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Fullerene Production from Pyrolysis of Chlorodifluoromethane

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Abstract

The formation of fullerenes and amorphous carbon from flame combustion (pyrolysis) of chlorodifluoromethane ($CHCLF_2$) was found to involve different systems of carbon clusters called fullerene intermediates. The correlation provides insight into the formation of fullerenes and other carbon clusters.Gas chromatograph mass spectrometer (GCMS) was employed for the separation and characterization of the reaction products, comprising C_{60} and other carbon clusters which molecular formula were determined from their special isotopic patterns. UV-spectrophotometer and Fourier transform infrared (FTIR) spectrophotometer revealed a strong absorption peak at wavelength range of 250nm-300nm.

Keywords*:* pyrolysis method of plasma; fullerene; chlorodifluoromethane.

1.0 Introduction

Nowadays, the interest in nanoscale materials has rapidly increased due to their positive use in various technical and scientific researches relating to the energy, environment, and biomedical fields. Carbon is one of the most amazing elements with a vast range of exotic properties. These properties have shed the light to carbon-based nanostructures, such as fullerenes [1], carbon nanotubes [2], and graphene [3]. Since its discovery in 1985 (Nobel Prize in 1996) [1], the subject of fullerene molecule C_{60}, the closed-cage carbon molecules, has attracted immense interest and incredible attention in enormous number of medical and technological applications due to its fascinating chemical and physical properties [4-7]. Indeed, the discovery of fullerenes has opened the gate for the production and investigation of much more suitable material building blocks which could be used as drug deliverers in medical applications and/or possible superconductors in technological applications. Therefore, fullerene provides an entirely new branch of chemistry, materials science, and physics. Electronically, solid fullerene is treated as a semiconductor material [8] and its structure is described in the face-centered-cubic lattice. Unlike the carbon nanotubes with only hexagonal cells, the fullerene consists of both pentagons and hexagons with two classes of bond lengths: a shorter bond length connecting two successive hexagons and a longer bond length joining a hexagon and pentagon. While the dependence of properties at nanoscale provides an opportunity to test and experiment with various materials, the fullerene is considered as a suitable candidate for nanotechnology since it has the right size and chemical stability. Therefore, the properties of fullerenes can be tuned and enhanced by doping various elements. Depending on the site of doping, fullerenes can be functionalized into three main categories: exohedral fullerenes with the dopant outside the cage, endohedral fullerenes with the dopant inside the cage, and on-site fullerene with the dopant replacing one C atom.
Having established the importance and suitability of fullerene C_{60} for different potential applications, the fullerene-based materials would be of extreme interest for promising applications in nanotechnology world [9].

2.0 Experimental Procedure

Fullerenes soot was produced in a quartz column reactor as shown in Figure1, which consist of plasma reactor, dust collector, quencher and vacuum pump, etc, When the reactor chamber was powered by a 250W, and pumped at a pressure of 0.005 Mpa, microwave was introduced along square wave-conduct pipe, stable plasma was observed in the reactor under a low pressure atmosphere. Chlorodifluoromethane ($CHClF_2$)gas fed at 0.15 g/min was vaporized into a reactor through the central nozzle of a transferred direct current plasma torch, which equipped on the top of the quartz column.

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At the plasma zone chlorodifluoromethane gas ($CHClF_2$) was dissociated to form carbon soot. By controlling the operating parameters, such as cooling temperature and reaction time, the product (carbon soot) were collected for extraction with chloroform using an ultrasonic bath and filtration at room temperature. A brownish solution of the extract containing dissolved fullerenes and other products was separated and analyzed by gas chromatography mass spectrometer (GCMS) detection, Fourier transform infra-red spectrometer (FTIR) and Ultraviolet-visible spectroscopy (UV-SPEC). This experiment was done at Sheda Science and Technology Complex (SHESTCO), Federal Ministry Of Science and Technology Abuja.

