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Development of Indigenous Underwater Arc Discharge Configuration for Gram Scale Growth of CNT

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Abstract
An indigenous arc discharge apparatus is designed for gram-scale carbon nanotube production. A detailed description of the system's design & construction the form, composition, &purity of carbon nanotubes were assessed using an indigenous setup with deionized water. The materials were characterized by SEM, XRD, and Raman spectroscopy. The arc discharge equipment samples show agglomerated tubed fibre-like structures in SEM pictures. XRD verified the hexagonal crystallographic phase. These bands also confirm. The presence of carbon nanotubes in samples and sp^2 hybridization

Key words: CNT, Indigenous arc discharge setup, XRD, SEM, RAMAN

1. INTRODUCTION

The research of novel smart material technologies has captivated scientists for decades [1]. In designing & developing sensors, electronics, energy storage, &nano catalysts [2], carbon-based nanostructures of 1 to 100 nm are very useful. Researchers have focused on carbon-based nanostructures since the discovery of buckminsterfullerene, carbon nanotubes, carbon nano fibers, and graphene [3]. Despite limited manufacturing and high costs, CNTs have captivated researchers owing to their unique features [4]. Despite decades of study on CNTs, a cost-effective method for producing CNTs on a wide scale is still absent. Due to a lack of knowledge of the CNT development process [5], the key barrier is insufficient manufacturing & the high cost of CNTs compared to other materials. Despite enormous advances in CNT research, large-scale production of CNTs with specified qualities has yet to be produced [6]. The electric arc discharge is used for the mass manufacture of CNTs [7]. Producing CNTs with an arc discharge requires modifying the setup and reaction chamber environment medium. Adjusting parameters such

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as pressurize, electrode geometry, temp., precursor, & catalyst improvement of quality and quantity of CNTs. [8]. According to Farhat et al., adjusting the ratio of gases may regulate the size of SWNTs [9]. List al. and Sun et al. improved the catalyst configuration and reached gram-scale CNT yields [10,11]. The current between the two electrodes has also increased synthesized nanotubes' quality, yield, & size [12,13]. Few studies have shown that the environment (liquid or gas) influences the quality & production of CNT products in an arc release. Another study has demonstrated CNT mixture in a liquid environment, particularly deionized water [14,15]. Researchers have studied the effects of electricity (AC or DC, voltage & frequency), catalyst type and concentration, carbon precursor (type), gas pressure & synthesis environment on product yields during the last 3–4 years, with yields ranging from micrograms to grammas per hour [16]. Several other groups have reported achieving yields of 2g/h &6.5g/h by employing arc discharge synthesis. The external magnetic field has been studied as a critical parameter in synthesizing CNTs using electric arc plasmas (EAP). An external magnetic field enhances arc plasma properties such as plasma density, temperature, and confinement, responsible for enhanced CNT quality (length, diameter, diameter distribution, and vertical and horizontal alignment defect-free development) and greater yield (2–5 g/h). The arc discharge method is the most helpful for scientific applications because of the high process temperature, which results in highly graphitized tubes. The primary problem with large-scale CNT synthesis using arc discharge and CVD is that the material quality degrades as yield grows, increasing cost. This widespread problem stymies the large-scale production of high purity CNTs. CNT properties are readily maintained in small reactors, but when the same technology is employed in big reactors, the purity declines, raising the cost. As a result, a more straightforward, complex, dependable, & long-lasting technique for mass-producing CNTs is needed. A thorough investigation into optimizing CNT growth parameters, low-cost renewable precursor and combination, nano catalyst concentration, and external magnetic field in an arc discharge might solve this problem. Over the last two decades, scientists have been researching the properties, manufacturing procedures, and potential applications of carbon nanotubes. CNTs are known for their exceptional electrical, mechanical, thermal, chemical, and optical properties. To synthesize carbon nanotubes, we developed an indigenous underwater arc discharge device. Two electrodes generate an arc with a high current and low voltage in a water medium in the arc discharge method. SEM, XRD, & Raman spectroscopy characterize the produced CNTs.

