Chemistry of Carbon Nanotubes

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Received July 12, 2005

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1. Introduction

The unidirectional growth of materials to form nanowires or nanotubes has attracted enormous interest in recent years. Within the different classes of tubes made of organic or inorganic materials and exhibiting interesting electronic, mechanical, and structural properties, carbon nanotubes (CNT) are extremely promising for applications in materials science and medicinal chemistry. The discovery of CNT has immediately followed the synthesis of fullerenes in macroscopic quantities,[1] and since then the research in this exciting field has been in continuous evolution.[2] CNT consist of graphitic sheets, which have been rolled up into a cylindrical shape. The length of CNT is in the size of micrometers with diameters up to 100 nm. CNT form bundles, which are entangled together in the solid state giving rise to a highly complex network. Depending on the arrangement of the hexagon rings along the tubular surface, CNT can be metallic or semiconducting. Because of their extraordinary properties, CNT can be considered as attractive candidates in diverse nanotechnological applications, such as fillers in polymer matrixes, molecular tanks, (bio)sensors, and many others.[3]

However, the lack of solubility and the difficult manipulation in any solvents have imposed great limitations to the use of CNT. Indeed, as-produced CNT are insoluble in all organic solvents and aqueous solutions. They can be dispersed in some solvents by sonication, but precipitation immediately occurs when this process is interrupted. On the other hand, it has been demonstrated that CNT can interact with different classes of compounds.[4−20] The formation of supramolecular complexes allows a better processing of CNT toward the fabrication of innovative nanodevices. In addition, CNT can undergo chemical reactions that make them more soluble for their integration into inorganic, organic, and biological systems.

The main approaches for the modification of these quasi one-dimensional structures can be grouped into three categories: (a) the covalent attachment of chemical groups through reactions onto the π-conjugated skeleton of CNT; (b) the noncovalent adsorption or wrapping of various functional molecules; and (c) the endohedral filling of their inner empty cavity.

As clearly visible from the high number of citations, this field is rapidly expanding. The information reported in this review on each literature citation will necessarily be limited in space. It is the aim of this review to consider the three approaches to chemical functionalization of CNT and to account for the advances that have been produced so far.

2. Covalent Approaches

2.1. Sidewall Halogenation of CNT

CNT grown by the arc-discharge or laser ablation methods have been fluorinated by elemental fluorine in the range

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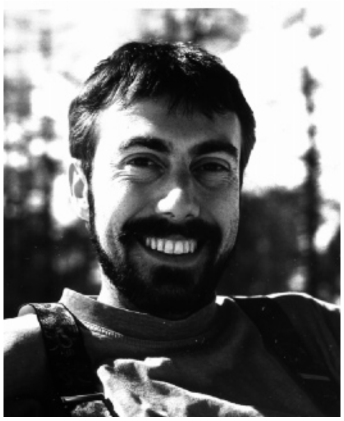
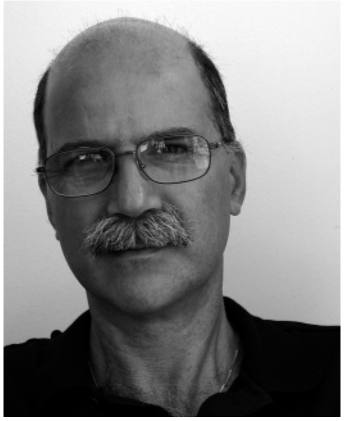
10.1021/cr050569o CCC: $59.00

Dimitrios Tasis was born in Ioannina, Greece, in 1969. He received his B.S. and Ph.D. degrees in Chemistry from the University of Ioannina in 1993 and 2001, respectively. In 2002, he moved to the laboratory of Prof. M. Prato at the University of Trieste, Italy, for two years as a postdoctoral fellow, working with carbon nanotubes and fullerenes. Since early 2004, he has been teaching in the Department of Materials Science at the University of Patras, Greece, as a lecturer (under contract). His research interests lie in the chemistry of nanostructured materials and their applications, focusing on carbon nanotubes and their polymer composites for advanced mechanical properties.

Nikos Tagmatarchis is at Theoretical and Physical Chemistry Institute (TPCI) at the National Hellenic Research Foundation (NHRF), in Athens, Greece. His research interests focus (i) on the chemistry and physics of carbon-based nanostructured materials for nanotechnological applications and (ii) on supramolecular assemblies of hybrid ensembles consisting of carbon-based nanostructured materials with organic and/or inorganic systems. He received his Ph.D. degree at the University of Crete, Greece, in 1997, in Synthetic Organic and Medicinal Chemistry with Prof. H. E. Katerinopoulos. At the end of the same year, he was introduced to fullerenes as a Marie-Curie EU TMR Fellow at Sussex University, U.K., in the solid-state chemistry group of Prof. K. Prassides, working on azafullerenes. In 1999, he moved to Nagoya University, Japan, and joined the group of Nanostructured Materials of Prof. H. Shinohara, where he investigated endohedral metallofullerenes with funds received from the Japan Society for the Promotion of Science (JSPS). From 2002 until 2004 he was in the group of Prof. M. Prato at the University of Trieste, Italy, active in the field of carbon nanotubes and nanotechnology. He is a member of the Editorial Boards of the journals *Mini Reviews in Medicinal Chemistry*, *Medicinal Chemistry*, and *Current Medicinal Chemistry*, edited by Bentham Science Publishers. In 2004 he received the European Young Investigator (EURYI) Award from the European Heads of Research Councils (EUROHORCs) and the European Science Foundation (ESF). Earlier this year he was invited by The Nobel Foundation to participate at the Alfred Nobel Symposium in Stockholm, Sweden.



Alberto Bianco received his Laurea degree in Chemistry in 1992 and his Ph.D. in 1995 from the University of Padova, under the supervision of Professor Claudio Toniolo, working on fullerene-based amino acids and peptides. As a visiting scientist, he worked at the University of Lausanne during 1992 (with Professor Manfred Mutter), at the University of Tübingen in 1996–1997 (with Professor Günther Jung, as an Alexander von Humboldt fellow), and at the University of Padova in 1997–1998 (with Professor Gianfranco Scorrano). He currently has a position as a Researcher at CNRS in Strasbourg. His research interests focus on the synthesis of pseudopeptides and their application in immunotherapy, solid-phase organic and combinatorial chemistry of heterocyclic molecules, HRMAS NMR spectroscopy, and functionalization and biological applications of fullerenes and carbon nanotubes.



Maurizio Prato studied chemistry at the University of Padova, Italy, where he was appointed Assistant Professor in 1983. He then moved to Trieste as an Associate Professor in 1992 and was promoted to Full Professor in 2000. He spent a postdoctoral year in 1986–87 at Yale University and was a Visiting Scientist in 1992–93 at the University of California, Santa Barbara. He was Professeur Invité at the Ecole Normale Supérieure, Paris, in July 2001. His research focuses on the functionalization chemistry of fullerenes and carbon nanotubes for applications in materials science and medicinal chemistry, and on the synthesis of biologically active substances. His scientific contributions have been recognized by national awards including the Federchimica Prize (1995, Association of Italian Industries), the National Prize for Research (2002, Italian Chemical Society), and an Honor Mention from the University of Trieste in 2004.

between room temperature and 600 °C (Figure 1).[21−25] Fluorinated nanotubes have been extensively characterized by transmission electron microscopy (TEM),[23] scanning tunneling microscopy (STM),[26] electron energy loss spectroscopy (EELS),[27] and X-ray photoemission spectroscopy (XPS),[28] whereas thermodynamical data were obtained using theoretical approaches.[29−32]

The structures of fluorinated CNT have been investigated both experimentally and theoretically. Controversy exists regarding the favorable pattern of F addition onto the sidewalls of CNT. On the basis of STM images and semiempirical calculations, Kelly et al.[26] proposed two possible addition patterns, consisting of 1,2-addition or 1,4-addition, and concluded that the latter is more stable. On the contrary, DFT calculations on a fluorinated tube predicted

