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A NOVEL GROWTH OF HIGH GRADE SWCNTS FROM GR (0.25 MM) GRAPHITE RODS FOR DNA TRANSPORT IN GENETIC TRANSFORMATION – A NEW FEASIBILITY STUDY

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ABSTRACT

A novel synthesis of high grade Single Walled Carbon Nano Tubes (SWCNTs) by the modified AC method – VSA methodology (with KRS or NTFDS theory) from natural organics. i.e, GR (0.25 mm) graphite rods was adopted in this present work for possible application towards DNA transport in genetic transformation. Parameters, as inferences from characterizations were calculated and graphically emphasized. Correlation studies between these characterization inferences (such as grain size, purity) and above optimization parameters were carried out with a high light on yield of high grade SWCNTs. Beyond all of these, we have carried out a novel feasibility study at first time, which comprises the possible usage of precursor graphite rods (under Specialized materials category) as organic carbon sources for high grade SWCNTs growth with high yield via a low cost technique and methodology as value in commercial efforts.

Keywords: *Modified AC method, VSA methodology, NTFDS theory, Natural organics, GR (0.25 mm) graphite rods, XRD, EDAX, SEM, HRTEM, SWCNTs, DNA transport in genetic transformation system.*

1. INTRODUCTION

The present work showed that SWCNTs obtained by open air annealing and cooling of GR (0.25 mm) graphite rods (under Specialized materials category). The present work made a new break through via plays as an alternative method to conventional, traditional methods such as arc discharge, laser ablation, metal catalysts, pyrolysis and varieties of CVD methods. It also removes the usage of synthetic chemicals. The modified AC method [Zhenhui Kang et al, 5] – VSA methodology (with KRS or NTFDS theory) was adopted in this present work for the fabrication of CNTs from natural organics i.e., GR (0.25 mm) graphite rods. The entire steps such as Precursor materials selection with cleaning, Annealing, Sudden cooling, Interaction between red hot natural organic carbon resource materials and Dipping solutions (DS) [including Nano Thermo Fluid Dynamics (NTFDS) and Nano-drilling process] involved in this process were explained. CNT acts as a present day king of nanomaterials in nano technology due to their peculiar physical and chemical properties. Similarly CNTs have numerous applications from day to day events to advanced one [1-5].

2. EXPERIMENTAL DETAILS

The precursor/carbon source materials i.e., GR (0.25 mm) graphite rods were collected from nature, cleaned in water and dried in open air. These materials were used without further purification. Then the materials were open air annealed (upto red hot) in a muffle furnace at various temperatures viz., 600^0C and 800^0C for various time of heating viz., 1- 5 minutes. After that they were immediately dipped into various types of solutions viz., Sodium hydroxide (NaOH), Hydrogen peroxide (H_2O_2), Nitric Acid (HNO_3), Sulphuric Acid (H_2SO_4), Hydrochloric Acid (Hcl), Mineral Water (MW), Salt Water (SW), Double Distilled Water (D2W), Ice Water (Ice W), Hot Water (HW), Pure Water (PW), Ice water mixed double distilled water (IceW+D2W) and Hot water mixed double distilled water (HW+D2W) solution with various solution temperature ranging from 0^0C to 100^0C, various time of dipping viz., 30 seconds, 45 seconds 60 seconds, 75 seconds, 90 seconds and 120 seconds. The final samples were dried in open air at room temperature for 5 hours and then packed for characterization with mentioning synthesis conditions.

The above process was optimized with 11 Physical parameters viz., 1. Nature (pH) of the dipping solutions (acidic, basic and neutral), 2. Volume of Dipping Solution ranges from 0.5 ml, 1.0 ml, 1.5 ml, 2.0 ml and 2.5 ml, 3. Various types of materials: fibrous: specialized: GR (0.25 mm) graphite rods, 4. Various Parts of the materials: leaves, stems, 5. Various Dipping solutions: Sodium hydroxide (NaOH), Hydrogen peroxide (H_2O_2), Nitric Acid (HNO_3), Sulphuric Acid (H_2SO_4), Hydrochloric Acid (Hcl), Mineral Water (MW), Salt Water (SW), Double Distilled Water (D2W), Ice Water (Ice W), Hot Water (HW), Pure Water (PW), Ice water mixed double distilled water (IceW+D2W) and Hot water mixed double distilled water (HW+D2W), 6. Various Dipping Timings : 30 seconds, 45 seconds, 60 seconds, 75 seconds, 90 seconds and 120 seconds, 7. Various Number of Dippings: 1, 8. Various Annealing Temperatures: 600^0C and 800^0C, 9. Various Time of Annealing : 1 minute to 5 minutes, 10. Various Dipping Solution temperatures: 0^0C to 100^0C, 11. Number of Annealing: 1