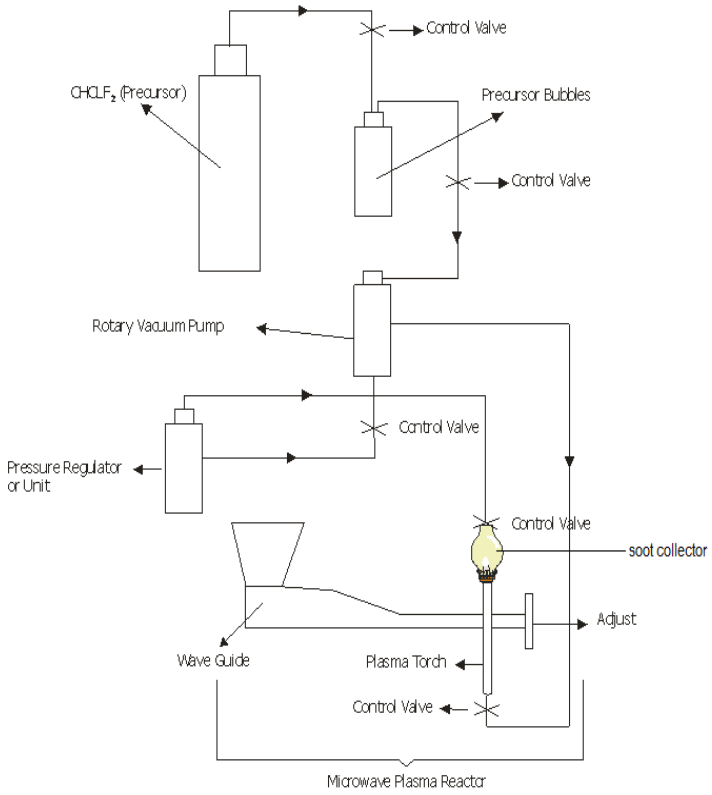

Fig. 1: Schematic process of Microwave Synthesis System of Fullerene/Intermediates.

3.0 Results and Discussion

The gas chromatography mass spectrometer analysis of the Microwave plasma synthesized fullerene and fullerene intermediates from chlorodifluoromethane ($CHClF_2$) produced a series of carbon clusters as shown in Figure 2, some of these carbon clusters can be identified from its mass and the isotopic patterns from mass spectrum as labeled in Figure 3. From the masses and the isotopic patterns of molecular ion peaks in the recorded mass spectra, chemical formulas of the products separated by gaschromatography mass spectrometer were determined, example $C_{18}H_{32}O_2$, $C_{18}H_{34}O_2$, $C_{21}H_{18}O_2$, $C_{21}H_{40}O_4$, $C_{18}H_{31}ClO$, etc. In the proposed microwave plasma reaction, all the products, fullerenes, and the fullerene intermediate, were produced from chlorodifluoromethane ($CHClF_2$), a compound containing a single carbon atom only. From the GCMS analysis in figure 2, although we could not observe a clear concentration peak for fullerene C_{60} due to its small yield in the synthesized sample, but on the other hand, some results on the optical measurements had made it plausible to infer the presences of C_{60} as compared with result in [10]. Hence the UV-spectra obtain in Figure 4 were virtually identical to those of standard $C_{60,}$ other carbon clusters were equally found to be present in large concentration, as revealed in Figure 2. Therefore, it is reasonable to say that these carbon clusters are the intermediates of fullerenes, and further investigation of their structures will shed light to the formation mechanism of fullerenes. The above experiment can be considered as a preliminary study for the production of C_{60} provided the necessary conditions are considered for fullerene production. Also it is very important to note that the above process (microwave synthesis) can be operated continuously and the reaction device can be easily scaled up, so the microwave synthesis reaction from chlorodifluoromethane ($CHClF_2$) might be applied to the economical production of fullerenes in the future.

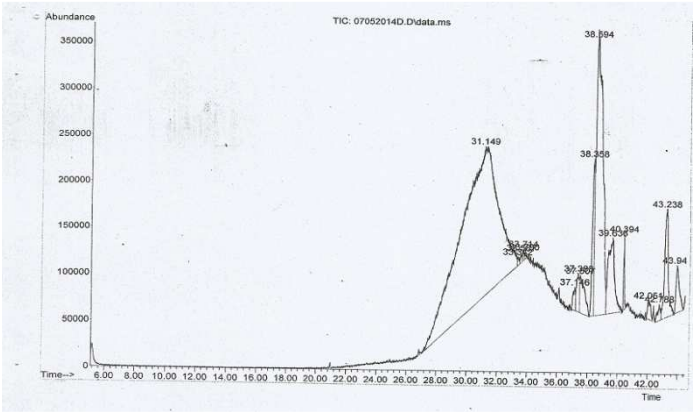

Fig 2: Gas chromatography mass spectrometer analysis of the fullerene sample, showing the different concentration of the various carbon clusters.

