

The preparation and characterization of liquefied wood based primary fibers

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ABSTRACT

The preparation of primary fibers were performed from liquefied wood in phenol by melt-spinning with different spinnerettes of length/diameter ratio (L/D) (0, 2, and 4), to investigate the effect of the size of spinnerette on the thermal stability and structural properties of primary fibers. High thermal transition temperatures and small weight loss percentages of resultant primary fibers representing good thermal stability were obtained with L/D of spinnerette increasing. Besides, few defective structure occurred on cross section and surface of primary fibers in comparison with those prepared with spinnerette of $L/D=0$. We found that these improvements were caused by the formation of hydrogen bond among phenolic hydroxyl groups in the period of flow entering the cylindrical die of spinnerette. Thus, the precursor, with either porous structure for preparing activated carbon fibers or defect-free structure for preparing carbon fibers with high performance, can be probably prepared by controlling the spinnerette system.

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1. Introduction

As typical biomass materials in nature, nowadays, both wood and its main components including cellulose, hemicelluloses, and lignin have attracted more and more attention concerning their effective utilization, due to the abundant and renewable characterization. Wood liquefaction, wherein wood flour is directly solvolyzed with the solvent of phenol or polyalcohol at the presence of small amount acid as catalyst, has been proved to be one of the effective techniques for preparing wood-based materials such as adhesives, foams, and moldings (Maldas, Shiraishi, & Harada, 1997; Lee, Teramoto, & Shiraishi, 2002; Lin, Yoshioka, Yao, & Shiraishi, 1995a, 1995b). Comparatively, preparation of carbon fibers (CFs) from liquefied wood seems to be a more facile and promising process because of higher value-added process (Ma & Zhao, 2010).

In general, the process for manufacturing CFs consists of several steps, such as wet or melt spinning depending on raw material, thermostabilized treatment for obtaining infusible fibers and carbonization in inert atmosphere at temperatures of 1000–2000 °C as well as the graphitization at 2000–3000 °C. As the first step, melt-spinning process, for mesophase pitch or liquefied wood as the starting material, is a key step to prepare excellent fibers for producing CFs with high performance. This step governs the macrostructure of spun fibers and eventually determines the

texture and properties of resultant CFs through several important melt-spinning parameters (Zha, Shi, Ji, Liu, & Qian, 1992). Among these parameters, the size and shape of spinnerette plays a quite significant role in forming transverse structure of the filament which is formed by rearrangement of spinning solution through the spinnerette capillary during spinning (White & Buechler, 1984). Additionally, pitch-based CFs with random transverse structure are less graphitizable than that with radial transverse structure, and occasionally leads to the formation of defects along the filament axis, resulting in the great reduction of mechanical properties of CFs (Bright & Singer, 1979; Hamada, Nishida, Furuyama, & Tomioka, 1988). Therefore, more attention should be paid to these problems for obtaining the desirable products.

However, there were few investigations on reporting the effect of melt-spinning parameters on the quality of resultant liquefied-wood based primary fibers. Accordingly, in this article we describe the efforts directed at the characterization and evaluation of primary fibers derived from liquefied wood in phenol by melt-spinning with different spinnerettes.

2. Materials and methods

2.1. Preparation of liquefied wood based primary fibers

Twenty grams of dried Chinese Fir (*Cunninghamia lanceolata*) powder (20–80 mesh) were placed in a round bottom flask together with 120 g of phenol and 9.6 g of phosphoric acid (8 wt% on the weight of phenol). The flask was immersed in an oil bath that was preheated at 160 °C for 150 min. The flask was then cooled to room

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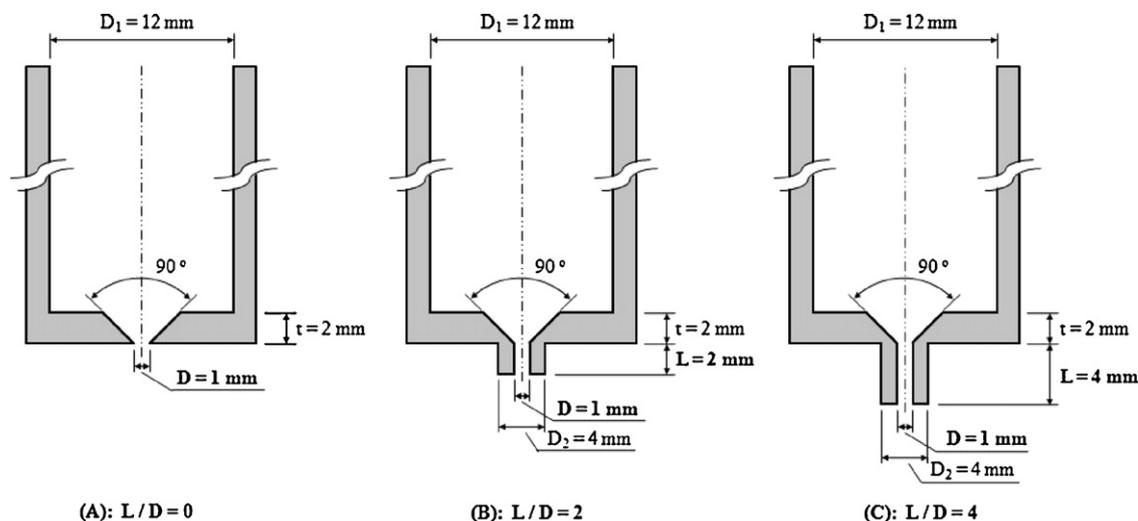


Fig. 1. Illustration of spinnerettes with different L/D .

temperature with cooling water, and then 0.75 L of acetone was added to the reaction product. The resultant mixture was filtrated through filter paper to separate filtrate and residue.