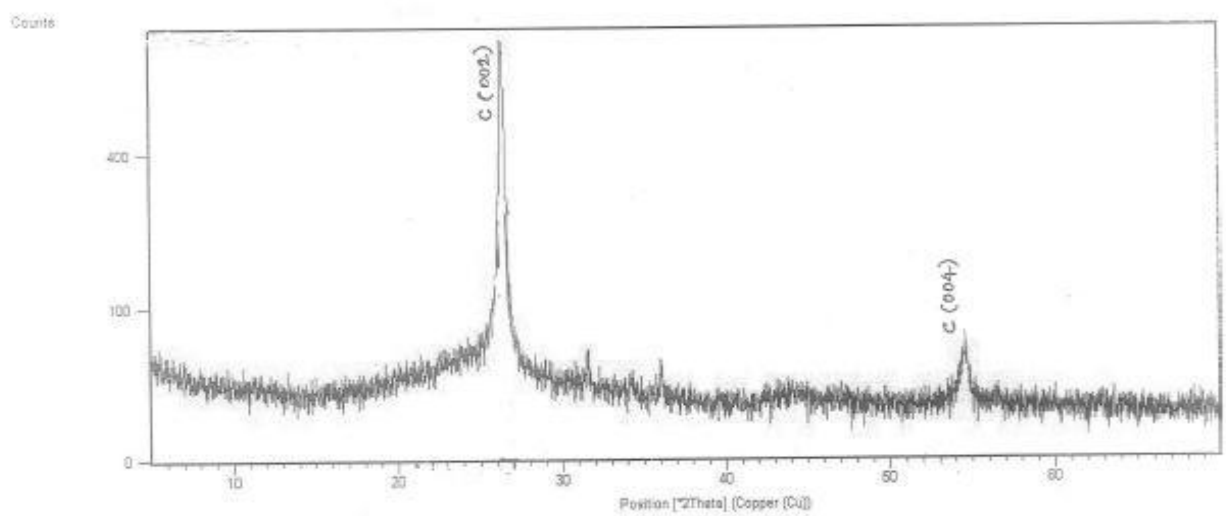
3. RESULTS AND DISCUSSION

Characterization of SWCNTs

3.1 Structural Characterization

GR (0.25 mm) graphite rods

Fig. [3.1.1] emphasized XRD studies on GR (0.25 mm) annealed at 800^{o}C (4 minutes) dipped in Hydrochloric acid (volume: 1 ml) for 120 seconds. The first order [C (002)] and second order [C (004)] diffraction peaks shows the single crystalline nature of the final product. Actually during GR (0.25 mm) graphite rods preparation process, a strong covalent bonding formation takes place inside the carbon matrix, which finally leads to the lattice formation i.e., single crystalline nature of the material.



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3.2 Compositional Characterization

GR (0.25 mm) graphite rods

Fig. [3.2.1] revealed the EDAX studies on GR (0.25mm) annealed at 800^{o}C (3 mints) dipped in double distilled water (DD water) (volume: 2.5 ml) for 30 seconds.

Presence of rich Carbon atoms (1st prominent carbon peak in EDAX spectrum) confirmed that the formed tubes are made up of carbon atoms. Which was authentically shows the formation of CNTs. Due to high temperature annealing, dehydration takes place, H_2 atom was removed. Which was evidentially shown from the EDAX spectrum (i.e., H_2 not found in the EDAX spectrum). Presence of O_2 atom in the EDAX spectrum evidentially shows that oxygen was injected / feed up during annealing. Presence of (Silicon) Si,(Oxygen) O shows that the leaf was enriched with SiO_2 (Soil) in which the tree was grown. S, Na, Cl, K, Fe, Se and Ca proved that the roots of parent tree or any organic material (from which GR (0.25mm) graphite rods were derived) sucked these essential elements (nutrients) from soil for its growth and stability.