2. EXPERIMENTAL SECTION

As previously stated, we devised an indigenous arc discharge system to synthesize CNTs. Two electrodes in an acrylic cylinder filled with deionized water from the arc discharge setup. Fig. 1 shows a schematic of a typical underwater arc discharge system with an experimental setup created in Solid Works 2017. In addition to the arc discharge system, there is a DC power supply, arcing reaction chamber, and micro spinning system. A micro spinning mechanism regulated the distance between the anode & cathode in the reaction chamber. The current study used three alternative approaches to the synthesis CNTs. The first method selects electrode materials such as iron, copper, & graphite. In a second way, the cathode size is kept constant while the anode size is varied. In the third approach, the function of the catalyst is investigated. A DC is sent between two

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submerged electrodes in each example, causing an arc discharge. The arc starts between the anode & the cathode. The discharge current was taken as 100 A at 15 volts. The experiment was done for many minutes to acquire enough carbon soot. The underwater arc discharge remained steady during the experiment, resulting in black soot sputtering in all directions. After the experiment, the deionized water was drained, & the carbon soot was collected, dried, and kept for examination [18,19].

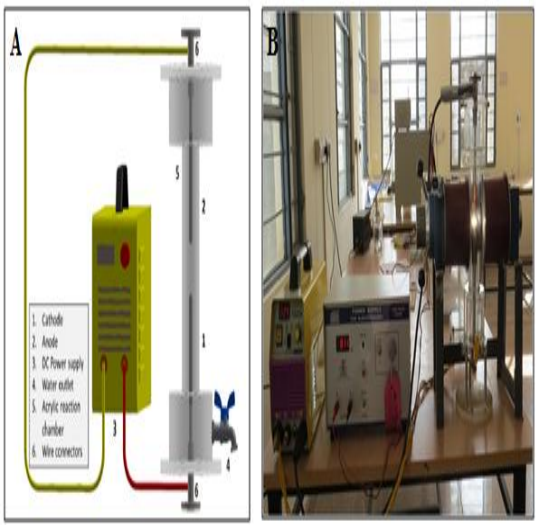

Fig.1:- Design & experimental setup of an indigenous arc discharge system.

2.1 Methodologies

The approach consisted of three steps: choosing the material, sizing the electrode, & studying the function of the catalyst.

A. Material Selected

The electrode material utilized in CNT synthesis is critical. The table below shows several electrode material combinations. Table 1 also displays the parameters used for each combination, such as electrode gap, DC, and DC voltage. The arc is formed in the arching chamber between two electrodes, initially graphite cathode and aluminum anode. Both electrodes are immersed vertically in deionized water with a 2mm micro spanning system. The cathode & the pure Al anode (6 mm tip diameter) form an electric arc (15 mm diameter). Anode & cathode rod lengths were 10 cm each. The discharge current was taken as 100 A at 15 volts. To obtain enough carbon soot, the experiment ran for ten minutes. The underwater arc discharge was steady during the experiment, & a sufficient quantity of black soot spewed in all directions in the reaction container. The deionized water was drained, & the bottom carbon soot was collected, dried, and kept for examination. The experiment is repeated in step two, but the anode is replaced with copper, & all other parameters stay the same (size, arcing current, gap, experiment period). We replaced the anode's substance with ferrous metal in step three while keeping all other settings and parameters the same. In step four, we use another anode material combination i.e. Graphite now. In this case, both electrodes are made of graphite. Each step of the experiment is repeated.

Table:1 Material for Electrode Selection Criteria

S. No	Material of Cathode	Material of Anode	Applied DC Current(A)	Applied DC Voltage(Volt)	Gap Between Electrodes(mm)	Time (min)
1	Graphite	Aluminium(Al)	100 A	15volt	2mm	10 min
2	Graphite	Copper(CU)	100 A	15volt	2mm	10 min
3	Graphite	Ferrous (Fe)	100 A	15volt	2mm	10 min
4	Gra	Graphi	100	15volt	2mm	10

	phite	te	A			min

B. Electrode Size Selection

The size of electrodes is critical in the synthesis of CNT. Finally, we chose the optimum material for the arching chamber electrodes. This stage of the experiment will determine electrode size. The cathode will be fixed, & the anode will be gradually increased. In this stage, the cathode & anode sides are 10cm, 8cm, and 5cm, respectively. Everything else is as in the material selection method. Here changes the distance between two electrodes. Collect the yield from the cathode & other components of the arching chamber for characterization. Some factors, such as material current, voltage, and cathode size, are fixed in Table 2. The anode size is the only variable. After analysis, we found that a 5 cm anode provided the greatest results. XRD and Raman spectroscopy studies demonstrate that the reaction time stays constant at 10 minutes. It's not only the arching chamber that changes.

