IOSR Journal of Applied Physics (IOSR-JAP)
e-ISSN: 2278-4861.Volume 7, Issue 6 Ver. I (Nov. - Dec. 2015), PP 77-83
www.iosrjournals

Fullerenes Synthesis Using Fabricated Arc Discharge System with Relatively Large Chamber Size

P. I. Kyesmen[1], A. D. Onoja[2] and A. N. Amah[3]
[1](Physics Department, University of Agriculture Makurdi, Nigeria)
[2](Physics Department, University of Agriculture Makurdi, Nigeria)
[3](Physics Department, University of Agriculture Makurdi, Nigeria)

Abstract: *Arc discharge technique is still one of the popular techniques for producing fullerenes. This work describes an arc discharge installation with a chamber size of 140 mm diameter and 500 mm length that is able to produce fullerenes. Fullerenes solid was extracted from carbon soot collected from the arc discharge system at various experimental conditions of discharge current and chamber pressure using the solvent extraction technique. Fullerenes yield of up to 6 to 8 % was extracted from the fabricated discharge system which was higher when compared to the 3-4 % yield produced by an arc discharge system of 100 mm diameter and 200 mm length. The fabricated discharge system gave the best fullerenes percentage yield of 8 % at arc current of 150 A and 100 Torr of chamber pressure. Increase in inert atmosphere from 100 to 200 Torr on the amount of carbon soot vapourization but inhibited the percentage fullerenes yield from 8 to 6 %. Scanning Electron Microscopy (SEM) and Ultraviolet Visible (UV-Vis) Spectroscopy analysis carried out on all fullerenes solid extracted revealed the presence of fullerenes. The fabricated arc discharge system suggests that large chamber size could promote the formation the synthesis of fullerenes.*

Keywords:– *Chamber, Discharge, Fullerenes, Graphite, Synthesis.*

I. Introduction

The discovery of the arc discharge method of mass producing fullerenes in 1990 has resulted in a revolution in materials physics and chemistry research. This method of producing Fullerenes utilizes carbon arc method in which graphite electrodes are vaporized in a low pressure helium atmosphere by passing an electrical current through the electrodes, thus generating an arc which produces carbon soot containing fullerenes [1]. After the discovery of arc discharge method for fullerenes production other interesting methods have emerged. However, the arc discharge method of graphite conversion to fullerene-containing carbon condensate in helium atmosphere remains one of the popular methods [2].

Fullerenes have cutting-edge applications in technology such as the solar cells technology and molecular electronics among many other areas [3]. So, the search for system designs and ways for synthesizing macroscopic quantity of fullerenes still constitute a problem [2]. Large arc discharge chambers may be better in fullerenes yield than smaller ones. This is because larger chamber may be better at coping with pressure changes due to the heating effect and therefore may give better yields [4]. This work presents a fabricated arc discharge system with a relatively large chamber size for fullerenes synthesis.

II. Method

2.1 Discharge system fabrication

High current feedthroughs, mechanical feedthrough and all items needed in the arc discharge chamber were constructed and welded to a stainless steel sheet of 3 mm. The stainless steel sheet was then attached to a stainless steel flange as shown on Fig. 1 below. The mechanical feedthrough was constructed such that it made contact with one of the ceramics holding one of the electrodes so as to make the electrode movable