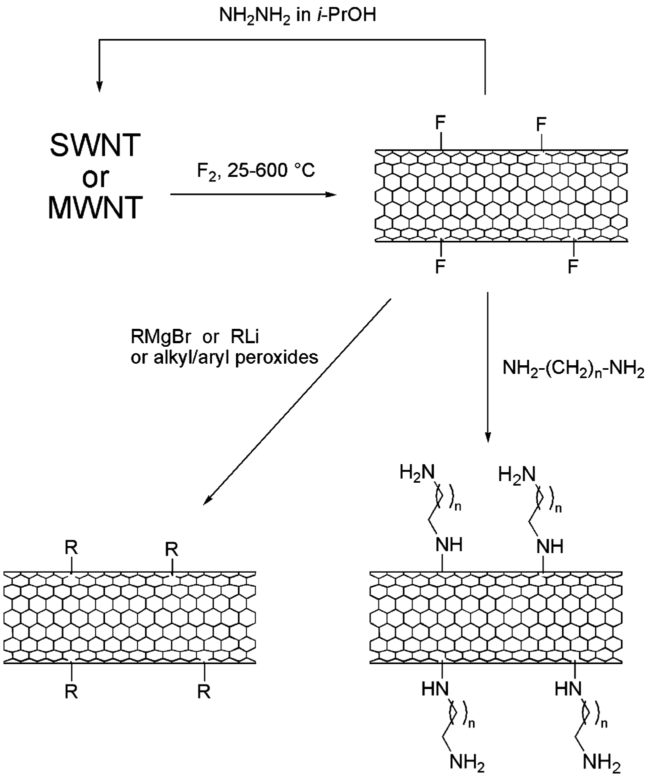


Figure 1. Reaction scheme for fluorination of nanotubes, defunctionalization, and further derivatization.

an energetic gain of 4 kcal/mol in favor of the 1,2-addition pattern.[29b] However, such a small energy difference between the two addition patterns implies that both types of fluorinated material probably coexist. The sidewall carbon atoms on which F atoms are attached are tetrahedrally coordinated and adopt sp^3 hybridization. This destroys the electronic band structure of metallic or semiconducting CNT, generating an insulating material.

The best results for the functionalization reaction have been achieved at temperatures between 150 and 400 °C,[23] as at higher temperatures the graphitic network decomposes appreciably. The highest degree of functionalization was estimated to be about C_2F by elemental analysis. However, when fluorination was applied to small diameter HipCO-SWNT (single-walled CNT), the nanotubes were cut to an average length of less than 50 nm.[33] Fluorinated nanotubes were reported to have a moderate solubility (~1 mg/mL) in alcoholic solvents.[34] The majority of the fluorine atoms could be detached using hydrazine in a 2-propanol suspension of CNT,[23,35] whereas heat annealing was used as an effective way to recover the pristine nanotubes.[36,37] In a different approach, defunctionalization of fluoronanotubes has been observed under electron beam irradiation in microscope observations.[38]

The fluorination reaction is very useful because further substitution can be accomplished.[39] It was demonstrated that alkyl groups could replace the fluorine atoms, using Grignard[40] or organolithium[41] reagents (Figure 1). The alkylated CNT are well dispersed in common organic solvents such as THF and can be completely dealkylated upon heating at 500 °C in inert atmosphere, thus recovering pristine CNT.

In addition, several diamines[42] or diols[43] were reported to react with fluoronanotubes via nucleophilic substitution reactions (Figure 1). Infrared (IR) spectroscopy allowed confirming the disappearance of the C−F bond stretching at 1225 cm^{-1} as a result of the reaction. Because of the presence of terminal amino groups, the aminoalkylated CNT are soluble in diluted acids and water. The amino-functionalized CNT were further modified, for example, by condensation with dicarboxylic acid chlorides.[42] The cross-linked nanotubes were characterized by Raman and IR spectroscopy. In additon, primary amines can be employed to further bind various biomolecules to the sidewalls of CNT for biological applications.

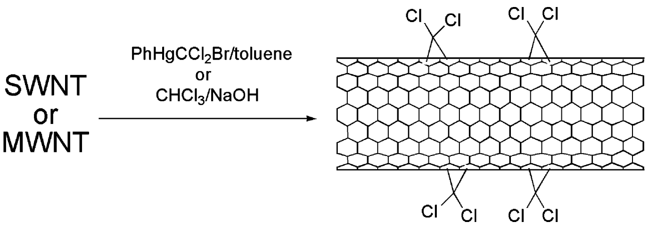


Figure 2. Cycloaddition reaction with *in situ* generated dichlorocarbene.

Using an alternative approach, the functionalization of fluoronanotubes with free radicals, thermally generated from organic peroxides, has been reported and the resulting material was characterized by FT-IR, Raman, thermogravimetric techniques, and microscopy.[44]

Chlorination or bromination reactions to CNT were achieved through electrochemical means.[45] The electrochemical oxidation of the appropriate inorganic salts afforded the coupling of halogen atoms on the graphitic network. The modified material was found to be soluble in polar solvents, whereas the carbon impurities were insoluble.

2.2. Hydrogenation

Hydrogenated CNT have been prepared by reducing pristine CNT with Li metal and methanol dissolved in liquid ammonia (Birch reduction).[46] Using thermogravimetry−mass spectrometry analysis, the hydrogenated CNT were found to have a stoichiometry of $C_{11}H$. The hydrogenated material was found to be stable up to 400 °C. TEM micrographs showed corrugation and disorder of the nanotube walls due to hydrogenation. Binding energies between carbon and hydrogen atoms were estimated with computational methods.[47] Moreover, CNT have been functionalized with atomic hydrogen using a glow discharge[48−50] or proton bombardment.[51] Supporting evidence for the covalent attachment was given by FT-IR spectroscopy.

2.3. Cycloadditions

Carbene [2+1] cycloadditions to pristine CNT were first employed by the Haddon group.[52−56] Carbene was generated *in situ* using a chloroform/sodium hydroxide mixture or a phenyl(bromodichloro methyl)mercury reagent (Figure 2).

The addition of dichlorocarbene functionality induced some changes in the XPS and far-infrared spectra, whereas chemical analysis showed the presence of chlorine in the sample. It was found that over 90% of the far-infrared intensity is removed by 16% CCl_2 functionalization. Such covalent modification exerted stronger effects on the electronic band structures of metallic SWNT.

Nucleophilic addition of carbenes has been reported by the Hirsch group.[6,57] In this case, zwitterionic 1:1 adducts were formed rather than cyclopropane systems (Figure 3, route a).

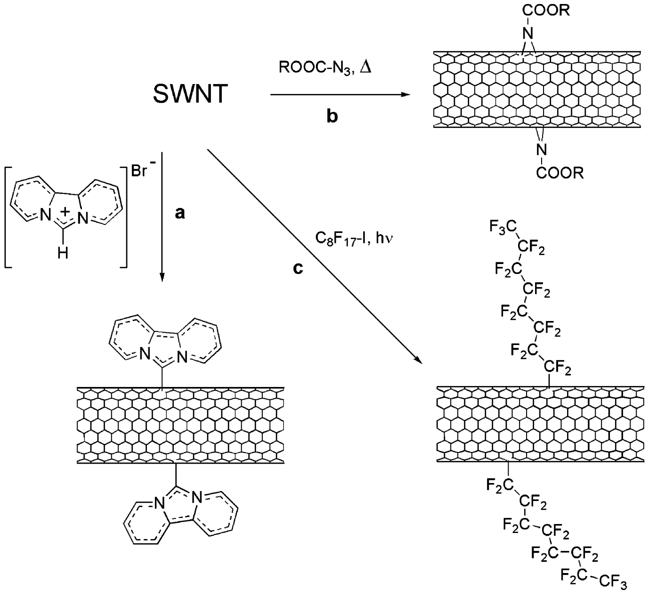


Figure 3. Derivatization reactions: (a) carbene addition; (b) functionalization by nitrenes; and (c) photoinduced addition of fluoroalkyl radicals.