Table2 Size of Electrode Selection Parameter

S. No	Material of Cathode	Material of Anode	Applied DC Current (A)	Applied DC Voltage(Volt)	Size of Cathode	Size of Anode	Time (min)
1	Graphite	Graphite	100 A	15volt	10cm	10 cm	10 min
2	Graphite	Graphite	100 A	15volt	10cm	8cm	10 min
3	Graphite	Graphite	100 A	15volt	10cm	5cm	10 min

C. Catalyst role in Arching Chamber

The catalyst in arc discharge CNT synthesis is generally a powdered metal with a carbon precursor on either side of the electrode. The metal should have a low boiling temperature & a rapid evaporation rate [10]. When an arc current is applied, the metal atoms and carbon precursor vaporise.

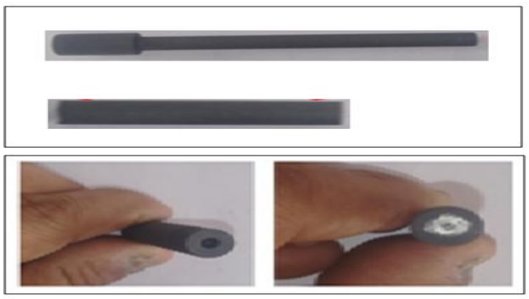

Fig:2 The iron-Cobalt composite catalytic nano powder anode rod

Carbon & metal particles from nuclei on the arc reactor wall. Figure 2 shows a pure graphite anode rod loaded with iron-cobalt composite catalytic nano powder. Together with the liquid metal, the carbon vapours may move towards the cathode in the plasma. Metal particles don't stick to the cathode's surface, so they fly away and create CNTs. Catalysts include Fe and Co, with Y, S, and Cr promoters. In this step, we employed a 1:1, 1:2, and 1:3 mixer of Fe and Co to boost the purity and yield.

2.2 Classification methods

A. Structural

It was used to investigate the crystallographic order, impurity content & flaws in synthetic materials. In this case, the XRD pattern is captured over 2 ranges from 0 to 80° at 0.5°/min. The sharp peak at 2 = 26° corresponds to the (002) hexagonal phase of a highly crystalline carbon crystal system. The two wide peaks at 2 = 43° and 54° relate to various carbon structures. [11].

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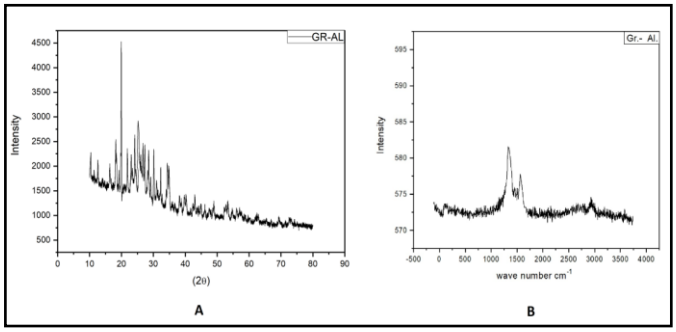
B. Spectroscopy

For researching the chemical & electrical properties of carbon nanostructured materials, Raman spectroscopy is used. One of the essential characterisation techniques for carbon nanotubes, single-walled and multi-walled, and graphene and its composites. Ideal CNTs feature three peaks at 1350, 1591, and 2657 cm-1. The D band is produced by flaw and disorder components in CNTs breathing style of sp2-hybridization. The D band's strength is related to the CNT's defect count. The G band's second peak is created by the in-plane vibration of sp2 carbon atoms. The 2D band (formerly known as the G band) is an overture of the D band created by two phonons with opposite momentum interacting. The 2D band's location and form are sensitive to the thickness and number of layers of graphitic material, and in this situation, it shows tubular layered synthesis [20]. This section compares each combination's Raman spectroscopy to the ideal CNT Raman analysis.

C. Morphology

A scanning electron microscope (SEM) examines the surface of a sample with a high-energy electron beam. Zeiss (20 kV) was used to characterise the morphology of the graphene-based nanostructured material in this study. Electrons interact with atoms in a sample to produce signals that reveal surface topography, composition, and other properties like electrical conductivity. Using an SEM instead of a light detector has various advantages. According to the SEM picture of CNTs synthesised by the underwater arc discharge setup, the morphology of synthesis materials demonstrates that the as-produced materials are CNTs.

Fig3 Graphite-Aluminum XRD and Raman Spectroscopy



3. RESULT AND DISCUSSION

3.1 Material selection

In this section of the experiment, we will discuss the characteristics analysis of yields found in the synthesis process. The analysis process includes structural analysis and spectroscopic analysis. The purpose of these two analyses is that we have taken four combinations of materials for selecting electrodes in the arcing chamber, and now we will identify which combination gives the best result by using these two analyses.

