the fibers has shown a good quality fiber, a high production rate, and relatively inexpensive for mass-scale production.

Keywords: Styrofoam, fiber, rotary forcespinning.

1. Introduction
Fibers have broad applications in various fields such as filtration, biosciences, medical, textiles, and energy [1–5]. Fibers also have unique structures and properties such as high surface area, submicron-sized diameter, high porosity, low-base weight, etc. Extensive applications and promising advantages make researchers focused on developing large-scale and efficient fibers production method. The most popular fibers synthesis technique is electrospinning. It is superior regarding the quality of the fibers produced but low in the rate of production and concerning the safety of high voltage [6]. On the other hand, rotary forcespinning is a low-cost technique utilizing centrifugal force to produce fibers with a high production rate with quite decent quality [7–9]. The speed of rotation, the distance between the nozzles to the collector, orifice diameter, solution concentration, temperature, and humidity are the parameters that control the morphology, size, and quality of the fibers [10]. Various materials such as ceramics, polymers, and composites fibers have been synthesized by the rotary forcespinning route [11–14].
Very recently, styrofoam waste has been used to produce the polymer electrolyte membranes for lithium battery, degrade wastewater with the support of photocatalysts TiO$_2$, create micro and nanoparticles polystyrene [15], and for adhesive [16]. In our previous study, the rotary forcespun styrofoam fibers membranes have been produced to be applied as a soilless growing medium [17,18]. In those studies, the styrofoam was melted in acetone, and the resulted fibers have an average diameter of 8.85 µm. To the best of our knowledge, there have been no other studies that have discussed in detail regarding the synthesis of styrofoam fibers using centrifugal force-based techniques. Therefore, in this study, we report the synthesis and characterization of styrofoam fibers produced using the rotary forcespinning technique. Styrofoam was completely dissolved in d-limonene—a natural solvent. The physical and chemical structure of styrofoam fibers was analyzed using a scanning electron microscope (SEM), an optical microscope, a Fourier Transform Infrared (FTIR) Spectrometer, and an X-ray Diffractometer (XRD). The relationship between the parameters of the styrofoam solution and the fibers morphology will be discussed.

2. Methods

2.1 The preparation of solution and rheological property of the solutions
Waste styrofoam (expanded polystyrene foam) was obtained from the remnants of styrofoam on a styrofoam craft industry in Bandung, Indonesia. It was cut into small pieces, then washed with water to remove the dirt, and finally dried with an air dryer. The waste styrofoam was dissolved in d-limonene with the solute to solvent ratios of 10, 15, 20, 25 and 30 wt %. D-limonene as a natural solvent has been used in styrofoam [18-19]. The styrofoam solution was stirred using a magnetic stirrer at 60 °C for 6 hours. During this process, the shredded pieces of styrofoam were slowly dissolving into the solvent. The viscosity of the solution was measured by using Fenske-Oswald Viscometer (Fisher) at 25 °C.

2.2 Rotary forcespinning parameters
Rotary forcespinning apparatus consists of several main parts, namely a syringe pump to provide a certain solution flow, a motor that rotates at a high speed, a solution reservoir, and a drum collector [17,19,20]. The solution was loaded into the syringe mounted on the syringe pump. The solution was driven to the reservoir via a silicone tube with a flow rate of 20 ml/h. The reservoir had orifices with a diameter of 0.4 mm at its bottom. When the reservoir was rotated at 12,000 rpm, the solution was discharged through the orifices as jets pulled toward the drum collector with the orifice-collector distance of 15 cm. The drum collector was coated with aluminum foil and rotated at a certain speed. The detailed parameters for each solution are shown in Table 1.

| Solution Concentration (wt%) | Rotation speed (rpm) | Orifice diameter (mm) | Solution flow rate (ml/h) | RH (%) |
|-----------------------------|----------------------|-----------------------|---------------------------|--------|
| 10                          | 12,000               | 0.6                   | 20                        | 70     |
| 15                          | 12,000               | 0.6                   | 20                        | 70     |
| 20                          | 12,000               | 0.6                   | 20                        | 70     |
| 25                          | 12,000               | 0.6                   | 20                        | 70     |
| 30                          | 12,000               | 0.6                   | 20                        | 70     |

2.3 Scanning electron microscopy (SEM) and optical microscope
The morphologies of the styrofoam fiber were analyzed by a scanning electron microscope (SEM) (JEOL, JSM-6510) and an optical microscope (Trinocular, XSZ-107E). The samples were coated with electric conductive carbon, and a high voltage of 10 kV with 1,000× optical magnification was employed to obtain the SEM images.
2.4 Fourier Transform Infrared (FTIR) and X-Ray Diffraction (XRD)

Functional groups of styrofoam bulk and styrofoam fibers were evaluated using a Fourier-transform infrared (FTIR) spectrometer (Bruker, Alpha). The FTIR spectra were recorded with the wavenumber in the range of 500 to 4000 cm$^{-1}$. The X-ray diffraction patterns of styrofoam fibers and styrofoam bulk were recorded using an X-ray Diffractometer (XRD) (Panalytical X’pert Pro). The FTIR and XRD examinations were carried out only on styrofoam fibers with a concentration of 20 wt%.

3. Results and Discussion

Figure 1 (a), shows a styrofoam mat attached to the collector coated with aluminum foil. Macroscopically, the mat produced has a smooth white texture, randomly deposited in the collector, and can be peeled from the aluminum foil. The rotary force spinning collector consists of 4 drums which rotate at a certain speed, with their position surrounding the reservoir. The rotation of the drum will force the fiber out of the orifice. The application of a rotating drum collector aims to make the fibers are collected into a mat because the resulted fibers are usually in the form of a lump. Figure 1 (b) shows the SEM image of styrofoam fibers. The morphologies of the resulted fibers were uniform, smooth, and having no beads. The diameter of the fibers ranged from submicron to micrometer. This result is relevant to our previous research; we had synthesized styrofoam fibers by dissolving styrofoam in acetone and obtained fine and uniform fibers with an average diameter of 8.85 µm [17]. At present, we have dissolved styrofoam completely in d-limonene and produced fibers with the same morphology but with a smaller diameter.

