



A Novel Synthesis of SWCNTs from Tapioca for Opto-Electronic Devices: 3D – High Efficiency Solar Cells, Flat Panel Displays, LEDs & Lasers, Magnetic Storage Devices: High Capacity Pen Drives and Nano Thermometers – A New Feasibility Study

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ABSTRACT

A novel modified AC method – VSA methodology (with KRS or NTFDS theory) was adopted in this present work for the preparation of SWCNTs from natural organics i.e., Tapioca leaves towards the possibility of application to Opto-electronic devices : 3D – high efficiency Solar cells, Flat panel displays, LEDs & Lasers, Magnetic storage devices and Nano thermometers. Structural, Compositional, Surface Morphological and Nano structural Characterizations were carried out on harvested products. 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 materials and on high grade SWCNTs growth with high yield were studied intensively. Inferences from characterizations were derived 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 new feasibility study at first time, which comprises the possible usage of precursor organic carbon source i.e., Tapioca leaves for high grade SWCNTs with high yield via a low cost technique and methodology as value in commercial efforts.

Keywords: 3D – high efficiency Solar cells; Flat panel displays; High capacity pen drives and Nano thermometers; HRTEM; LEDs & Lasers; Magnetic storage devices; Modified AC method; Natural organics; NTFDS theory; Opto-electronic devices; SWCNTs; Tapioca leaves; VSA methodology.

1. INTRODUCTION

The present work showed that SWCNTs obtained by open air annealing and cooling of leaves of Tapioca. The present work made a break through via plays an alternative method to conventional, traditional methods such as arc discharge, laser ablation, metal catalysts, pyrolysis, and varieties of CVD methods and also removes the usage of synthetic chemicals. Due to that dehydration (removal of H₂O molecules from C-H-O matrix) takes place. Finally it allows carbon atoms only present in the material part. The modified AC method (Zhenhui Kang *et al.* 2005) – VSA methodology (with KRS or NTFDS theory) was adopted in this present work for the preparation of CNTs from natural organics. 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 in detail. The characterizations performed on outcome products were internationally accepted, standard methods as followed by field specialists through out the world (Iijima, 1991; Iijima, 1993; Ajayan and Ebbesen, 1997; Ajayan and Iijima, 1993; Yakobson and Smalley, 1997; Dresselhaus, 1996; Ebbesen, 1997; Dekker, 1999; Dujardin *et al.* 1994; Collins and Zettl, 1996; Dean and Chalamala *et al.* 1999; Winter *et al.* 1998; Dresselhaus *et al.* 1999; Calvert, 1999; Shaoping Xiao and Wenyi Hou, 2006; Zhiyang Rong, 2008; Rostam Moradian and Ali Fathalian, 2006; Chun Li *et al.* 2007; Rossi *et al.* 2004; Majumder *et al.* 2005; Mattia *et al.* 2007; Guoyong Xu *et al.* 2007; Teh-Hwa *et al.* 2007; Kuromochi *et al.* 2007; Bond *et al.* 2007; Xue, 2006; Wang *et al.* 2006; Chiu *et al.* 2002; Wang *et al.* 2005; Shiren Wang *et al.* 2006; Safarova *et al.* 2007; Bellucci *et al.* 2007; Zhenhui Kang *et al.* 2005).

2. EXPERIMENTAL DETAILS

Materials i.e., Tapioca leaves was 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 °C and 800 °C for various time of heating viz., 1 minute to 5 minutes. After that they were immediately dipped into various types of solutions viz., Sodium hydroxide (NaOH), Hydrogen peroxide (H₂O₂), Nitric Acid (HNO₃), Sulphuric Acid (H₂SO₄), 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 °C to 100 °C, 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: conventional: varieties of plants, trees: Tapioca,
4. Various Parts of the materials: leaves, stems,
5. Various Dipping solutions: Sodium hydroxide (NaOH), Hydrogen peroxide (H₂O₂), Nitric Acid (HNO₃), Sulphuric Acid (H₂SO₄), 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 °C and 800 °C,
9. Various Time of Annealing: 1 minute to 5 minutes,
10. Various Dipping Solution temperatures: 0 °C to 100 °C,
11. Number of Annealing: 1

3. RESULTS & DISCUSSION

Characterization of SWCNTS

3.1 Structural Characterization Tapioca Leaves

Fig. 1 delivered X-ray diffract gram (XRD) of Tapioca leaves annealed at 600 °C (2 minutes) dipped in NaOH solution @ 45 °C (volume: 1.5 ml) for 90 seconds. Fig. 2 defined X-ray diffract gram (XRD) of Tapioca leaves annealed at 800 °C (4 minutes) dipped in Hot water mixed with DD water @ 100 °C (volume: 0.5 ml) for 30 seconds. The XRD spectrums showed amorphous (non-crystalline) nature.

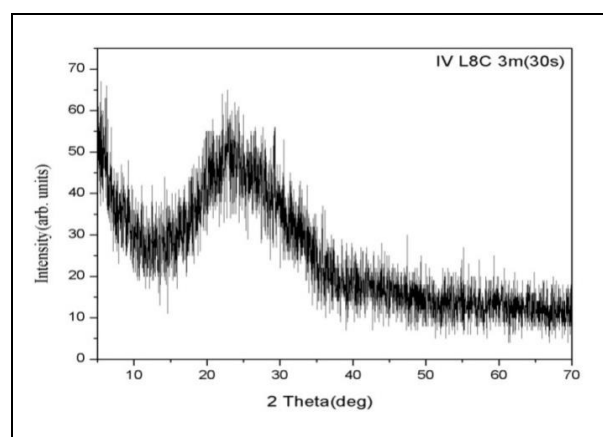


Fig. 1: X-ray diffract gram (XRD) of tapioca annealed at 600°C (2 minutes) dipped in NaOH solution @ 45 °C (Volume: 1.5 ml) for 90 seconds.