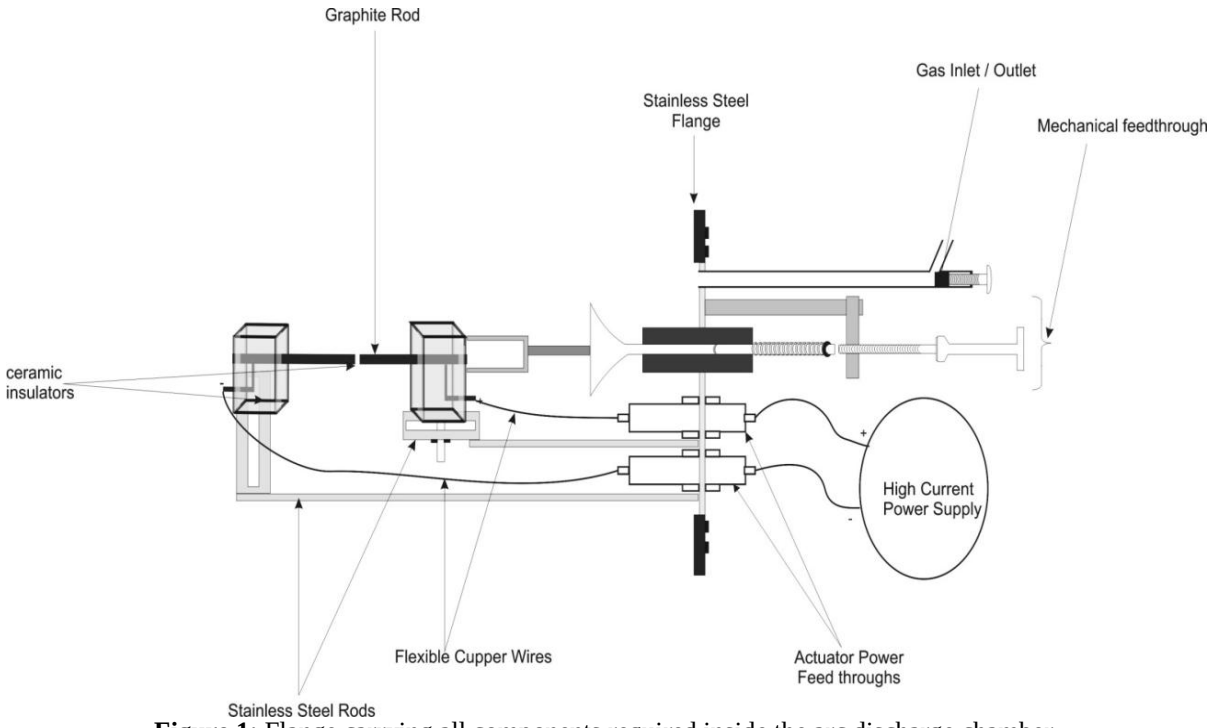

Figure 1: Flange carrying all components required inside the arc discharge chamber

In another phase of the discharge system fabrication, stainless steel cylinder of 140 mm diameter and 500 mm length which had a flange at one end was used to form the arc discharge chamber. A viewing window to the chamber was constructed using a stainless steel cylinder. Also, galvanized and foldable Iron sheet of 1.5 mm thickness was used to form the outer layer of the doublr wall cylinder with water inlet and outlet as shown in Fig 2 below.