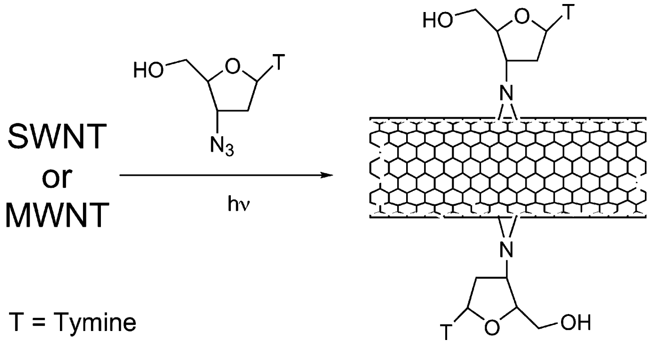


Figure 4. Photoinduced generation of reactive nitrenes in the presence of nanotubes.

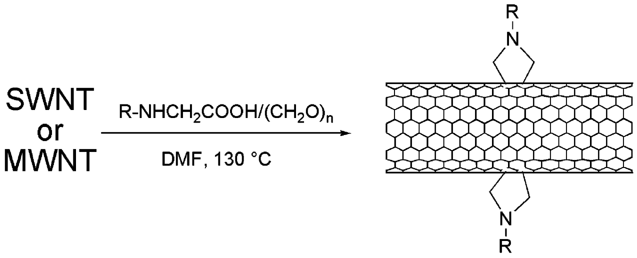


Figure 5. 1,3-Dipolar cycloaddition of azomethine ylides.

In another [2+1] cycloaddition reaction, the thermal functionalization of CNT by nitrenes was extensively studied (Figure 3, route b).[6,57−59] The first step of the synthetic protocol was the thermal decomposition of an organic azide, which gives rise to alkoxycarbonylnitrene via nitrogen elimination. The second step consisted of the [2+1] cycloaddition of the nitrene to the sidewalls of CNT, affording alkoxycarbonylaziridino-CNT. A variety of organic functional groups, such as alkyl chains, dendrimers, and crown ethers, were successfully attached onto CNT. It was found that the modified CNT containing chelating donor groups in the addends allowed complexation of metal ions, such as Cu and Cd.[58] The [2+1] cycloaddition reaction resulted in the formation of derivatized CNT, soluble in dimethyl sulfoxide or 1,2-dichlorobenzene. The final material was fully characterized by ^{1}H NMR, XPS, UV−vis, and IR spectroscopies,[58] while chemical cross-linking of CNT was demonstrated by using α,ω-bifunctional nitrenes.[59]

In a similar approach, the sidewalls and tips of CNT were functionalized using azide photochemistry.[60] The irradiation of the photoactive azidothymidine in the presence of nanotubes was found to cause the formation of very reactive nitrene groups in the proximity of the carbon lattice. In a cycloaddition reaction, these nitrene groups couple to the nanotubes and form aziridine adducts (Figure 4).

The free hydroxyl group at the 5′ position of the deoxyribose moiety in each aziridothymidine group was used as the site of modification from which DNA strands could be further attached.[60a] Theoretical studies have supported the feasibility of the reactions of CNT with carbenes (or nitrenes) from a thermodynamic point of view.[61,62]

A simple method for obtaining soluble CNT was developed by our group.[63,64] The azomethine ylides, thermally generated *in situ* by condensation of an α-amino acid and an aldeyde, were successfully added to the graphitic surface via a 1,3-dipolar cycloaddition reaction, forming pyrrolidine-fused rings (Figure 5).

In principle, any moiety could be attached to the tubular network, in an approach that has led to a wide variety of functionalized CNT. After the first report,[63] various aspects have been extensively explored including applications in the fields of medicinal chemistry, solar energy conversion, and selective recognition of chemical species. The amino-functionalized CNT were particularly suitable for the covalent immobilization of molecules or for the formation of complexes based on positive/negative charge interaction.[65] Various biomolecules have been attached on amino-CNT, such as amino acids, peptides, and nucleic acids (Figure 6).[65−70] Several applications in the field of medicinal chemistry can be envisaged, including vaccine and drug delivery, gene transfer, and immunopotentiation.

One of the central aspects in CNT chemistry and physics is their interaction with moieties via electron tranfer. Intramolecular electron-transfer interactions between nanotubes and pendant ferrocene groups showed that this composite material can be used for converting solar energy into electric current upon photoexcitation.[71] In another application, a SWNT−ferrocene nanohybrid was used as a sensor for anionic species as a result of hydrogen bond interactions.[72] The complexation of the functionalized CNT with phosphates was monitored by cyclic voltammetry. The detection of ionic pollutants is very important in the field of environmental chemistry. By an analogous approach, glucose could be detected by amperometric means.[73]

The organic functionalization of CNT with azomethine ylides can be used for the purification of raw material from metal particles and amorphous carbonaceous species.[74a] Three main steps were followed: (a) the chemical modification of the starting material, (b) the separation of the soluble adducts and reprecipitation by the use of a solvent/nonsolvent technique, and (c) the thermal removal of the functional groups followed by annealing at high temperature. The final material was found to be free of amorphous carbon whereas the catalyst content was less than 0.5%.

Water-soluble, functionalized, multiwalled carbon nanotubes (MWNT) have been length-separated and purified from amorphous material through direct flow field-flow fractionation (FlFFF). In this context, MWNT subpopulations of relatively homogeneous, different lengths have been obtained

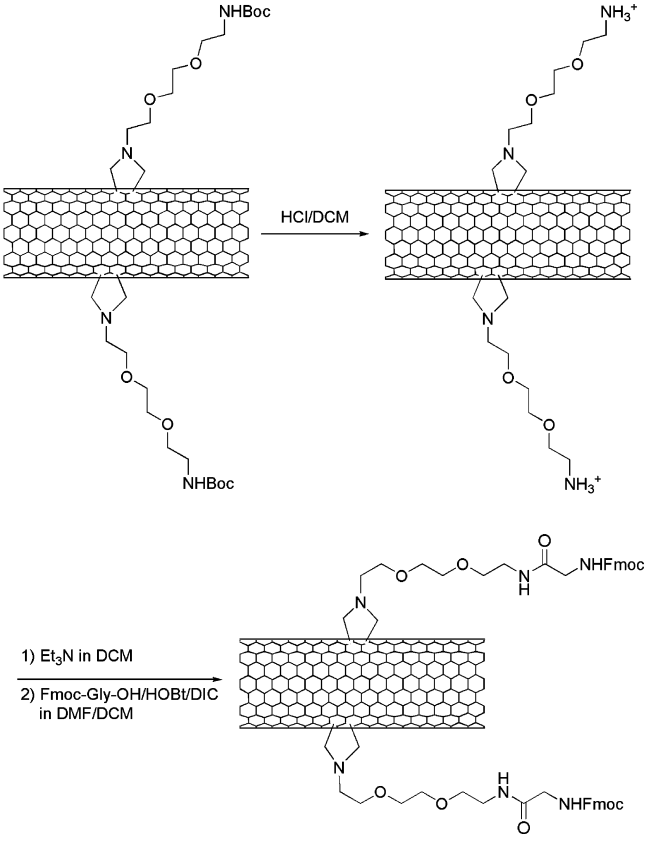


Figure 6. Reaction pathway for obtaining water-soluble ammonium-modified nanotubes. The latter can be used for the delivery of biomolecules.

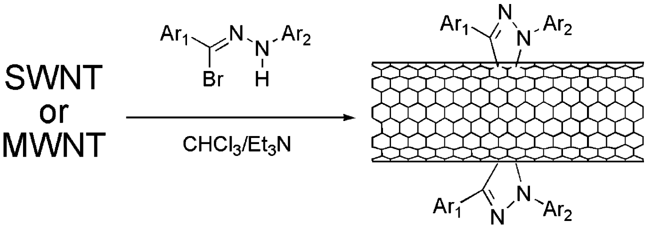


Figure 7. 1,3-Dipolar cycloaddition of nitrile imines to nanotubes.

from collecting fractions of the raw, highly polydispersed (200−5000 nm) MWNT sample.[74b] Although the resulting length-based MWNT sorting was performed on a micro-preparative scale, the isolation of purified and relatively uniform-length MWNT is of fundamental importance for further characterization and applications requiring monodisperse MWNT material.

