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High temperature and high pressure extraction of fullerenes from KH carbon

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The extraction of fullerenes from KH carbon at high temperature was studied by using a low-boiling-point solvent in a high-pressure soxleh extractor. However, the high temperature extarction resulted the high yields of fullerenes, the polymerization of solvents was observed.

1.INTRODUCTION

High-boiling-point solvent such as quinoline extraction at ambient-pressure is favorable in terms of solvating the fullerenes [1]. We supposed that solvent temperature and possibly pressures are key parameters in extraction of the fullerenes, especially of the giant fullerenes. We report the extraction of fullerenes from KH carbon (soot made by arc-discharge methods) using a low-boiling-point solvent in a high-pressure soxleh extractor. An extracted sample was analyzed by HPLC and mass spectrometry.

2. EXPERIMENTAL

The starting material was KH carbon that was synthesized by arc-discharge method by using VMC type 3 carbon cluster synthesizer. The conditions of synthesis were as following, a He pressure was 50 torr, diameter of anode carbon rod was 15 mm, dc current was 300 A and dc voltage was 24 volts. Approximately 1g of KH carbon had first been solvent-extracted with toluene at ambient pressure. Then we tried extraction by using high-pressure soxleh extractor (Figure 1) at temperatures from 200° to 350°C. HPLC analysis was done by using Waters μ-Bondsphere C_{18} column (3.9x159mm). A mobile phase was toluene/methanol (55:45) and flow rate was 1.0 ml/min. The JEOL DX303 mass spectrometer was used to obtain FAB mass spectrum and m-nitrobenzylalchol was used as matrix.

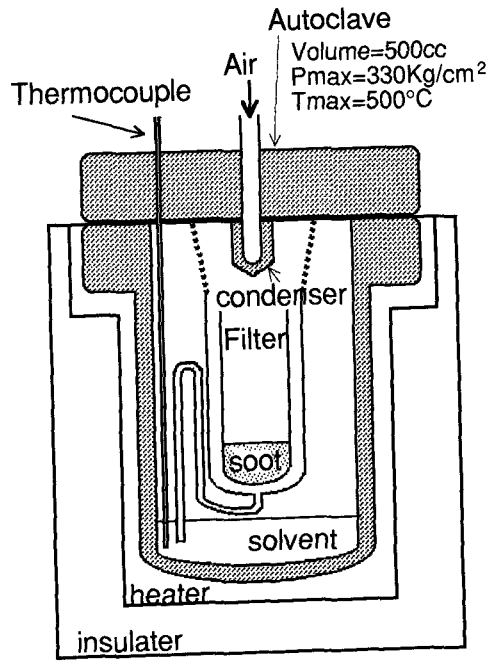

Figure 1 High pressure soxleh extractor

3. RESULTS AND DISCUSSION

The yield of ambient pressure solvent extraction was approximately up to 9% and main component was the C_{60} and C_{70}. These extra yields of high-pressure soxleh extraction were approximately up to 5% (Table 1).

Table 1 Results of extraction

Temp(°C)	110	250	300
		(After Extraction at 110°C)	
Yields (wt%)	8.91	5.88	4.98

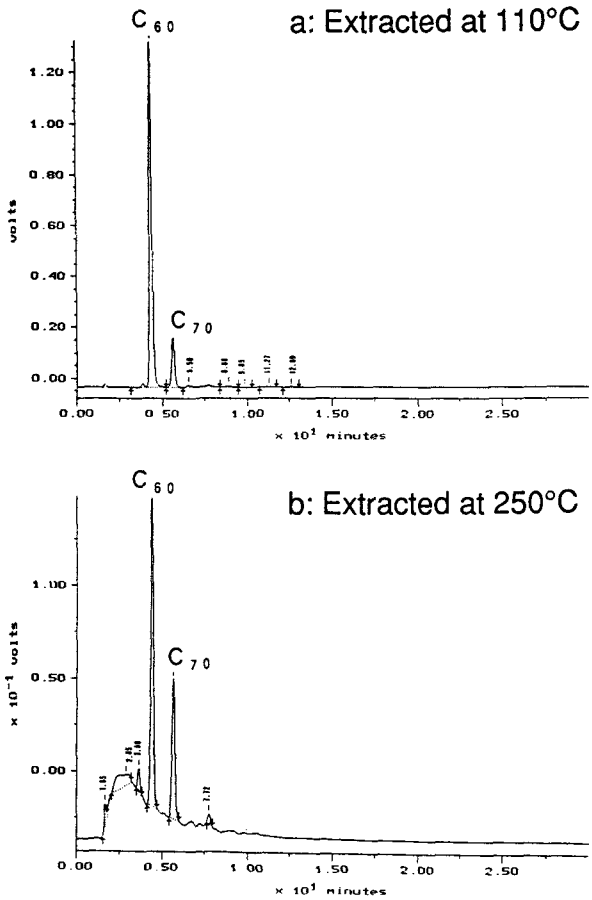

Figure 2 HPLC analysis of extracts

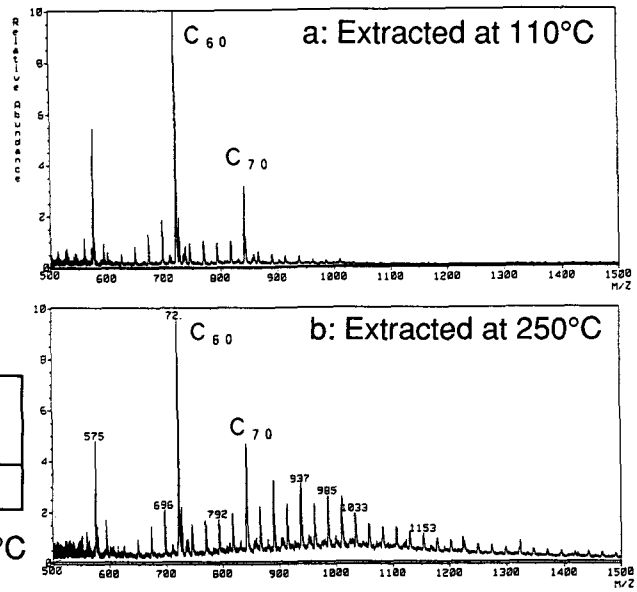

Figure 3 FAB mass spectrum of extracts

HPLC results (Figure 2) showed that a yield of C_{70} at the higher temperature extraction of fullerenes is 4 times higher than that of 110°C and there was an obviously large production of hydrocarbon compounds probably the result of solvent polymerization or reaction of solvent and fullerenes. FAB mass spectra (Figure 3) showed that the product of high temperature extraction of fullerenes contains much giant fullerenes (up to C_{120}). The disadvantage of high-pressure soxleh extractor was that the extracted sample was kept high temperature in a bottom of soxleh extractor during long extraction time. Therefore, we developed high-pressure flow extractor that combined with photo-diode array UV detector. In this extractor, the extracted sample was cooled quickly after extraction and we monitored UV absorption spectra directly. By using this extractor, we are studying characteristic of extraction of the fullerenes along with temperature.

REFERENCE

1) M. Shinohara et al, J.Phys.Chem. 95 (1991) 8449