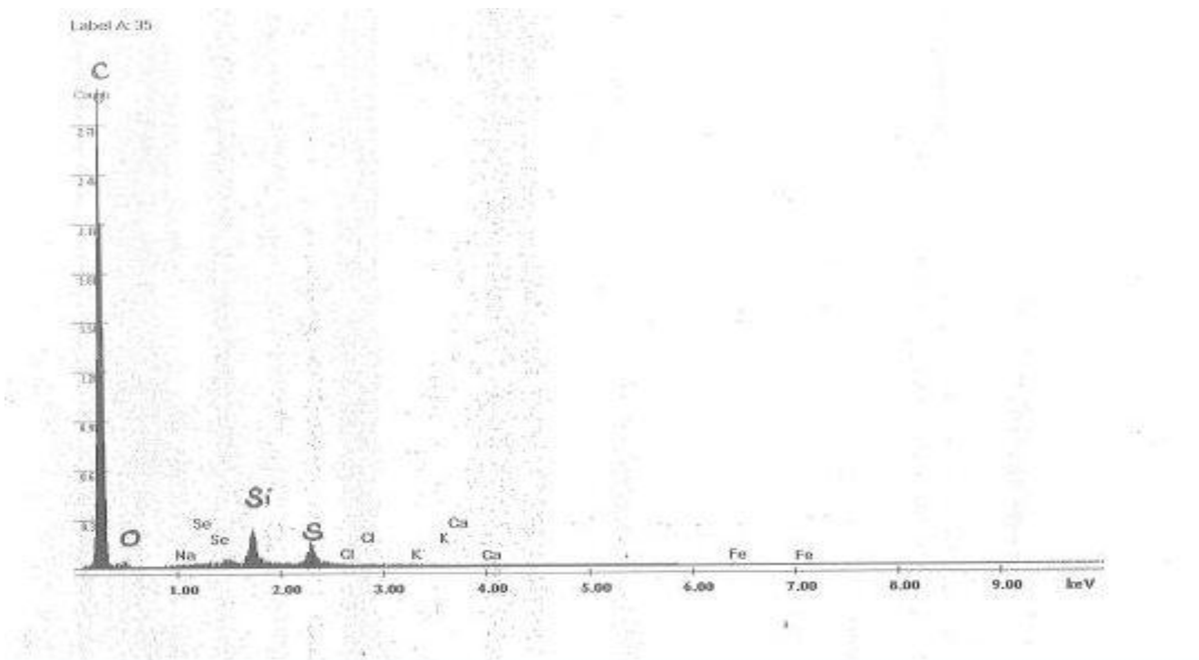

Fig. [3.2.1] EDAX studies on GR (0.25mm) annealed at 800^{o}C (3 mints) dipped in double distilled water (DD water) (volume: 2.5 ml) for 30 seconds.

3.3 Surface Morphological Characterization

SEM studies were carried out with a JEOL JSM-840 operated at 20 KV.

GR (0.25 mm) graphite rods

In Fig.[3.3.1], Surface morphology (SEM) of 800^oC (5 minutes) annealed red hot GR (0.25 mm) dipped in Hydrochloric acid (Hcl) solution (volume: 2.5 ml) for 30 seconds emphasized the clear evidence for the high grade super matured horizontal SWCNTs – perfectly aligned tubular structure of equal diameters (as rolled graphene layer which composed of hexagonal carbon matrix networks) (longitudinal cross sectional view) as individual fibres exhibit a middle hallow empty space (an eagle insight view). In Fig.[3.3.2], Surface morphology (SEM) of 800^oC (1 minute) annealed red hot GR (0.25 mm) dipped in Hydrochloric acid (Hcl) solution (volume: 0.5 ml) for 120 seconds, shows very poor growth of damaged, inclined horizontal SWCNTs – tubular structure as closed individual fibres exhibit a middle hallow empty space (longitudinal cross sectional view). In Fig.[3.3.3], Surface morphology (SEM) of 800^oC (4 minutes) annealed red hot GR (0.25 mm) dipped in Hydrochloric acid (Hcl) solution (volume: 2 ml) for 45 seconds shows a damaged portion of the semi-matured stage of [eagle view], of vertical SW-CNTs. (cross-sectional view). In Fig.[3.3.4], Surface morphology (SEM) of 800^oC (3 minutes) annealed red hot GR (0.25 mm) dipped in Hydrochloric acid (Hcl) solution (volume: 1.5 ml) for 60 seconds shows intermediate, damaged distribution of (tubular) rolled, scattered graphene layers belongs to horizontal SWCNTs (longitudinal cross sectional view). Due to poor coagulation, more star like patterns (composed of needle like grains) were obtained.

In Fig.[3.3.5], Surface morphology (SEM) of 600^oC (5 minutes) annealed red hot GR (0.25 mm) dipped in Sulphuric acid (H_2SO_4) solution (volume: 2.5 ml) for 30 seconds shows imperfect, earliest growth, distribution, scattering and coagulation of hexagonal carbon matrices (basic building blocks of graphene layer) derived from core species. In Fig.[3.3.6], Surface morphology (SEM) of 600^oC (4 minutes) annealed red hot GR (0.25 mm) dipped in Sulphuric acid (H_2SO_4) solution (volume: 2.0 ml) for 45 seconds, shows imperfect, just after initial budding growth, distribution, scattering and coagulation of hexagonal carbon matrices (basic building blocks of graphene layer).

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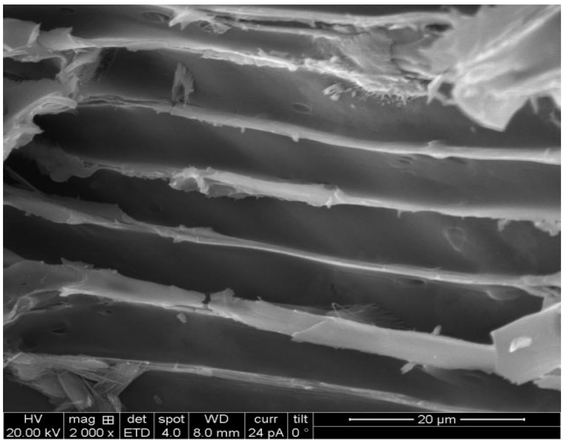

