Examination of carbon fiber production from the extracts produced from the solvent treatment of biomass

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Abstract:
A production of carbon fiber by using a Soluble-based precursor, a low-molecular-weight compound extracted from a degradative solvent extraction process of rice straw, was examined in this study. The procedure of carbon fiber production started from improvement of softening point of the Soluble, melt spinning, followed by stabilization and carbonization steps. During spinning, three collecting speeds were varied; 600, 800 and 1000 rpm. Based on 100 wt% of Soluble, it was found that the average yields of the spun fibers after experiencing stabilization step and carbonization step were around 69.7-70.0 wt% and 33.7-34.2 wt%, respectively. The average diameters of the spun fibers were found to reduce with increasing spinning speed; 11.6, 11.0 and 9.6 μm for the fibers spun at 600, 800 and 1000 rpm, respectively. The tensile strength distributions of each spun fiber condition were shown to vary showing in the ranges of 578.2 MPa, 551.6 MPa and 626.3 MPa for the spinning speeds of 600, 800 and 1000 rpm, respectively. These preprimary results were judged to show a good potential for the carbon fiber production from the Soluble extracted from rice straw.

Keywords: *carbon fiber production; biomass extraction; solvent treatment; degradative solvent extraction*

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1. Rationale

Carbon fiber is a well-known advanced material that has unique properties such as high strength to weight ratio, good rigidity, corrosion resistance, etc. Nowadays, the utilization of carbon fiber as a precursor of material productions has been expanded in several applications. In general, about 90% of the overall carbon fiber productions uses polyacrylonitrile (PAN) as a main precursor, while others use pitch or coal tar-based materials, including by-product from petroleum industry. Few of carbon fiber productions use bio-based material, such as rayon as a precursor (Huang, 2009). In this study a possibility of carbon fiber production by using a novel precursor, a low-molecular-weight compound obtained from a solvent treatment process developed by the author's research group, has been examined. This extract called Soluble was derived from an extraction of rice straw. From the previous works, the authors showed that the Soluble can be produced in high yield (55.0% of carbon based yield) and has attractive properties, such as, high carbon content, low oxygen content, and moisture- and ash-free. Additionally, Soluble, in particular, showed a unique melting behavior that it completely melts at around 100°C before starting decomposition (Wannapeera et al., 2012). These properties are therefore very attractive for the Soluble to be used as a precursor for making a carbon fiber. In this study, the conventional examination of the carbon fiber production from Soluble precursor without using any chemicals was conducted. The properties of the obtained carbon fiber were preliminary investigated.

2. Experimental and analyses

The Soluble produced from a solvent extraction process of rice straw was used as a raw material in this study. The extraction process was performed at 350°C using 1-methylnapthalene as a solvent. The extraction procedure was explained in the previous works (Wannapeera et al., 2012). The

elemental composition heating values and ash contents of rice straw and Soluble are shown in Table 1.

Table 1 Elemental analyses, heating values and ashes of rice straw and Soluble reported in d.a.f. basis

Sample	Elemental composition [wt%]				O/C	H/C	HHV	Ash
	C	H	N	O (diff.)			[MJ/kg]	[wt%]
Rice straw	47.0	6.5	0.6	45.9	0.73	1.66	16.99	15.8
Soluble	82.0	6.6	1.1	10.3	0.09	0.97	35.38	0.0

In this study, due to the relatively low softening point of Soluble, a pretreatment of Soluble prior to be introducing in the melt spinning system was conducted. Fig. 1 shows the apparatus used for removing the light-fraction compounds in order to raise its softening point.

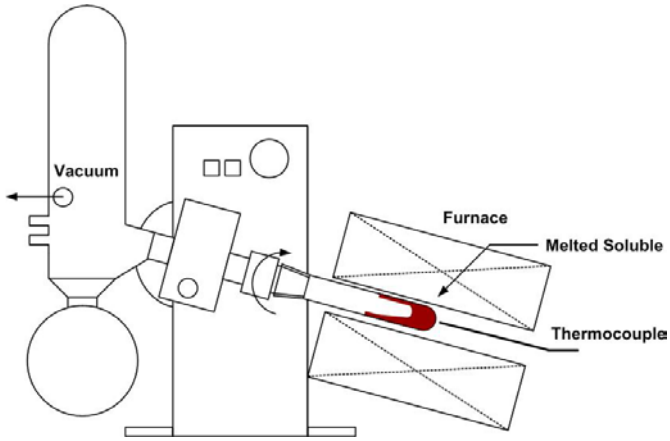

Fig. 1 The apparatus used for removing of light-fraction compounds from Soluble.