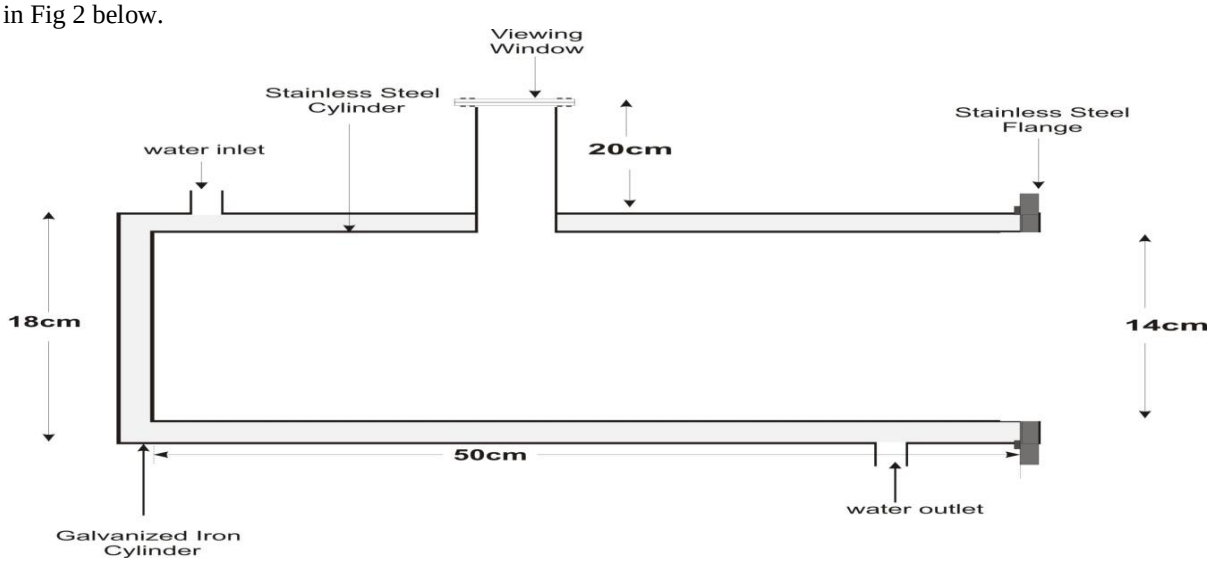

Figure 2: fabricated double walled cylinder with a viewing window

The systems as fabricated in Fig. 1 and Fig. 2 above were brought together and bolted firmly at the flanges as shown in figure 3 below. An arc welding transformer was connected to the high current feedthroughs to serve as high current source. The system as set up in Fig. 3 below forms a complete system for arc discharge method of fullerenes synthesis with a chamber dimension of 140 X 500 mm.