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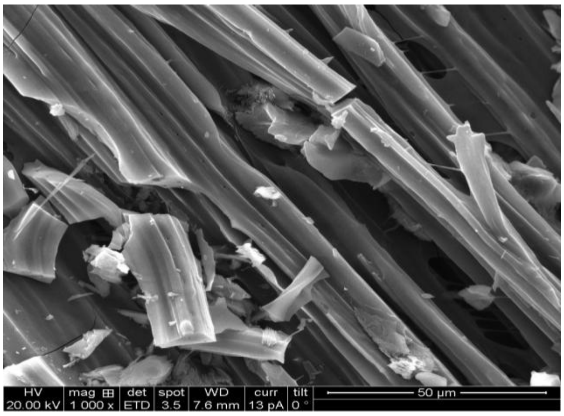
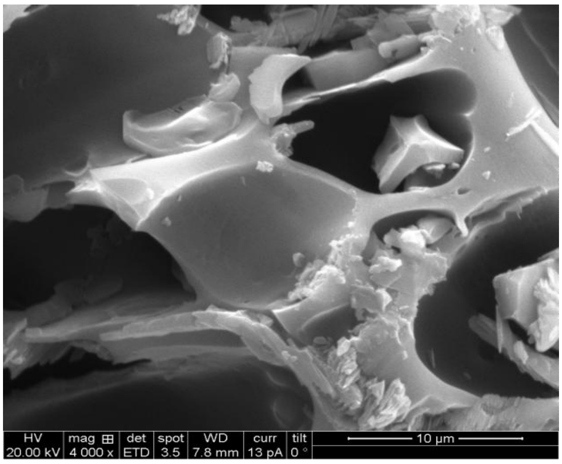


Fig.[3.3.2] Surface morphology (SEM) of 800^{o}C (1 minute) annealed red hot GR (0.25 mm) dipped in Hydrochloric acid (Hcl) solution (volume: 0.5 ml) for 120 seconds, shows very poor growth of damaged, inclined horizontal SWCNTs – tubular structure as closed individual fibres exhibit a middle hallow empty space (longitudinal cross sectional view).



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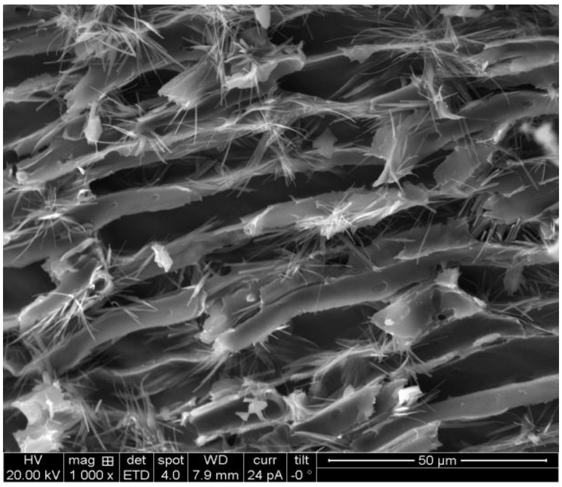
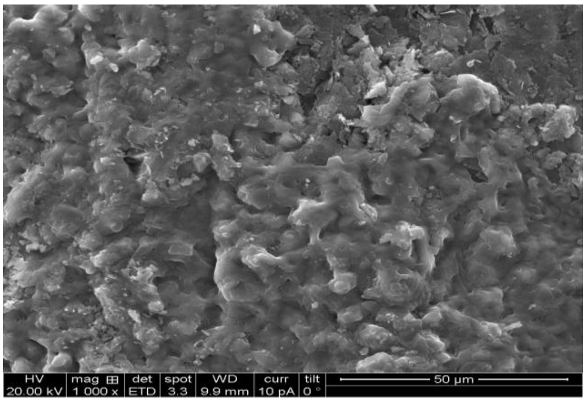


Fig.[3.3.4] Surface morphology (SEM) of 800^{o}C (3 minutes) annealed red hot GR (0.25 mm) dipped in Hydrochloric acid (Hcl) solution (volume: 1.5 ml) for 60 seconds shows intermediate, damaged distribution of (tubular) rolled, scattered graphene layers belongs to horizontal SWCNTs (longitudinal cross sectional view). Due to poor coagulation, more star like patterns (composed of needle like grains) were obtained.



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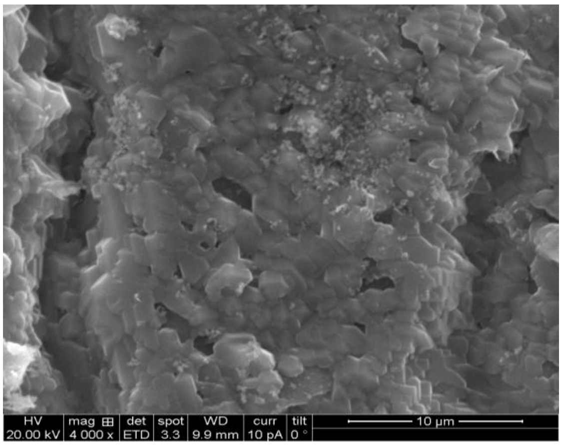

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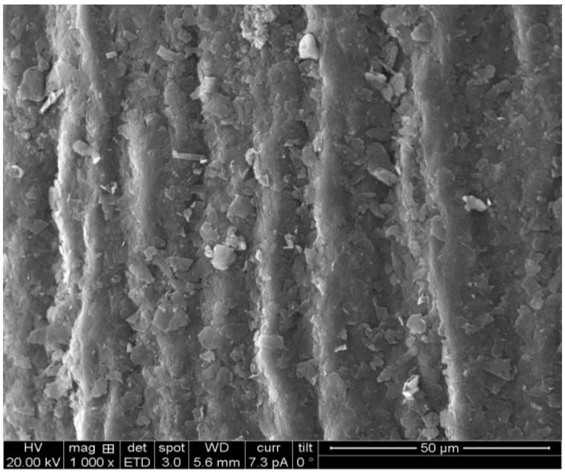


Fig.[3.3.7] Surface morphology (SEM) of 800^{o}C (3 minutes) annealed red hot GR (0.25 mm) dipped in Nitric Acid (HNO_3) solution (volume: 1.5 ml) for 45 seconds, shows an eagle view of perfectly aligned vertical SWCNTs – tubular structure as closed individual fibres exhibit a middle hallow empty space (longitudinal view).