In another approach, Alvaro et al.[75a] modified nanotubes by thermal 1,3-dipolar cycloaddition of nitrile imines, whereas the reaction under microwave conditions afforded functionalized material in 15 min (Figure 7).[75b] The pyrazoline-modified tubes were characterized by UV−vis, NMR, and FT-IR spectroscopies. Photochemical studies showed that, by photoexcitation of the modified tubes, electron transfer takes place from the substituents to the graphitic walls.[75a] The applicability of the 1,3-dipolar cycloadditions onto the sidewalls of CNT has been supported by theoretical calculations.[76]

The so-called Bingel [2+1] cyclopropanation reaction was also reported recently.[77] In this reaction, diethylbromomalonate works as a formal precursor of carbene. The [2+1] addition to CNT dispersed in 1,8-diazobicyclo[5,4,0]undecene (DBU) afforded the modified material. In a subsequent step, CNT reacted with 2-(methylthio)ethanol to give thiolated material. The functional groups on the nanotube surface could be visualized by a tagging technique using chemical binding of gold nanoparticles (Figure 8). The degree of functionalization by the Bingel reaction was estimated to be about 2%.

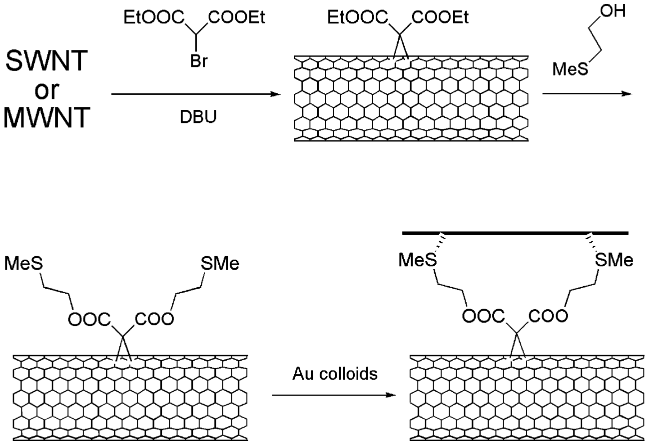


Figure 8. Bingel reaction on nanotubes and subsequent attachment to gold nanoparticles.

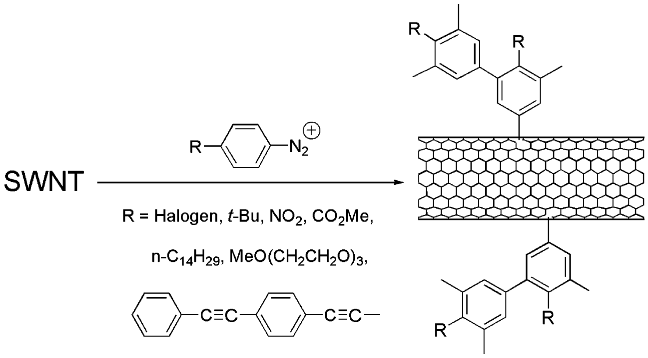


Figure 9. Derivatization scheme by reduction of aryl diazonium salts.

A Diels−Alder cycloaddition was performed on the sidewalls of CNT.[78a] The reaction involves four π-electrons of a 1,3-diene and two π-electrons of the dienophile. The active reagent was *o*-quinodimethane (generated *in situ* from 4,5-benzo-1,2-oxathiin-2-oxide), and the reaction was assisted by microwave irradiation. The modified tubes were characterized by Raman and thermogravimetric techniques. The feasibility of the Diels−Alder cycloaddition of conjugated dienes onto the sidewalls of SWNT was assessed by means of a two-layered ONIOM(B3LYP/6-31G*:AM1) molecular modeling approach.[78b] While the reaction of 1,3-butadiene with the sidewall of an armchair (5,5) nanotube was found to be disfavored, the cycloaddition of quinodimethane was predicted by observing the possible aromaticity stabilization at the corresponding transition states and products.

2.4. Radical Additions

Classical molecular dynamics simulations have been used to model the attachment of CNT by carbon radicals.[79] These simulations showed that there is great probability of reaction of radicals on the walls of CNT. A simple approach to covalent sidewall functionalization was developed via diazonium salts (Figure 9).[80−88]

Initially, derivatization of small diameter CNT (HipCO) was achieved by electrochemical reduction of substituted aryl

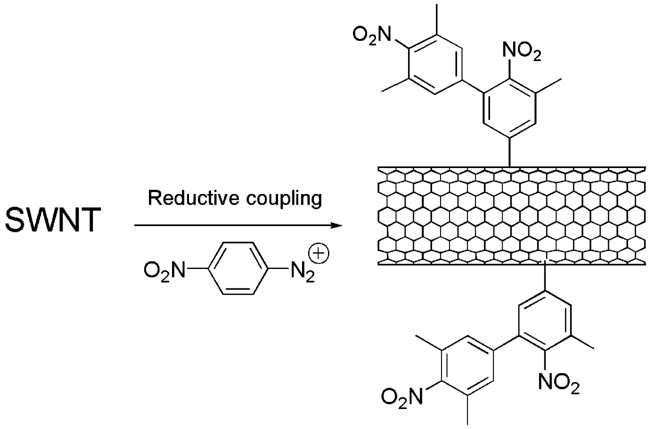


Figure 10. Electrochemical functionalization resulting in C−C bond formation.

diazonium salts in organic media,[80−82] where the reactive species was supposed to be an aryl radical. The formation of aryl radicals was triggered by electron transfer between CNT and the aryl diazonium salts, in a self-catalyzed reaction. A similar reaction was later described, utilizing water-soluble diazonium salts,[83,84] which have been shown to react selectively with metallic CNT.[83,84a] Additionally, the methodology gave the most highly functionalized material by using micelle-coated CNT. The micelles were generated using the surfactant sodium dodecyl sulfate (SDS).[84a] The micelle-coated material was made of noncovalently individually wrapped SWNT. Functionalization of this type of CNT material occurred very easily according to UV−vis spectroscopy, and the tubes were heavily functionalized according to Raman spectroscopy and TGA (one functional group every 10 carbon atoms). Analysis by AFM of the modified CNT, dispersed in DMF, showed a dramatic decrease in bundling. This profoundly increased the solubility of CNT in DMF (0.8 mg/mL).

In situ chemical generation of the diazonium salt was found to be an effective means of functionalization, providing well-dispersed nanotubes in DMF[85,86] or aqueous solutions.[87] The same reaction can also be performed under solvent-free conditions, offering the possibility of an efficient scale-up with moderate volumes.[88]

Electrochemical modification of individual CNT was demonstrated by the attachment of substituted phenyl groups.[89−91] Two types of coupling reactions were proposed, namely the reductive coupling of aryl diazonium salts (Figure 10) and the oxidative coupling of aromatic amines (Figure 11). In the former case, the reaction resulted in a C−C bond formation at the graphitic surface whereas, in the latter, amines were directly attached to CNT. Commercial fabrication of field-effect transistors (FETs) using electrochemically modified CNT was recently reported by Balasubramanian et al.[91] The authors utilized electrical means for the selective covalent modification of metallic nanotubes, resulting in exclusive electrical transport through the unmodified semiconducting tubes. To achieve this goal, the semiconducting tubes were made nonconducting by application of an appropriate gate voltage prior to the electrochemical modification. The FETs fabricated in this manner display good hole mobilities and a ratio approaching 10^6 between the currents in the on/off states.

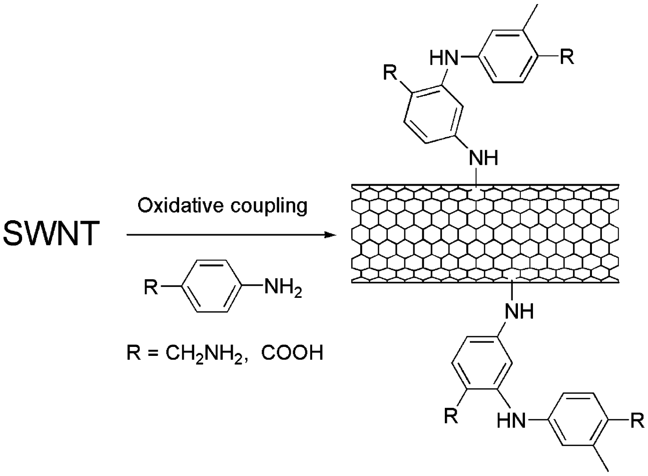


Figure 11. Electrochemical functionalization by oxidative coupling resulting in C−N bond formation.

