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Natural Precursors for Synthesis of Carbon Nano Materials by Chemical Vapor Deposition Process: A Review

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Abstract: ***This review article deals with the Carbon nano materials derived from natural precursors using chemical vapour deposition(CVD) method and its various modified forms like pyrolysis, spray pyrolysis as well as traditional method of deposition of carbon nano materials. The range of natural precursors used including leaves, stem, oils, seeds, dried sap etc. have been discussed in relation to the type of nano material obtained. The major parameters affecting the synthesis process, namely, temperature, catalyst, carrier gas have been discussed. A correlation between applications of the carbon nano materials in relation to their structure and size have also been explored.***

Keywords: Natural precursor, Chemical vapour deposition, pyrolysis, temperature, catalyst, carrier gas, carbonnano materials

1. Introduction

Harold Krotos' Nobel prize winning discovery of carbon nano materials(CNMs)[1] and Ijima's subsequent work on graphitic carbon[2]opened a floodgate in the field of research on CNMs and their applications. There were numerous studies on syntheses, characterization and applications of CNMs especially using petroleum derived precursors. Keeping in mind the depleting nature of petroleum derived precursors Sharon et.al. [3-6] have successfully synthesized CNMs using plant derivatives. Since then a host of studies involving a whole spectrum of plant based precursors have been published which has firmly established the role of plant based precursors as an alternative raw material for deriving CNMs.

It is widely accepted that CNMs of various different morphologies are synthesized from plants parts as well as plant derivatives by the process of Chemical Vapor Deposition (CVD) under pyrolytic conditions [7-11]. Researchers have also successfully prepared carbon nanotubes (CNTs) from vegetable sources by a modified traditional process[12-14]. In all these processes, there are various parameters which have played a definitive role in the type of CNM being synthesized. This article attempts to review these parameters and also the relation between morphology of the synthesized CNMs and their applications.The parameters found to have a significant impact are being discussed, namely: (i) Type of precursor (ii)Temperature during CVD (iii) Catalyst and (iv) Carrier gas.

2. Chemical Vapor Deposition (CVD) Process For Synthesis Of Carbon Nano Material (CNM)

The set up used for CVD process is composed of single or double zone furnace (Fig. 1). Double zone furnace is used when a catalyst is used and placed at a different temperature for the synthesis of CNM. The CVD method involves thermal decomposition of the carbon containing material into carbon vapors. The carbon vapors are depositedon the catalyst, usually transition metals, leading to formation of various forms of carbon nanomaterials, including CNTs[15-26]. The reaction transformation takes place in a horizontalquartz or stainless steel furnace in inert atmosphere, the temperature of furnace is an important parameter that determines the type of carbon nanomaterial to be formed. It has been observed by Qi et.al [27] , that the reactivity inside a stainless steel furnace lead to higher yield of CNMs, whereas that in quartz furnaces lead to better selectivities [28-29]. The researchers have pointed out the effect of iron in stainless steel to be the main factor for these results. Hence, the CVD and its various modifications enable researchers to customize the deposition of the CNTs in terms of quantity, thickness, aligned growth. The CVD furnace being relatively easy to fabricate and customize has led to several modifications to aid the production and yield of CNMs.

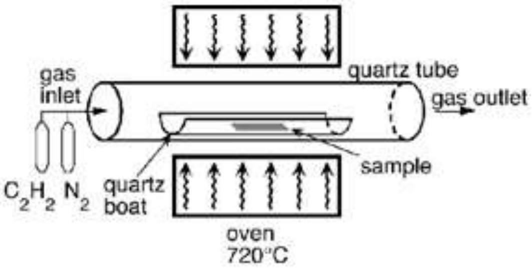


Figure 1: Schematic representation of a single zone furnace

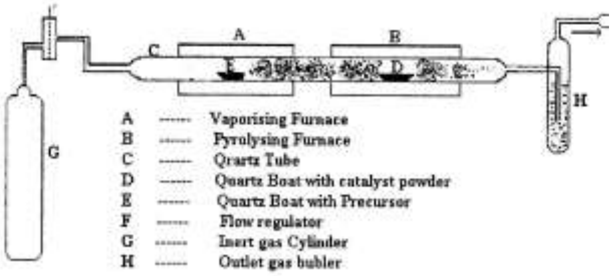


Figure 2: Schematic representation of a double zone furnace

The modified CVD method uses a clay pot and highly cleaned cotton medium to pyrolyze the precursor(vegetable oil) without excluding oxygen or ensuring an inert atmosphere.An extremely hygienic atmosphere is maintained inside the laboratory. To direct the flame and control it, an aluminum perforated cylinder is kept on top of the lamp at an approximate height of one meter. There is continuous supply of oxygen when the carbon nanomaterial is collected from the flame on a glass collector. The researchers have reported controlling the diameter of the CNM and surface density by controlling the flame temperature [12-14].