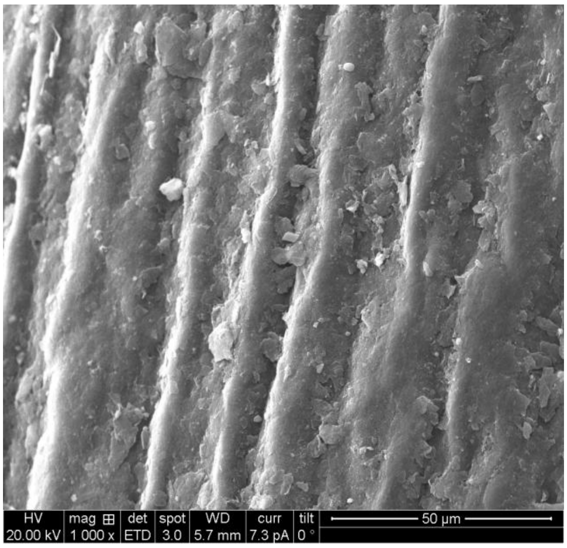


Fig.[3.3.8] Surface morphology (SEM) of 600^{o}C (5 minutes) annealed red hot GR (0.25 mm) dipped in Nitric Acid (HNO_3) solution (volume: 2.5 ml) for 30 seconds, shows shows an eagle view of perfectly aligned super matured vertical SWCNTs – tubular structure as closed individual fibres exhibit a middle hallow empty space (longitudinal view).

In Fig.[3.3.7], Surface morphology (SEM) of 800^{o}C (3 minutes) annealed red hot GR (0.25 mm) dipped in Nitric Acid (HNO_3) solution (volume: 1.5 ml) for 45 seconds, shows an eagle view of perfectly aligned vertical SWCNTs – tubular structure as closed individual fibres exhibit a middle hallow empty space (longitudinal view). In Fig.[3.3.8], Surface morphology (SEM) of 600^{o}C (5 minutes) annealed red hot GR (0.25 mm) dipped in Nitric Acid (HNO_3) solution (volume: 2.5 ml) for 30 seconds, shows an eagle view of perfectly aligned super matured vertical SWCNTs – tubular structure as closed individual fibres exhibit a middle hallow empty space (longitudinal view).

Generally SEM images revealed the earliest formation of hexagonal carbon lattice network (horizontal view) SWCNTs. The image picture out the formation of various phases of carbon atom network due to sudden cooling, after high temperature annealing in GR (0.25 mm). SEM images showed that NaOH solution acts as a suitable medium (any solution which has pH > 7) for the formation of (suffled) vertical growth of SWCNTs with middle hole surmounted by single wall in GR (0.25 mm). SEM showed tubular structure of SWCNTs as individual fibres exhibit a middle hallow empty space with single wall outer cover in Banyan leaf. SEM images informed that NaOH acts as a optimum medium (any solution which has pH >7) for earliest, intermediate stage formation and rolling of graphene sheets to form longitudinal SWCNTs in GR (0.25 mm). SEM images depicts the earliest and matured rolling of graphene sheets to form SWCNTs, which shows that mineral water is an optimum medium for SWCNT growth. Also SEM Photos interpreted the formation of hexagonal carbon (lattice network) matrix. From the SEM studies, we know that, the single time dipping and minimum time of dipping in particular solution leads to the formation of SWCNTs. 600^0C and 800^0C both act as suitable growth environment for SWCNTs.

3.4 Nanostructural Characterization

HRTEM studies were carried out with a JEOL JSM-840 operated at 20 KV.

GR (0.25 mm) graphite rods

In Fig.[3.4.1], HRTEM image pointed out super matured, perfectly aligned SWCNTs grown from 800^0C (3 minutes) red-hot GR (0.25 mm) when dipping in Sulphuric Acid (H_2SO_4) (Volume:2.5 ml) for 30 Sec. In Fig.[3.4.2], HRTEM image delivers super matured, perfectly aligned SWCNTs grown from 800^0C (3 minutes) red-hot GR (0.25 mm) when dipping in Nitric Acid (HNO_3) (Volume: 2.5 ml) for 30 Sec.

In Fig.[3.4.3], HRTEM image focused un-matured intermediate stage of a graphene sheet from 600^0C (1 minute) red-hot GR (0.25 mm) when dipping in Hydrochloric Acid (Hcl) (Volume:1 ml) for 90 Sec. In Fig.[3.4.4], HRTEM image flashed out preliminary tubular formation of SWCNTs grown from 800^0C (2 minutes) red-hot GR (0.25 mm) when dipping in Nitric Acid (HNO_3) (Volume: 1.5 ml) for 120 Sec. In Fig.[3.4.5], HRTEM image flourished un-matured intermediate stage of SWCNTs grown from 800^0C (2 minutes) red-hot GR (0.25 mm) when dipping in Nitric Acid (HNO_3) (Volume: 2 ml) for 90 Sec. In Fig.[3.4.6], HRTEM image showed matured SWCNTs grown from 800^0C (4 minutes) red-hot GR (0.25 mm) when dipping in Nitric Acid (HNO_3) (Volume:2 ml) for 60 Sec.