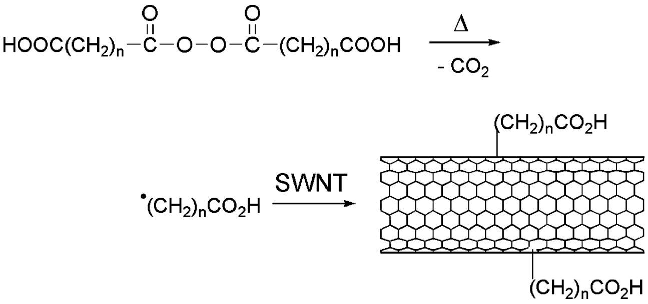


Figure 12. Derivatization reaction with carboxyalkyl radicals by a thermal process.

Electrochemically modified CNT with amino groups were shown to act as potential grafting sites for nucleic acids.[92a] Covalent attachment of DNA strands was accomplished by first immersing the nanotubes into a solution of the heterobifunctional cross-linker sulfo-succinimidyl 4-(*N*-maleimidomethyl)cyclohexan-1-carboxylate to expose the reactive maleimido groups for the selective ligation with a thiol-modified DNA. The specificity of the DNA-modified CNT was tested in the presence of a mixture of four complementary DNA molecules, each of which was labeled at the 5′-end with a different fluorescent dye. Emission spectra showed that the DNA molecules are able to recognize their appropriate complementary sequences with a high degree of selectivity. Each sequence was able to hybridize only with the complementary sequence bonded to the CNT. Similarly, Zhang et al.[92b] have electrografted poly(*N*-succinimidyl acrylate) by *in situ* polymerization onto the surface of SWNT. In a subsequent step, glucose oxidase was covalently attached to the nanotube-polymer assembly through the active ester groups of the polymer chain. The authors explored the potential application of this composite for the electrocatalytic oxidation of glucose.

Thermal and photochemical routes have also been applied to the successful covalent functionalization of CNT with radicals. Alkyl or aryl peroxides were decomposed thermally and the resulting radicals (phenyl or lauroyl) added to the graphitic network.[93,94] In an alternative approach, CNT were heated in the presence of peroxides and alkyl iodides or treated with various sulfoxides, employing Fenton's reagent.[95]

The reaction of CNT with succinic or glutaric acid acyl peroxides resulted in the addition of carboxyalkyl radicals onto the sidewalls (Figure 12).[96] This acid-functionalized material was converted to acid chlorides and then to amides with various terminal diamines.

The reductive intercalation of lithium ions onto the nanotube surface in ammonia atmosphere[97a,c] or in polar aprotic solvents[97b,c] has been studied. The negatively charged tubes were found to exchange electrons with long chain alkyliodides, resulting in the formation of transient alkyl radicals.[97a,c] The latter were added covalently to the graphitic surface, and the resulting modified nanotubes were characterized by FT-IR, Raman, and TEM.

Addition of perfluoroalkyl radicals to CNT was obtained by photoinduced reactions (Figure 3, route c).[6,57,60b,98] The precursor used in this case was an alkyl iodide which dissociated homolytically upon illumination.

In another approach, it was shown that H, N, NH, and NH_2 radicals could be added to CNT using a cold plasma method.[99] The authors used ammonia plasma generated by microwave discharge as a precursor. By using amino-functionalized multiwalled CNT as a starting material, chemical bonds were shown to form by covalent attachment of ^{13}C-enriched terephthalic acid.[100] The characterization of these modified tubes was achieved using ^{13}C NMR spectroscopy.

2.5. Electrophilic Additions

Electrophilic addition of chloroform to CNT in the presence of a Lewis acid was reported followed by alkaline hydrolysis.[101] Further esterification of the hydroxy groups to the surface of the nanotubes led to increased solubility, which allowed the complete spectroscopic characterization of the material.

2.6. Addition of Inorganic Compounds

Osmium tetroxide is among the most powerful oxidants for alkenes. The base-catalyzed [3+2] cycloaddition of the oxide with alkenes readily occurs at low temperature, forming osmate esters that can be further hydrated to generate diols.[102] In light of these features, the covalent linkage of osmium oxide to the double bonds of CNT lattices was theoretically studied.[103] The calculations predicted that the cycloaddition of osmium oxide could be viably catalyzed by organic bases, giving rise to osmylated CNT. In practice, the sidewall osmylation of CNT has been achieved by exposing the tubes to osmium tetroxide vapors under UV irradiation.[104a] The proposed mechanism for the photostimulated osmylation of CNT involved photoinduced charge transfer from nanotubes to osmium oxide and subsequently quick formation of the osmate ester adduct. The cycloaddition product can be cleaved by UV light *in vacuo* or under oxygen atmosphere whereby the original electronic properties are restored. Concerning the effect of the oxide vapor on MWNT, the tips of the tubes were opened after treatment with the inorganic reagent.[104b]

Using a solution-phase approach, Banerjee et al.[104c] suggested that the reaction is highly selective to the metallic tubes. The phenomenon of chemoselective reactions with metallic versus semiconducting CNT was confirmed by Lee and co-workers using Raman spectroscopy.[105] The authors observed the selective disintegration of metallic tubes by stirring them in a solution of nitronium (NO_2^+) salt, while semiconducting tubes remained intact.

CNT were allowed to react with *trans*-$IrCl(CO)(PPh_3)_2$ to form nanotube−metal complexes.[106a] The coordination of the inorganic species to the graphitic surface was confirmed by FT-IR and ^{31}P NMR spectroscopies. The reactivity of the SWNT sidewalls toward metal coordination was not straightforward. It was found that coordination mainly occurred at defect sites.[106b,c] The development of this chemistry was crucial for applications of SWNT as reusable catalyst supports.

Carbon nanotube interconnects were obtained by covalent attachment of an inorganic metal complex, such as [ruthenium-(4,4′-dicarboxy-2,2′-bipyridine)(2,2′-bipyridyl)$_2$](PF_6)$_2$, to CNT which were previously treated in ammonia atmosphere.[107] Cross-linking was visualized by microscopy imaging, while emission spectroscopy showed significant changes between the starting components and the resulting ruthenium−nanotube complex.

The coordination chemistry of CNT with the inorganic complex $Cr(CO)_3$ was studied by density functional theory calculations.[108,109] It was suggested that the metal fragment coordinates to the walls of the nanotube. The synthesis of the nanotube adduct had been attempted by Wilson et al.[110] However, experimental difficulties in the manipulation of nanotubes rendered impossible the characterization of the final product.

2.7. Ozonolysis

Single-walled CNT have been subjected to ozonolysis at −78 °C[111] and at room temperature,[112] affording primary CNT-ozonides. Pristine CNT were subjected to cleavage by chemical treatment with hydrogen peroxide or sodium borohydride,[111a] yielding a high proportion of carboxylic acid/ester, ketone/aldeyde, and alcohol groups on the nanotube surface. This behavior was supported by theoretical calculations.[113] By this process, the sidewalls and tips of the nanotubes were decorated with active moieties, thus substantially broadening the chemical reactivity of the carbon nanostructures. Banerjee et al.[111c] found that the chemical reactivity in this sidewall addition reaction is dependent on the diameter of the nanotubes. Smaller diameter nanotubes have greater strain energy per carbon atom due to increased curvature and higher rehybridization energy. The radial breathing modes in the low wavenumber region of the Raman spectra of CNT indicate that, after functionalization, the features corresponding to small diameter tubes were relatively decreased in intensity as compared to the profile of larger diameter tubes.

Cai et al.[114] demonstrated the attachment of ozonized nanotubes to gold surfaces by the use of appropriate chemical functionalities, namely conjugated oligo(phenyleneethynylenes). The derivatized materials were characterized by means of SEM and TEM, and spectroscopically, using Raman, UV−vis−NIR, and XPS.