3. Type of Precursor

Almost all parts of the plants (Stem, Roots, Leaves, Seeds etc), and plant derived products (camphor, Pinene, Latex, Oil, Juice) have been used as precursors of CNMs. The choice of precursor is primarily based on wide availability as per the concept of green chemistry (use of renewable resources). The next criterion is the percentage of hydro--carboncontentsuch as oil or fatty acid, cellulose, polysaccharide, ligninetcof the chosen plant precursor.

It has been observed that oils from seeds of plants which are high in concentration of fatty acids serve as excellent precursors for the synthesis of CNMs. Paul et al have worked on a number of plant species from the north east of India[30] while there are several other studies which have used plant based oils as precursors [31-35]. Sharon's group[36-40]have worked with camphor, oil, seed, fibres and plant waste materials etc. with or without pretreatment which have served as excellent precursors. Researchers have also used sugarcane juice [41], an algae Euglena[42], cotton fibre[43] rice straw, corncob[44], Latex of Calotropis[45],bamboo(46), pretreated rice straw [47]as raw materials with promising results.The use of palm oil has yielded aligned CNT arrays [34]. Spray pyrolysis of biodiesel has yieldedwell definedmultiwalled carbon nanotubes, as reported byKarthikeyan et.al [48].The nature of precursor determines to a large extent the type of CNMs obtained. This will be discussed in detail in a later section. The anatomical structure of plant organs have also played an important role is deciding the unique morphology of CNM [49].

Table 1: Oil content of various plants that have been used as precursor for synthesis ofCNM

Sr. No.	*Precursor*	*% Oil Content*	*Used as CNM Precursor by*
1	*Cocosnucifera*	63.4	S. Paul(30)
2	*Brassica nigra*	34.6	S. Paul(30)
3	*Sesamumindicum*	49.2	S. Paul(30)
4	*Messuaferrea*	63.7	S. Paul(30)
5	*Ricinuscommunis*	45.9	S. Paul(30)
6	*Azadirachtaindica*	48.7	S. Paul(30)
7	*Gossipyumbarbadense*	19.7	S. Paul(30)
8.	*Pongamiapinnata*	29.2	S. Paul(30)
9.	*Pongamiaglabra*	31	S. Tripathi et.al (40)
10.	*Ritha/Sapindusmukorossi*	51.8	M. Sharon et.al (36)
11.	*Jatrophacuracas*	36	S.Karthikeyan et.al (48)
12.	*Helianthus annus*	22-36	H.Y. Zhang et.al (62)

4. Impact of Temperature on Different Precursors

Temperature is one of the most significant parameterswhich controls the yield and nature of CNMs. In every study it has been emphasized that yield of CNM at higher temperature is significantly higher than at lower temperatures. This is because at higher temperature carbonaceous compounds are completely cracked into carbon. Moreover, reformation of graphene like honeycomb structure of carbon arrangement into different form on the CNM also depends on the CVD temperature e.g. according to [6-7],[9],[50] at 500^{o}C to 900^{o}C yielded MWCNTs and carbon nano beads (CBD) whereas 900^{o}C to 1400^{o}Cproduced SWCNTs.

4.1 Plant parts as precursor

In the study involving pyrolysis of bamboo[46], Zhu et.alreportedMWCNT formation only in the range 1200 to 1400^{o}C, with increasing yield at higher temperature. In fact for this study the temperature range 1200 to 1400^{o}C is particularly specific as at temperatures less than or equal to 1200^{o}C, $CaSiO_3$(one of the minerals present in bamboo) cannot melt and help in dissolution of Carbon from ethanol. On the other hand, at temperatures greater than or equal to 1500^{o}C, there is no CNT formation as nature of $CaSiO_3$ changes. So in this case the temperature for obtaining better yield depends upon the melting temperature of the mineral $CaSiO_3$ which effectively acts as the catalyst.

There have been efforts to pre-treat plants prior to pyrolysis. Rajalakshmi et.al have pre-treated corncob [45] by calcining it at 500^{o}C after impregnation with concentrated H_3PO_4 They obtained porous CNMs which gave good hydrogen adsorption capacity.

Pyrolysis of hydrocarbon rich waste plant parts of maize using CVD process , at 600, 800 and 1000^{o}C temperatures was preferred [38]. High temperature ensures greater volatilization of carbon with lower quantity of ash being formed from this solid precursor.

4.2 Algae as precursor

The study onusing Euglena as precursor for CNM [42] shows the growth temperature to be between 850 to 1200^{o}C. In the temperature range 600 to 900^{o}C,diameter distribution of MWCNTs increases with increasing temperature. Lower the temperature, narrower is the diameter distribution. At higher temperature the diffusion of Carbon particles in the catalyst or vice versa is more facile. In this study, Carbon nano flakes consisting of spheres and rhombohedral morphologies were obtained at 900^{o}C with dimension ranging from 50 to 100 nm.