The effects of optimizations parameters like pH of the various dipping solutions (acidic, basic and neutral), volume of dipping solutions, various types and parts of the materials, various dipping timings, number of annealing and dipping, various annealing temperature, various time of annealing and various dipping solution temperatures on structural, compositional, surface morphological, nano-structural characterizations of various types of materials and on high grade SWCNTs growth with high yield were studied intensively. Parameters, as inferences from above characterizations were calculated and graphically emphasized. Correlation studies between these characterization inferences (such as grain size, purity) and above optimization parameters were carried out with a high light on yield of high grade SWCNTs.

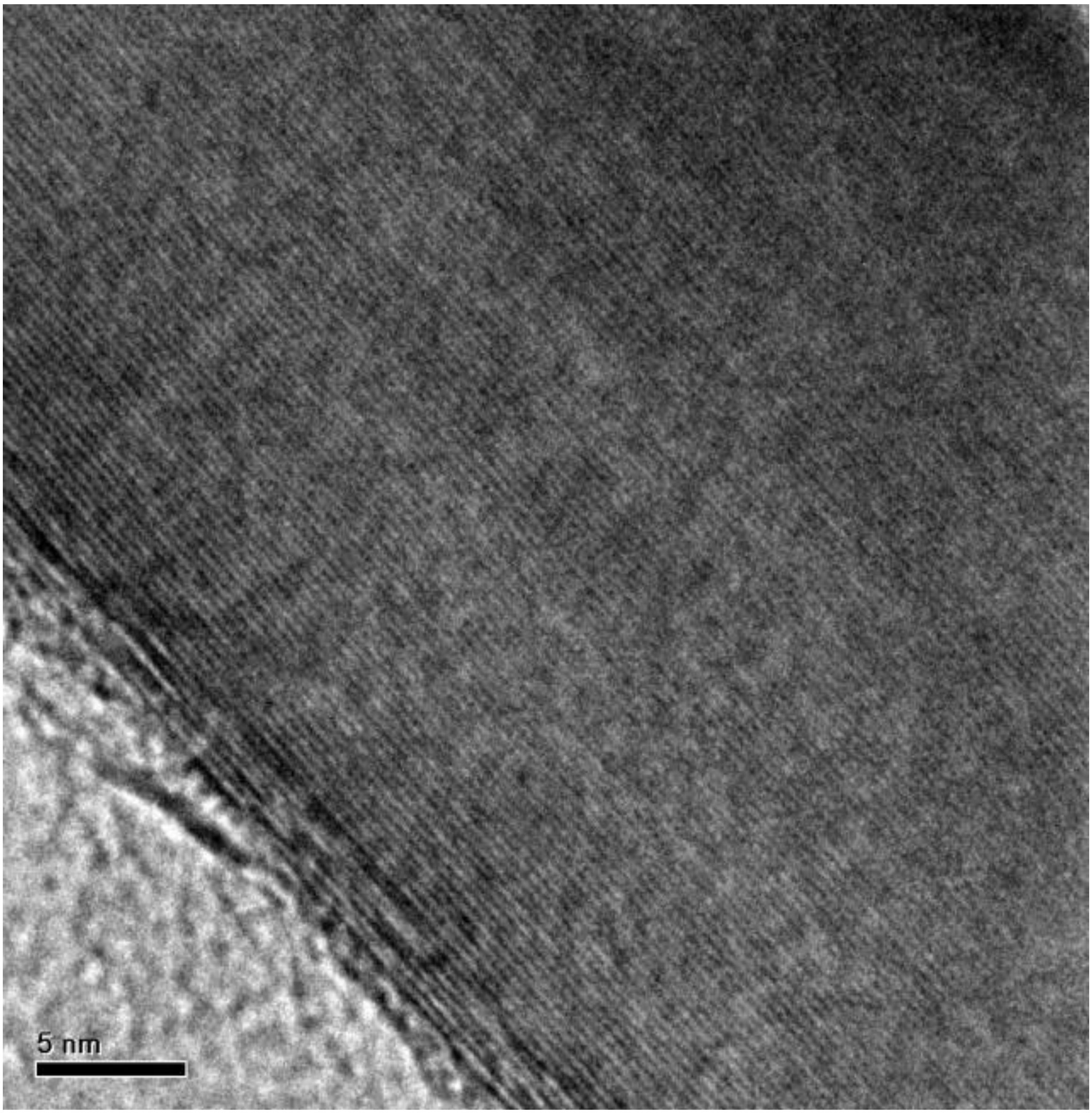


Fig.[3.4.1]. HRTEM image of super matured, perfectly aligned SWCNTs grown from 800^0C (3 minutes) red-hot GR (0.25 mm) when dipping in Sulphuric Acid (H_2SO_4) (Volume: 2.5 ml) for 30 Sec.

