Chapter 8

Summary

The work carried out, in line with the objectives of the proposed research as spelt out in the beginning (Chapter 1), are described in the Chapters, 3-7. The following section summarizes the outcome of the research chapter-wise. The Table 8.1 provides a glimpse of the work accomplished with regards to type of precursors, synthesis parameter, morphology of the nanoparticles accessed and their dimension.

An internal comparison has been drawn at the end.

Table 8.1A comprehensive list of type of precursor used and synthesis parameter, morphology and dimension of nanomaterial

Sl. No.	Precursor	CVD/Pyrolysis parameter	Morphology	Dimension
1.	Turpentine oil	CVD, Catalyst free, 700^{0}C	MWCNT	≥8µm longCNTs with outer and inner diameters 49.0 and 24.0 nm
		CVD, Fe nanoparticles, 700^{0}C	MWCNT	CNTs with outer and inner diameters 35.8 and 16.5nm
2.	Sesame oil	CVD, Catalyst free, 600^{0}C	MWCNT	≥3µm long hollow spiral nanotubes, the outer and inner diameters 54.6 and 22.94 nm
		CVD, Ni nanoparticles, 600^{0}C	CNW	CNW of diameter 60-80 nm
3.	Soybean oil	CVD, Catalyst free, 650°C	MWCNT	≥10µm long CNT, with inner and outer diameters 19.4 and 44.8nm.
		CVD, Ni nanoparticles, 650^{0}C	MWCNT	Outer and inner diameters of the nanotube 40.3 and 12.0 nm
4.	Sun flower oil	CVD, Ni nanoparticles, 650^{0}C	MWCNT	Entangled multiwalled carbon nanotubes with outer and inner diameters 33.6 and 8.6 nm
5.	Refined Mustard oil	CVD, Ni nanoparticles, 750^{0}C	MWCNT	Outer and inner diameters of the nanotubes16.4 and 6.2 nm

6.	Ghee	CVD, Catalyst free,750^{0}C	MWCNT	Intertwined bundles of CNTs with outer and inner diameters 27.45 and 13.68 nm
7.	Palmolein oil	CVD, Ni nanoparticles, 750°C	Nanowhisker / Necklace	Whiskers arranged in the form of necklace. The lattice fringes between two adjacent planes are 0.34 nm apart
		CVD, Ni nanoparticles, 650^{0}C	Nano rings	Carbon nanorings with ~65 layers of graphene sheets. Lattice fringes were 0.34 nm
8.	D-Fructose	CVD, Ni nanoparticles, 650°C	MWCNT	Outer and inner diameters of the nanotubes were 20 and 6 nm, respectively with open end.
		CVD, Ni nanoparticles, 550°C	CNW	Carbon nanowhiskerwith interlayer distance of 0.34 nm
9.	1-Butanol	CVD, Co nanoparticles, 750^{0}C	MWCNT	Entangled noodle-like densely packed MWCNT of length more than 10µm. The inner and outer diameter 4.0 nm and 17.0 nm
		CVD, Ni nanoparticles, 750^{0}C	CNW	Carbon nanowhisker, lattice fringes between the two adjacent planes are 0.33nm
10.	Vinyl alcohol	CVD, Ni nanoparticles, 750^{0}C	MWCNT	Micrometer long nanotubes, inner and outer diameters were 5.0 nm and 20.7 nm and interlayer distance is about 0.33 nm.

11.	Gulmohar (*Delonixregia)* seeds	Pyrolysis, 800^0C	Carbon nanospheres	Monodispersed carbon nanospheresof size ~ 40 nm.
12.	Bean *(Phaseolus sp.)* seeds	Pyrolysis, 800^0C	Carbonnanocubes	Quasi cubic particles in the nano dimension with coarse surface. The size of the carbon nano particles are 150nm.
13.	Castor oil (*Ricinuscommunis*) seed coat	Pyrolysis, 800^0C	Carbon nanoparticles	Agglomerated quasi-spherical nanoparticles of size in the range of 15-25 nm.
14.	Castor oil (*Ricinuscommunis*) seed	Pyrolysis, 800^0C	Carbon nanoparticles	Non-uniform porous blocks of dimension 200µm. Small particles of irregular shape and size were found to be scattered over the surface of the blocks
15.	Lai (*Brassica juncea*) seeds	Pyrolysis, 800^0C	Carbon nanoparticles	Particles are mono-dispersed with no agglomeration and irregular in shape having sizes in the range 20-30nm.
16.	Denga (*Amaranthus spinosus*)s eeds	Pyrolysis, 800^0C	Carbon nanoparticles	Quasi-spherical nano-particles having sizes in the range 20-25 nm.