4.3 Plant-Oil as precursor

Oil being pure hydrocarbons are accepted as suitable precursor for synthesis of CNM. Pyrolysis of Castor oil at 900^{0}C produced CNT and CNS[39]. Spray pyrolysis of Kalonji seed oil [51] in the presence of Nickel catalyst at 800^{o}C yielded CNTs. Synthesis of aligned CNTs (ACNTs) was observed using Olive oil[52] at temperatures of 750 to

850^{o}C. Similar results were obtained using Neem oil [35] where growth of ACNTs occurred in the temperature range of 750-850^{o}C, however they usedspray pyrolysis technique. Characterization results indicated that 825^{o}C is ideal temperature under the given conditions for the growth of dense ACNTs with lengths of the order of 20-50**μ**. A change of temperature to 775^{o} or 875^{o}C shows a significant lowering in the quality and quantity of yield. While reaction at 775^{o}C does not lead to complete decomposition of neem oil into graphitic carbon synthesis at 875^{o}C leads to a lowering of yield with thicker CNTs.It is probable that the solubility of the carbon vapors in the given catalyst is highest at the particular temperature range, which dictates the yield. Also the stability of the graphitic carbon is affected at higher temperatures which narrow down the temperature range at which CNTs are obtained in scalable amounts. This factor obviously depends on the type of precursor used.

Since CVD synthesis of CNM depends on many parameters such as precursor type, carrier gas, pyrolysis temperature, catalyst; each having 3-4 levels; Sharon's group [53] optimized the parameters by applying "Taguchi Method". The best parameter regarding the yield of carbon, varied for each type of precursor oil. From the Taguchi Optimization method, opting for "larger the better", the optimum temperatures for neem oil was found to be 500^{o}C, castor oil 700^{o}C and Karanja oil 900^{o}C. But the researchers found CNMs at all the observedtemperatures though the quality of CNMs varied. For neem oil lower temperatures, 500^{o} and 700^{o}C yielded bead like structures which elongated to tubular structures at higher temperatures, 900^{o}C. Karanja oil yielded tubular structures at all temperatures, while castor oil gave tubes only with a particular catalyst at 900^{o}C.

The most dramatic effect of temperature on the quantity and quality of CNMs is given by the study of Karthikeyan et al[32] who have synthesized CNMs using pine oil, methyl ester of *Jatrophacuracas* oil and methyl ester of *Pongamiapinnata* oil at comparatively lower temperatures of 550^{o}, 650^{o} and 750 ^{o}C. The study shows a relatively low yield of CNTs at lower temperature of 550^{o}C while at 650^{o}C, the formation of well defined MWCNTs is high as the decomposition of vapors into carbon over catalyst is more effective. Conversely the studies show a rise in yield of amorphous carbon on raising the temperature to 750^{o}C. Hence the intermediate temperature of 650^{o}C is found to be the optimum for producing graphitic carbon.

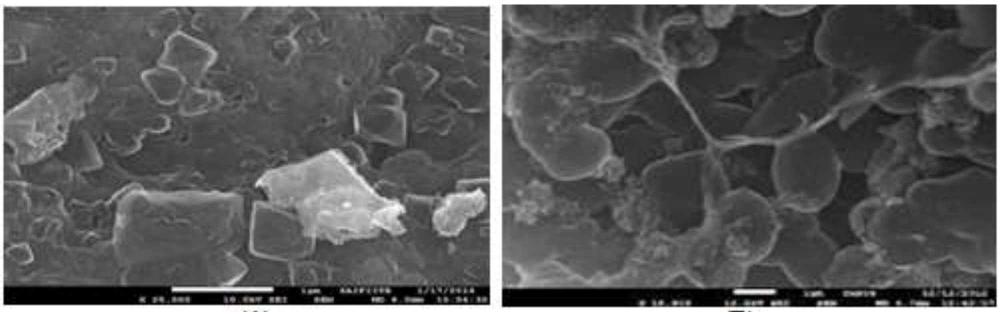
Another study on *Madhucalongifolia* oil [54] shows the optimum temperature for CNT production to be 650^{o}C while at 550^{o} and 750^{o}C a marked production of amorphous carbon was reported.

Palm oil and waste palm oil [31], [34] could produce vertically aligned CNTs at 750^{o}C. Here the flow rate of oils was also found to have an impact on the nature of CNMs formed, with a slow flow rate the role of temperature is clearly very important.