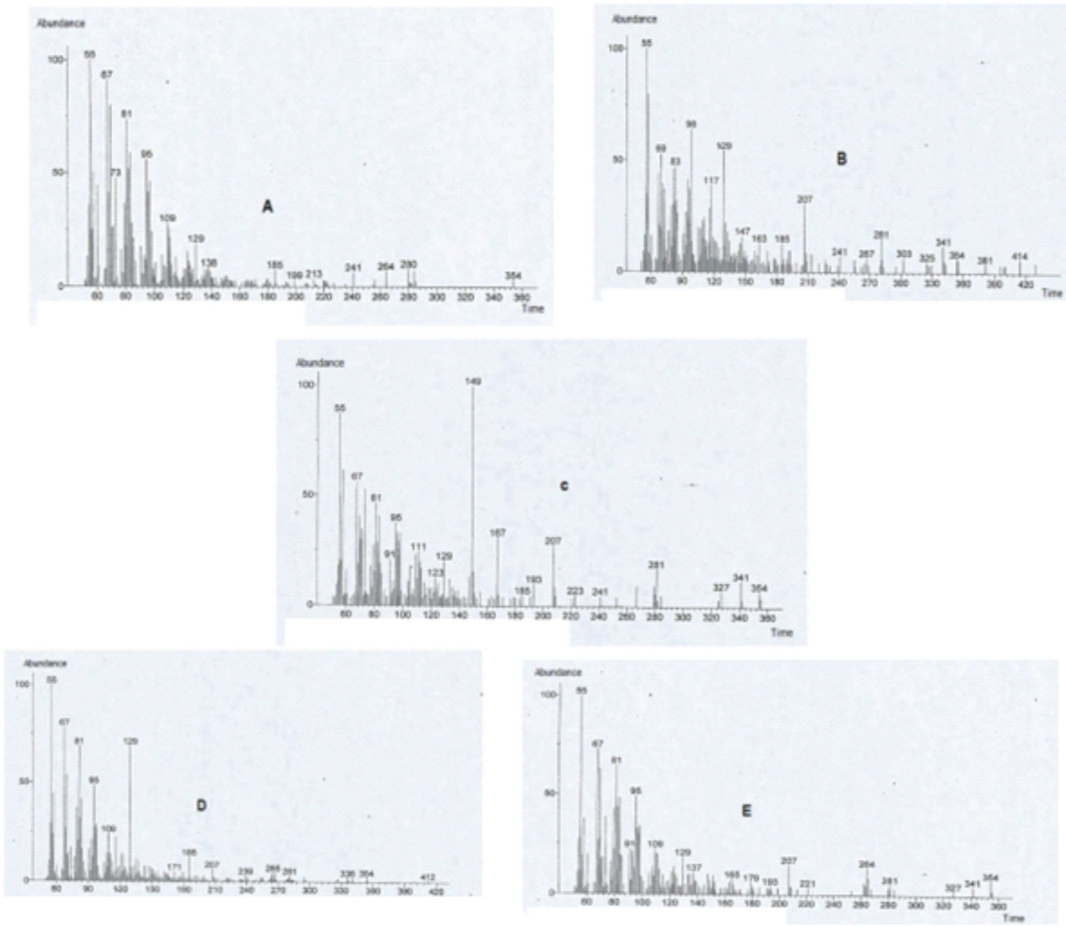

Fig. 3: Isotope Distribution of selected carbon cluster present in the fullerene sample as recorded in the mass spectra A: 9, 12-octadecadienoi acide ($C_{18}H_{32}O_2$), B: Oleic acide ($C_{18}H_{34}O_2$), C: [1,1-bicyclopropyl]-2-octanoic acide 2-hexyl-methyl ester ($C_{21}H_{38}O_2$), D: 9-Octadecenoic acid-2-hydoxy-1-hydroxymethyl ethyl ester ($C_{21}H_{40}O_4$), E: 9,12-Octadecadienoyi chloride ($C_{18}H_{31}ClO$)

3.1 Ultraviolet-Visible Spectroscopy (UV-Spec) and Fourier Transform Infrared Spectrometer Analysis of Fullerenes

Figure 4 shows the plot of absorbance versus wavelength of the fullerene samples, from the graph, a strong absorption peak was observed at wavelength range of 250nm-300nm. This result is in a close agreement with the result of [10] who reported a strong absorption of (300nm-350nm) range of wavelength. The above result is virtually identical to those of standard $C_{60;}$ hence the result clearly indicates that the material has potential application in fabrication of solar cell. Also Figure 5 shows the Fourier transform infrared spectrophotometer (FTIR) analysis of the fullerene samples, from the diagram, up to six characteristics absorption peaks can be observed, with wave number 434, 840.99, 1410.98, 1638.58, 2847.03, and 3430.51 cm^{-1} respectively, but because the carbon soot was dissolved with a chloroform, the amorphous carbon, graphite and other hydrocarbons were removed, as a result, the characteristic absorption peaks stand out obviously.