Figure 1. (a) Styrofoam mat composed of fibers on aluminum foil collector, (b) SEM image of styrofoam fibers with 1000× magnification

Figure 2 gives the relationship between the concentration and the viscosity of the styrofoam solution. Styrofoam fibers were formed from the solution with the viscosity ranging from 65 to 128 cP. The concentration and viscosity relationship was plotted using a linear function. This result showed the viscosity of styrofoam solution depended linearly on its solution concentration. The increase in concentration caused an increase in the viscosity.
Figure 2. The relationship between solution concentration and viscosity of the styrofoam solution

Figure 3 shows the relationship between the concentration of styrofoam solution and the average diameter of the fibers. The inset shows the optical microscope images at 1000× magnification. Table 2 correlates the solution concentration, average fiber diameter, and fiber homogeneity. For all variations of solution concentration, the fibers produced were all straight, uniform, free of beads, with the average diameter ranging from 556 to 2318 nm, and coefficient of variation (CV) below 0.3. According to the reference, if the CV is smaller than 0.3, the fibers are categorized as uniform [21].

Figure 3. The effect of the solution concentration on the average fiber diameter

SEM morphology analysis confirmed that the increase in the solution concentration caused the increase in the average fiber diameter. This result is relevant to Lu et al. which reported an increase in diameter of PAN fibers along with the increase in the solution concentration synthesized by forcespinning [22]. Upson et al. also confirmed the same results on the synthesis of PHBV fibers with centrifugal spinning. In this study, the lowest concentration (10 wt%) and the highest (30 wt%) produced fibers without any beads, indicating that the range of the concentrations used in the experiment was the most suitable range for obtaining the fine fibers. The styrofoam solution with the high concentration and viscosity has longer stress relaxation time, faster evaporation of solution and resists jet fracturing, elongation, and thinning [22]. Jets coming out of the orifice will experience elongation and evaporation of the solution. A higher concentration of solution contains a fewer solvent, which makes the evaporation process occurs faster. The evaporated solvent will discontinue the elongation process so that the fiber size cannot be smaller. From this process, it can be explained how the increase in concentration and viscosity results in greater fiber diameter. Even at the concentration of 30 wt%, the diameter sizes of styrofoam fibers were above 2 μm.
Table 2. The effect of concentration on styrofoam fibers diameter and morphology

| Solution Concentration (wt%) | Fibers diameter (nm) | Standard deviation | Coefficient of variance | Fibers homogeneity |
|-----------------------------|----------------------|--------------------|------------------------|-------------------|
| 10                          | 556                  | 49.1               | 0.09                   | Uniform           |
| 15                          | 734                  | 97.8               | 0.13                   | Uniform           |
| 20                          | 1178                 | 125.7              | 0.11                   | Uniform           |
| 25                          | 1680                 | 255.7              | 0.15                   | Uniform           |
| 30                          | 2318                 | 380.4              | 0.16                   | Uniform           |

Figure 4 (a) displays the IR spectra of styrofoam. Generally, styrofoam fibers and styrofoam bulk have similar peaks at certain wavenumbers. The dominant peak at the wavelength of 696 cm\(^{-1}\) was assigned to monosubstituted benzene C-H bending. There are aromatic C=H stretching vibrations at 1493 cm\(^{-1}\) and 1452 cm\(^{-1}\). The peak at 2924 cm\(^{-1}\) was associated with asymmetric stretching vibration of CH\(_2\) [23–25]. This IR spectrum of styrofoam fibers has the same peak value as the electrospun IR polystyrene recycled fibers IR reported by Zander et al. [20]. The peak of d-limonene solvent 2900, 1700, and 1100 cm\(^{-1}\) did not appear. This means that during the spinning process, d-limonene evaporates completely. The XRD pattern of styrofoam fibers and bulk is presented in Figure 4 (b). The main peak was at 18.5\(^{\circ}\). From the XRD pattern, there were no sharp peaks in styrofoam bulk and fibers. These results indicate that both samples were amorphous. This result was also confirmed in other studies, which proved that polystyrene fibers are amorphous [27,28].

![Figure 4](image-url)

**Figure 4.** (a) IR spectra of styrofoam fibers and bulk, (b) XRD patterns of styrofoam fibers and bulk

4. **Conclusion**

We have succeeded in synthesizing styrofoam fibers using waste styrofoam dissolved in a d-limonene solvent using a rotary force spinning technique. The fibers were accumulated to form a mat on the rotating drum collector. SEM and optical microscope images showed fine fiber morphology with no beads and the fiber diameter distribution were all uniform on all variations of concentrations. The viscosity of styrofoam solution depended on the concentration with a linear relationship in which the greater the concentration, the greater the viscosity. The concentration of the solution also affected the average diameter of the fibers. The increase in concentration caused an increase in the average diameter of the fibers. A variation in solution concentration from 10 to 30 wt% produced the fibers with the average diameter ranging from 516 to 2318 nm. An increase in the concentration resulted in an increase in the diameter of the fiber. IR spectra of styrofoam bulk and fibers showed similar peaks, and the d-limonene peaks were not detected on styrofoam fibers sample. Therefore, the solvent evaporation was
confirmed by the FTIR spectra analysis. The XRD patterns obtained showed that styrofoam fibers were amorphous because there were no sharp peaks in the patterns.

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