

Fullerenes: C₆₀ from camphor – A novel approach

Kingsuk Mukhopadhyay, Kalaga Murali Krishna and Maheshwar Sharon

Department of Chemistry, Indian Institute of Technology, Bombay 400 076, India

C₆₀ and other members of the fullerene family have been isolated by vacuum evaporation technique from the toluene extract of ether insoluble camphor soot for the first time. Also, with the help of hot filament chemical vapour deposition technique, under the optimized conditions, we are able to avoid the tedious chromatographic separation method. Fast atom bombardment mass spectroscopic studies and scanning electron micrographs reveal the presence of C₆₀, a few smaller fullerenes and fullerene-based tubules of various sizes (0.14–1.15 mm)

THE pioneering work of Kroto and co-workers¹⁻⁴ on Buckminsterfullerene by laser vaporization of graphite and its consequent applications as a superconducting material^{5,6} led scientists to find an economical process

for its production. Fullerenes have largely been produced from graphite^{1-4,7}, burning of benzene⁸ and hydrocarbon combustion⁹. Here we report the generation of fullerenes from a natural source: camphor (which is an extract of a tree, mostly found in Asia, particularly in India and China) for the first time by the vacuum evaporation of toluene extract of ether insoluble camphor soot.

The apparatus for burning camphor and collecting its soot consists of a 1 m long and 10 cm diameter glass tube, over which another tube (diameter 4 cm and length 1 m) is kept with the help of a conical funnel. The lower and the upper glass tubes are held by clamps. A slow gas exhaust is connected to the upper end of the upper tube. Both tubes are externally cooled by water circulation. Camphor is burnt at the bottom and soot is collected simply by scratching the wall of the tube with soft cotton.

The soot collected is treated with ether for 24 h in a Soxhlet apparatus. The ether insoluble product is again Soxhlet-extracted with toluene, to remove low molecular weight carbon clusters. The toluene is distilled off under argon atmosphere and the solid residue (hereafter referred to as toluene extract) is collected.

