

Discharge Characteristics on the Synthesis of Carbon Nanostructures through Arc-Plasma in Water

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ABSTRACT

Simple arc discharge technique was used for formation of carbon nonmaterials without using any type of gases. The used arc discharge technique between the pure graphite electrodes with different diameters for anode submerged in water at room temperature. Arc plasma is produced using D.C power supply with current (30-90amp), and voltage (5-25volt). The discharge characteristics of the arc plasma generation in water and, with Ni catalyst and in NaCl solution were examined with the change in working parameters of electrode diameters and related arc current. The nanostructures obtained of these experiments were examined by scanning electron microscope (SEM).

Keywords: Electrical discharge, Arc plasma, Plasma in water, Carb nanopartical.

خصائص التفريغ عند تكوين التراكيب النانوية بتقنية قوس البلازما في الماء

الخلاصة:

استخدام تقنية بسيطة لتكوين مواد كاربونية نانوية باستخدام التفريغ بالقوس. تقنية التفريغ القوس تمت باستخدام أقطاب كاربونية (قطب الانود) ذات أقطار مختلفة مغمورة بالماء وبدرجة حرارة الغرفة.

تم توليد البلازما باستخدام مجهز قدره بتيار (30-100) امبير و فولتيه (5-25) فولت. خصائص التفريغ درست في الماء وبوجود بلورة النيكل وكذلك في محلول كلوريد الصوديوم مع تغيير قطر الانود المرتبط بتغير تيار القوس. ان المواد النانوية المنتجة بهذه التجارب تم فحصها باستخدام مجهر الماسح الالكتروني .

INTRODUCTION

Electric arc plasma was the first available method to fabricate carbon nanotubes. An electric arc discharge is an electrical breakdown of a gas producing a plasma discharge, very similar to a spark, which is the flow of current through a nonconductive medium such as the air and gases or distilled water. Usually this method creates nanotubes through arc-vaporization of two carbon rods placed end to

end separated by approximately 1mm, in an enclosure that is usually filled with distilled water. In this method carbon nanotubes are produced at the core in the cathode deposit[1]. A direct current of (50 to 100 A) driven by approximately 25 V creates a high temperature discharge between the two electrodes. The discharge vaporizes one of the carbon rods and forms a small rod shaped deposit on the other rod. Producing nanotubes in high yield depends on the uniformity of the plasma arc and the temperature of the deposit form on the carbon electrode[2]. Electric arc discharge in liquid environment was first used by Hsin et al [3] in water 1991 in order to synthesize CNTs. This simplified method does not require expensive noble gases, high temperature furnace and vacuum equipments [4]. Since then, benzene, toluene [5] and the liquid nitrogen ([6]-[8]) were also used as liquid environment. Sano et al[6] produced a fine quantity of carbon nano onions and CNTs from arc discharge in deionised water ([9]-[11]). Arc discharge was not stable in deionised water due to its electrical insulating characteristic. Therefore, salt solution was used as the liquid environment in order to improve electrical conductivity of solution [12].

In this paper, synthesis, and structural characterization of carbon nanoparticles based on arc discharge in liquid media are discussed. In addition, several parameters such as: voltage difference between electrodes, current, catalysts, electrical conductivity, and temperature of plasma solution are investigated.

Experimental procedure

The arc plasma was generated between two pure graphite electrodes in water. The experimental setup of water plasma generation system is shown in Fig. 1. These electrodes emerged in the container and they were horizontally aligned on the same axis with about 1mm gap. One of the brass electrode holders was free to move(anode) forward and backward using a micrometer, which enables proper electrodes gap adjusted during arc discharge. The power supply is turned on the anode electrode is then moved gradually towards the cathode electrode using a manual micrometer. Once the electrodes are in touch to generate the discharge the anode turned red hot subsequently, the anode electrode is moved backwards to maintain the gap about 1mm. simultaneously the plasma in spherical shape is formed. When the arc discharge stabilized the rod are kept at about 1mm a part while the carbon deposited on the cathode. The power supply is turned off after (5-10min) and left for a while for cooling.

