

POSTER

PREPARATION OF ANISOTROPIC/ISOTROPIC PITCH AND CARBON FIBER FROM NCC-PFO

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INTRODUCTION

Carbon materials are widely used in industry due to their unique physical and chemical properties. Although all compounds containing carbon can be used as a raw material basically, the real application is restricted by the required properties of the final material and the production cost. Petroleum pitch and coal tar pitch have been evaluated as a suitable feedstock among carbon compounds, because of their low price and high coking yield. Especially, much attention has been given to FCC-DO (fluidized catalytic cracking decant oil) and NCB (naphtha cracking bottom) because of their high aromaticity, low sulfur content and low toluene insoluble content. In this study, we have tried to evaluate the feasibility of the PFO (pyrolyzed fuel oil), which is the bottom oil from a NCC (naphtha cracking center), as a raw material of carbon fiber. The physical and chemical properties of PFO were studied and spinnable pitch and carbon fiber were produced from PFO.

EXPERIMENTAL

Since heavy oils like PFO are composed of various kinds of molecules, it is very difficult to find out their exact compositions. Therefore, we inferred the compositions of PFO and PFO pitch from several analysis data obtained by $^1\text{H-NMR}$, FT-IR, GPC, TGA, EA, etc. Ashland 240 pitch (A240), which is known as a good feedstock for the carbon material, was also analyzed and compared with the PFO and PFO pitch. Analytical data are summarized in Table 1. PFO was initially heat-treated in a batch type reactor to remove the volatile matter, which consists of 30-40 wt% of the feedstock, and then thermally pyrolyzed in a bubble type reactor. Isotropic pitch

obtained by pyrolysis at 370°C was made into isotropic carbon fiber by the successive procedure of melt-spinning, oxidation and carbonization.

RESULTS AND DISCUSSION

The properties required as a feedstock for the production of carbon fiber are high aromaticity, high C/H ratio, narrow molecular distribution and low sulfur content. The comparison in Table 1 shows that the properties of the precursor pitch (PFO-II) were improved by the heat-treatment. The aromaticity of PFO-II was comparable to that of A240 and the sulfur content was a lot lower. Since molecular weight distribution and C/H ratio (or TGA residual yield) of the precursor can be improved, PFO pitch would be an excellent feedstock of carbon fiber. To remove the volatile matters, we initially heat-treated PFO at 300°C for 2hr under N_2 atmosphere, and obtained the PFO precursor pitch with the softening point of 88.1°C and no toluene insolubles at the yield of 63 wt%. The nonvolatile precursor pitch derived from the PFO was thermally pyrolyzed in the temperature range from 350°C to 450°C for 3 hr under 500 cc/min of N_2 blowing condition. Spinnable mesophase pitch with the softening point of 255°C and the toluene insolubles of 28 wt% was obtained at 370°C, and then was successfully spun through a capillary spinneret with 0.5 mm diameter nozzle. After spinning, an isotropic carbon fiber of 25 μm diameter was obtained via oxidation and carbonization procedures. Mesophase spherules began to be observed from the second stage product pitch pyrolyzed at 410°C, and bulk mesophase with a flow texture was observed at 430°C and 450°C.

CONCLUSIONS

It was revealed that PFO could be a good feedstock for carbon fiber by the appropriate modification of molecular weight distribution and coking yield. Spinnable isotropic pitch could be prepared by the heat-treatment of PFO under the optimum temperature which was determinant to the properties of the resultant pitch.

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Table 1. Analysis of the raw material(PFO-I), the precursor-pitch(PFO-II) and A-240 pitch

	PFO-I	PFO-II	A-240
C/H Ratio	1.02	1.08	1.41
Aromaticity (%)	69.9	72.1	84.6
Sulfur content (wt%)	0.1	0.1	2.4
Molecular weight			
Mn	473	662	133
Mw	1564	1190	311
Dispersity	3.31	1.80	2.33
TGA residual (wt%)			
at 200°C	73.6	92.9	99.6
at 500°C	14.6	28.8	55.2
at 900°C	7.6	5.8	18.6

Table 2. Thermal pyrolysis results of the PFO precursor-pitch for preparation of isotropic/anisotropic pitches at different temperatures for 3 hr with 500cc/min N₂ blowing

	PFOII	350 °C	370 °C	390 °C	410 °C	430 °C	450 °C
Pitch yield (wt%)	63.0	38.9	31.9	27.2	26.4	24.9	25.9
Softening point (°C)	88	215	255	315	355	>43	>45
Toluene insol. (wt%)	0	4.6	28.4	54.1	84.1	99.9	97.5
Mesophase content (%)	-	0	0	<1	10	100	100
Pitch type	-	← Isotropic →		← Anisotropic →			

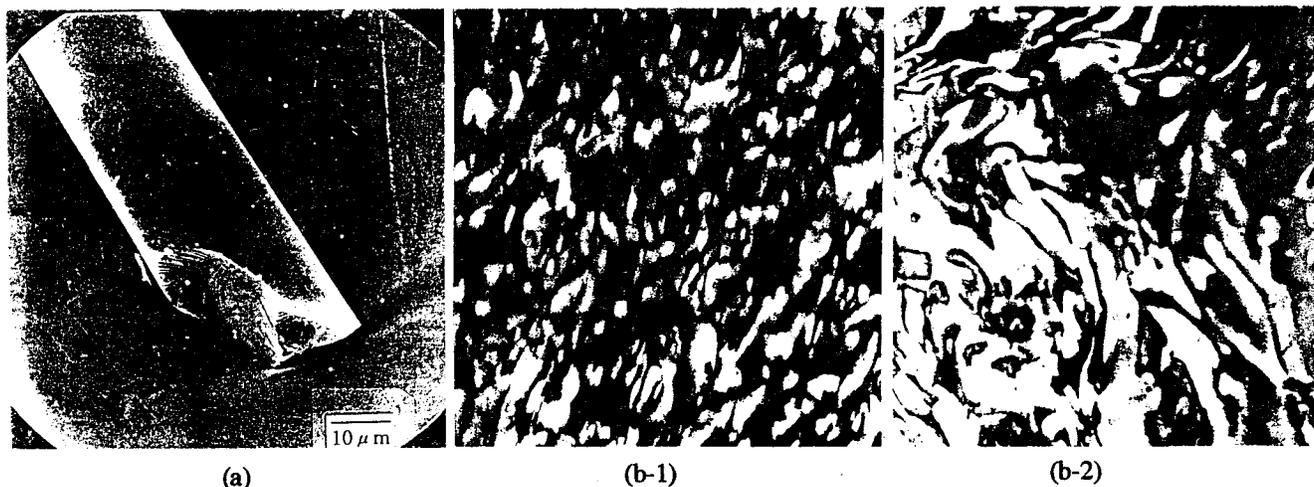


Figure 1. (a) SEM micrograph of the isotropic carbon fiber prepared from the PFO pitch and Polarization microphotograph of PFO isotropic pitches prepared at 430°C(b-1) and 450°C(b-2) for 3 hr under 500cc/min N₂ blowing