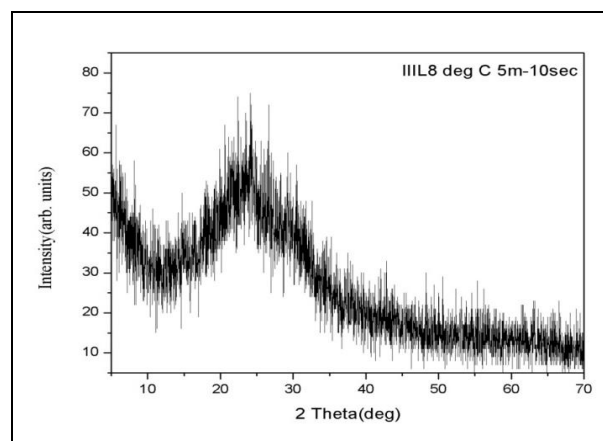


Fig. 2: X-ray diffract gram (XRD) of Tapioca annealed at 800 °C (4 minutes) dipped in Hot water mixed with DD water @ 100°C (volume: 0.5 ml) for 30 seconds.

Open air dry of tapioca fresh leaves after taken out from field and annealing at 600 °C (2 min) and 800°C (4 minutes) leads to complete dryness of leaf. Due to that dehydration (removal of H₂O molecules from C-H-O matrix) takes place in the leaf. Finally it allows carbon

atoms only present in the leaf. After annealing, we have to immediately undergone that leaves by dipping into NaOH solution with solution temperature 45 °C and Hot water mixed with DD water with solution temperature 100 °C. Due to sudden cooling, the carbon lattices present in the leaves were cracked, split up into individual atoms. Finally that lattice formation is disappeared, leads to amorphous (non-crystalline) nature.

3.2 Compositional Characterization Tapioca Leaves

Fig. 3 confirmed the EDAX studies on Tapioca leaves annealed at 800 °C (2 mints) dipped in Mineral water (volume: 2.5 ml) for 60 seconds. Fig. 4 interpreted the EDAX studies on Tapioca leaves annealed at 800 °C (2 mints) dipped in Sodium Hydroxide (NaOH) (volume: 2.5 ml) for 60 seconds. The EDAX spectrums showed compositional elements present in the Tapioca leaves.

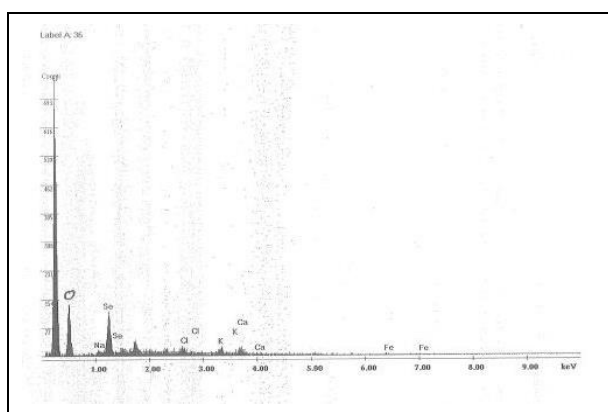


Fig. 3: EDAX studies on Tapioca leaf annealed at 800 °C (2 mints) dipped in Mineral water (volume: 2.5 ml) for 60 seconds.

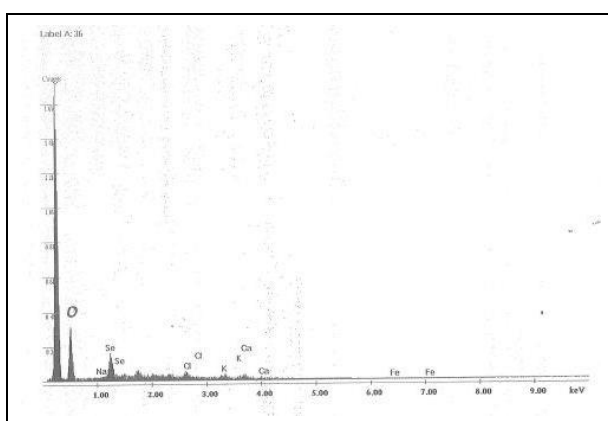


Fig. 4: EDAX studies on Tapioca leaf annealed at 800 °C (2 mints) dipped in Sodium Hydroxide (NaOH) (volume: 2.5 ml) for 60 seconds.

Presence of rich Carbon atoms (1st prominent 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, H₂ atom was removed. Which was evidentially shown from the edax spectrum (i.e., H₂ not found in the EDAX spectrum) Presence of O₂ 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 SiO₂ (Soil) in which the plant (herb) was grown. Na, Se, K, Cl, Ca and Fe proved that the Soil was enriched with fertilizer and roots of plant sucked these essential elements (nutrients) from soil for its growth and stability.

