

POSTER

The Characterization and Utilization of Fullerene Soot

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1. Introduction

Arc-discharge of graphite electrode have been reported and widely applied to produce toluene-soluble C₆₀ and C₇₀ of large interest, however their yields are very limited to less than 15wt%. The major product is toluene-insoluble soot like materials¹. In the present study, such soot was characterized by solvent solubility, structural analysis by X-ray and SEM, and TGA reactivity against oxygen and graphitizability in comparing with conventional carbon blacks. Application of soot as the carbon catalyst and electrode will be described.

2. Experimental

Fullerene soots were obtained bought from MER Corp(U.S.A.), or prepared in our laboratory by arc-discharge. Fullerene soots were produced under some conditions(discharge voltage, pressure and flow rate of inert gas). Two types of carbon black, KB and MA were used for comparison.

Fullerene soots were then extracted with toluene and then quinoline, separated into three fractions, toluene-soluble(TS), toluene-insoluble but quinoline-soluble(TI-QS), and quinoline-insoluble(QI). QI was further separated by its density. The structural characterization of QI and heat-treated QI were carried out by means of X-ray diffraction, high resolution SEM. The oxidation reactivities of QIs were also measured by TGA under the O₂(20%)/N₂.

3. Results

Extraction yields of as received MER-soot are shown in **Table 1**. The yield of TS was only 11wt%. The major product was QI of 72wt%. It should be noted that the yield of TI-QS which may contain giant fullerene such as C₃₀₀² was 17wt%.

Fig.1 shows the high resolution SEM photograph of as received MER soot, MER-QI and carbon blacks. All of them basically consist of very small particles of 10~100 nm except some stucked particles in as received MER soot. Such particles perfectly removed by quinoline extraction as shown in MER-QI.

TGA profiles of MER-QI under N₂ were compared with carbon blacks in **Fig.2**. MER-QI lost its weight sharply up to 600°C and then gradually up to 1000°C. About 25wt% of MER-QI evaporated at 1000°C, almost corresponding to the sum weight of TS and TI-QS. In contrast, no weight loss was observable in carbon blacks.

Fig.3 shows the X-ray diffraction patterns of MER-QI and heat-treated one. MER-QI exhibited two peaks, very broad peak around 16° and rather sharp peak around 26.5°, corresponding amorphous and graphite-like structure. The former peak shifted to higher angle of about 26.3° by the heat-treatment at 2500°C although this still located at lower angle than latter, indicating that MER-QI consists of two types of carbons of different graphitizably.

TGA profiles of MER-QI in 20% O₂ are illustrated in **Fig.4**. Definite weight decrease of both carbon blacks began at higher temperature than 550°C, whereas MER-QI began to lose its weight at lower temperature of 300°C. It is clearly indicated that the weight loss profile of MER-QI consists of two steps, lower than 600°C and higher than 600°C, again indicating that MER-QI consists of two types of carbons of different reactivity against oxygen.

Density distribution of QI is shown in **Fig.5**. This profile indicates that QI consists of two fractions, their densities being 1.6 g/cm³ and 2.15 g/cm³.

4. Discussion

Carbons produced by arc-discharge of graphite electrode can be largely classified into two types. One is non-graphitizable carbon and the other is graphitizable one. The latter may consists of flat sp² hexagonal network of carbons. In contrast, the former partially contains pentagonal rings and formed non-flat structure. Although both carbons of smaller molecules evaporate during the heat-treatment, larger molecules condensed and remained as graphitizable and non-graphitizable carbons, respectively, by heat-treatment at 2500°C. Such carbons in QI showed different gravity and reactivity against oxygen, indicating separation and concentration possibility.

References

1. W.Kratschmer, L. Lamb, K. Foristopoulous and D. Huffman, *Nature*, **347**, 354(1990)
2. H. Shinohara, H. Sato, Y. Sato, M. Takayama, A. Izuoka and T. Sugawara, *J. Phys. Chem.*, **95**, 8449(1991)