2.8 Mechanochemical Functionalizations

The ball-milling of MWNT in reactive atmospheres was shown to produce short tubes containing different chemical functional groups such as amines, amide, thiols and mercaptans.[115] The solid material obtained after treatment with different gases contained functional groups in rather high quantity. The introduction of the functional groups was confirmed by IR and XPS.

In an analogous strategy, SWNT have been reacted with potassium hydroxide through a simple solid-phase milling technique.[116] The nanotube surface was covered with hydroxyl groups, and the derivative displayed an increased solubility in water (up to 3 mg/mL). Using the same approach, fullerene-C_{60} could also be attached to the graphitic network of nanotubes.[117] The featureless absorption spectrum of SWNT-C_{60} and the increased intensity of the disordered mode in the Raman spectrum were indicative of the successful functionalization of SWNT.

2.9. Plasma Activation

An alternative approach to chemical modification of CNT involving radiofrequency glow−discharge plasma activation was developed.[118] Nanotubes were treated with aldehyde-plasma, and subsequently aminodextran chains were immobilized through the formation of Schiff-base linkages. The resulting material possessed a highly hydrophilic surface due to the presence of polysaccharide-type moieties.

2.10. Nucleophilic Additions

Solvent-free amination of closed caps of MWNT with octadecylamine was attempted recently by Basiuk et al.[119a] It was suggested that the addition takes place only on five-membered rings of the graphitic network of nanotubes and that the benzene rings are inert to the direct amination. Thermogravimetric analysis revealed a high content of organic groups attached on the nanotube surface. To covalently modify CNT with both alkyl and carboxylic groups, Chen et al.[119b] treated pristine material with *sec*-BuLi and subsequently with carbon dioxide. The resulting CNT have lengths ranging between 100 and 200 nm, which can be individually dispersed in water at the concentration of 0.5 mg/mL.

Georgakilas et al.[120] studied the alkylation of single-walled nanotubes catalyzed by layered smectite minerals. The alkyl-modified tubes were found to intercalate between the clay layers, and the resulting composite was characterized by FT-IR, Raman, TGA, XRD, and microscopy techniques. In the presence of functionalized tubes, the spacing of the clay layers was increased by about 2.5 nm, indicating partial exfoliation of the inorganic component.

2.11. Grafting of Polymers

The covalent reaction of CNT with polymers is important because the long polymer chains help to dissolve the tubes into a wide range of solvents even at a low degree of functionalization. There are two main methodologies for the covalent attachment of polymeric substances to the surface of nanotubes, which are defined as “grafting to” and “grafting from” methods. The former relies on the synthesis of a polymer with a specific molecular weight followed by end group transformation. Subsequently, this polymer chain is attached to the graphitic surface of CNT. The “grafting from” method is based on the covalent immobilization of the polymer precursors on the surface of the nanotubes and subsequent propagation of the polymerization in the presence of monomeric species.

2.11.1. “Grafting to” Method

Koshio et al.[121a] reported the chemical reaction of CNT and PMMA using ultrasonication. The polymer attachment was monitored by FT-IR and TEM. As a result of this grafting, CNT were purified by filtration from carbonaceous impurities and metal particles.[121b] A nucleophilic reaction of polymeric carbanions with CNT was reported by Wu et al.[122] Organometallic reagents, like sodium hydride or butyllithium, were mixed with poly(vinylcarbazole) or poly(butadiene), and the resulting polymeric anions were grafted to the surface of nanotubes. An alternative approach was reported by the group of Blau.[123] MWNT were functionalized with *n*-butyllithium and subsequently coupled with halogenated polymers. Microscopy images showed polymer-coated tubes while the blend of the modified material and the polymer matrix exhibited enhanced properties in tensile testing experiments.

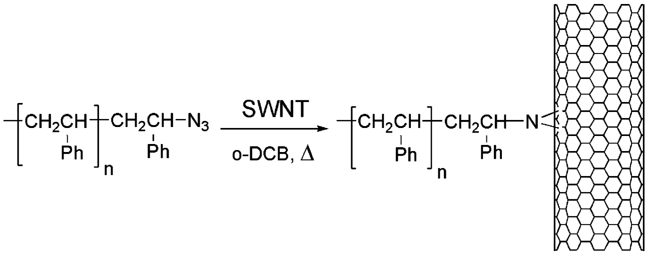


Figure 13. “Grafting to” approach for nanotube−polystyrene composites.

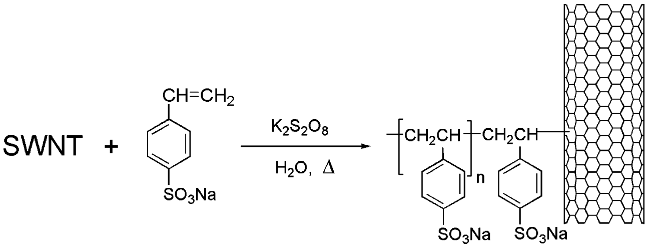


Figure 14. Grafting of a polyelectrolyte by an *in situ* process for obtaining water-soluble nanotubes.

Qin et al.[124a] reported the grafting of functionalized polystyrene to CNT via a cycloaddition reaction. An azido-polystyrene with a defined molecular weight was synthesized by atom transfer radical polymerization and then added to nanotubes (Figure 13). In a different approach, chemically modified CNT with appended double bonds were functionalized with living polystyryllithium anions via anionic polymerization.[124b] The resulting composites were soluble in common organic solvents.

Using an alternative method, polymers prepared by nitroxide-mediated free radical polymerization were used to functionalize SWNT through a radical coupling reaction of polymer-centered radicals.[125] The *in situ* generation of polymer radical species takes place via thermal loss of the nitroxide capping agent. The polymer-grafted tubes were fully characterized by UV−vis, NMR, and Raman spectroscopies.

2.11.2. “Grafting from” Method

CNT−polymer composites were first fabricated by an *in situ* radical polymerization process.[126] Following this procedure, the double bonds of the nanotube surface were opened by initiator molecules and the CNT surface played the role of grafting agent. Similar results were obtained by several research groups.[127] Depending on the type of monomer, it was possible not only to solubilize CNT but also to purify the raw material from catalyst or amorphous carbon. Qin et al.[127d] studied the grafting of polystyrene-sulfonate (PSS) by *in situ* radical polymerization (Figure 14). Through the negative charges of the polymer chain, the composite could be dispersed in aqueous media, whereas the impurities were eliminated by centrifugation.

In a subsequent work, the same authors fabricated films consisting of alternating layers of anionic PSS-grafted

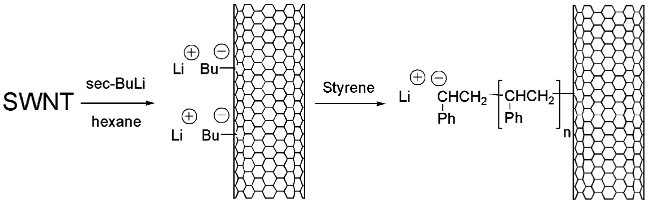


Figure 15. Grafting of polystyrene chains by anionic polymerization.

nanotubes and cationic diazopolymer.[127e] The ionic bonds in the film were converted to covalent bonds upon UV irradiation, which improved greatly the stability of the composite material. Ford and co-workers[127f] prepared polyvinylpyridine (PVP)-grafted SWNT by *in situ* polymerization. Solutions of such composites remained stable for at least 8 months. Layer by layer deposition of alternating thin films of SWNT-PVP and poly(acrylic acid) resulted in free-standing membranes, held together strongly by hydrogen bonding.

Assemblies of PSS-grafted CNT with positively charged porphyrins were prepared via electrostatic interactions.[128] The nanoassembly gave rise to photoinduced intracomplex charge separation that lives for tens of microseconds.[128a] The authors have demonstrated that the incorporation of CNT−porphyrin hybrids onto indium tin oxide (ITO) electrodes leads to solar energy conversion devices. This system displayed monochromatic photoconversion efficiencies up to 8.5%.[128b]

Viswanathan et al.[129] demonstrated the feasibility of *in situ* anionic polymerization and attachment of polystyrene chains to full-length pristine nanotubes. The raw material was treated with *sec*-butyllithium, which introduces a carbanionic species on the graphitic surface and causes exfoliation of the bundles. When a monomer was added, the nanotube carbanions initiate polymerization, resulting in covalent grafting of the polystyrene chains (Figure 15).