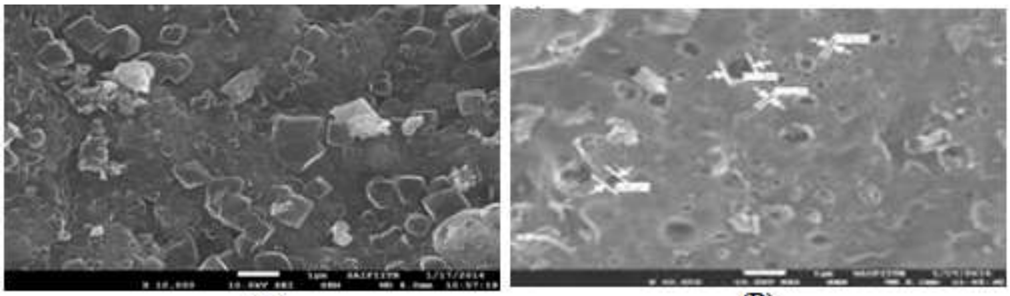
4.4 Plant product as precursor

When sugarcane juice and syrup[41]mixed with an Anodic Aluminum Oxide template were pyrolyzed at 500^{o}, 555^{o} and 600^{o}C in an inert atmosphere of nitrogen it lead to the formation of MWCNTs. The study shows increasing external diameter with increase in temperature which is as exhibited in the earlier studies[55] [56]. Though the results with sugarcane juice and sugarcane syrup are distinctly different. Higher concentration of syrup gave better yields of CNTs at higher temperatures. The external diameters of CNTs obtained with the syrup are distinctly greater than that fromthe juice. Also it has been observed that there is a limiting temperature as far as CNT production is concerned with no CNTs being synthesized with juice at 600^{o}C.

Our study conducted on usingdried latex of Calotropis as a precursor [45,57],which is a rich source of hydrocarbon, involved pyrolysis of the dried latex at 800, 900 and 1000^{o}C. This precursor yielded unique graphitic square plate like structures of 110-250nm diameter at 800^{o}C. At higher temperature of 900^{o}C a mixture of carbon nanobeads (CNB) and CNTs were obtained.While at 1000^{o}C, porous sheet like structures were obtained which were very different from theCNMs obtained at lower temperatures. Hence, effect of temperatures on growth, yield and morphology of graphitic carbon is corroborated (Fig. 3).



(A) (B)



(C) (D)

Figure 3: Showing impact of temperature on the morphology of CNM obtained frompyrolysis of Calotropis latex using Nickel as catalyst; (A) at 800^{0}C graphitic square plate like structures (B) at 900^{o}C a mixture of carbon nano beads (CNB) and CNTs and (C) at 1000^{o}C porous sheets of carbon

Table 2: Optimum temperature for synthesis and yield of CNM from various plants and plant derivatives that have been used as precursor for synthesis of CNM

Precursor	*Source Plant*	*Optimum Temperature (^{o}C) for CNM Production*
Plant Parts	Bamboo	1200-1500
	Grass	250
	Maize	600,800,1000
	Bagasses,	800
	Soyabean,	800
	Cashew nut	800
	Ritha seed	800
	Euglena	900
Plant Derivatives	Calotropis Latex	900
	Sugarcane juice and syrup	500
	Sugar syrup	550
Plant Oil	Neem oil	825
	Madhucalongifolia Oil	650
	Olive oil	750-850
	methyl ester of JatrophaCuracas oil	650
	Pine oil	650
	Castor oil	900
	Kalonji seed oil	800
	methyl ester of Pongamiapinnata oil	650
	Palm oil	750
	Waste palm oil	750
	Karanja oil	900

5. Catalyst

Transition metals serve as excellent catalysts for synthesis of CNMs as they enable catalytic decomposition of carbon source, form metallic carbides easily and enable diffusion or dissolution of carbon through bulk or through the surface of the metal leading to precipitation of carbon nanomaterial. The phase diagram of carbon and the transition metals, namely Iron, Cobalt and Nickel indicate solubility. The growth of the CNM starts at the catalytic surface hence it is imperative that the size of the catalyst should be in the nanometer scale range , i.e. 1-100nm [58]. Koziol et.al. [8] have proposed two mechanisms for catalyst fuelled growth ofCNM by CVD method. (i) The Tip growth method, it is stated that after decomposition of the carbon species, the carbon dissolves in the catalyst and diffuses through it and finally precipitates at the end to form carbon nanotubes. The catalyst is predicted to be always present at the top of the growing nanotube. (ii) The Base growth model involving bottom carbon diffusion through catalytic particle, the catalyst particle is proposed to be present at the base of the growing nanotube.

The dissolution of the carbon at one part of the catalyst and subsequent precipitation at another part is aided by a temperature gradient which might develop due to the exothermic nature of the decomposition of hydrocarbons.The carbon diffusion parameter depends on the dimensions of the catalyst particles, nature of metal catalyst, temperature and hydrocarbons and gases involved in the process. It has also been proposed that when the substrate –catalyst interaction is strong, CNT grows up with the catalyst particle rooted at its base. Conversely when the substrate –catalyst interaction is weak, the catalyst particle is lifted up by thegrowing nanotube. The driving force for carbon diffusion and subsequent CNM formation is the difference in solubility of carbon at the gas-catalyst interface and the catalyst-CNMinterface, determined by the affinity for carbon formation in the gas phase and the thermodynamic properties of the CNM respectively[26].

Though transition metals and their alloys like Co, Ni, Fe, Ti, Zn, Mg, Mnact as excellent catalysts for CNM synthesis some other catalysts like Zeolites,ferrocene etc. have also been used with good results. Most studies involving petroleum based precursors report the effectivity of catalysts in the order Fe<Co<Niwith Ni affording better aligned and more dense MWCNTs[59] with ethanol as the precursor.In an extensive review Qi et.al. [27] highlighted the effect of not only catalyst but their method of preparation, presence of catalyst support and catalyst promoters.