17.	Sandal wood (*Santalumsp.*) seeds	Pyrolysis, 800^0C	Carbon nanoparticles	Evenly dispersed uniform nanospheres of diameter in the range 50-60 nm.
18.	Kanchan (*Bauhinia acuminata*) seeds	Pyrolysis, 800^0C	Carbon nanoparticles	Non agglomeratedquasi spherical nano particles and having sizes about 20-30nm
19.	Tishi (*Linumusitatissimun*) seeds	Pyrolysis, 800^0C	Carbon nanoparticles	Agglomerated particles of irregular shape, and their size distribution is not uniform and size around 50-150nm.
20.	Mahogany (*Swieteniamehogoni*) seed coats	Pyrolysis, 800^0C	Carbon nanoparticles	Nanoparticles are of irregular shape and size distribution is not uniform. The particle size is in the range of 70-200 nm.
21.	*Crassocephalumcrepidio ides*seed hairs	Pyrolysis, 800^0C	Carbon nanoflakes	Nanoflakes are of thickness less than ~10nm
22.	Palmyra *(Borassusflabellier*) fibres	Pyrolysis, 800^0C	Carbon nanofibres	The fibres are spider web like and the length is found to be more than 1.8 mm. The diameter of the fibres was ~70 nm.

23.	Palmyra *(Borassusflabellier*) seeds	Pyrolysis, 800^0C	Carbon nanopaticles	The particles are quasi-spherical agglomerated particle of size ~ 100nm.
24.	Blady grass *(Imperatacylindrica)* inflorescences	Pyrolysis, 800^0C	Carbon nanoflakes	The flakes are of irregular shape and the thickness was in the range 37-41 nm
25.	Purol *(Luffa cylindrica)* fibres	Pyrolysis, 800^0C	Carbon nanofibres	Spider web like morphology of the nanofibre of length more than 650µm. The diameter of the fibres was found to be ~24 nm
26.	*Pajanelialongifolia* seeds	Pyrolysis,800^0C	Carbon nanofibres (Honeycomb)	Highly porous honeycomb like fibres. The pore size is 7µm x 7µm. The pore separation of the channel is 500nm
27.	Daisy *(Tridaxprocumbens)* seed hairs	Pyrolysis, 800^0C	Carbon nanofibres	The surface of the fibre is well decorated with evenly spaced interconnected beads with micrometers dimensions
28.	*Eulalia fastigiata* inflorescence	Pyrolysis, 800^0C	Hollow carbon nanofibres	Uniform hollow fibres. The length of the fibres is in the range of 0.3- 1 millimeter. The outer and inner diameters are found to be ~ 8µm and 6µm

29.	Psyllium (*Plantago* sp.) seed husk	Pyrolysis, 800^0C	Porous Carbon nano materials	The porous carbon nanomaterials with disorganized micrometric cavities
30.	Papaya (*Carica papaya*) stems fibre	Pyrolysis, 800^0C	Carbon nanosieve	Highly porous with symmetric oval shaped pores of dimension in the range ~2-4µm. The wall thickness of the nanosieve is ~700 nm. The porous carbon framework is decorated with homo-aligned interconnected thread like extensions of length ~50µm and cross section ~2µm.
31.	Papery bracts of *Bougainvillea spectabilis*	Pyrolysis, 800^0C	Carbon nanosheets	Nanosheets are of thickness ~ 150nmand size ranging from 2-10µm.
32.	Bontula (*Bombax insigne)*	Pyrolysis, 800^0C	Carbon nanoflakes	The flakes are of irregular shape. The thickness of the flakes is ~700nm.
33.	Takpalong *(Spinaciaoleracea*) seed coats	Pyrolysis, 800^0C	Carbon nanoflakes	The carbon nanomaterials show wavy glacier patterned flakes of length more than 220µm. The flakes are arranged in layers with layer thickness around 30nm.

34.	Betel nut (*Areca catechu*) fibres	Pyrolysis, 800^0C	Carbon nanofibres	Millimeter long carbon nanofibres with unique surface texture. Fibres are having some depressions on its surface at almost uniform distance apart along its length. The diameter of the shell around 9µm containing clusters of beads of irregular shape, and size in the range of 500-700nm
35.	Blue-green alga (*Scytonemaguyanense)*	Pyrolysis, 600^0C	Carbon- silica nanocomposites	The thicknesses of the flakes are ~3 nm. Quasi-spherical five-fold multiply twinned silica nanoparticles (25-40 nm) are embedded.
36.	Green alga *(Trentepohliaaurea)*	Pyrolysis, 600^0C	Carbon- silica nanocomposites	The spherical silica nanoparticles are of diameter ~20 nm embedded in carbon nanoflakes having thicknesses of ~1.5 nm.
37.	Green alga (*spirogyraneglecta)*	Pyrolysis, 600^0C	Carbon- silica nanocomposites	The spherical silica nanoparticles are of diameter ~5nm embedded in ~4nm thick carbon nanoflakes and the nanoparticles are almost spherical.
38.	Plant Charcoal	Bunsen burner flame to red hot 2 hour	Calcium sulphate nanocomposites	Particles are spherical,nano-scaled domains forming micro-scale aggregates and the size~600 nm

39.	Waste bond paper	Calcined in muffle furnace 600^0C	Calcium carbonate nanoflakes	The thicknesses of the flakes are recorded to be ~1.5nm and are polycrystalline
40.	*Dalbergiasissoo* leaves	Calcined in muffle furnace 500^0C	Calcium carbonate nanoflakes	Highly crystalline nanobeads having dimension ~91nm and the lattice fringes are at interplanar distance of 0.38nm
41.	Gulmohar *(Delonixregia)* leaves	Calcined in muffle furnace 500^0C	Calcium carbonate nanoflakes	Nanospheres are of diameter 21 nm with interplanar distance of 0.384nm
42.	*Oroxylumindicum* seeds	Pyrolysis, 800^0C	Carbon nanofibres	Nanofibres of length more than 600µm with layer thickness ~15nm.