Table 1. The Yield of Extraction

MER-TS (C ₆₀ , C ₇₀)	11%
MER-TI-QS	17%
MER-QI (carbon form)	72%

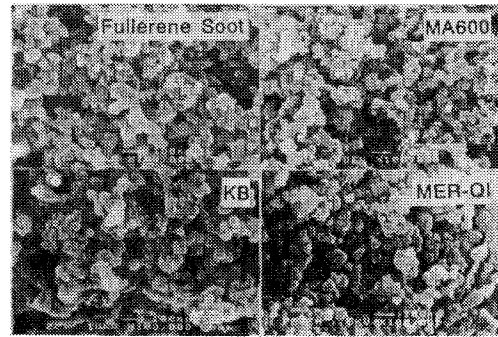


Fig.1. SEM Photographs of Fullerene soot and Carbon Blacks

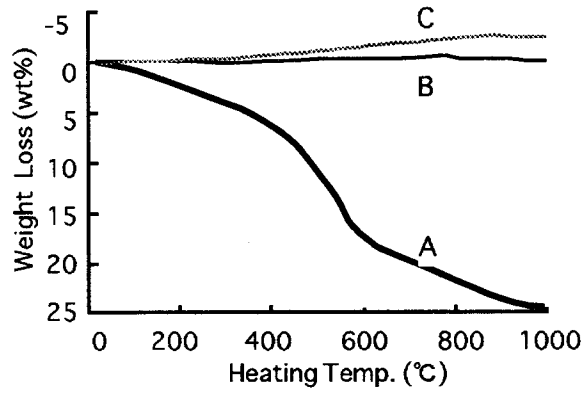


Fig.2. TGA Profiles of QI and Carbon Blacks in N₂
A. MER-QI B. MA600 C. KB

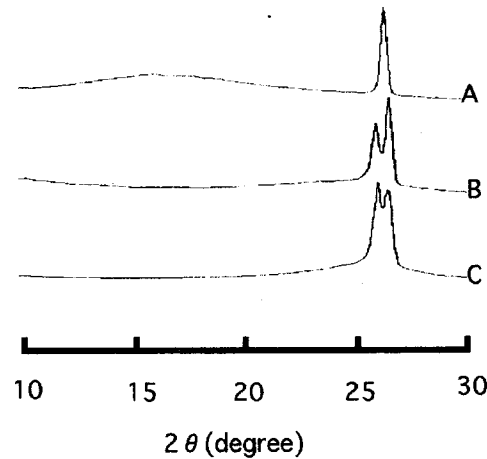


Fig.3 X-Ray Diffraction Patterns of QI and its Heated Materials
A. QI B. 2000°C Heated C. 2500°C Heated

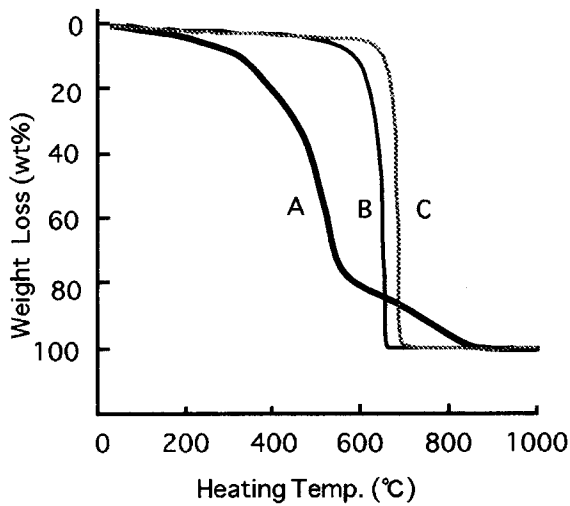


Fig.4. TGA Profiles of QI and Carbon Blacks in O₂(20%)/N₂
A. MER-QI B. MA600 C. KB

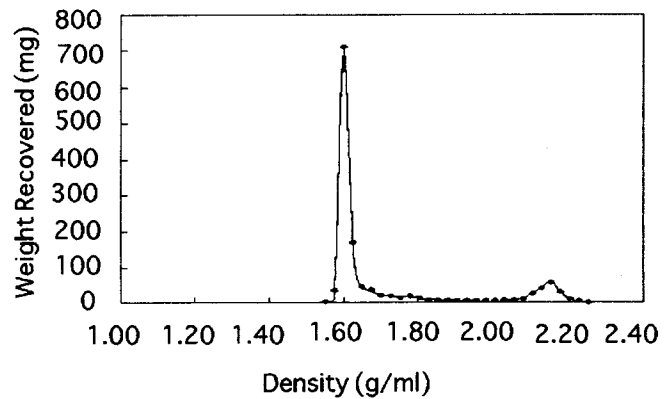


Fig.5. Density Distribution of QI
(Total Weight: 1.7 g)