3.3 Surface Morphological Characterization

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

Tapioca Leaves

In Fig. 5, Surface morphology (SEM) of 800 °C (5 minutes) annealed red hot Tapioca dipped in Hot Water mixed with Double Distilled Water (HW+D2W)solution (volume: 2.5 ml) for 30 seconds, shows close up view of earliest high grade SWCNTs threading out (earliest tubular growth of graphene layer rolling) from carbon core. In Fig. 6, Surface morphology (SEM) of 800 °C (3 minutes) annealed red hot Tapioca dipped in Hot Water mixed with Double Distilled Water (HW+D2W) solution (volume: 1.5 ml) for 60 seconds, shows close up view of intermediate stage of free standing, shuffled high grade SWCNTs already threaded out from carbon core (an authentication, also a better evidence for SWCNTs growth) .

In Fig. 7, Surface morphology (SEM) of 800 °C (2 minutes) annealed red hot Tapioca dipped in Hot Water mixed with Double Distilled Water (HW+D2W) solution (volume: 1 ml) for 90 seconds, shows close up view of pre-final stage of free standing, semi-matured, shuffled high grade SWCNTs already threaded out from carbon core (an authentication, also a better evidence for SWCNTs growth) . In Fig. 8, Surface morphology (SEM) of 800 °C (2 minutes) annealed red hot Tapioca dipped in Mineral Water(MW) (volume: 1.0 ml) for 90 seconds, shows close up view of clustering of hexagonal carbon matrices due to poor coagulation during sudden cooling-dipping.

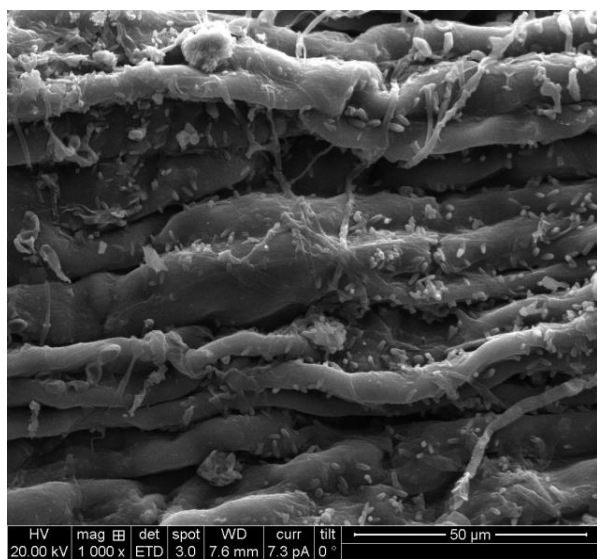


Fig. 5: Surface morphology (SEM) of 800 °C (5 minutes) annealed red hot Tapioca dipped in Hot Water mixed with Double Distilled Water (HW+D2W) solution (volume: 2.5 ml) for 30 seconds, shows close up view of earliest high grade SWCNTs threading out (earliest tubular growth of graphene layer rolling) from carbon core.

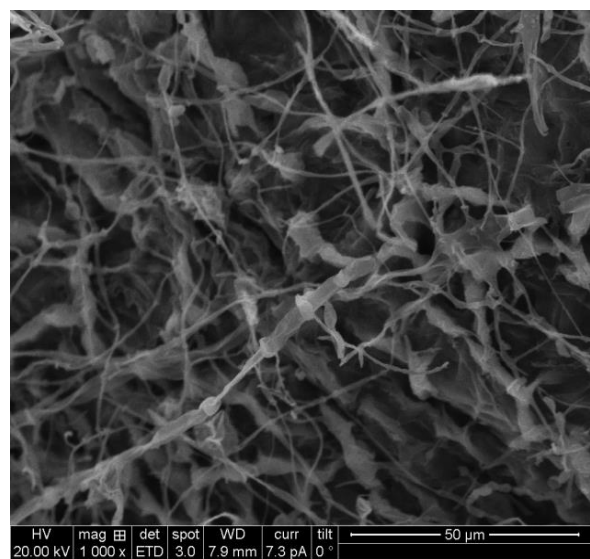


Fig. 6: Surface morphology (SEM) of 800 °C (3 minutes) annealed red hot Tapioca dipped in Hot Water mixed with Double Distilled Water (HW+D2W) solution (volume: 1.5 ml) for 60 seconds, shows close up view of intermediate stage of free standing, shuffled high grade SWCNTs already threaded out from carbon core (an authentication, also a better evidence for SWCNTs growth).

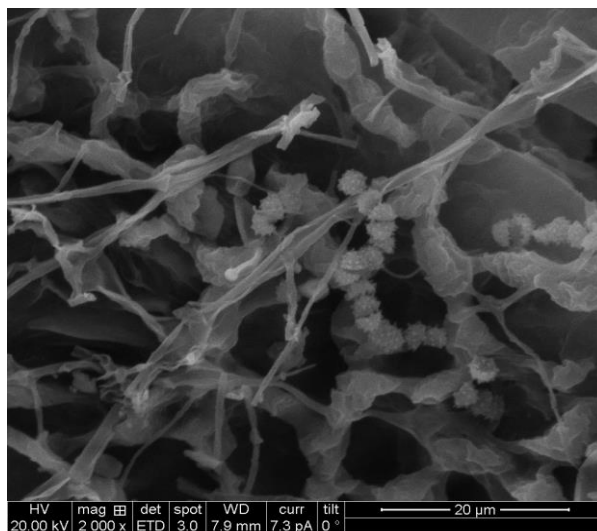


Fig. 7: Surface morphology (SEM) of 800 °C (2 minutes) annealed red hot Tapioca dipped in Hot Water mixed with Double Distilled Water (HW+D2W) solution (volume: 1 ml) for 90 seconds, shows close up view of pre-final stage of free standing, semi-matured, shuffled high grade SWCNTs already threaded out from carbon core (an authentication, also a better evidence for SWCNTs growth).