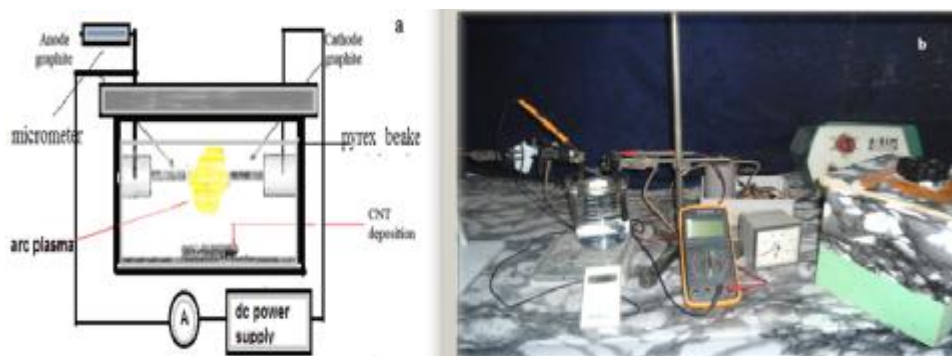


Figure1: (a): The schematic feature and (b): set up of arc discharge system

Results and Discussion

The electrical characteristics include of the study of the I-V in different diameters of electrodes at different arc time. The current and voltage effects on the production of nanostructures by a variety of voltage values in different experiments are investigated. The discharge current and voltage for all experiments sustained at 5-25 Volt and 50-100 Amp. Respectively during which the arc shown to be limited stable according to the inter electrode spacing. Fig.2 and table(1) shown the experimental I-V characteristics and data of non catalytic and catalytic process when arc plasma are generated. We noticed that I-V data there is stable for arc plasma in different arc time and there is new behavior depending on the electrodes diameter, electrical power and on the solution of arc container. The arc current appears to be stable and having small fluctuations around its mean value, however, the potential drop across the discharge and the light intensity vary considerably, revealing the occurrence of dynamical effects in the plasma. The electrode anode diameter related to the cathode electrode effects on the arc discharge current and time as shown in the table(1). The table (1) of arc discharge experiments shows the formation of maximum arc current (90-96Amp) at electrodes diameters of (Ad) (3,6mm) and(Cd) (6,6mm). When the arc discharge using the Ni catalyst in the diameter and Ad=(6,Cd=6mm) the arc current intentionally reduced to approximately 80A and, simultaneously, the current rises to the fixed value of 80A and the arc became unstable at the arc time 6min. Our results are good agreement with the previous study using catalyst percentage of 1mol% Fe[12].

Table(1): Experimental arc discharge data with and without catalyst in different diameters of electrodes where cd=cathode diameter, Ad=anode diameter.

Electrodes Diameter (mm)	V Volt	I Amp	V volt	I Amp	V volt	I Amp	V volt	I Amp	V volt	I Amp	V volt	I Amp	V volt	I Amp	V volt	I Amp	V volt	I Amp
Cd=6, Ad=1 Arc time 4min	22	38	8.5	76	8	78	10	76	14	64	17	60	18	58				
Cd=6, Ad=1.5 Arc time 5min	17	44	9	80	11	74	10	76	13	62	17	50	15	55				
Cd=6, Cd=2.5 Arc time 5min	15	45	8.5	85	10	80	12	75	11	74	13	68	15	63	17	58		
Cd=6, Ad=3 Arc time 6min	14.5	47	5.2	96	6	92	7.5	90	9	86	9.5	82	9.7	80	10	78	12	72
Cd=6 Ad=6 Arc time 8min	11	60	6	90	7	84	7.3	80	10	78	12	68	15	55				
Cd=6 Ad=3 Arc time 6 min, Ni catalyst	20	46	7.4	86	8.2	84	9.5	80	10	76	11	72	12	66	12.7			
Cd=6 Ad=3 Arc time 6min, Ni catalyst in NaCl Solu.	13	40	8	84	8.6	80	9.6	76	11	72	11.8	68	12.5	62	14	54		