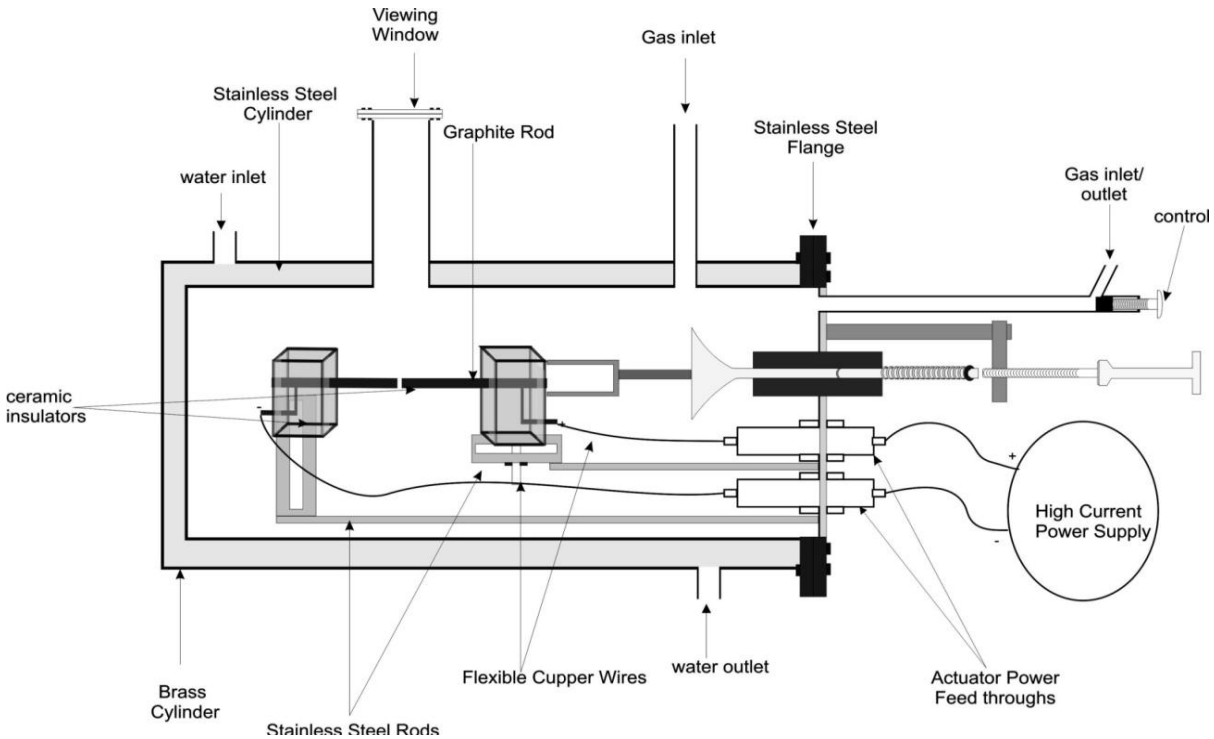

Figure 3: Arc discharge system for fullerenes synthesis

2.2 Experimental procedure

The fabricated arc discharge system for fullerenes synthesis was setup as shown in Fig. 3 above. The two electrodes in the stainless steel chamber were set up such that they are about 1 to 2 mm apart and one of the electrodes was movable. The movement of the movable electrode was such that it can be control from outside the stainless chamber by a mechanical feedthrough. First, air inside the stainless steel chamber was pumped out of the chamber using a vacuum pump to a pressure of $\leq 10^{-3}$ Torr. Helium gas was then introduced into the chamber from the helium gas cylinder to a pressure of 100 Torr. High current of 100 Ampere was supplied to the electrodes using the Arc Welding Transformer power supply. The mechanical feedthrough was used to push the movable electrode such that the two electrodes were just touching each other and then released. This initiated an arc discharge between the electrodes which resulted to the vaporization of the graphite electrodes. The mechanical feedthrough was then used to control the movable electrode such that the 1 to 2 mm gap between the electrodes, required to sustain the arc discharge was maintained. The arc discharge was made to run for about 2 minutes. While the arc discharge occurs in the chamber, water was made to circulate round between the walls of the stainless steel chamber and the galvanized iron cylinder through the water inlet and outlet channels to provide cooling for the stainless chamber. During the arc discharge, part of the carbon rods vapourized and deposited carbon soot on the water cooled wall surface of the stainless steel chamber. The arc discharge that took place inside the stainless chamber was viewed through the viewing window of the fabricated system with the aid of a camera. This helped in the control of the movable electrode in the chamber through the mechanical feedthrough. The same procedure was repeated but for different conditions of arc discharge current and stainless chamber pressures of 100 Ampere at 200 Torr, 150 Ampere at 100 Torrr and 150 A at 200 Torr.

2.3 Soot collection

After each of the experimental procedure in section 2.2 above, the discharge system was allowed to cool down for about 10 minutes. Also, the helium gas in the chamber was pumped out for about 10 minutes. By pumping out the gas, first; any toxic gases which may have been produced during the experimental procedure would be safely evacuated. Secondly, removing the gas helped compact the soot into a crusty layer and made the soot collection easier. After this, the flange that carried all the components in the stainless steel chamber was unbolted and removed. The soot deposited on the walls of the stainless steel chamber was scrapped off using a stiff brush and a long spatula and collected into a clean container.

2.4 Solvent extraction of fullerenes

First, about 250 mg (milligram) of the carbon soot that was collected at each of the arc discharge system operating condition of discharge current and chamber pressure was mixed with 100 mg of boiling toluene (fullerenes are soluble in toluene) and stirred for 4 hrs. The mixture was then filtered using a Whatman-

2 filter paper and an orange-yellow like colored solution was obtained. Re-crystallization of fullerenes from toluene solution was carried out by concentrating the solution with a water bath. The concentrate was then allowed to evaporate at room temperature and brown crystals of fullerenes were collected. The crystals were then washed with diethyl ether to remove any hydrocarbon that might be present.

2.5 Fullerenes characterization

All fullerenes solids obtained from the extraction process above were analyzed using Scanning Electron Microscopy (SEM) and Ultraviolet Visible (UV-Vis) Spectroscopy.

III. Results

3.1. Carbon Soot Collection Results

TABLE 1 below presents the quantity of carbon soot collected after every 2 minutes of arc discharge run at different experimental conditions. From the carbon soot collected from all the arc discharge operating conditions, the highest soot of 43.67 mg was collected when the discharge current was 100 A at 200 Torr of chamber pressure while the lowest soot of 26.67 was collected when the discharge current was 100 A at 200 Torr of chamber pressure.

3.2 Fullerenes Extraction Results

The quantity of fullerenes solid extracted from 250 mg of each carbon soot collected at different arc discharge system operating conditions are presented on Table 1 below. The highest fullerenes solid extracted was 20 mg about 8 % of the raw carbon soot obtained from the arc discharge operating condition of 150 A of discharge current and 100 Torr of chamber pressure while the lowest was 15 mg about 6 % of the raw carbon soot obtained at arc discharge system operating condition of 150 A and 200 Torr of discharge current and chamber pressure respectively.