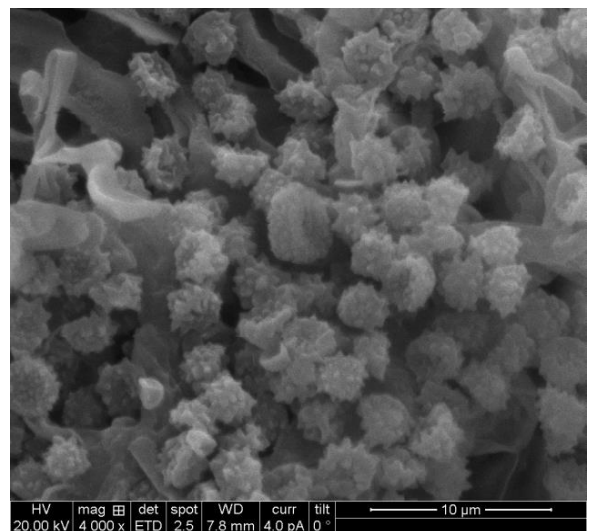


Fig. 8: Surface morphology (SEM) of 800 °C (2 minutes) annealed red hot Tapioca dipped in Mineral Water (MW) (volume: 1.0 ml) for 90 seconds, shows close up view of clustering of hexagonal carbon matrices due to poor coagulation during sudden cooling-dipping.

In Fig. 9, Surface morphology (SEM) of 800 °C (1 minute) annealed red hot Tapioca dipped in mineral water (MW) (volume: 0.5 ml) for 120 seconds, shows close up view of very poor coagulation of hexagonal carbon matrices due to sudden cooling-dipping. In Fig. 10, Surface morphology (SEM) of 800 °C (3 minutes) annealed red hot Tapioca dipped in Mineral Water (MW) solution (volume: 1.5 ml) for 60 seconds, shows close up view of intermediate stage of free standing, shuffled moderate grade tubular SWCNTs threaded out from clustering of hexagonal carbon matrices (originated due to poor coagulation during sudden cooling-dipping). (an authenticated better evidence for SWCNTs growth).

In Fig. 11, Surface morphology (SEM) of 800 °C (5 minutes) annealed red hot Tapioca leaf dipped in Mineral Water (MW) solution (volume: 2.5 ml) for 30 seconds, shows close up view of final stage of free standing, shuffled, matured moderate grade tubular SWCNTs threaded out from clustering of hexagonal carbon matrices (originated due to poor coagulation during sudden cooling-dipping). (an authenticated better evidence for SWCNTs growth). In Fig. 12, Surface morphology (SEM) of 800 °C (1 minute) annealed red hot Tapioca leaf dipped in Sodium Hydroxide (NaOH) Solution (volume: 0.5 ml) for 120 seconds, shows very poor coagulation of basic carbon core regions.

In Fig. 13, Surface morphology (SEM) of 800 °C (3 minutes) annealed red hot Tapioca dipped in Sodium Hydroxide (NaOH) Solution (volume: 1.5 ml) for 75 seconds, shows close up view of (intermediate stage) scattering of hexagonal carbon networks which were already derived from inner carbon core. In Fig. 14, Surface morphology (SEM) of 800 °C (4 minutes) annealed red hot Tapioca dipped in Sodium Hydroxide (NaOH) Solution (volume: 2.0 ml) for 45 seconds, shows close up view of tubular textured high grade SWCNTs threading out [via graphene sheet rolling] from base carbon core.

In Fig. 15, Surface morphology (SEM) of 800 °C (5 minutes) annealed red hot Tapioca dipped in Sodium Hydroxide (NaOH) Solution (volume: 2.5 ml) for 30 seconds, shows close up view of well advanced, free standing, super matured SWCNTs which were already threaded out from (via graphene layer rolling) base carbon matrices. In Fig. 16, Surface morphology (SEM) of 800 °C (1 minute) annealed red hot Tapioca dipped in Hot Water mixed with Double Distilled Water (HW+D2W) solution (volume: 0.5 ml) for 120 seconds, shows a clear evidence for deriving of high grade SWCNTs from carbon core species.

In Fig. 17, Surface morphology (SEM) of 800 °C (3 minutes) annealed red hot Tapioca dipped in Hot Water mixed with Double Distilled Water (HW+D2W) solution (volume: 1.5 ml) for 75 seconds, shows an intermediate growth stage of high grade SWCNTs from carbon core species via hexagonal carbon clusters. In Fig. 18, Surface morphology (SEM) of 800 °C (4 minutes) annealed red hot Tapioca dipped in Hot Water mixed with Double Distilled Water (HW+D2W) solution (volume: 2.0 ml) for 45 seconds, shows close up view of pre-final, matured high grade SWCNTs-tubular texture threaded out from core species via graphene rolling.

In Fig. 19, Surface morphology (SEM) of 800 °C (4 minutes) annealed red hot Tapioca dipped in Mineral Water (MW) (volume: 2.0 ml) for 45 seconds, shows close up view of in-homogeneously distributed moderately grown hexagonal carbon networks originated from carbon core species due to explosion during sudden cooling-dipping. In Fig. 20, Surface morphology (SEM) of 800 °C (3 minutes) annealed red hot Tapioca leaf dipped in Mineral Water (MW) (volume: 1.5 ml) for 75 seconds, shows eagle view and evidential proof for the origination of tubular SWCNTs from hexagonal carbon network distribution in an intermediate stage.