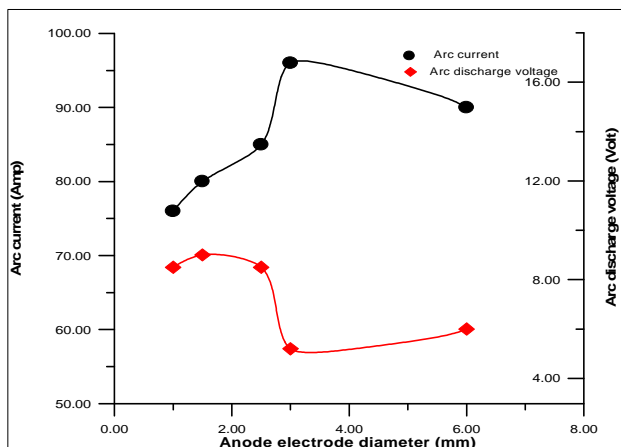


Figure (2): the I-V characteristics in different diameters of electrodes of arc discharge in water.

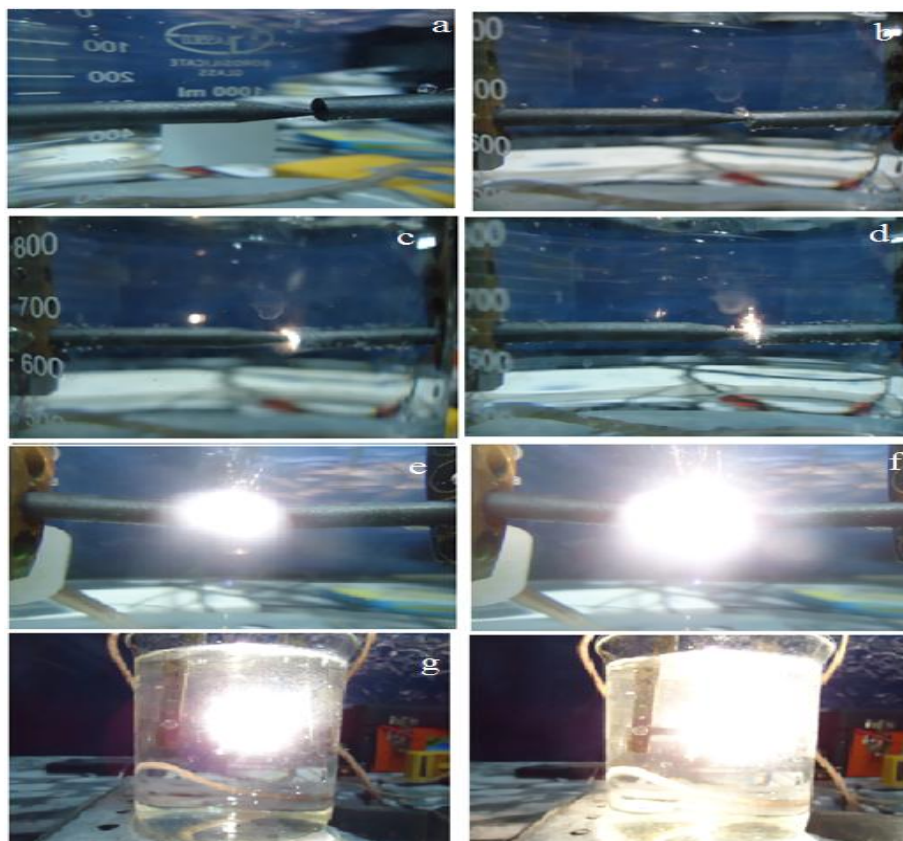
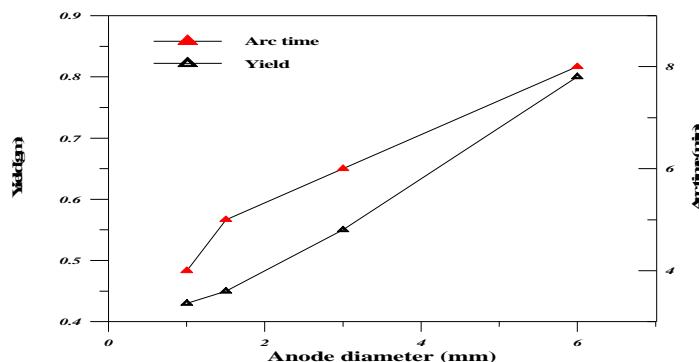