Table 1: Carbon Soot Collection and Fullerenes Yield at all arc Discharge System Operation Conditions

Arc discharge system operating condition	Carbon soot collected for 2 minutes of arc discharge (mg)	Fullerenes Solid Extracted from 250 mg of carbon soot (mg)	Percentage of fullerenes solid extracted from carbon soot (%)
100 A, 100 Torr	38.00	19	7.60
100 A, 200 Torr	26.67	16	6.40
150 A, 100 Torr	29.50	20	8.00
150 A, 200 Torr	43.67	15	6.00

3.3 Fullerenes Characterization Results

3.3.1 Scanning electron microscopy of fullerenes

Scanning Electron Microscopy (SEM) micrographs of fullerenes solid extracted from carbon soot collected from arc discharge system operating conditions of arc discharge current and chamber pressure of 100 A and 100 Torr, 100 A and 200 Torr, 150 A and 100 Torr and 150 A and 200 Torr are shown in figures 4a, 4b, 4c and 4d respectively. The SEM micrograph of Fig. 4a shows a solid material with a regular morphological and polycrystalline pattern.The structural pattern of the SEM micrograph of Fig. 4b below could not be ascertained. Lastly, the SEM micrographs of Fig. 4c and 4d below shows tubule like whiskers with closed ends. All SEM micrographs showed materials average dimension which falls within 10 µm – 100 µm.