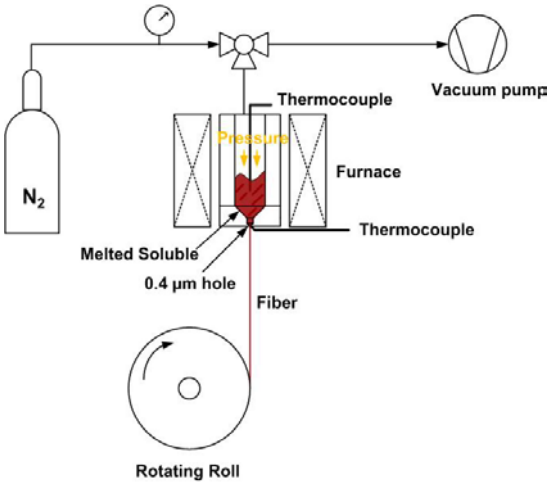

Fig. 2 Schematic diagram of melt-spinning system.

In this step, Soluble was treated under reduced pressure at around 250 to 340°C to by using a film distillation technique. After cooling down to the room temperature, around 4 g of treated Soluble was introduced to the sample holder of compacted melt-spinning system, which was equipped with a single hole 0.4 µm dia. nozzle at the bottom of sample holder. A Fig. of the melt-spinning system is shown in Fig. 2. Before starting spinning, the Soluble was re-treated under vacuum at 290°C for 30 min, in order to reduce the viscosity of the melted Soluble, while the nozzle hole was tightly closed with the cap. After reaching the treatment condition, the sample holder was cooled down to around 260°C and the nozzle hole was opened. The pressurized nitrogen was then charged to the sample holder constantly at 0.6 MPa to push the Soluble throughout the spinning process. During spinning, single fiber was continuously generated and quenched by air and further collected by the rotating roller. In this study, three spinning speeds of the collecting roll were examined; 600, 800 and 1000 rpm, respectively.

Then, the spun fiber was stabilized under an air stream at 300°C with a very low heating rate of 5°C/min and held for 1 h by using the horizontal tube furnace. Finally, the stabilized fiber was then carbonized under an inert atmosphere at 800°C to enrich the carbon content, i.e. to become a carbon fiber. After carbon fiber production process, the properties of fibers in the terms of average diameter and tensile strength including the fiber images were carried out.

3. Results and discussion

Fig. 3 shows the yields of removed light-fraction compounds against the softening point of Soluble

treated at elevated temperature. It was found that removing light-fraction compounds resulted in higher softening points of the Soluble. In this case, around 20.6 wt% of light-fraction compounds was removed at the treatment temperature of 340 °C and the final softening point of treated Soluble was raised to 226°C

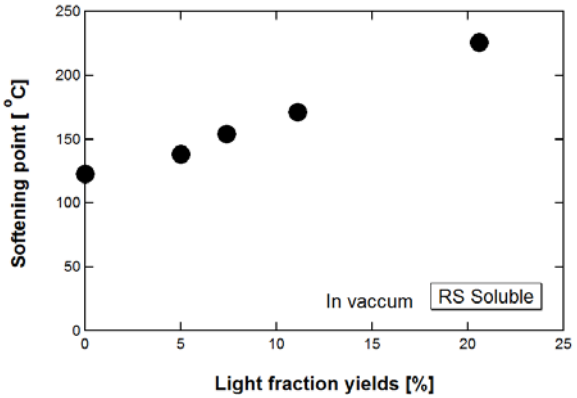

Fig. 3 Light fraction yields against the melting point of Soluble.

Table 2 shows the yields of treated spun fibers after experiencing stabilization step and carbonization step, respectively. Based on 100 wt% of Soluble, it shows that around 69.7-70.0 wt% of the yields remains after stabilization and around 33.7-34.2 wt% remains after carbonization.

