Preparation of Hollow Carbon Fiber from a Rice Straw-derived Extract

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Highlights

- Hollow carbon fiber was successfully produced from a biomass-derived extract.
- The hollow was found to form during melt-spinning process.
- The treatment and spinning conditions were the factors affecting hollow formation.
- The yield of carbon fiber was around 34% on Soluble basis.

1. Introduction

We have presented a process called degradative solvent extraction for producing a high quality extracts from several types of low-rank coals and biomasses [1,2]. In this process, raw material was dewatered and upgraded through the degradation reactions in a non-polar solvent such as 1-methylnapthalene at 350 °C. Three main solid extracts, Residue, Deposit, and Soluble, were obtained by the process. The carbon basis yield of the smallest molecular weight fraction, Soluble, reached as high as 70 wt % by using biomass as raw material. Soluble is free from water and ash and its carbon content and oxygen content are respectively higher than 80 wt % and lower than 10%. Soluble melted completely below 100 °C before starting decomposition. Soluble having such unique properties is expected to be used as a precursor of advanced carbon materials such as carbon fiber. In this study the production of carbon fiber from a Soluble obtained from the degradative solvent extraction of a rice straw was examined.

2. Methods

A rice straw (RS) Soluble was initially treated by the N_2 purge method at around 280-320 °C to increase its melting point. Then the treated Soluble was spun to fiber by the melt-spinning technique through the single 0.8 mm dia. nozzle at around 250-290 °C to obtain pitch fibers. The pitch fibers were stabilized by heating them from room temperature to 300 °C at 0.5 °C/min in an air stream. Then, the stabilized fibers were heated at 10 °C/min to 900 °C and kept 1 h at the temperature in a nitrogen stream to be a carbon fiber. The physical and chemical properties of the treated Soluble were analyzed by a thermomechanical analyzer (TMA50, Shimadzu), a FTIR spectrometer (JIR-WINSPEC50, JEOL) and a CHN analyzer (JM10, J-Science). Properties of the obtained carbon fibers were characterized by a tensile testing machine (EZ-LX, Shimadzu), a scanning electron microscope (JCM-6000, JEOL) and an adsorption analyzer (BELSORP-mini II, BEL Japan)

3. Results and discussion

Figure 1 shows the melting behaviors of Solubles treated at three conditions: 280°C-60min, 300°C-80min, and 300°C-100min. Melting points of the Solubles increased respectively to around 144°C, 270°C, and 185°C. It was found that the pitch fibers prepared from the Soluble treated at 300°C-80 min and 300°C-100 min with a spinning speed of 180 m/min had hollow inside each fiber as shown in **Figure 2**. On the other hand, the pitch fiber prepared from the Soluble treated at 280°C-60 min did not have such hollows. This difference seems to be explained by the differences in rheological property and viscosity among the treated Solubles. Phase separation during spinning of fiber may also affect the hollow formation. The hollow may

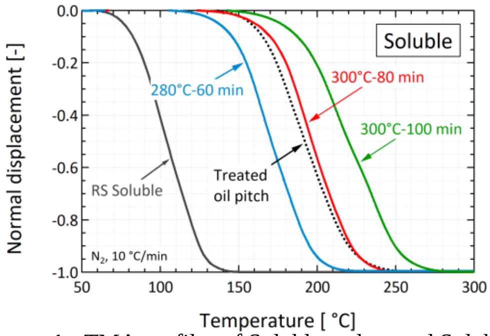

Figure 1. TMA profiles of Soluble and treated Solubles

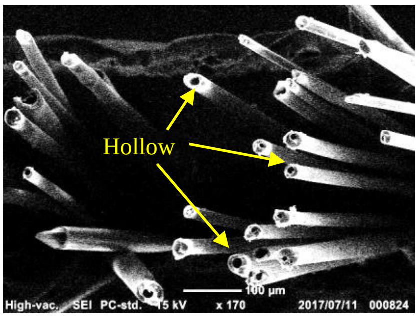

Figure 2. Pitch fiber obtained after melt-spinning process

form due to the thermal expansion of treated Soluble during the spinning process. When the fiber diameter is relatively large, temperature is not uniform inside the fiber. Then we varied the spinning speed to control the diameter of pitch fiber and to examine the effect of the diameter on the formation of hollow. Spinning at the spinning speed over 250 m/min for the Soluble treated at 300°C-80min gave the hollow free pitch fiber with 14.8 µm of diameter. On the other hand, spinning at the spinning speed at 90 m/min for Soluble treated at 280°C-60min gave the hollow pitch fiber with 19.1 µm of diameter. These examinations showed clearly that the spinning speed is one of the parameters controlling the formation of hollow in the pitch fiber.

Figure 3 shows the carbon fibers produced at 180 m/min of spinning speed from the Solubles treated at different treatment conditions: 280°C-60min, 300°C-80min, and 300°C-100min. It is clearly shown that hollows formed during the spinning process are retained in the carbon fibers with larger diameter. The average diameters of the carbon fibers prepared at the three conditions were 9±2, 20±3, and 24±4 µm, and the tensile strengths of the carbon fibers were respectively 79±36, 148±36, and 378±85 MPa. Their yields were around 33.6-34.0% irrespective of treatment conditions and their elemental compositions were: C = 85.9-88.0%, H=1.7-2.0%, N=2.3-2.7%, and O=7.7-9.7%.

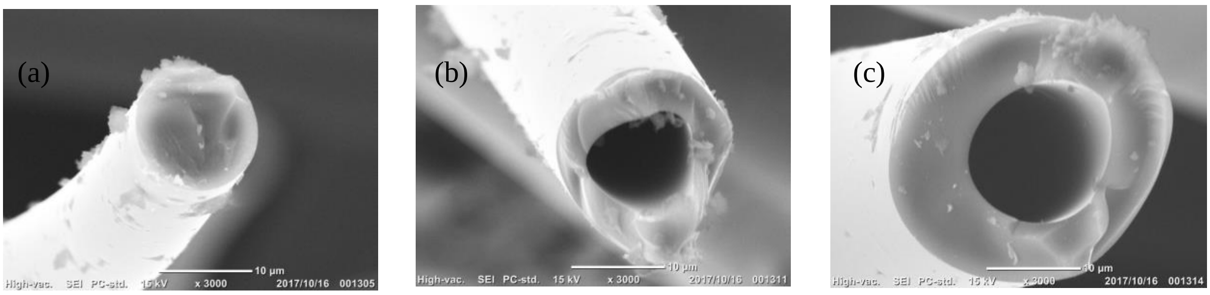

Figure 3. Carbon fiber produced from Soluble prepared at (a) 280°C-60min, (b) 300°C-80min, and (c) 300°C-100min

4. Conclusions

It was fond that unique hollow carbon fiber can be produced from a rice straw derived extract, Soluble. The hollow was found to be formed during the spinning process preparing pitch fiber. The rheological properties of the treated Solubles were one of the major factors affecting the formation of hollow. The spinning speed during preparing pitch fiber changes the rheological properties of the treated Solubles and hence could control the hollow formation and the diameter of pitch fiber. The yield of carbon fiber reached 34 wt% on Soluble basis. The tensile strength of carbon fiber was varied by controlling the treatment condition of Soluble.

References

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Keywords
Hollow carbon fiber, biomass based carbon fiber, biomass-derived extract, degradative solvent extraction