The acetone in the filtrate was removed by evaporation. The resulting liquefied wood mixed with hexamethylenetetramine (HMTA) (5 wt% on the weight of liquefied wood) were transferred into the metal tube equipped with different sizes of spinnerette [Length/Diameter ratio (L/D) = 0, 2, and 4] (as shown in Fig. 1). The mixture was heated from room temperature to 120 °C with the heating rate of 2.5 °C/min, and then the final temperature of 120 °C was maintained for 5 min to obtain the spinning solution. Subsequently, the melt-spinning process was conducted to prepare the primary fibers at the nozzle temperature of 120 °C and the winding rates of 14 m/min under a nitrogen pressure.

2.2. Characterizations

2.2.1. Thermogravimetry (TG)

The weight loss behaviors and decomposition temperature (T_d) of liquefied wood based primary fibers were measured by using thermogravimetric analysis (TGA) and differential thermal analysis (DTA) on a simultaneous thermal analyzer (DTG-60, Shimadzu, Japan) from room temperature to 600 °C with the heating rate of 10 °C/min in high pure nitrogen stream (30 mL/min).

2.2.2. Thermomechanical analysis (TMA)

TMA was performed on a TMA-4000S System (MAC Science System 010, MAC Science, Yokohama, Japan) at the temperature ranging from 30 to 300 °C under compression loading of 5 g under a N_2 stream at 0.15 L/min. The glass transition temperature (T_g) and thermal-flow starting temperature (T_f) were estimated as a first and a second transition points in TMA profiles (Kubo, Uraki, & Sano, 1996).

2.2.3. Scanning electronic microscopy (SEM)

Morphologies of primary fibers were observed by scanning electronic microscopy (SEM; S-3400N, Hitachi, Japan) using gold-coated samples, at magnification factors of 1500–5000, and at an accelerating voltage of 10 kV, respectively.

2.2.4. Fourier transform infrared spectroscopy (FTIR)

Fourier transform infrared (FTIR) spectroscopy was carried out on an FTIR spectrophotometer (SENSOR 27, Bruker, Germany), using the KBr disk method.

2.2.5. Mercury intrusion porosimetry (MIP)

Fiber pore structures were characterized by mercury porosimetry (AutoScan-33, Quantachrome, USA) with the pressure ranging from 0.2 to 60,000 psia and equilibration time of 10 s (Leon, 1998).

3. Results and discussion

3.1. Thermal stability of primary fibers

Melt-spinning processes were successfully conducted to prepare liquefied wood based primary fibers from spinning solution at 120 °C with different L/D spinnerettes. The primary fibers exhibited significant differences in thermal stability by TGA measurements as shown in Fig. 2. With the temperature increasing, all the primary fibers weights decreased to various degree, and the weight loss processes could be partitioned into two stages (stage 1: sharp weight loss varying from 150 °C to 400 °C; stage 2: slow weight loss ranging from 400 °C to 600 °C) excluding the temperature lower than 150 °C because of the loss of adsorbed water molecules, which derived from ambient environment.

In the case of stage 1, high weight loss process was mainly induced by the thermal degradation starting from the decomposition temperature (T_d), which was defined as a temperature that give a 5% weight loss (Uraki, Kubo, Nigo, Sano, & Sasaya, 1995). As shown in Table 1, high L/D of spinnerette gave rise to the high T_d of primary fibers. Especially for the $L/D = 4$, around 20 °C increase of T_d was obtained in comparison with that of primary fibers prepared with spinnerette of $L/D = 0$, resulting in the decrease of weight loss percentage from 39.2% to 33.3% in this stage. For stage 2, similarly, decrease in weight loss percentage took place with the L/D of spinnerette increasing, which was probably caused by the complete decomposition (Zheng, Zhou, Du, & Zhang, 2002). Accordingly, the highest residual ratio of 53.2% was obtained by using

Table 1

Thermal transition temperatures and residual ratio of primary fibers prepared with different L/D spinnerettes.

L/D	Transition temperature (°C) ^a			Residual ratio (%)
	T_g	T_f	T_d	
0	97	140	210	45.3
2	95	142	215	45.3
4	104	149	229	53.2

^a T_g , T_f , and T_d denote glass transition temperature, thermal-flow starting temperature, and decomposition temperature, respectively.

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