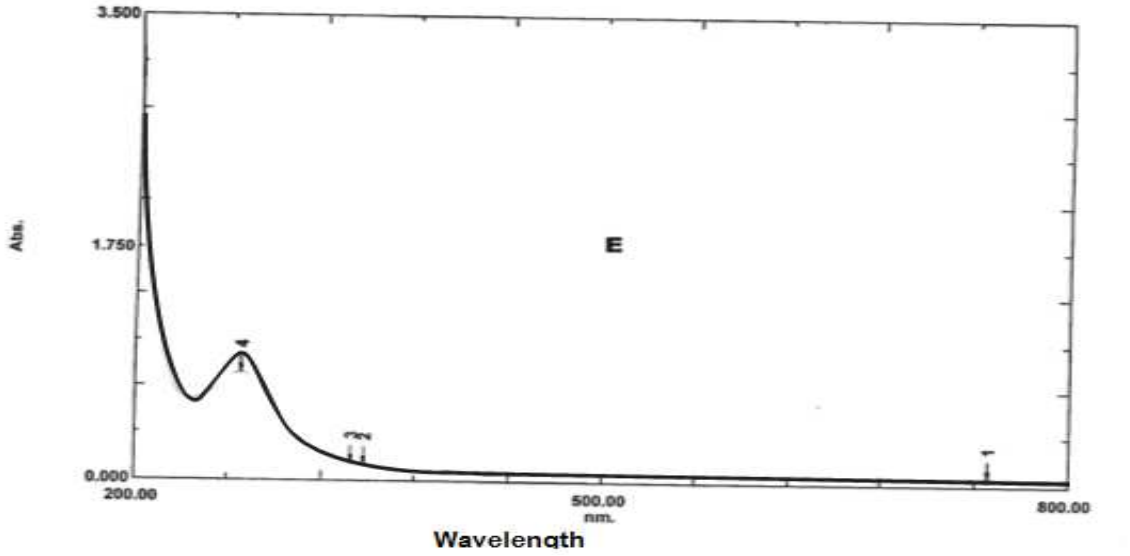

Fig 4: UV- spec of the fullerene samples showing the wave length of the absorption peaks.

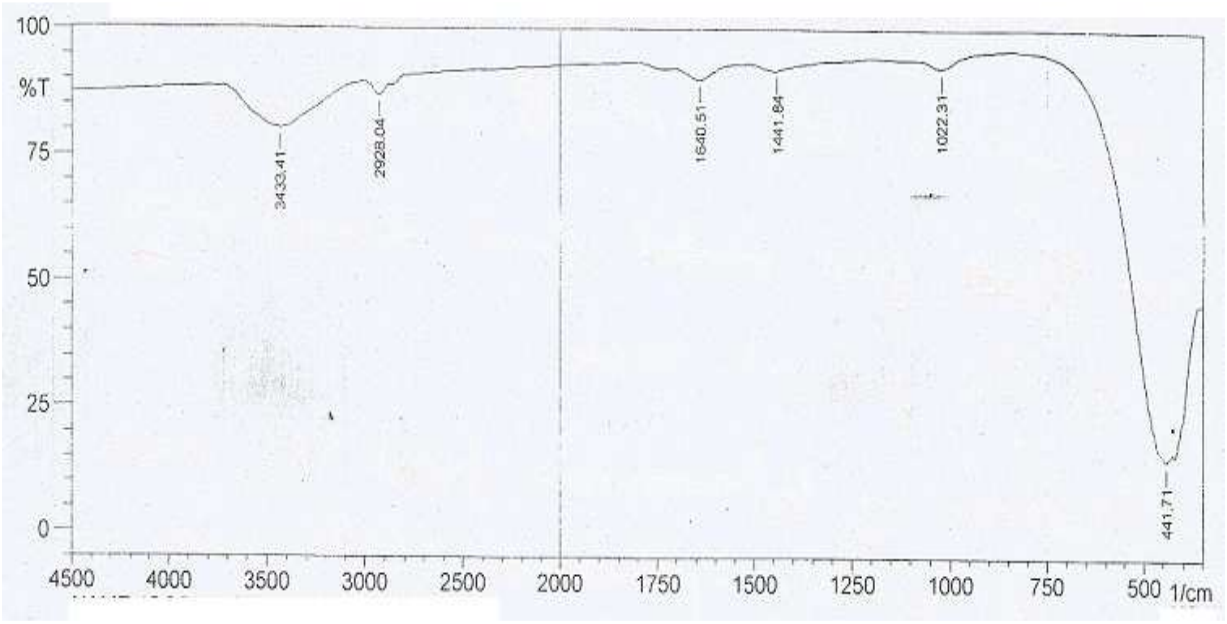

Fig 5: The FTIR analysis of the fullerene sample

5.2 Conclusion

A micro wave plasma reactor has been used for the synthesis of fullerene and other carbon clusters from chlorodifluoromethane ($CHClF_2$). Therefore, it is reasonable to say that these carbon clusters are the intermediates of fullerenes, and further investigation of their structures will shed light to the formation mechanism of fullerenes. Also, the above experiment can be considered as a preliminary study for the production of C_{60} from chlorodifluoromethane ($CHClF_2$) provided the necessary conditions are considered for fullerene production. But is also very important to note that the above process (microwave synthesis) can be operated continuously and the reaction device can be easily scaled up, so the microwave synthesis reaction from chlorodifluoromethane ($CHClF_2$) might be applied to the economical production of fullerenes in the future[11].

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