Fig.3 shows the Graphite-Aluminum Combination XRD and Raman Spectroscopic Analysis the XRD analysis reveals the first peek at an angle of 19o. So, because this material combination doesn't work, we must go on to the next material combination. The existence of the D band, G band, and 2D band in the graph suggest that this combination is not up to the standard. Thus, we must go on to other combinations. The first peak indicates the D band & the number of faults in CNT. However, for this combination, the optimal band placement is 1350 cm-1, showing that CNT has several faults that make it inappropriate for us. The G band exhibits sp2 carbon atoms in-plane vibration. It should be at 1591 cm-1, yet close to 1470 cm-1, indicating that this combination is not optimal.

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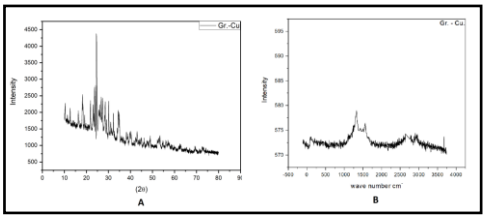

Fig4 Graphite-Copper Combination XRD and Raman Spectroscopy

Fig 4 shows the Graphite-Copper Combination XRD & Raman Spectroscopic Analysis: This combination's XRD examination shows the first peek at an angle of 22^{o}. Thus, we must go on to the next material combination. The Raman analysis lacks D, G, and 2D bands. The first peak indicates the D band & the number of faults in CNT. However, for this combination, the optimal band placement is 1350 cm-^{1}, showing that CNT has several faults that make it inappropriate for us. The G band exhibits sp^{2} carbon atoms in-plane vibration. This combination of electrodes likewise does not meet the criterion for CNTs at 1591 cm-^{1}.

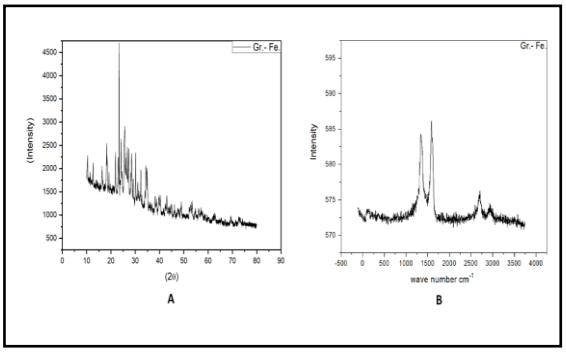

Fig5 Graphite – Ferrous Combination XRD and Raman Spectroscopy

Fig. 5 shows the Graphite – Ferrous Combination XRD and Raman Spectroscopy With this combination of materials, the XRD analysis indicates the first peak on the angle 21^{o}; hence this material combination is not ideal. We must go on to the next material combination. D, G, and 2D bands in Raman the first peak of the graph represents the D band & the number of defects in CNT. The D band should be around 1350 cm-1; however, with this combo, it is at 1210 cm-1. The existence of the G Band demonstrates the plane vibration of sp2 carbon atoms, which is not acceptable for humans. For CNTs, it should be on 1591 cm-1, yet it is close to 1670 cm-1.fig.6 shows the Graphite-Graphite Combination XRD & Raman Spectroscopic Analysis, this combination's XRD examination display the first peek at 26o. Thus, we chose this combination for further investigation. The first peak of the graph represents the position of the D band & reveals the existence of faults in CNT. The existence of the G band reveals the plane vibration of sp2 carbon atoms; it should be at 1591 cm-1, but it comes close to 1590 cm-1. The third peak displays the 2D band, which shows the layer in CNT.

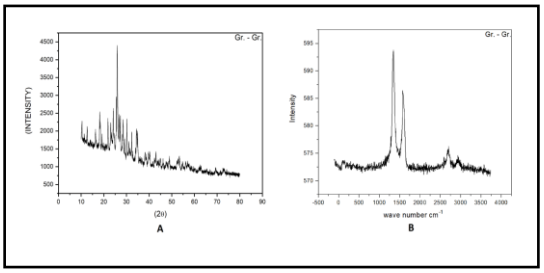

Fig6 Graphite - Graphite Combination XRD and Raman Spectroscopy

3.2 Size Choice

After deciding on the electrode material, we must now concentrate on the electrode form and size. The cathode size remains constant while the anode size changes from 10cm to 8cm to 5cm.