3.4 Nanostructural Characterization

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

Tapioca Leaves

In Fig. 21, HRTEM image shows poor Coagulation of Scattered pentagonal, hexagonal carbon networks, which forms Graphene sheets (due to sudden cooling inside dipping solution) from 800 °C (2 minutes) red-hot Tapioca leaf when dipping in Mineral Water (MW) (volume: 0.5 ml) for 120 seconds, kept at room temperature based on single time annealing and single time dipping process. In Fig. 22, HRTEM image shows an intermediate stage of very poor coagulation of scattered pentagonal, hexagonal carbon networks, which forms graphene sheets from 600 °C (2 minutes) red-hot Tapioca leaf when dipping in NaOH solution (volume: 1 ml) for 90 Seconds (the dipping solution was kept at room temperature) based on single time annealing and dipping.

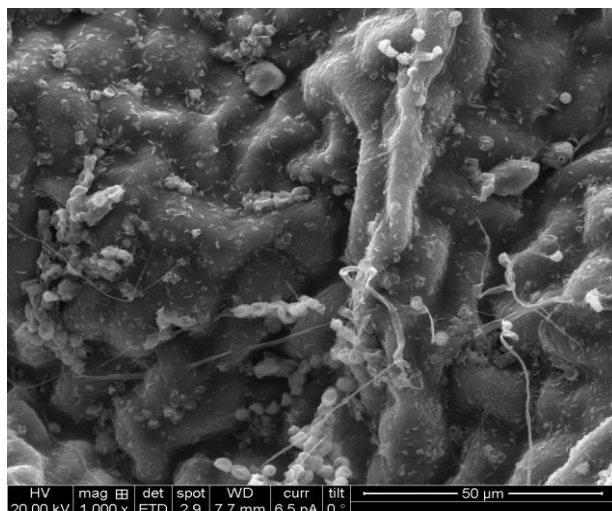


Fig. 9: Surface morphology (SEM) of 800 °C (1 minute) annealed red hot Tapioca dipped in Mineral Water (MW) (volume: 0.5 ml) for 120 seconds, shows close up view of very poor coagulation of hexagonal carbon matrices due to sudden cooling-dipping.

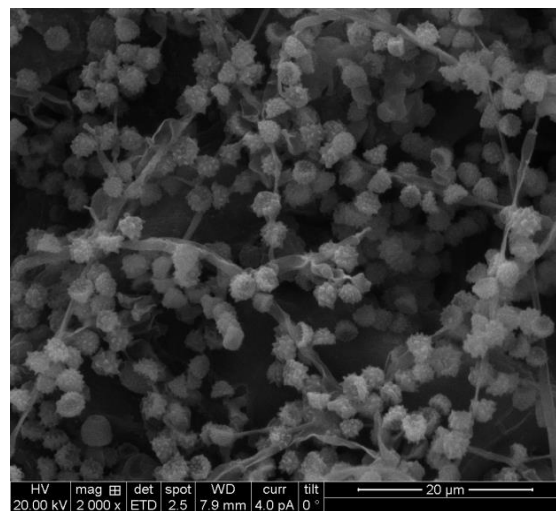


Fig. 10: Surface morphology (SEM) of 800 °C (3 minutes) annealed red hot Tapioca dipped in Mineral Water (MW) solution (volume: 1.5 ml) for 60 seconds, shows close up view of intermediate stage of free standing, shuffled moderate grade tubular SWCNTs threaded out from clustering of hexagonal carbon matrices (originated due to poor coagulation during sudden cooling-dipping). (an authenticated better evidence for SWCNTs growth).

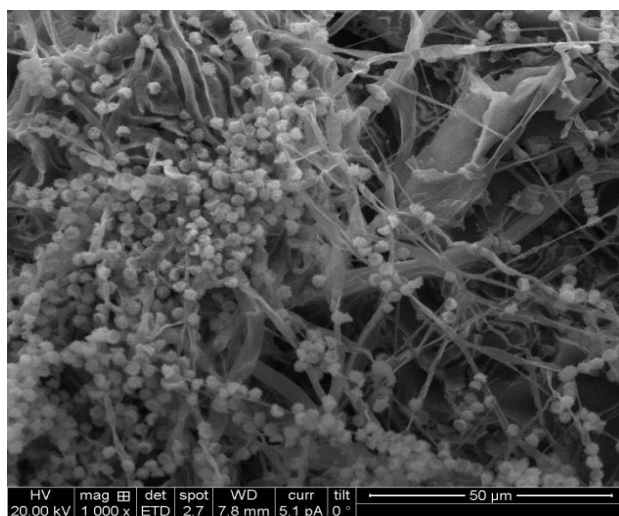


Fig. 11: Surface morphology (SEM) of 800 °C (5 minutes) annealed red hot Tapioca leaf dipped in Mineral Water (MW) solution (volume: 2.5 ml) for 30 seconds, shows close up view of final stage of free standing, shuffled, matured moderate grade tubular SWCNTs threaded out from clustering of hexagonal carbon matrices (originated due to poor coagulation during sudden cooling-dipping). (an authenticated better evidence for SWCNTs growth).