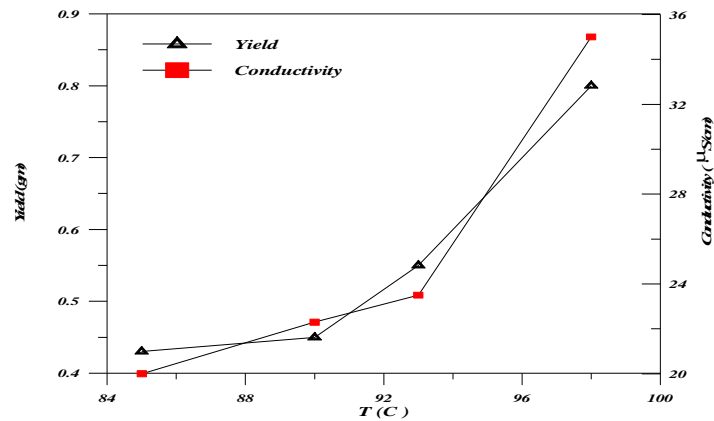
Figure (3): A plasma during the arc discharge corresponding current and emitted Light intensity (a-38A, b-46A, c-70A, d-72A, e-86A, f-96A, g-96A, h-96A).

The area between the electrodes is called the plasma region. The plasma can also be seen to surround the anode, indicating the direction of plasma expansion. The light emitted by the arc was recognized by digital Sony camera, through a glass window on the side of the beaker. The side views of the plasma arc for (Ad, Cd, 6, 3mm) are shown in Fig(3), that the arc current and the light intensity rise in synchronization, showing that change in the ionization and excitation reaction arc intimately related. The plasma frame becomes larger at higher current of the arc. The frame of the arc plasma in water can be divided into three thermal regions on the base of the temperature distribution along the plasma arc, such as arc region, plasma region and downstream region. The arc region was defined as a region between the electrodes, the plasma region was the higher temperature region about (6000k) and the downstream region was the lower temperature region about (1000k) on the top of the plasma body [13,14].

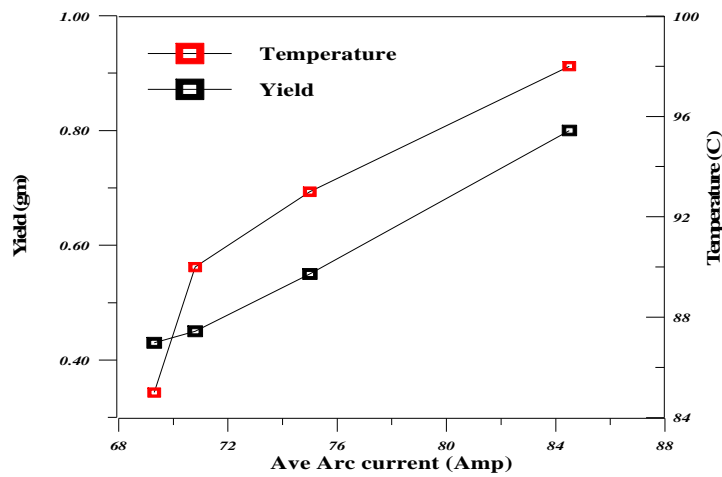
In the parametric study, the weight change of the electrodes with time which related to the amounts or yields of collected nanoparticles (production rate) for each run are monitored to optimize the data related to carbon nanoparticles production. Since the major in the parametric study is associated with the carbon nanoparticles synthesis optimization, the yield of the collected nanoparticle is discussed in detail. The effect of anode diameter of graphite and the arc time on the nanostructures yield generated by arc discharge are shown in Fig(4). We noticed that the increase in anode diameter led to increase arc time, then increase in the plasma reaction result to increase the nanostructures yield. In this case as shown in Fig (5), the electrical conductivity of colloidal increase because of the reaction inside the container is increase, so that the solution temperature increases directly (Fig.6). It is considered that the solution temperature is relatively low compared with the plasma and electrode temperatures under arc discharge. The increasing of anode diameter related to the arc current, so the plasma have large numbers of charged particles led to increase the arc discharge solution temperature, consequently the nanostructures yield and conductivity are increased. In Fig(7) the yield of collected nanoparticle versus number of runs is plotted for the four different currents. It is found that at higher values of arc current, the yield increases with time during the arc-discharge. It is well known that the plasma temperature increases with an increase in the current. Therefore, for higher water temperature, the yield of nanoparticles increases with time.