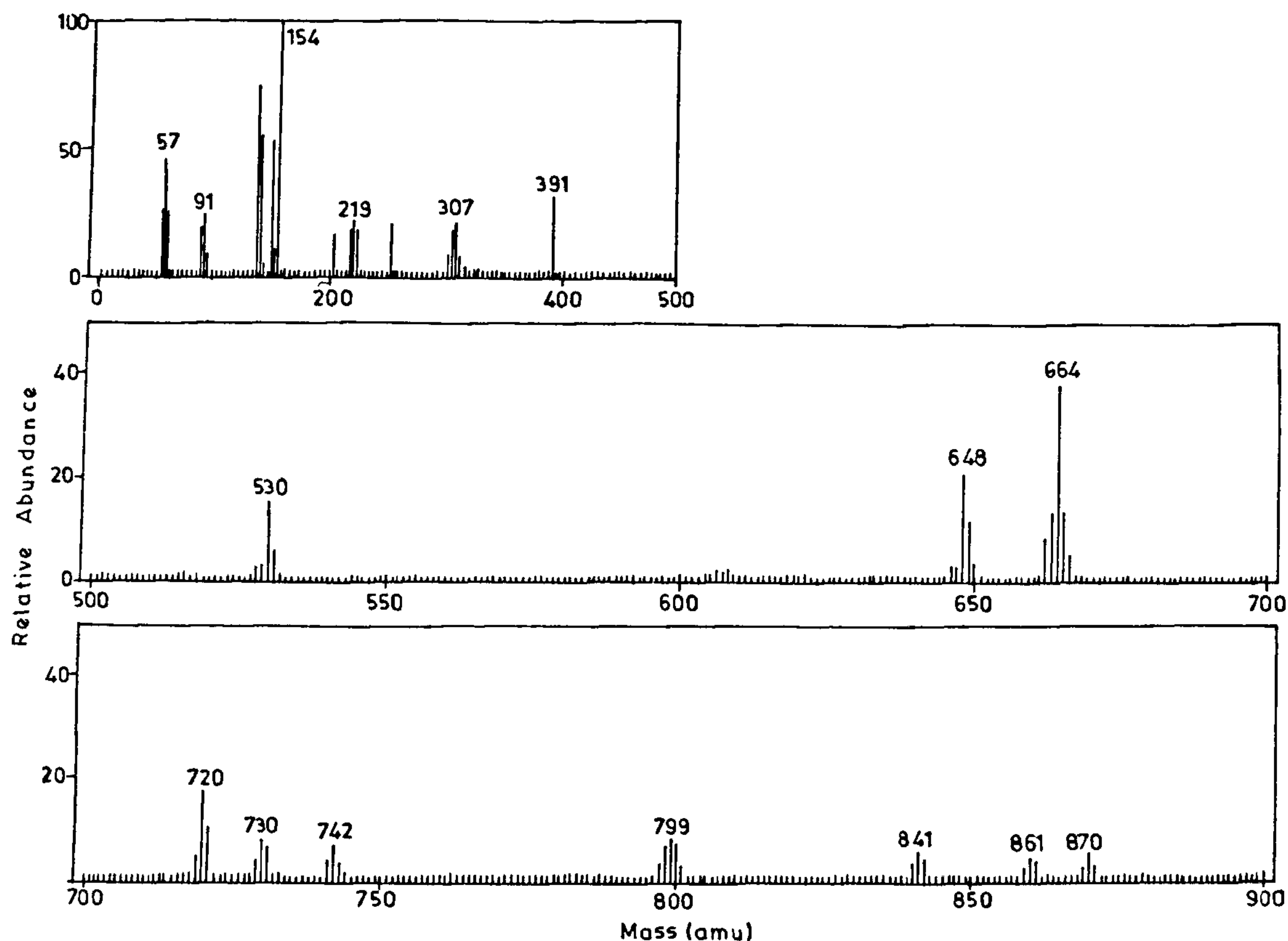


Figure 1. FAB mass spectrum of the vacuum deposited toluene extract of ether insoluble camphor soot on Si substrate confirming the presence of C₆₀. The spectrum also indicates the presence of other fullerenes.