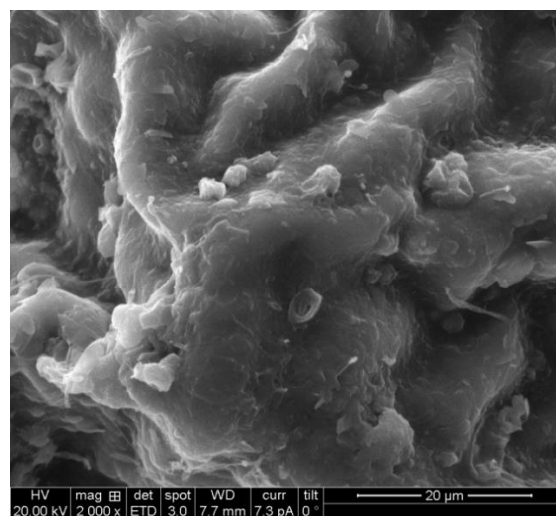


Fig. 12: Surface morphology (SEM) of 800 °C (1 minute) annealed red hot Tapioca leaf dipped in Sodium Hydroxide (NaOH) Solution (volume: 0.5 ml) for 120 seconds, shows very poor coagulation of basic carbon core regions.

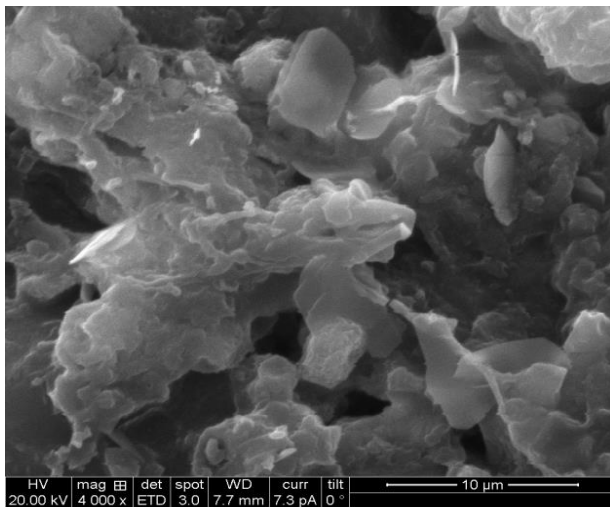


Fig. 13: Surface morphology (SEM) of 800 °C (3 minutes) annealed red hot Tapioca dipped in Sodium Hydroxide (NaOH) Solution (volume: 1.5 ml) for 75 seconds, shows close up view of (intermediate stage) scattering of hexagonal carbon networks which were already derived from inner carbon core.

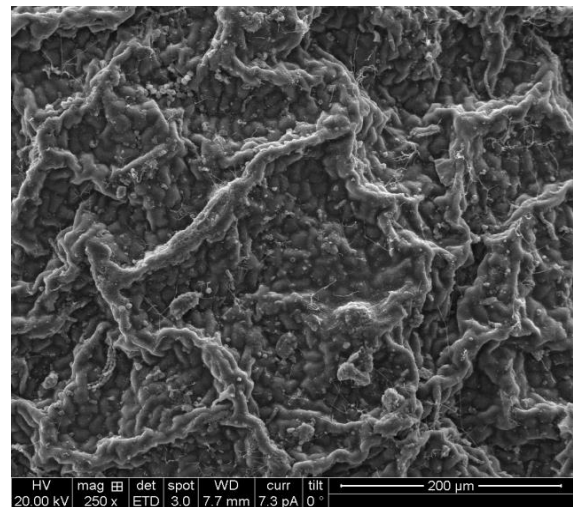


Fig. 14: Surface morphology (SEM) of 800 °C (4 minutes) annealed red hot Tapioca dipped in Sodium Hydroxide (NaOH) Solution (volume: 2.0 ml) for 45 seconds, shows close up view of tubular textured high grade SWCNTs threading out [via graphene sheet rolling] from base carbon core.

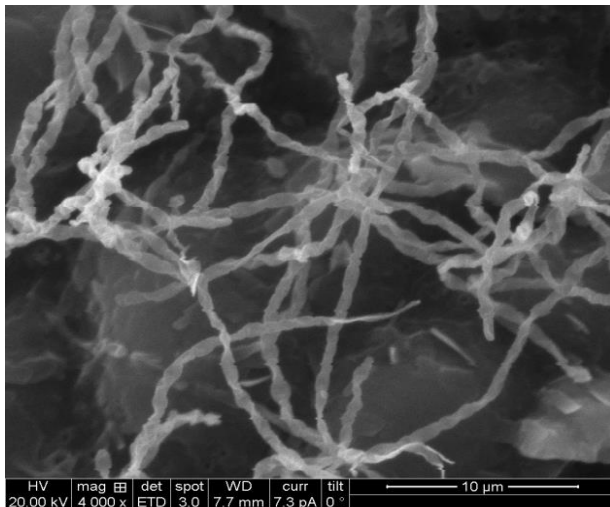


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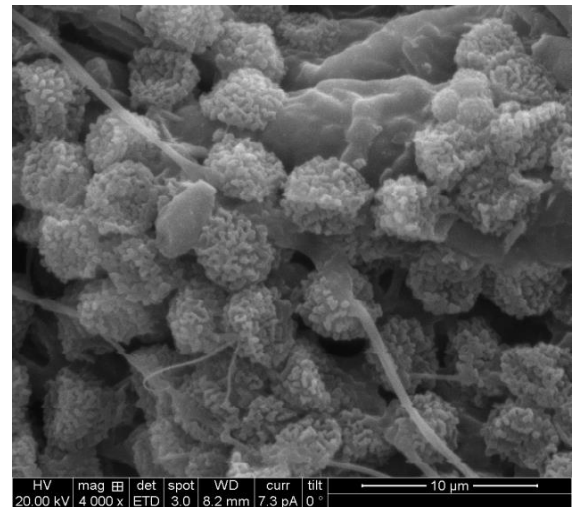


Fig. 16: Surface morphology (SEM) of 800 °C (1 minute) annealed red hot Tapioca dipped in Hot Water mixed with Double Distilled Water (HW+D2W) solution (volume: 0.5 ml) for 120 seconds, shows a clear evidence for deriving of high grade SWCNTs from carbon core species.