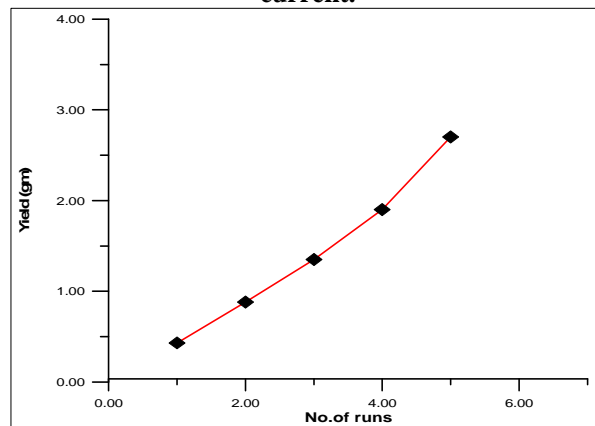
Figure(4): The arc time of discharges and nanostructures yield according to the anode diameter



Figure(5): The nanostructures yield and solution conductivity of arc discharge according to the water temperature.

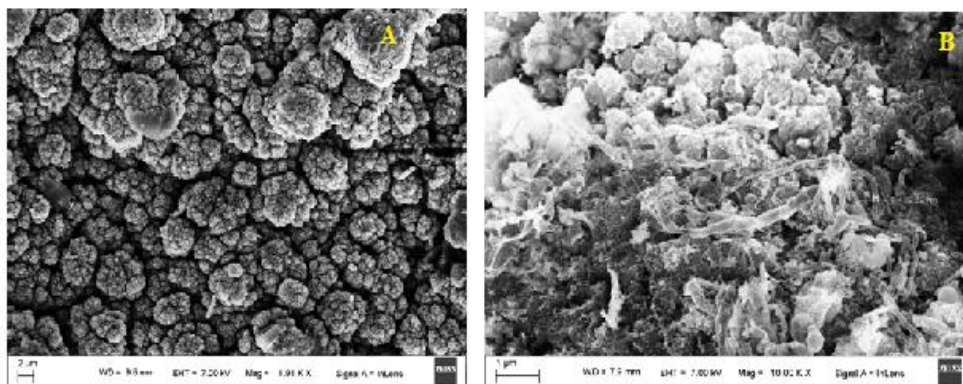


Figure(6): The yield of arc discharge and water temperature according to the arc current.



Figure(7): The yield of collected nanoparticles versus number of arc discharge runs.

Fundamental for morphology study of the samples is microscopy in particular electron microscopy. Using scanning electron microscopy (SEM), it is possible to observe the external structures of nanotubes or nanofiber and nanoparticles, but it is not possible to distinguish SWCNTs (single wall carbon nano tubes) from MWCNTs (multilayer wall carbon nano tubes). A study conducted by SEM is conducive to the characterize the texture as well as the structure (crystalline) of the deposited carbon. It is shown that the dc arc discharge plasma process in water is a versatile process for growing a wide range of carbon nanostructures. Fig (8b and 8a) shows the cathode rod after arc discharge process with and without catalyst and illustrate the typical micrograph of the deposited carbon on the electrode end which correspond to the arc discharge current of 90A. Stacked graphite layers which is formed due to the sudden quenching of plasma and high current densities used during arcing process. The SEM image of electrode rod with Ni catalyst Fig (8b) on the top surface indicated that the CNFs (carbon nano fibers) were formed between the nanocarbon layers and aggregates.



Figure(8,a,b): SEM image for cathode rod after arc discharge A: without catalyst and B: with Ni catalyst respectively

Conclusion

An electrical discharge characteristics on the arc-discharge in water was successfully carried out to correlate the parametric data from the synthesis method, so that nanostructures particles can be synthesized in a controllable manner. It was found that a steady rate of synthesis can be achieved at higher values of the applied current .

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