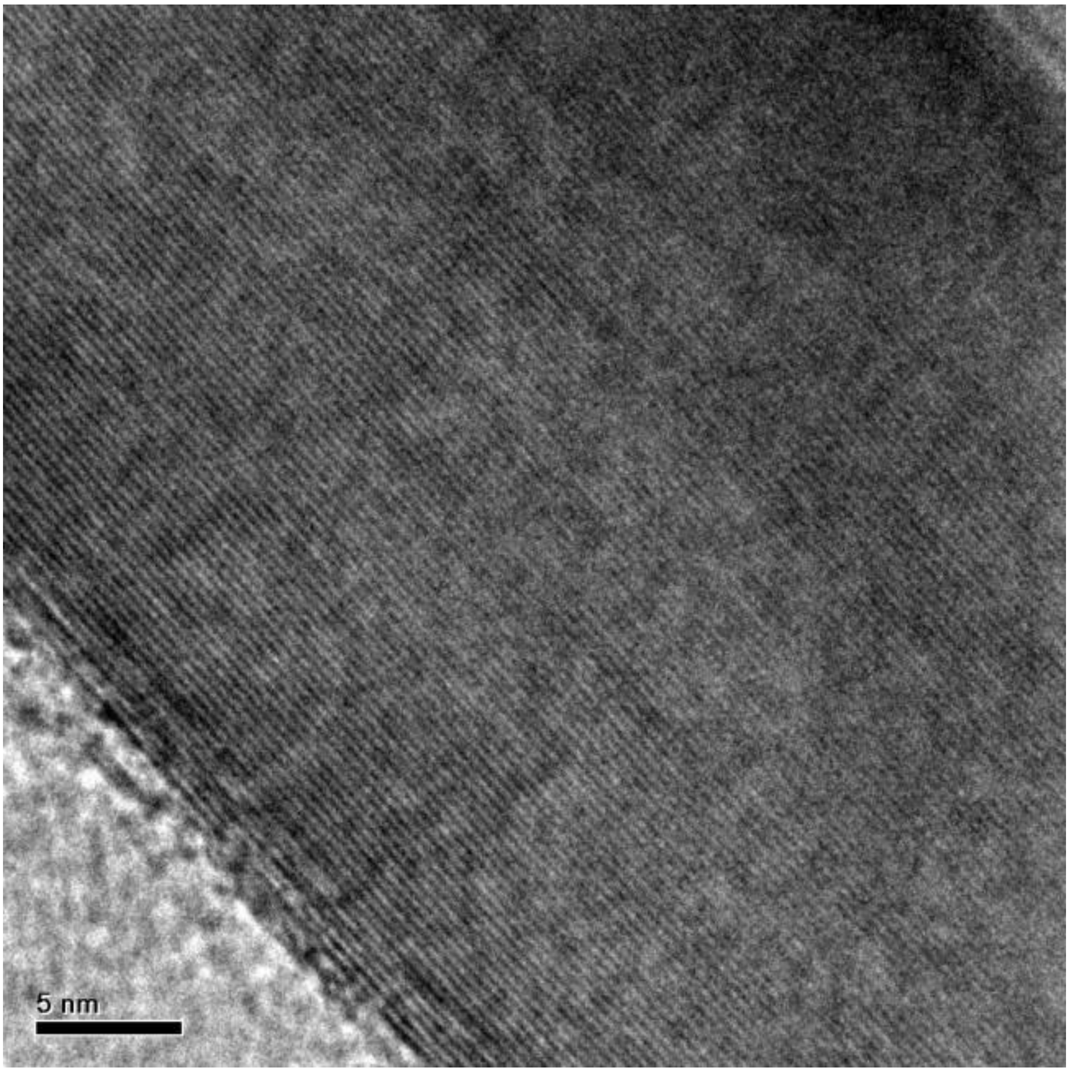


Fig.[3.4.2]. HRTEM image of super matured, perfectly aligned SWCNTs grown from 800^0C (3 minutes) red-hot GR (0.25 mm) when dipping in Nitric Acid (HNO_3) (Volume: 2.5 ml) for 30 Sec.

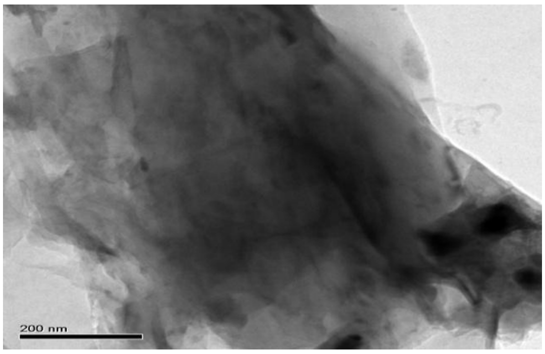


Fig.[3.4.3]. HRTEM image of un-matured intermediate stage of a graphene sheet from 600^0C (1 minute) red-hot GR (0.25 mm) when dipping in Hydrochloric Acid (Hcl) (Volume: 1 ml) for 90 Sec.

