INFLUENCE OF NAPHTHALENE BASED COAL TAR DERIVED OILS ON MESOPHASE CHARACTERISTICS

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Introduction

There are several proposals of materials and methods useful in conversion of isotropic pitches to anisotropic mesophase [1,2]. Some emphasize heat treatment equipment and processing parameters [1]. Others report on the effects on mesophase yield and quality of physico-chemical treatments of pitches [2]. A great deal of research shows a definite interest in primary quinoline insolubles fraction (QI) effects over mesophase nucleation, growth and coalescence.

The main purpose of this work is to study the solvent effectiveness of coal tar derived oils on the soft coal tar pitch, to remove the QI fraction for improving mesophase production yield [3,4].

Experimental

A commercial soft pitch produced by USIMINAS and its derivatives produced by solvent extraction process and their characteristics are displayed in Table I. They were converted to mesophase by heat treatment.

Table I. Properties of raw materials.

Material	SP*(°C)	QI**(%)	TI***(%)
soft pitch	46.0	4.6	13.8
soft pitch/QUI	47.0	0.7	5.6
soft pitch/WO	45.3	0.2	9.7
soft pitch/NO	49.7	0.3	10.7

SP : softening point temperature; **QI : quinoline insoluble fraction;** TI : toluene insoluble fraction.

Mesophase conversion heat treatment was performed at a semi-pilot polymerization reactor [4] operating at the following conditions : isothermal temperature of 480°C, nitrogen injection at 20 Nl/min; sampling intervals varying from 1.0 to 8.0 h.

All samples transformations induced by the process were followed by characterization (SP, QI, TI, Anisotropic phase content) of samples collected after each 1.0 h of processing at isothermal temperature.

Final mesophase pitch samples were named as MP, MP_{QUI}, MP_{NO} and MP_{WO} , and were produced at different processing times. These samples were analyzed by means of SP, fixed carbon, elementary hydrogen and carbon content ratio H/C, optical microscopy and X-Rays Diffraction. Results were analyzed in comparison with those obtained from the commercial mesophase AR-24 (Mitsubishi Gas Chemical Company).

Results and Discussion

Time dependent results of SP, QI, TI and Aniso are presented in figure 1. Physicochemistry and structural analysis are collected in Table I and II. Those results show that characterization parameters SP, TI, QI and Aniso increased more gradually with soaking time, for all solvent pre-treated samples. A higher increase rate of insoluble matter and the anisotropic phase content is observed for soft pitch, soft pitch/QUI and soft pitch/WO samples. This fact is believed to induce a faster increase of the softening temperature, leading to the loss of thermoplastic characteristic.

A smooth and homogeneous transformation is observed in soft pitch/NO sample, probably due to the chemical modification induced by naphthalene base solvents (NO) and the high yield of QI extraction presented by this oil. The primary influence of this chemical modification of pitch is observed as a mesophase sphere growth agent in detriment of the nucleation rate. This complex formation and growth of the mesophase spheres in the isotropic media of soft pitch/NO, leads to a very high mesophase conversion rate (70,5%).

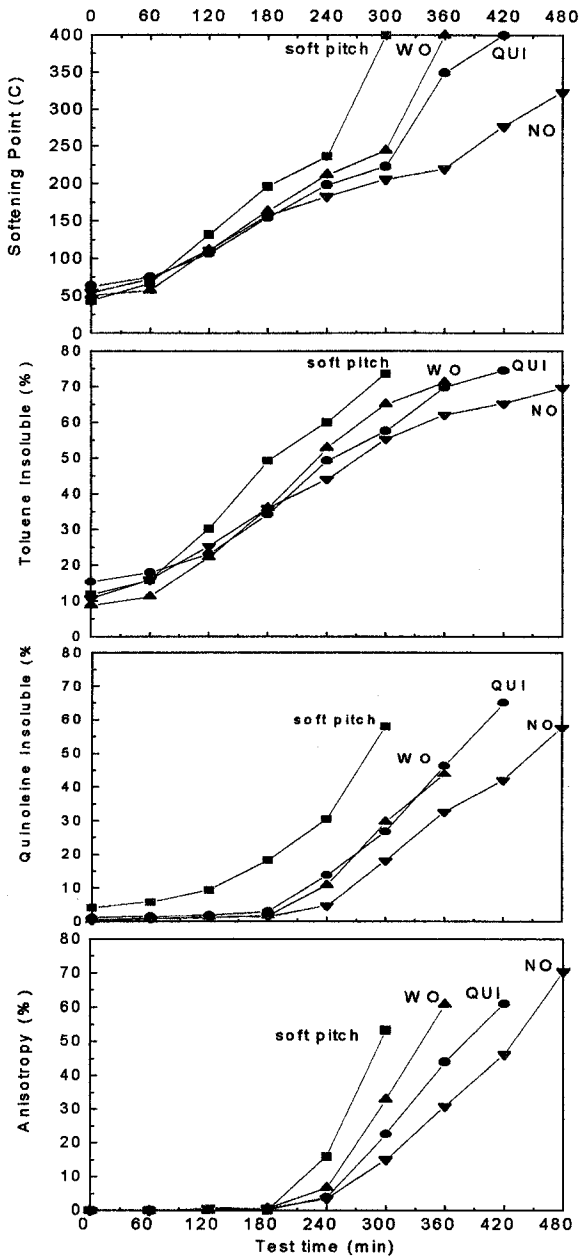

Figure 1. Time-dependent characteristics of samples heat-treated at 480°C; WO = soft pitch/WO; QUI = soft pitch/QUI and NO = soft pitch/NO.

Table I - Physicochemistry characteristics of the mesophase pitches.

	SP (oC)	QI (%)	TI (%)	FC (%)	H/C
AR24		55.6	73.0	82.9	0.60
MP_{QUI}		65.0	74.6	89.6	0.48
MP_{WO}		43.8	71.2	90.4	0.49
MP_{NO}		57.7	69.8	88.2	0.50

Table II - Structural analysis of mesophase pitches.

	Aniso (%)	d_{002} (Å)	Lc (Å)
AR24	100.0	3.536	35.924
MP_{QUI}	61.0	3.455	24.354
MP_{WO}	60.8	3.467	23.449
MP_{NO}	70.5	3.457	26.395

The MP_{NO} sample showed a thermoplastic characteristic, even for treatments as long as 8 hs, which in application as a precursor of carbon materials may be decisive. Its general characteristics (SP, QI, TI, FC) are quite close to those presented by AR24, as are its optical microscopy texture that revealed to be of flow type, similar to that showed by AR24, as can be observed in figure 2. The H/C, Aniso contents and crystallographic parameters values, on the other hand, are significantly different, as a consequence of their characteristic chemical structure.

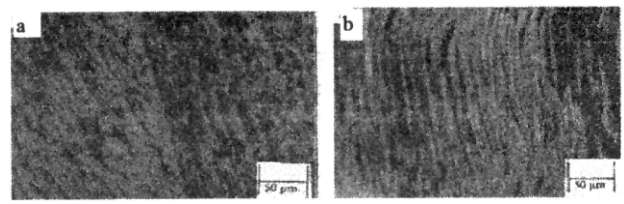

Figure 2. Micrographs of (a) MP_{NO} and (b) AR24.

Conclusions

Among the solvents investigated, the NO was found to be a chemically active agent which improves the yield and quality of final mesophase products.

The MP_{NO} with relatively low contents of insoluble matter, low aromaticity and high anisotropy associated with low softening temperature may be suitable as raw material for specialty carbon processing.

Acknowledgment

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References

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