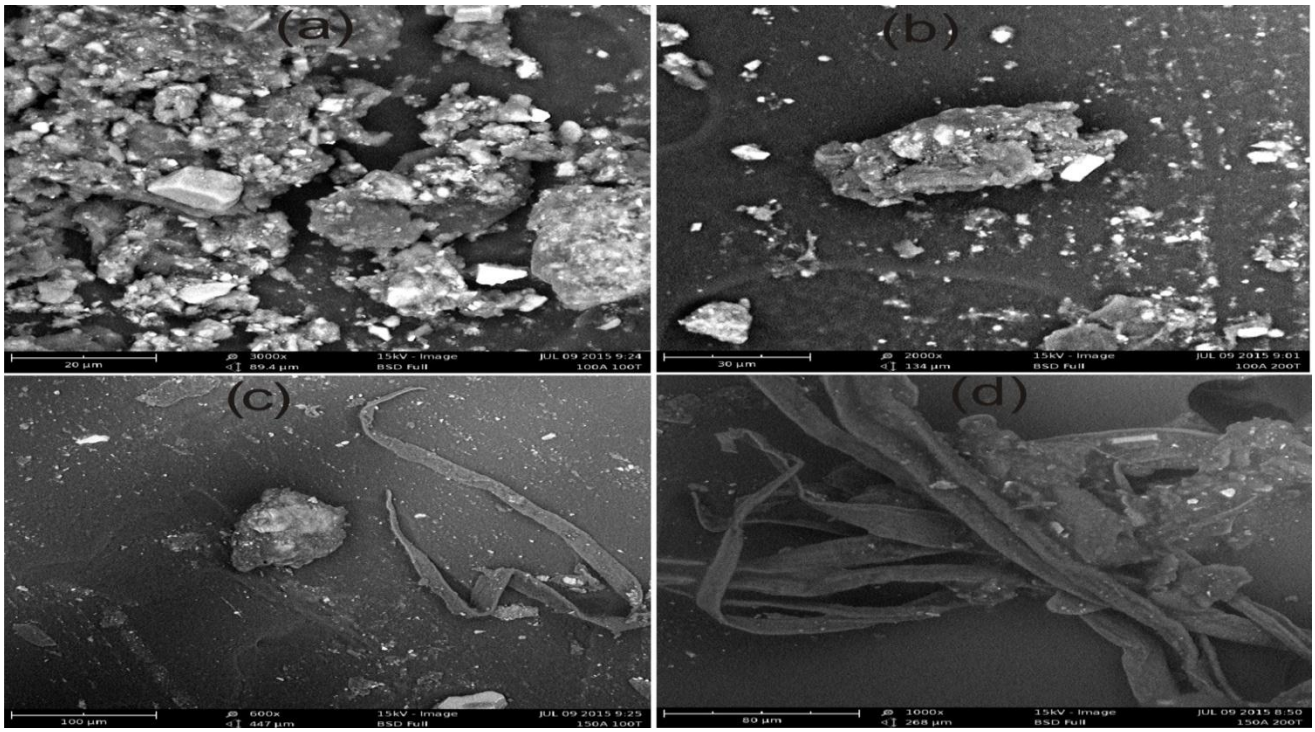

Figure 4: SEM Images of fullerenes solid extracted from carbon soot collected at different arc discharge operating conditions of discharge current and chamber pressure of **(a)** 100 A and 200 Torr **(b)**100 A and 200 Torr **(c)** 150 A and 100 Torr **(d)** 150 A and 200 Torr.

3.3.2 Ultraviolet visible (UV-Vis) spectroscopy of fullerenes

The UV-Vis Spectroscopy of fullerenes solid extracted from the carbon soot collected at different system condition of discharge current and chamber pressure of 100 A and 100 Torr, 100 A and 200 Torr, 150 A and 100 Torr and 150 A and 200 Torr are shown in Fig. 5a, 5b, 5c and 5d respectively. The UV-Vis Spectra of Fig. 5a recorded absorbance in Abs (Absorption units) with absorption peaks at 433.20, 479.00, 509.02, 516.80, 540.80, 653.20 and 673.80 nm. The UV-Vis Spectra Fig. 5b below recorded absorption peaks at 473.60, 526.80, 587.80, 629.20 and 663.40 nm. Also, the UV-Vis Spectra shown on Fig. 5c recorded absorption peak at 474.80 while the one shown on Fig. 5d recorded peaks at 479.80 and 574.00 nm.

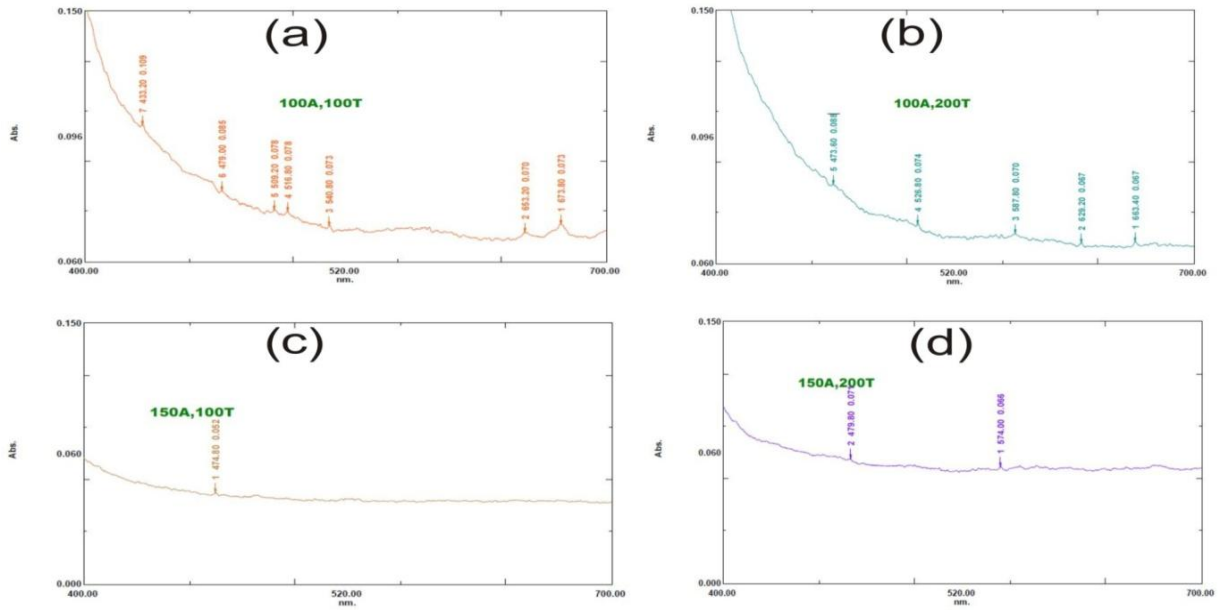

Figure 5: UV-Vis Spectra of fullerenes solid extracted from carbon soot collected at different arc discharge operating conditions of discharge current and chamber pressure of **(a)** 100 A and 200 Torr **(b)**100 A and 200 Torr **(c)** 150 A and 100 Torr **(d)** 150 A and 200 Torr.