5.1 Synthesis of CNM without using any external Catalyst

Sometimes when the precursor contains certain minerals which aid in the formation of CNMs the presence of any external catalyst is not required. These minerals aid in the growth of the nanoforms by adjusting pore size and modifying the pyrolytic process. This is especially evident in the study conducted on bamboo charcoal[60] for synthesizing CNTs without anymetal catalyst [46]. These interesting studies involved minerals present in agricultural waste(palm kernel shell, coconut and wheat straw) and bamboo charcoal as catalysts, which eliminate the need for an externalcatalytic reagent. Chen et al.[61] synthesized carbon nanofibers (CNFs) using activated carbon produced from agricultural waste by chemical vapor deposition. They reported that the activated carbons containing iron can be used directly to synthesize CNFs. This eliminates the need for the preparation of iron catalyst.

In their work on synthesizing CNTs from sunflower seed hulls and sago, Zhang et.al [62] reported pyrolysing the precursors in a quartz tube in the absence of any external catalyst. From the well defined CNTs obtained from both the precursors it is evident that minerals inherently present in their composition aid in the formation of the CNTs.

In an interesting study Zhao et.al. [63] conducted a study using black Jews ear fungus and black sesame seeds as catalyst precursor for growing arrays of CNTs. The seeds contain iron,among other elements, as metal complex which can act as catalyst. Since the amount of iron is uniform in each cell of the seeds, the catalyst active sites arealso equally distributed all over the surface of the seeds and promotes growth of aligned CNTs. The diameter of CNTs are proportional to the iron content in the sesame seeds.

Zhu et.al have emphasized the catalytic role of minerals on the synthesis of SiC nanowires in bamboo [46]. The large availability of bamboo plants renders them attractive biomass resources for activated carbon. The ICP-AES results for minerals present in bamboo charcoal pyrolyzed at 1000^{o}C indicate the presence of the following elements; K, Mg, Ca, Si, O, Fe, S, P. The EDS(energy dispersive spectroscopy) of bamboo charcoal at high temperatures show spheres containing Ca, Si, O which have also been shown to be of non uniform distribution.Studies indicate the carbon solubility in molten (CaO-SiO-Al_2O_3) increased with increasing CaO-SiO content. The temperature dependent nature of the silicate catalyzed reaction shows better CNT production at higher temperatures(1200-1400^{o}C) but lower than 1500^{o}C. The study on synthesizing CNM from sugarcane juice and concentrated sugar syrup[41], involvespyrolysis of these liquids without catalyst. But elemental analysis of sugarcane juice reveals the presence of K, Na, Mg, P, Ca, Cl, Fe, which may catalyze the pyrolysis. Sharon' group [36] in their effort to synthesize CNM having high surface area so that it can adsorb more hydrogen; synthesized CNM from plant parts such as bagass, soybean, cashew-nut and ritha seed by pyrolysis without using any catalyst. Hence from this and previous studies it can be proposed that in case of certain categories of biomass namely bamboo, wheat grass, bagasse, oil seeds etc as the precursor, the inherent presence of minerals helps to catalyze their transformation into graphitic carbon in the absence of external catalysts. The solubility of carbon is high in these transition metals which is evident from their phase diagrams.

5.2 Transition Metal Nickel, Cobalt, Iron as Catalyst for synthesis of CNM

In the study on synthesis of CNT from cotton fibres[43] by pyrolysis, the precursor was pretreated with 0.1M Nickel nitrate and ferric nitrate for 3hours at room temperature in order to enhance the surface area so as to increase its hydrogen adsorption capacity.

During the study on Calotropis sap as precursor for CNM [45,57] catalyst powder was sprinkled over the dried sap in order to enable efficient pyrolysis catalyzed by the metal nano particles(Fe, Co, Ni).The CNMs obtained were CNB and CNTs. The metal catalysts used were Ni, Co and Fe. The SEM and TEM images indicatedwell formed CNB and CNTs were obtained when Nickel was used as catalyst. The results of this study reconfirm the conclusions offered by Xiaosi Qi et.al [27].A study was also conducted to synthesize CNMs from sap of Calotropis without using anycatalysts and it produced graphitic sheets with perforations.

In another studies involving parts of maize plant (hair, calyx, stem), Nickel has been used as catalyst[38]. The activity of Ni as catalyst was found to be superior to Co and Fe. Ni was been found to be a more potent catalyst whenCalotropis sap was used as precursor and the CNM produced were found to be graphitized to a greater extent than Co and Fe catalyzed ones.

In the study on Pine oil, Methyl ester of *Jatrophacurcas*oil and Methyl ester of *Pongamiapinnata*oil for synthesis of CNM by spray pyrolysis [32] method, silica supported Fe, Co and Mo catalyst were used at different reaction temperatures. Whereas for synthesis of CNM from oils [30]of coconut, black mustard, nagakesara, castor, neemiron oxide was used as catalyst, leading to formation of MWCNTs. The iron oxide nanoparticles were prepared by treating ferric nitrate with urea in the presence of oleic acid as the surfactant in a muffle furnace at 650^{o}C temperature.