In chapter3, synthesis of MWCNT by chemical vapour deposition from renewable precursors has been accomplished. A total of six plant based oils (turpentine oil, sesame oil, soybean oil, sun flower oil, refined mustard oil, palmolein oil), one animal fat (ghee of cow's milk), one carbohydrate (D-fructose) and two alcohols (1- butanol, vinyl alcohol) were used as precursors for the production of CNTs and related carbon nanostructures such as nanowhiskers, nanorings etc. under catalyst free and catalytic conditions. Antioxidant efficacy and electrochemical behaviour of a few select synthesised materials were also demonstrated.

In chapter4, synthesis of carbon nanomaterials(CNMs) with various morphologies such as nanospheres, nanocubes and nanoparticles by pyrolysis in a Chemical Vapour Deposition chamber from plant seeds available in North- Eastern Region of India were performed. A total of eight plant seeds [Krishnachura or gulmohar *(Delonixregia)*, Bean *(Phaseolus*sp.*)*,Castor *(Ricinuscommunis)*, Lai (*Brassica juncea*), Denga (*Amaranthus spinosus*),Chandan or Sandalwood(*Santalumsp*.), Kanchan (*Bauhinia acuminata*), Tishi (*Linumusitatissimun*)] and two seed coats [Castor *(Ricinuscommunis)*,Mahogany (*Swieteniamehogoni*)] were used as precursors for the production of CNMs under catalyst free conditions. Antioxidant efficacy and electrochemical behaviour of a few select synthesisedCNMs were observed.

In chapter 5, synthesis of carbon nanoflakes and fibrous nanostructuresby pyrolysis in chemical vapourdeposition furnace from plant fibres were achieved. A total of fourteen plant fibres [Crassocephalum *(Crassocephalumcrepidioides)*, Palmyra *(Borassusflabellier*), Blady grass *(Imperatacylindrica)*, Luffa *(Luffa cylindrica)*, Pajanelia *(Pajanelialongifolia)*, Daisy *(Tridaxprocumbens)*,Eualia*(Eualiafastigiata)*,Psyllium (*Plantago* sp.), Papaya stem (*Carica papaya*), Honur (*Oroxylumindicum)*, Paper flower (*Bougainvillea spectabilis)*, Bontula (*Bombax insigne)*,Thunga or Takpalong *(Spinaciaoleracea*),Betel nut (*Areca catechu*)] were used as precursors for the production of carbon nanomaterials under catalyst free condition. Antioxidant efficacy, photocatalytic behaviour and electrochemical behaviour of a few select synthesised materials were also studied.

In chapter 6, synthesis of carbon-silica nanocomposites from algal biomass by pyrolysis at relatively lower temperature has been accomplished. A total of three algae (*Scytonemaguyanense,Trentepohliaaurea, Spirogyra neglecta*) were used as precursors for the production of carbon-silica nanocomposites. Antioxidant efficacy of the synthesisednanocomposites was monitored using DPPH as free radical source.

In chapter 7, synthesis of metal based nanomaterials by calcination and roasting of plant based precursors were discussed. A total of four plant based precursors (plant charcoal, waste bond paper, *sisoo* leaves, and *gulmohar* leaves) were used for the production of metal based nanoparticles. Antimicrobial efficacy, photocatalytic activity, electrochemical behaviour of the synthesised materials was investigated.

Thus a close scrutiny of research work reported spanning Chapter 3-7, reveal that the type and morphology of nanomaterial obtained vary depending on the type of precursor used. Pyrolysis of the plant based precursors at a temperature range of 500 – 800^{0}C afforded the carbon nanomaterial. Use of fibrous plant precursors, in particular, led to the formation of novel porous carbon nanomaterials that are anticipated to have potential for different applications.Process development to scale up the production of such material holds lot of future scope and prospects.

A number of carbon nanomaterial accessed as a part of the present PhD program have exhibited antioxidant activities and some of these materials showed good electrochemical behavior. Carbon nanomaterials derived from biological resources are anticipated to be non-toxic, inexpensive and biocompatible and hence are likely to be of practical relevance. The porous CNM, in particular, might be useful in catalysis, gas absorption phenomena etc. Besides carbon nanomaterial, calcium sulphate composite and calcium carbonate nanomaterial could be obtained in few cases.