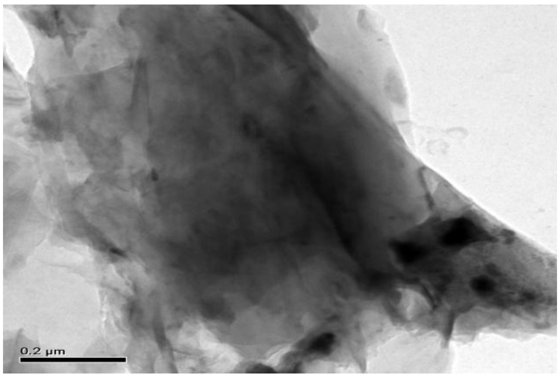
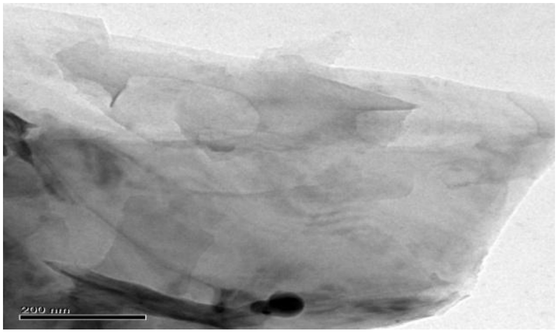


Fig.[3.4.4]. HRTEM image shows preliminary tubular formation of SWCNTs grown from 800^0C (2 minutes) red-hot GR (0.25 mm) when dipping in Nitric Acid (HNO_3) (Volume: 1.5 ml) for 120 Sec.



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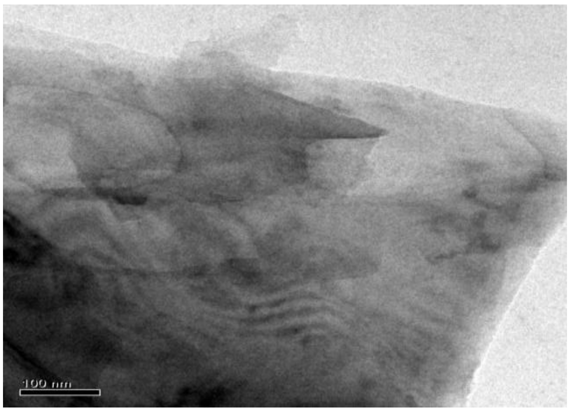

Fig.[3.4.6]. HRTEM image shows matured SWCNTs grown from 800^0C (4 minutes) red-hot GR (0.25 mm) when dipping in Nitric Acid (HNO_3) (Volume: 2 ml) for 60 Sec.

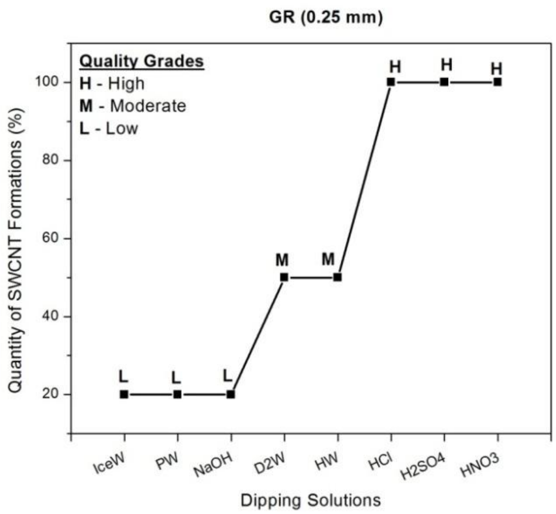

Fig. [4.1] Changes of Quantity of SWCNTs formation (%) with various Dipping solution with a note on Quality grades of developed SWCNTs from GR (0.25mm).

Beyond all of these, we have carried out a novel feasibility study at first time, which comprises the possible usage of precursor graphite rods as organic carbon sources for high grade SWCNTs growth with high yield via a low cost technique and methodology as value in commercial efforts.

4. CONCLUSION

In this over all study, Graphite Rod i.e., GR (0.25 mm) plays as a most optimum material for high grade SWCNTs growth. Generally, we may understand that specialized materials play as most optimum materials than fibrous and conventional materials. Also any Dipping Solution (of Volume: 2.5 ml) having pH=7, i.e., neutral solution (normally maintained at room.temp.,RT) (with 30 Seconds as optimum time of dipping) act as an optimum medium which provides suitable environment for high grade, large quantity SWCNTs growth. Similarly 800^0C (having annealing time: 3 minutes) provides suitable background thermal history for high grade, large quantity SWCNTs growth based on single time annealing and dipping process. High purity precursor material yield high grade SWCNTs.

GR (0.25 mm) graphite rods

Fig.[4.1] related Changes of Quantity of SWCNTs formation (%) with various Dipping solution with a note on Quality grades of developed SWCNTs from GR (0.25mm). The Fig. revealed that acidic dipping solution (having pH < 7) provides a suitable environment for high grade and high quantity SWCNTs growth in GR (0.25 mm) graphite rods varities than neutral solution (having pH =7) and basic solutions (having pH >7). All of these works have value in nanotechnology, nano-materials processing and device fabrication efforts either as a technical or scientific basis, also as a contribution to the present day state of the art of DNA transport in genetic transformation system.

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