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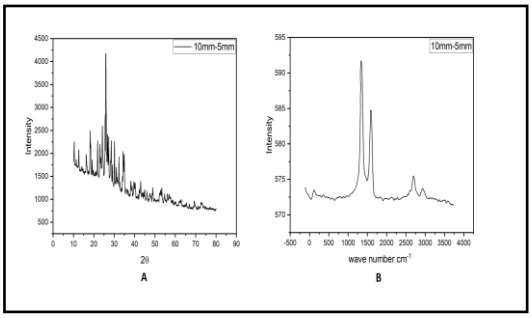

Fig7 5cm Anode XRD and Raman Spectroscopy

After collecting all the yields, we discovered that a 5cm anode boosts yields but also impurities. As shown in Fig. 7, we used a 5cm anode with pure graphite as anode & arced continuously for 10 minutes, resulting in a few grammas of shot (excluding large particles). Purification decreased fine carbon shot to 390-670mg.

3.3 Role of Catalyst

This section of the experiment uses pure graphite as anode and mixes iron-cobalt composite catalytic nano powder in 1:1, 1:2, and 1:3 ratios. The optimum result is achieved by combining Fe+Co at a ratio of 1:3. Raman spectroscopy is an essential tool for characterizing carbon materials possessing sp2 and sp3 hybridization of carbon atoms.fig. 8 shows Iron-Cobalt Composite Catalytic Nano Powder-Filled Pure Graphite Anode Rod XRD & Raman Spectroscopy

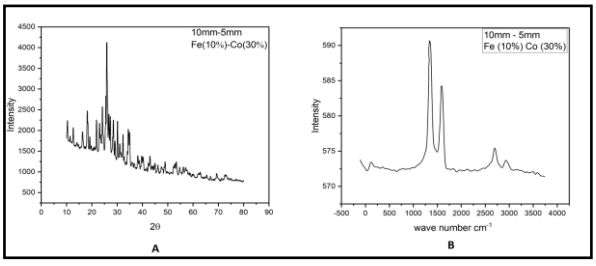

Fig8 Iron-Cobalt Composite Catalytic Nano Powder-Filled Pure Graphite Anode Rod XRD & Raman Spectroscopy

It also offers information on the number and orientation of layers, quality, kind of edge, doping, disorder, & presence of functional groups in carbon nanostructures. The Raman spectra show two peaks at 1362 cm1 and 1584 cm1, corresponding to the D and G bands. The G band is present in all sp2 carbon systems owing to the in-plane vibration of sp2 carbon atoms. The D band is caused by disordered sp2-hybridization, defect, and impurity structure in graphite sheets. The strength of the D band relates to the sample's defect count. The peak at 2740 cm1 corresponds to the 2D band, created by a two-phonon double resonance Raman process. Figure 9 shows SEM analysis for pure graphite anode rod loaded with iron-cobalt composite catalytic nano powder, which validates the tubular morphology of the materials.

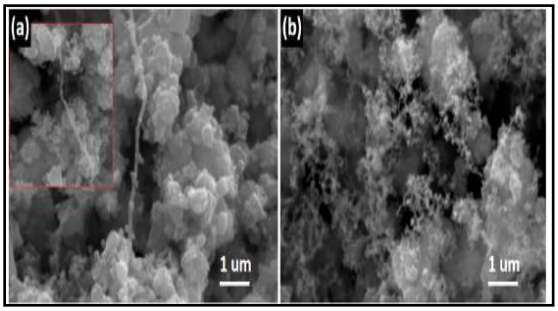

Fig9 SEM Inclusion of Iron-Cobalt Composite Catalytic Nano Powder in Graphite Anode Rod

4. CONCLUSION

Indigenous systems for the bulk production of CNTs have been designed, developed, and assembled at the Renewal Energy Laboratory, Vivekananda Global University, Jaipur.The underwater arc discharge system featured an acrylic reaction chamber and a quartz tube with high current and low voltage. Role of Catalyst, material, size, described during CNT synthesis. The authenticity of the produced CNTs was

verified using powder XRD, Raman spectroscopy, and SEM. Graphitized CNTs are visible in the Raman spectra of newly produced CNTs. SEM verifies the tubular shape of the materials produced here. Moreover, the produced CNTs do not need additional purification, allowing their usage in research or industrial applications. Indigenous Discharge mechanism for gramme scale Growing CNTs is simple and saves energy.

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