5.3 Ferrocene as Catalyst for synthesis of CNM

Ferrocene is found to be a suitable catalyst when oil is used as precursor. Aligned CNTs were prepared from turpentine oil by mixing it with ferrocene and spraypyrolyzing[33] at 700^{o}C. The ferrocene is believed to dissociate into iron which catalyses the deposition of CNTs.The coalescence kinetics involving Fe and C containing ferrocene helps in the alignment of the CNTs. Two substrates, quartz and silica have been used by the researchers for growth of the CNTs.

For synthesizing CNM from waste and virgin palm oil also ferrocene was used in the floating CVD set up [34,31]. The authors have suggested a mechanism for formation of vertically ACNTs. According to them the ferrocene catalyst undergoes decomposition to iron at 450^{o}C and mixes intimately with the palm oil which also disintegrates to a mixture of hydrocarbon molecules which settle on the silicon substrate. At 750^{o}C, the catalytic decomposition of the hydrocarbons takes place leading to dissolution of the carbon in the iron with elimination of oxygen and hydrogen as vapor.Literature survey shows a profusion of studies conducted on both edible and non edible oils. A tabular format of the same with the correspondingprecursors obtained is given in table 3.

Table 3: Catalyst used for different precursors and type of CNM formed

Precursor (Oil)	*Catalyst used*	*Type of CNM obtained*
Coconut, Mustard,Gingelly	Nil	CNB & CNS
Castor oil	Ni	CNS
	Co	Coiled CNTs & CNS
Kalonji seed	Ni	CNT
Olive oil	Ferrocene	CNT
Turpentine oil	Ferrocene	Aligned CNTs
Virgin pail oil and Waste palm oil	Ferrocene	Vertically aligned CNTs
Pine oil, Methyl ester of *Jatrophacurcas* oil and Methyl ester of *Pongamiapinnata*oil	Si supported Fe, Co &Mo	MWCNT
Coconut, black mustard, nagakesara, castor, and neem	Iron oxide	MWCNT

6. Carrier Gas

A survey of the published data on plant precursors shows the most common carrier gases used during CVD method and pyrolysis are Argon, Nitrogen and Hydrogen. The most important function of a carrier gas is to remove all traces of oxygen, which might oxidize the CNM produced and to provide an inert atmosphere for the production of CNMs. Providing a clean atmosphere is predominant as carbon nanomaterials have been synthesized using oxides and ferrocene as catalyst amongst others[30,34,64]. According to the observations of Khorrami et.al [56] who conducted a study on impact of carrier gas flow rates on growth of CNTs over copper catalyst, a higher gas flow rate showed a decrease in the growth of CNTs from ethanol precursor. This study involved the deposition of a copper nano-layer on mirror polished Si wafer by sputtering technique. The results led to the conclusion that equilibrium is established between the flowing gas molecules and the adsorbed gas molecules on the catalyst nano-layer. With increase in carrier gas flow rates most of the catalyst sites are occupied by the carrier gas molecules, hence unavailable for the precursor molecules. This leads to a drop in production of CNTs at higher carrier gas flow rates. Also it has been observed that a better yield is obtained with Hydrogen gas since it helps in the reduction of hydrocarbons present in the precursor [38, 45, 57].

Table 4: Various carrier gases used for different precursors

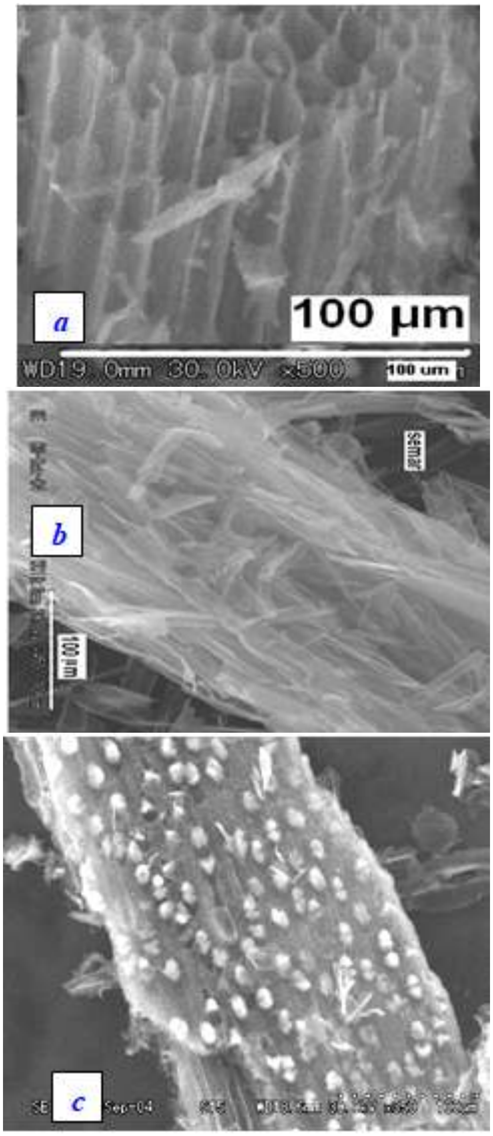
Plant precursor	*Carrier gas used*
Cotton fibre	Ar
Euglena	Ar
Parts of maize plant	Ar,H, N
Coconut	Ar, H, N
Black mustard,	Ar, H, N
Nagakesara,	Ar, H, N
Castor	Ar, H, N
Neem	Ar, H, N
Kalonji seed oil	H
Castor oil	Ar
Olive oil	N
Pine oil	N
Turpentine oil	N
Methyl ester of *Jatrophacurcas*oil	N
Methyl ester of *Pongamiapinnata*oil	N
Sap of Calotropis	Ar, H
Sunflower seed hulls, sago	N