IV. Discussion

4.1 Carbon Soot Yield

From TABLE 1 above, the carbon soot collected for two minutes of arc discharge run at discharge current of 100 A gave a better yield at a lower pressure of 100 Torr than 200 Torr (about 1.42 times better). At discharge current of 150 A, the carbon soot collected for two minutes of arc discharge run gave a better yield at a higher pressure of 200 Torr than at 100 Torr (about 1.47 times higher). Over all, the highest carbon soot yield of 43.67 mg was collected for two minutes of arc discharge run at discharge current of 150 A and chamber pressure of 200 A which was about 1.64 times higher than the lowest carbon soot collected at 100 A of discharge current and chamber pressure of 100 Torr.

4.2 Fullerenes Yield

From TABLE 1 above, fullerenes solid extracted from carbon soot collected at all arc discharge operating conditions using the direct heating extraction technique were between 6 % to 8 % yield. [4] This yield is better than 3-4 % fullerenes yield obtained after using soxhlet extraction technique to extract fullerenes from carbon soot collected from an arc discharge system of 100 mm diameter and 200 mm length of chamber size and at 100 A and 100 Torr of arc discharge current and chamber pressure respectively. If soxhlet extraction technique is use for fullerenes extraction, the carbon soot obtained from the arc discharge system fabricated in this work could give fullerenes yield above 8 %.

Also, from the extraction process carried out, the highest fullerenes solid was extracted from carbon soot collected at discharge current of 150 A and 100 Torr of chamber pressure with 8 % of fullerenes yield followed 7.60 % fullerenes yield extracted from carbon soot collected at discharge current of 100 A and 100 Torr. In both cases, the chamber pressures was at 100 Torr and were the only cases that lower chamber pressure was used. At both 100 and 150 A of arc discharge current, the arc discharge system fabricated supported the formation of fullerenes at lower chamber pressure of 100 Torr better than higher pressure of 200 Torr.

4.3 Scanning Electron Microscopy (SEM) Analysis of Fullerenes Extracts

The SEM micrograph of fullerenes solid obtained from carbon soot collected at arc discharge system operating condition of 100 A of discharge current and 100 Torr of chamber pressure shown on Fig. 4a, revealed a morphological and polycrystalline pattern of C60 thin film [5]. The SEM micrograph of fullerenes solid obtained from carbon soot collected at 100 A of discharge current and 200 Torr of chamber pressure shown in Fig. 4b above did not reveal an identified pattern. Although, the average dimension of the materials is some few tens of micrometer which is within the range of those of fullerenes whiskers [6]. The SEM micrograph of fullerenes solid obtained from carbon soot collected at 150 A of discharge current and at 100 Torr and 200 Torr of chamber pressure shown in Fig. 4c and 4d respectively all revealed some tubule-like whiskers. The length of the whiskers falls within 40 μm – 15 mm corresponding to those of fullerenes whiskers [7].

4.4 Ultraviolet Visible (UV-Vis) Spectroscopy of Fullerenes Extracts

The UV-Vis Spectra of fullerenes solid extracted from carbon soot collected at arc discharge system operating condition of 100 A of discharge current and 100 Torr of chamber pressure shown on figure 5a above, the absorption peak recorded at 540.80 correspond to the absorption band for C_{60} [8], while the peak recorded at 673.80 correspond to the absorption peak for C_{84} [9]. Also, the UV-Vis Spectra of fullerenes extract of carbon soot collected at 100 A of discharge current and 200 Torr of chamber pressure shown in Fig. 5b recorded absorption peak at 663.40 nm which correspond to the weak absorption peak for C_{70} of ~665 nm [8]. UV-Vis Spectra of fullerenes solid extracted from carbon soot obtained at 150 A of discharge current and 100 Torr of chamber pressure shown in Fig. 5c recorded absorption peak at 474.80 corresponding to the absorption peak of C_{78} of ~474.40 nm [10]. Lastly, fullerenes solid extracted from carbon soot collected at 150 A of discharge current and 200 Torr of chamber pressure shown in Fig. 5d recorded absorption peak at 574 nm which correspond to the weak absorption band for C84 of ~574 nm [9].