Xia et al.[130a] studied the fabrication of composites by *in situ* ultrasonic induced emulsion polymerization of acrylates. It was not necessary to use any initiating species, and the polymer chains were covalently attached to the nanotube surface. MWNT grafted with poly(methyl methacrylate) were synthesized by emulsion polymerization of the monomer in the presence of a radical initiator[130b] or a cross-linking agent.[130c] CNT were found to react mostly with radical-type oligomers. The modified tubes had an enhanced adhesion to the polymer matrix, as could be observed by the improved mechanical properties of the composite.[130b]

A different approach to composite preparation involves the attachment of atom transfer radical polymerization (ATRP) initiators to the graphitic network. These initiators were found to be active in the polymerization of various acrylate monomers. Adronov and co-workers[131] prepared and characterized composites of nanotubes with methyl methacrylate and *tert*-butyl acrylate. The former composites were found to be insoluble in common solvents, while the latter were soluble in a variety of organic media.

The fabrication of nanotube−polyaniline composites via *in situ* chemical polymerization of aniline was studied by many groups.[132,133] Initially, a charge-transfer interaction was suggested,[132] whereas a covalent attachment between the two components was described.[133]

The surface modification of SWNT was reported recently via *in situ* Ziegler−Natta polymerization of ethylene.[134] The exact mechanism of nanotube−polymer interaction remains unclear, although the authors suggested that a possible cross-linking could take place between the two components.

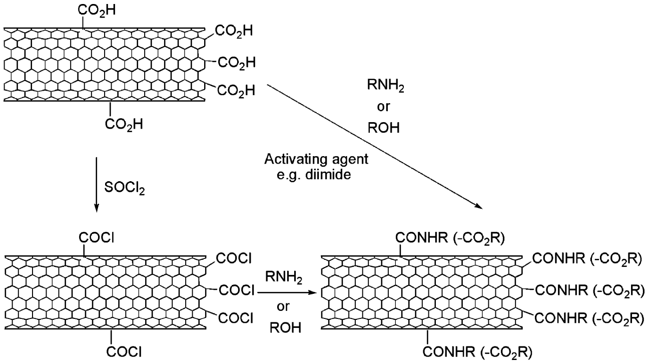


Figure 16. Derivatization reactions of acid-cut nanotubes through the defect sites of the graphitic surface.

The development of an integrated nanotube−epoxy polymer composite was reported by Zhu et al.[135] In the fabrication process, the authors used functionalized tubes with amino groups at the ends. These moieties could react easily with the epoxy groups and act as curing agents for the epoxy matrix. The cross-linked structure was most likely formed through covalent bonds between the tubes and the epoxy polymer.

Multiwalled CNT were successfully modified with polyacrylonitrile chains by applying electrochemical polymerization of the monomer.[136] The surface-functionalized tubes showed a good degree of dispersion in DMF while further proofs of debundling were obtained by TEM images.

3. Defect Site Chemistry

3.1. Amidation/Esterification Reactions

Up to now, all known production methods of CNT also generate impurities. The main byproducts are amorphous carbon and catalyst nanoparticles. The techniques applied for the purification of the raw material, such as acid oxidation,[137,138] induce the opening of the tube caps as well as the formation of holes in the sidewalls. The final products are nanotube fragments with lengths below 1 μm, whose ends and sidewalls are decorated by oxygenated functionalities, mainly carbonyl and carboxylic groups. Many groups have studied the chemical nature of these moieties through IR spectroscopy, thermogravimetry, and other techniques. In the seminal work of Liu et al.[138] it was demonstrated that the groups generated by the acid-cut nanotubes were carboxylates, which could be derivatized chemically by thiolalkylamines through amidation reaction. The resulting material could be visualized by AFM imaging after tethering gold nanoparticles to the thiol moieties. Lieber and co-workers[139] demonstrated that nanotube tips can be created by coupling basic or biomolecular probes to the carboxylic groups that are present at the open ends. These modified nanotubes were used as AFM tips to titrate acids and bases, to image patterned samples based on molecular interactions, and to measure binding forces between single protein−ligand pairs.[139c]

Chen et al.[53] treated oxidized nanotubes with long chain alkylamines via acylation and made for the first time the functionalized material soluble in organic solvents (Figure 16).

Further studies showed that 4-alkylanilines could also give soluble material,[140] whereas the presence of the long alkyl

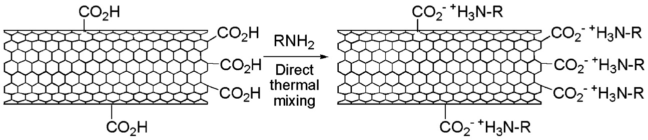


Figure 17. Direct thermal mixing of nanotubes and long chain amines.

chain played a critical role in the solubilization process. Direct thermal mixing of oxidized nanotubes and alkylamines produced functionalized material through the formation of zwitterions (Figure 17).[140,141] The length-fractionation of shortened (250 to 25 nm), zwitterion-functionalized, SWNT has been demonstrated via gel permeation chromatography.[142a] The UV−vis spectrum of each fraction indicates an apparent solubilization, as evident by the direct observation of all predicted optically allowed interband transitions. This nondestructive and highly versatile separation methodology opens up an array of possible applications for shortened SWNT in nanostructured devices.

In a subsequent work, the same group[142b] suggested that stable dispersions of SWNT with octadecylamine (ODA) in tetrahydrofuran originate not only from the first proposed zwitterion model[140,141] but also from physisorption and organization of ODA along the nanotube sidewalls. The affinity of amine groups for semiconducting SWNT,[143] as opposed to their metallic counterparts, provides a way for the selective precipitation of metallic tubes upon increasing dispersion concentration, as indicated by Raman investigations.

Esterification reactions resulted also in soluble functionalized nanotubes (Figure 16).[144] The photochemical behavior of soluble alkyl ester-modified nanotubes gave rise to measurable photocurrents after illuminating solutions of these tubes.[145] By using time-resolved spectroscopies (laser flash photolysis), the transient spectrum of the charge separated state could be detected.

Size and shape are very important issues in CNT chemistry. The change in shape from straight form to circles can have interesting implications in electronics.[146a] The condensation of carboxylate and other oxygenated functions at the ends of the oxidized SWNT allowed Shinkai and collaborators to produce perfect rings.[146b]

Sun and co-workers[147−150] attached lipophilic and hydrophilic dendrimers to oxidized CNT via amidation or esterification reactions (Figure 16). The modified material was characterized by NMR and electron microscopy. To provide evidence about the existence of ester linkages in the functionalized tubes, acid- or base-catalyzed hydrolysis was performed.[150] This resulted in the recovery of starting CNT, which were again insoluble in any solvent. Using deuterated alcohols as coreactants in esterification reactions, the same group demonstrated the attachment of deuterium to the nanotubes.[148] The attachment of fluorescent pyrene moieties to the surface of nanotubes induced some interesting photophysical properties. It was demonstrated that the planar pyrene groups interact with CNT after photoexcitation.[149,151] Photophysical experiments indicated that energy transfer is the main reason for the fluorescence quenching of pyrene groups.

Modified porphyrins were also attached at the defect sites of oxidized nanotubes for the fabrication of novel photovoltaic devices.[152a,b] Photophysical studies of the porphyrin-tethered nanotubes showed that fluorescence quenching of the dye is dependent on the length of the spacer linking the two components. It is believed that the structural arrangement between the nanotube and the porphyrins is critical for the photophysical behavior of the composites. In independent works, oxidized nanotubes with phthalocyanine moieties linked by amide bonds have been prepared.[15,152c] The resulting composites were characterized by UV−vis, IR, and TEM.