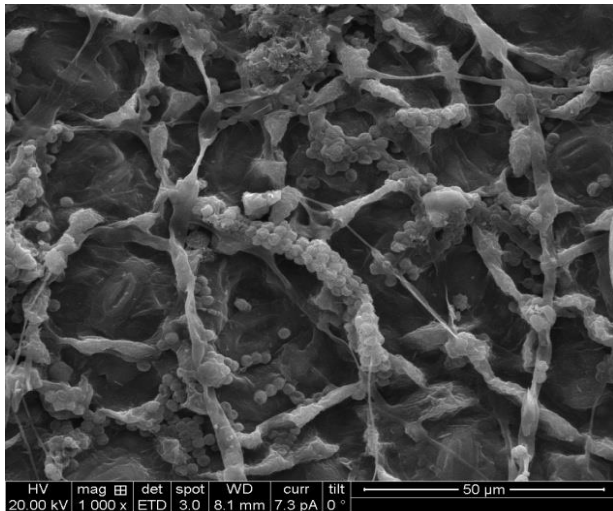


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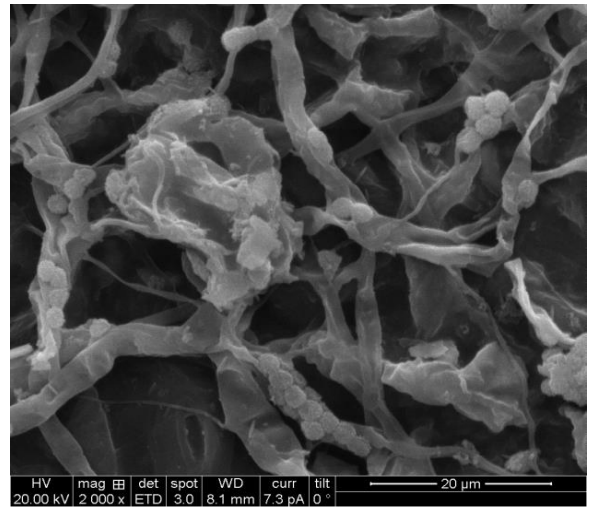


Fig. 18: Surface morphology (SEM) of 800 °C (4 minutes)annealed red hot Tapioca dipped in Hot Water mixed with Double Distilled Water (HW+D2W)solution (volume: 2.0 ml) for 45 seconds, shows close up view of pre-final, matured high grade SWCNTs-tubular texture threaded out from core species via graphene rolling.

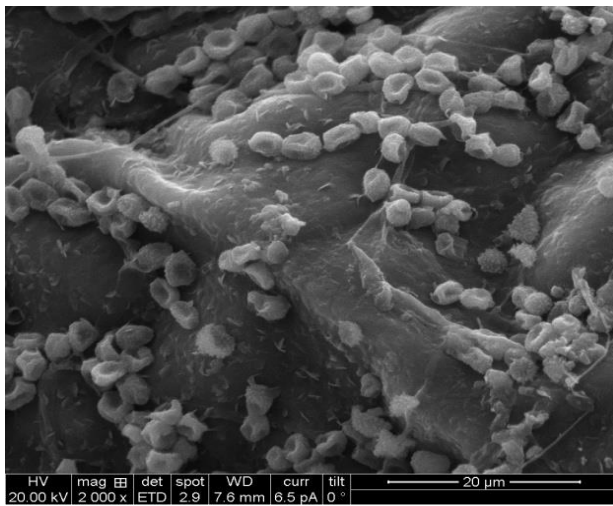


Fig. 19: Surface morphology (SEM) of 800 °C (4 minutes) annealed red hot Tapioca dipped in Mineral Water (MW) (volume: 2.0 ml) for 45 seconds, shows close up view of in-homogeneously distributed moderately grown hexagonal carbon networks originated from carbon core species due to explosion during sudden cooling-dipping.

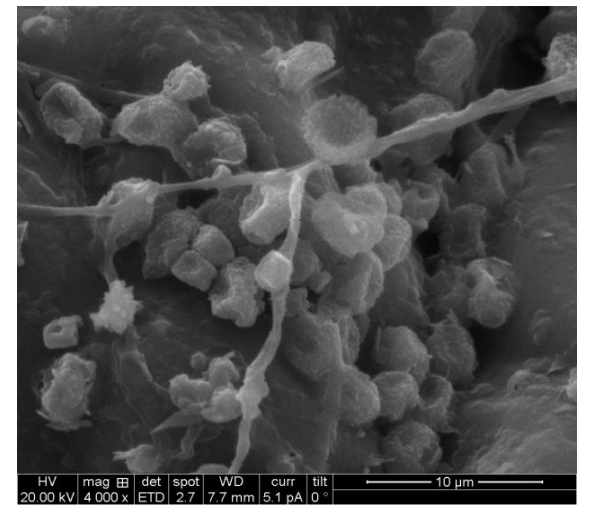


Fig. 20: Surface morphology (SEM) of 800 °C (3 minutes) annealed red hot Tapioca leaf dipped in Mineral Water (MW) (volume: 1.5 ml) for 75 seconds, shows eagle view and evidential proof for the origination of tubular SWCNTs from hexagonal carbon network distribution in an intermediate stage.