Table 2 Yields of spun fibers after treating in stabilization and carbonization steps

Sample	Yield (wt% as spun fiber)	
	Stabilization step	Carbonization step
No. 1 (600 rpm)	88.2	42.4
No. 2 (800 rpm)	87.8	42.7
No. 3 (1000 rpm)	88.2	43.1

The diameter of the fibers was also measured for 15 fiber samples for each spinning speed. It was found that average diameters decreased with the increasing spinning speeds; 11.6, 11.0 and 9.6 μm for the sample No. 1(600 rpm), No. 2(800 rpm) and No. 3(1000 rpm), respectively. Then, together with the tensile testing, the tensile strengths of the fibers were determined, as shown in Fig. 4. It is illustrated that the tensile strength distributions of each spun fiber condition were also found to vary showing in the ranges of 578.2 MPa, 551.6 MPa and 626.3 MPa for a fiber spun at 600 rpm, 800 rpm and 1000 rpm, respectively.

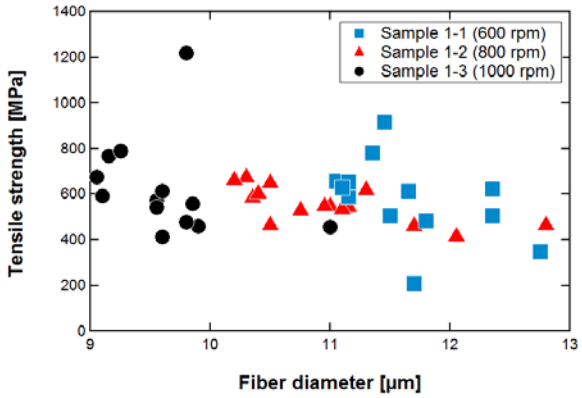

Fig. 4 Tensile strength distributions of carbon fiber prepared at several collecting speeds.

Fig. 5 shows the images obtained from a scanning electron microscope (SEM) of the carbon fiber produced. It is shown that the fiber diameters are rather uniform. A relatively smooth surface and a cross-section of the fiber are also observed.

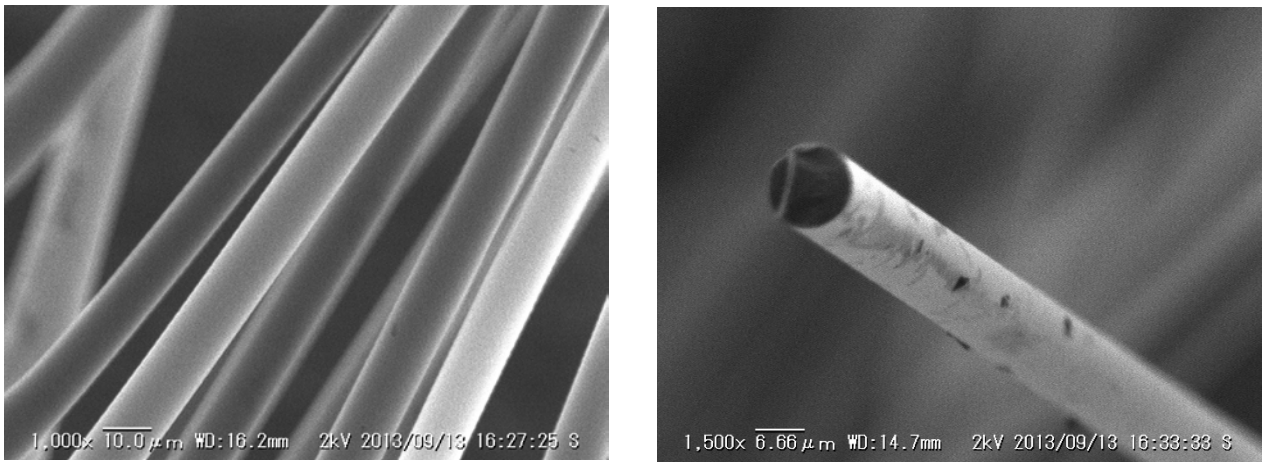

Fig. 5 SEM images of the carbon fiber (sample No.1-2).

4. Conclusion

The production of carbon fiber from the Soluble, which was produced from a solvent extraction process of rice straw, was successfully examined in this study. Throughout the production procedure, the first removing of light-fraction compounds, followed by melt spinning step, stabilization step and finally carbonization step, were performed. The final yield after experiencing carbonization step was around 33.7 to 34.2 wt% based on Soluble weight. From the physical observation, it was found that the average diameters of the spun fibers were in a range of 9.6 to 11.6 μm. The mechanical test showed the tensile strength distributions that were in a range of 551.6 to 626.3 MPa. These preprimary results showed a good potential for producing carbon fiber from the Soluble extracted from rice straw

5. Acknowledgment

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6. References

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