7. Applicability of CNM Synthesized from Plant Precursors

The properties and applications of the CNMs synthesized from plant precursors are varied and there appears to be a strong correlation between the morphology of the obtained CNM and its application.

7.1 Plant derived CNM for Hydrogen storage

Carbon fibres with channel type structures(Fig. 4) were found to have high *hydrogen storage* capacity. It has been reported by several researchers that carbon materials possessing lesser density, larger surface area, more graphitic nature and having pore size ~ 10**μ**favored hydrogen adsorption. Carbon fibres synthesized from bagasse [36] (Fig. 4a) of channel type CNM and cotton fibres (Fig. 4b)[43]were found to have channel like structures with graphitic nature, hence suitable for H_2 adsorption. Carbon fibres from bagasse showed a H_2 adsorption capacity of 2.05wt% at 1kg/m^2 pressureand that from cotton fibres at 7.32wt%. Broken CNTs obtained from Kalonji seed oil have optimal hydrogen storage capacity at 0.8 wt. % at 11 Kg/cm^3 and 2.5wt% at 100 Kg/cm^3[51].



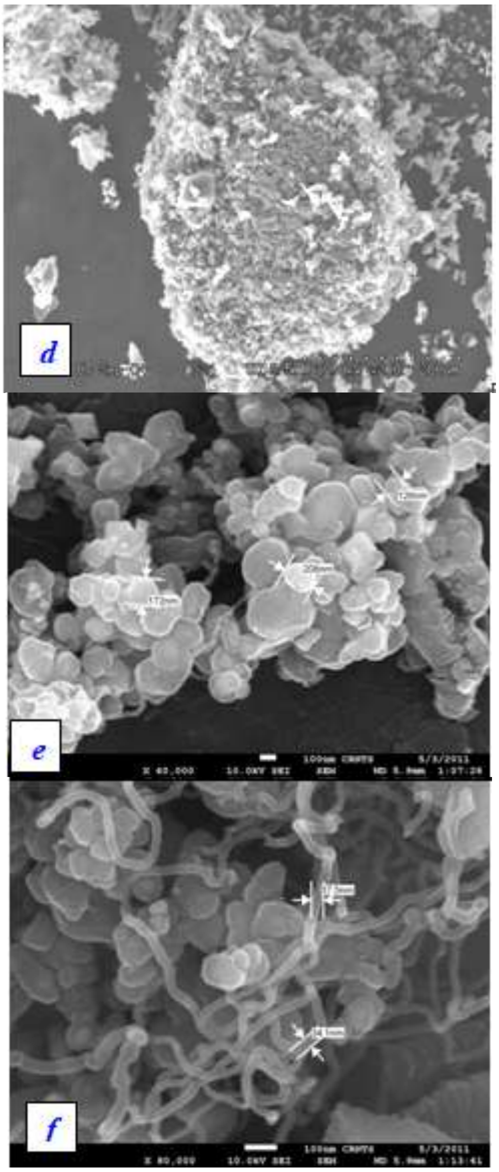


Figure 4: Morphology of CNM derived from various plant precursors

a) Channel type CNM from bagasse
b) Channel type CNM from semur cotton; that were used for hydrogen adsorption studies
c) CNMs from rice straw show channel type pitted structure and
d) CNM from Jackfruit deeds showing porous block structure both were used for supercapacitor
e) Spherical CNM from Castor oil using Ni as catalyst and
f) CNT from castor oil using Co as catalyst used for arsenic absorption [Source: Sharon's group]

7.2 Plant derived CNM for Supercapacitor

Another important application of CNMs is development of supercapacitor. Among the CNM obtained from seeds of various plants, it was found in the study by Khairnar et.al [37], that jackfruit seeds gave the highest capacitance of 92F/g. It has been observed that though the CNM from rice straw has a higher surface area(140.15 m^2/g) than those obtained from jack fruit seeds(114.02 m^2/g), the capacitance obtained from the CNM of rice straw was lower (83F/g) than that of CNM from jack fruit seed. This apparent anomaly has been resolved by analyzing the SEM micrographs of both the CNMs which shows that CNMs from rice straw show channel type pitted structure (Fig. 4c) while that from jackfruit seeds show porous but block type structure(Fig. 4d). The cotton fibre like structure of the CNM from jackfruit helps in forming the electrical double layer which is not so facile in the CNMs from rice straw.