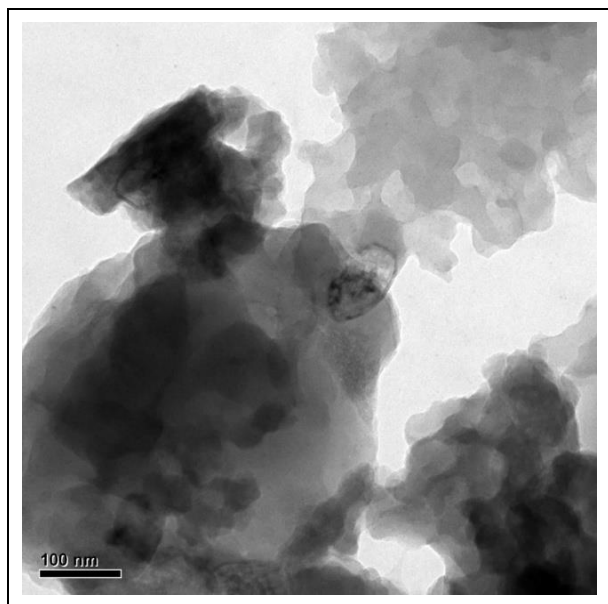


Fig. 21: HRTEM image shows poor Coagulation of Scattered pentagonal, hexagonal carbon networks, which forms Graphene sheets (due to sudden cooling inside dipping solution) from 800 °C (2 minutes) red-hot Tapioca leaf when dipping in Mineral Water (MW) (volume: 0.5 ml) for 120 seconds, kept at room temperature based on single time annealing and single time dipping process.

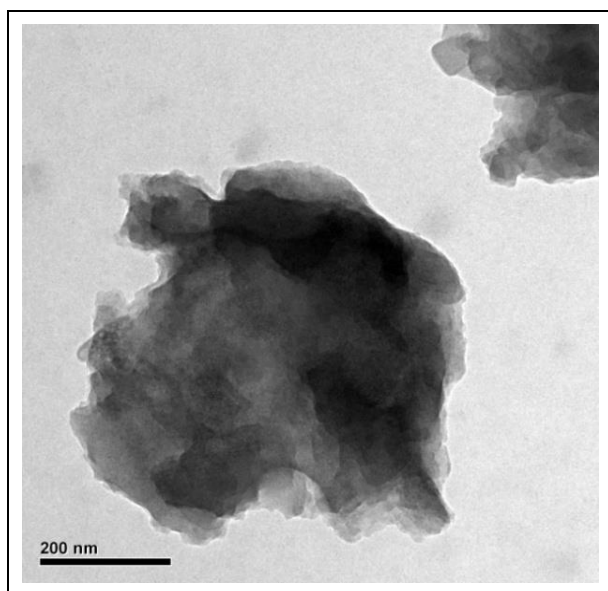


Fig. 22: HRTEM image shows an intermediate stage of very poor coagulation of scattered pentagonal, hexagonal carbon networks, which forms graphene sheets from 600 °C (2 minutes) red-hot Tapioca leaf when dipping in NaOH solution (volume: 1 ml) for 90 Seconds (the dipping solution was kept at room temperature) based on single time annealing and dipping.

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 materials and on high grade SWCNTs growth with high yield were studied intensively. Parameters, as inferences from above characterizations were calculated, tabulated and graphically emphasized. Correlation studies between these characterization inferences (such as grain size, purity) and optimization parameters were carried out with a high light on yield of high grade SWCNTs. Beyond all of these, we have carried out a new feasibility study at first time, which comprises the possible usage of precursor organic carbon source i.e., Tapioca leaves for high grade SWCNTs with high yield via a low cost technique and methodology as value in commercial efforts.

4. CNCLUSION

In this over all study, Tapioca leaves (under conventional materials) plays as a suitable material for high grade SWCNTs growth. 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 °C (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.

Tapioca Leaves

Fig. 23 delivered the Profile of Quantity of SWCNTs formation (%) with different dipping solution with an over view on Quality grades of grown SWCNTs, from Tapioca leaves. Also it confirms that neutral (having pH=7) and basic (having pH.>7) dipping solutions provide suitable environment for high yield of high grade SWCNTs. 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 energy efficient opto-electronic devices such as 3D – high efficiency Solar cells, Flat panel displays, LEDs & Lasers, Magnetic storage devices and Nano thermometers.

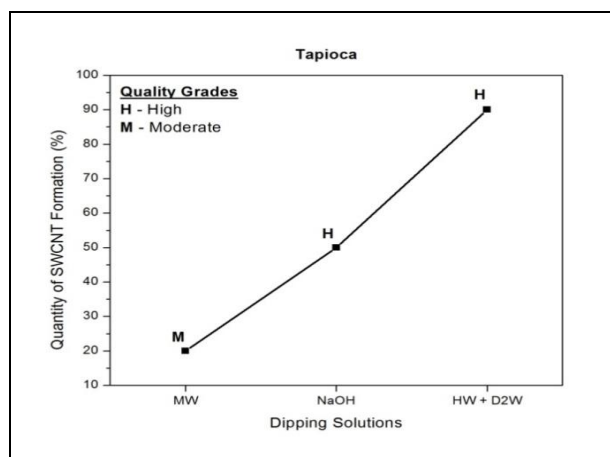


Fig. 23: Profile of Quantity of SWCNTs formation (%) with different dipping solution with an over view on Quality grades of grown SWCNTs, from Tapioca leaves.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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