V. Conclusion

The arc discharge system fabricated in this work with chamber size of 140 mm diameter and 500 mm length gave better fullerenes yield of 6 – 8 % when compared to the 3 – 4 % yield obtained from an arc discharge system of 100 mm diameter and 200 mm length. Also, if are exploited, the arc discharge system fabricated in this work could produce higher fullerenes yield. Hence, the positive impact of large arc discharge system chambers in increase fullerenes yield is obvious and worthy of further research. More so, other extraction techniques other than the direct heating technique used in this work could give higher fullerenes than the 6 – 8 % obtained in this work. Also, At both 100 and 150 A of arc discharge current, the arc discharge system fabricated supported the formation of fullerenes at lower chamber pressure of 100 Torr better than higher pressure of 200 Torr.

All the analysis carried out on fullerenes solid extracted from carbon soot collected in this work indicates the presence of fullerenes. The SEM analysis carried out on fullerenes solid extracts indicates polycrystalline pattern of C60 thin film or the presences of fullerenes whiskers. Also, the absorption peaks for C_{60}, C_{70}, C_{78} and C_{84} were observed from the UV-Vis Spectroscopy carried out for fullerenes solid extracts.

Reference

[1]. M. L. Charlse and C. Chia-Chun (1994). Preparation of fullerenes and fullerene-based materials, in Solid State Physics. Ehrenreich, H and Spaepen, F. (eds). USA, Academic Press Incorporation. 148pp.

[2]. G. N. Churilov. Synthesis of Fullerenes and Other Nanomaterials in Arc Discharge. Journal of Fullerenes, Nanotubes and Carbon Nanostructures. 16(5), 2008, 395-403.

[3]. Z. Yuming and C. Guang. C_{60} Fullerene Amphiphiles as Supramolecular Building Blocks for Organized and Well-Defined Nanoscale Objects. Structure and Bonding. 159, 2014, 23–54.

[4]. J. Hare (2008). The Design of a Fullerene Generator. [online] Available from: http://www.creative-science.org.uk/design.html [Accessed: 5 March 2015].

[5]. D.-W. Yan, W. Liu, H.-Z. Wang and C.-H. Wang. Preparation of Fullerene Polycrystalline Films on Different Substrates by Physical Vapor Deposition. Materials Transactions. 48(4), 2007, 700-703.

[6]. S. Marappan and M. Kun'ichi. Synthesis and Characterization of Fullerene Nanowhiskers by Liquid-liquid Interfacial Precipitation: Influence of C60 Solubility. Molecules. 17(4), 2012, 3858-3865.

[7]. M. Kingsuk, M. K. Kalaga and Maheshwar (1994). Fullerenes: C60 from Camphor – A Novel Approach. Current Science Journal. 67(8), 1994, 602-604.

[8]. J. P. Hare, H. W. Kroto and R. Taylor. Preparation and 'UV/visible spectra of fullerenes $C_{60}$and C_{70}. Chemical Physics Letters. **177**(4**), 1991, 394-398.**

[9]. K. Kikuchi, N. Nakahara, M. Honda, S. Susuki, K. Saito, H. Shiromaru, K. Yamauchi, I. Ikemoto, T. Kuramochi, S. Hino and Y. Achiba. Seperation, Detection, and UV/ Visible Absorption Spectra of Fullerenes; C_{76}, C_{78} and C_{84}. Chemistry Letters. 20(9), 1991, 1607-1610.

[10]. K. Simeonov. Higher fullerenes – Isolation, halogenation and structural studies. Ph.D. thesis. Department of Chemistry. Germany, University of Stuttgart. 2009.