This study established that for getting higher capacitance, the carbon materials in addition to large surface area should have a large number of pores in a plane parallel to the plane of the electrode for which the surface of CNM should be fluffy like a cotton ball.

The above facts have been also supported by the study of Zhang et.al [62] wherein the porous, curved and hollow CNTs derived from sunflower seed hulls and sago have been found to have a radii of 30nm and 20nm respectively. Their application as capacitors have shown promising results as evident from cyclic voltammogram and related studies. The large surface area of the CNTs as well as porous nature play an important role in directing the scope of applications.

7.3 Plant derived CNM for Removal of Arsenic

Sharon et.al. [39] have proposed *Arsenic removal*as another application of CNMs. Spherical CNMs obtained from castor oil (Fig. 4e) using Nickel as catalyst have been reported to adsorb 43% Arsenic, whereas CNTs from castor oil using cobalt (Fig 4f) as catalyst led to 49% Arsenic adsorption. This difference in adsorption was attributed by the researchers to presence of dangling bonds on the surface of CNTs.It was felt that CNTs having dangling bonds on the surface can uptake more arsenic.

7.4 Plant derived CNM for Solar Cell

CNTs obtained from olive oil were adhered to each other forming assemblies. It was found that the efficiency of solar cells with these CNTs as catalyst was markedly greater than solar cells using platinum as catalyst [52].

Vertically ACNTs (Fig. 5) from waste palm oil [34] were proposed to be suitable for applications in flat panel displace and flat lamps from their measured field electron emission properties from cathodes of VACNT synthesized using waste palm oil yield field enhancement, ¢, of 2740, turn on electric field of 2.25 V/¯m, threshold electric field of 3.00 V/¯m and maximum current densities of a few mA/cm^2.

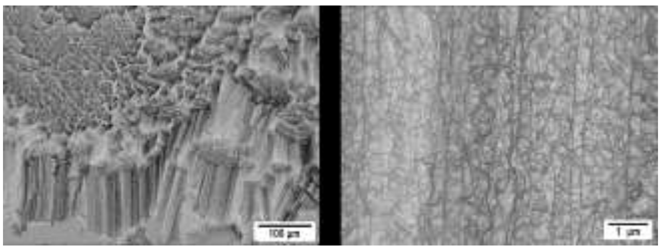


Figure 5: Vertically aligned CNTs from waste palm oil (Source Sharma et al 2012)

7.5 Plant derived CNM to be used in coatings

The study on CNM from burnt grass (*Desmostachyabipinnata*) provides a platform for the development of new environment friendly coatings[65]. It is found that a miniscule incorporation of the ash improves the overall property of the composite manifold which is a positive aspect towards generating green technology.

Study of properties and applications of various CNMs synthesized from plant precursor is still in progress. Hence it may be concluded that CNM synthesized from plant precursors has very high potential for significant applications in various fields. More research in this field will be in keeping with the principles of green chemistry.

8. Conclusion

Natural precursors have become a well researched alternative to the synthesis of CNMs apart from petroleum hydrocarbon based precursors. In this effort theeffect of factors like temperature, catalyst is well observed. Just as the CVD process has evolved into different methods like floating CVD, spray pyrolysis assisted CVD;Temperature remains the most important parameter for Carbon nanomaterial synthesis. A wide range of temperatures have been observed which lead to formation of CNTs and other CNMs. Higher temperatures lead to better yield in terms of quality and quantity. Also solubility of carbon in the catalyst is believed to increase at higher temperatures. In some precursors like neem oil, bamboo the yield is particularly high between certain ranges of temperature tapering off at both ends.Similar conclusions are drawn from the study on Pine oil, methyl ester of *Jatrophacurcas*, methyl esters of *Pongamiapinnata*, etc by Karthikeyan et.al [32], which concludes that 650^{o}C is the optimum temperature as opposed to 550^{o} and 750^{o}C. While at lower temperatures the yield is low, at temperatures higher than the optimum, the yield of amorphous carbon increases significantly emphasizing the importance of arriving at the optimumtemperature for CNM synthesis for a particular precursor. The temperature is found to be most important in deciding the yield and quality of the CNM.

Catalyst and catalyst support systems enhance the rate of growth of CNMs. Most synthesis methods have used the nanoforms of Ni, Co and Fe with varied results. Nickel emerges in some cases as a preferred catalyst like in the study on CNMs from different parts of maize, and the study on latex of Calotropis. In other studies, particularly when oil is the precursor, ferrocene acts as a catalyst for CNM production.

The effect of carrier gas is not very conclusive yet Hydrogen seems to have a marked influence on superior CNM synthesis.

The CNMs obtained from fibrous precursors like cotton, bagasse have been found to have applicability in hydrogen storage as they possessed tube like structures as opposed to porous plate like CNMs obtained from jackfruit seeds which were used as supercapacitors. Apart from these major applications CNMs have also been used as water purifiers, solar cells and ecofriendly dyes.

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