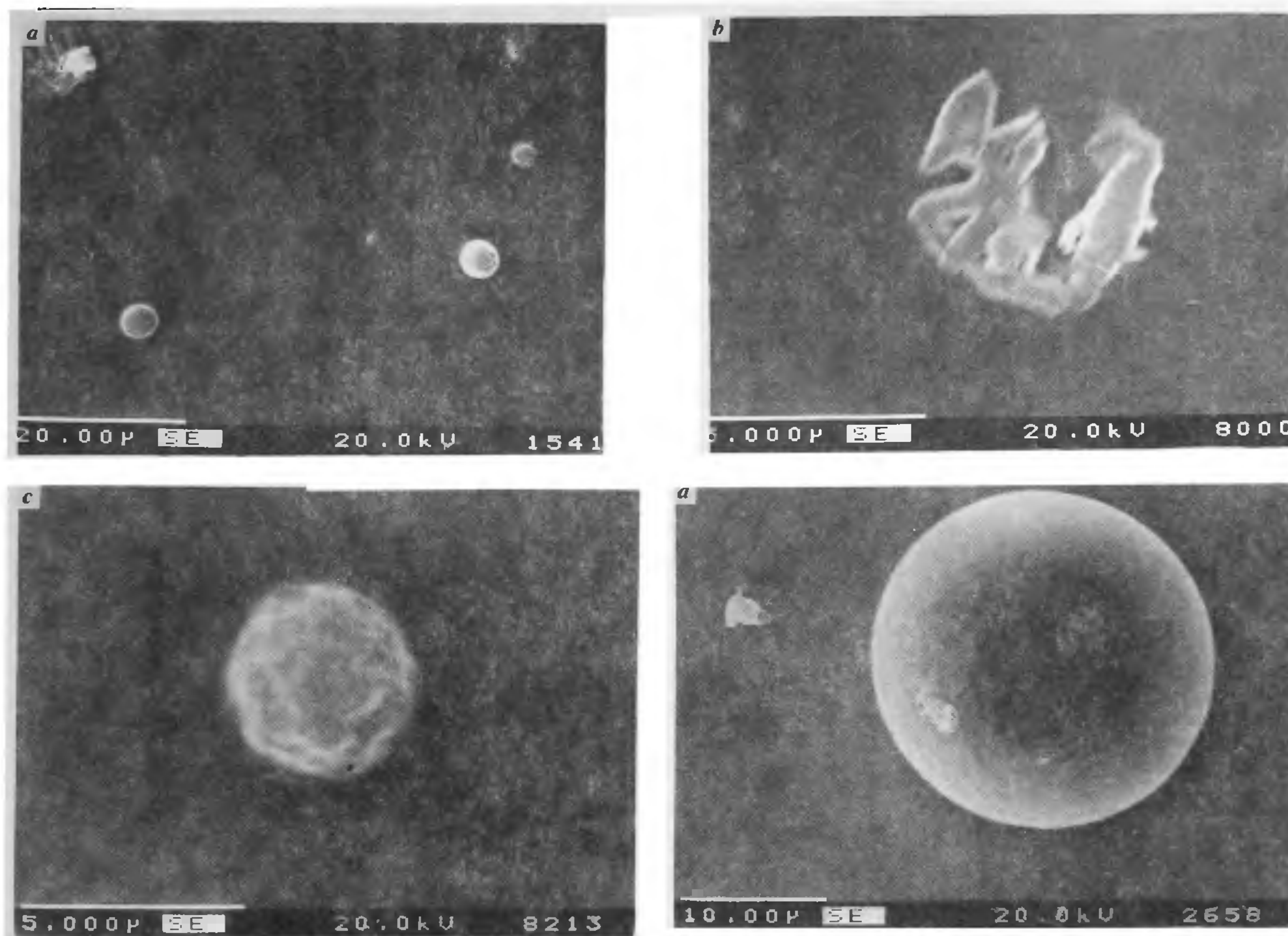


Figure 2. SEM images of C_{60} present in the film *a*, few C_{60} clusters, *b*, incomplete growth of C_{60} , *c*, fully grown C_{60} soccerball and *d*, magnified image of C_{60} selected from *a*

A small quartz boat is used as sample holder. The toluene extract is then transferred into the boat and subjected to hot filament chemical vapour deposition under 10^{-5} torr at substrate temperature of 140–150°C. Deposition of fullerenes is made over glass, Si wafer and Ti substrates. The temperature of the filament is maintained in the range of 600–700°C. The distance between the filament and the substrate heater is kept at about 2.5–3.75 cm. A thin yellow film of fullerenes is obtained after 30 min of deposition. The residual material left in the boat is shiny, conducting, granular and insoluble in toluene, whereas, the deposited material on the substrate is soluble in benzene or toluene.

The fast atom bombardment (FAB) mass spectrum of the thin film is shown in Figure 1. The peaks show the presence of C_{60} (720 amu) and a few smaller fullerenes such as C_{54} (648 amu). This is further confirmed by Fourier-transform infrared spectroscopy which gave strong absorption peaks (consistent with those reported¹⁰ for C_{60}) along with other additional peaks. This is also followed by the UV-visible spectrometry and detailed thermal analysis of the deposited material, and the

findings are in agreement with the literature^{10, 11}. EDAX analysis shows the absence of sulphur and microanalysis confirms the presence of pure carbon. Also, X-ray diffraction studies confirmed the crystal structure of C_{60} as hcp¹².

Figures 2 and 3 show the electron micrographs of the film deposited on Si substrate. Figure 2*a* shows the surface distribution of ball-shaped clusters ($\approx 5\mu\text{m}$) on the substrate. Under the same conditions, Figure 2*b* shows the growth process of an unfinished cluster which is about to attain the spherical shape while the fully grown spherical-shaped cluster with facets like a soccer ball is shown in Figure 2*c*. Figure 2*d* is a magnified image of one of the C_{60} clusters present in Figure 2*a*. Figure 2 and the peak at 720 amu of mass spectrum (see Figure 1) confirm the soccer ball cluster as C_{60} .

Figure 3 shows the SEM pictures of fullerene-based tubules. These whiskers were 40 μm –1.15 mm in length and 10–100 μm in diameter. These tubules are comparable with the SEM images of C_{60} whiskers of the previous work¹¹. This is also obvious from



Figure 3. SEM images of the fullerene-based whiskers *a*, a long tubule with both ends closed; *b*, a small C_{60} ball seen underneath carbon whisker and a layer structured whisker is shown in *c*.

Figure 3 *b*, which shows a small partially hidden ball of C_{60} along the whisker.

These results suggest that fullerenes (C_{60}) can be obtained from a natural source – camphor. Hot filament vacuum deposition technique can replace the cumbersome chromatographic separation technique. Although 100% purified C_{60} has not been achieved, with better optimized conditions one would be able to produce much more C_{60} (both quantitatively and qualitatively) in the near future and efforts along these lines are under way in our group. At this stage, we would also like to mention an important observation, the residue obtained after the vacuum evaporation of the toluene extract is very shiny, granular, conducting and insoluble in toluene. Work is also in progress to characterize this material.

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ACKNOWLEDGEMENTS. We (MS and KM) thank DST, New Delhi, for financial support. We also thank Dr K. P. Madhusudhanan of CDRI, Lucknow, for assistance in obtaining mass spectra, and Mr B. Kiran of Chemical Engineering Department of IIT Bombay for helping us in taking the SEM pictures.

Received 18 February 1994, revised accepted 1 July 1994