The effects on the photocurrent−voltage characteristics of solar cells were thoroughly studied by anchoring ruthenium dye-linked CNT to TiO_2 films.[152d] In comparison to the case of the unmodified TiO_2 cell, the open-circuit voltage (V_{oc}) increased by 0.1 V, possibly due to the presence of the NH groups of the ethylenediamine moieties in the TiO_2-linked nanotubes. In an analogous study, Haddon and co-workers[152e] demonstrated that photoinduced charge separation within chemically modified SWNT results in persistent conductivity of semiconducting carbon nanotube films. Carboxylated tubes reveal negative persistent photoconductivity that could be quenched by infrared illumination. The authors found that the covalent attachment of $Ru(bpy)_3^{2+}$ to SWNT makes carbon material sensitive to the light that is absorbed by $Ru(bpy)_3^{2+}$ and persistently photoconductive, thus opening opportunities for the selective light control of conductivity in semiconducting SWNT. Persistently photoconductive SWNT have potential uses as nanosized optical switches, photodetectors, electrooptical information storage devices, and chemical sensors.

The amidation or esterification of oxidized nanotubes has become one of the most popular ways of producing soluble materials either in organic solvents or in water. Gu and co-workers[153] showed that the solid-state reaction between oxidized nanotubes and taurine (2-aminoethanesulfonic acid) afforded water soluble material. Pompeo et al.[154] succeeded in solubilizing short-length nanotubes by attaching glucosamine moieties, whereas the groups of Kimizuka[155a] and Sun[155b] prepared galactose- and mannose-modified nanotubes. The grafting was obtained by producing the acyl chlorides or by carbodiimide activation, and the adducts were found to be water soluble. Carbohydrated carbon nanotubes were used to capture pathogenic *Escherichia coli* in solution.[155b]

By analogous coupling reactions, various fluorescent probes were attached at the acid-cut ends for photophysical studies,[156] whereas solid catalysts have been fabricated by grafting of organic complexes of metal ions.[157]

Following the method developed by the Haddon group, Cao et al.[158] condensed dodecylamine with the oxidized ends of tubes, while others studied the octadecylamine-modified tubes by optical spectroscopy.[159]

Kahn et al.[160] showed the possibility to modify oxidized nanotubes with an amine, bearing a crown ether. The chemical interaction between the nanotubes and the amine was suggested to be noncovalent (zwitterion formation). By using the carbodiimide approach, Feng et al.[161] prepared crown ether-modified full-length CNT.

The gas-phase derivatization procedure was employed for direct amidation of oxidized SWNT with aliphatic amines. The procedure includes treatment of the tubes with amine vapors under reduced pressure.[162]

Zhu and co-workers[163] studied the modification of MWNT by the reaction of a secondary alkylamine with the chlorinated acidic moieties of the tubes, following the Haddon approach. The adduct exhibited good optical limiting properties. The authors demonstrated that the amine-modified

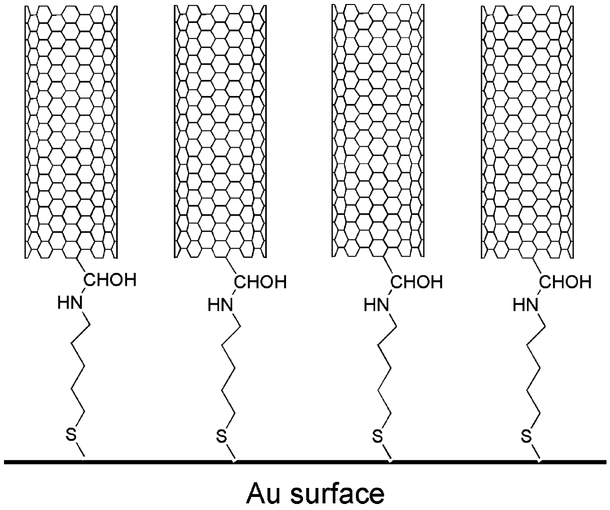


Figure 18. Controlled deposition of oxidized nanotubes onto gold surfaces by using aminothiols as chemical tethers.

soluble CNT could be linked with gold nanoparticles, by using a thiol-pyrene derivative as the cross-linker.[163d] Being a bifunctional molecule, the cross-linker can be bound to the surface of the CNT by $\pi-\pi$ stacking, while at the same time the thiol groups can react covalently with the gold nanoparticles. The nanotube−metal interaction was studied by fluorescence and Raman spectroscopies.

The groups of Castano[164] and Shaffer[165] independently described a method of silylating oxidized MWNT by reacting the carboxylic acids with the appropriate silanes.

Similar to the acylation−esterification approach, the carboxylic groups of oxidized nanotubes were converted to carboxylate salts by treatment with a base.[166] Subsequently, the carboxylates reacted with alkyl halides in the presence of a phase transfer agent to give alkyl-modified nanotubes. The solubility of the adducts was found to be a function of the chain length of the alkyl group.

Intermolecular junctions between CNT were reported by coupling oxidized material with the appropriate linkers.[167a,b] Acyl chloride-terminated nanotubes reacted with aliphatic diamines, and the resulting adduct was characterized by Raman spectroscopy. Such amino-functionalized tubes are perfect scaffolds for the covalent binding of polymers and biomolecules.[167c]

The issue of the controlled deposition and alignment of CNT on different types of surfaces has been studied extensively in the last few years. In principle, by attaching acidic moieties to the graphitic surface, one can guide the assembly on any substrate. Important progress concerning the controlled deposition of CNT on gold surfaces was achieved by the thiolization reaction of carboxyl-terminated CNT.[138,168,169] Short-length oxidized CNT were treated with the appropriate thiol derivative, and the resulting material was tethered chemically to a gold substrate (Figure 18).

Alternatively, gold substrates have been shown to interact with the appropriate tethering agents and subsequently assemble into oxidized tubes by forming amide bonds. Typically, the molecular bridges can be α,ω-aminomercaptans.[114,170,171] In a subsequent step, different macromolecules can be attached at the free ends of the oxidized CNT.

Deposition of oxidatively shortened nanotubes on a silver surface was based on spontaneous adsorption of the COOH groups onto the suface.[172] Various spectroscopies have been used to characterize the assembly, including Raman, AFM, and TEM.

The formation of organized CNT onto silicon wafers was shown to proceed through metal-assisted assembly.[173] The substrate was chemically modified using Fe^{3+}, which was subsequently transformed into its basic hydroxide form. The oxidized nanotubes bearing acidic groups were assembled onto the modified substrate by electrostatic interactions.

3.2. Attachment of Biomolecules

The integration of CNT with biological systems to form functional assemblies is a new and little explored area of research.[65a,174] CNT have been studied as potential carriers that transport and deliver various bioactive components into cells.[65] The combination of the conducting properties of CNT and the recognition properties of the biomaterials can give rise to new bioelectronic systems (e.g. biosensors). Nanotube−protein conjugates were prepared by the group of Sun[175] via diimide-activated amidation reaction. The tubes were functionalized with bovine serum albumine[175a−c] or horse spleen ferritin,[175d] and the composites were found to be soluble in aqueous media. The majority of the proteins remained active when conjugated to the nanotubes, as confirmed by microdetermination assays.[175c] Alternatively, the same proteins can be covalently bound to nitrogen-doped multiwalled nanotubes.[176]

In other cases, CNT were functionalized with poly-L-lysine, a polymer that promotes cell adhesion.[177] The biomolecule provided an environment for further derivatization. By linking peroxidase to this assembly it was found that hydrogen peroxide could be detected in relatively low concentrations.[177a]

Similarly, streptavidin was attached to nanotubes and the resulting composite was studied in biorecognition applications.[178a] The group of Dai covalently attached biotin at the carboxylic sites of oxidized nanotubes, and the resulting conjugate was incubated with streptavidin.[178b] The uptake of the nanotube−protein composite into mammalian cells was monitored by fluorescence confocal imaging and flow cytometry. It was found that streptavidin could enter inside the cells when complexed with the nanotube−biotin transporter.

Gooding et al.[171] studied the covalent immobilization of a redox protein (MP-11) at the oxidized ends of aligned CNT on a gold electrode surface. The reversible electrochemistry of the enzyme originated from the electron transfer through the bridging nanotubes. Wang et al.[179] have fabricated a nanotube−enzyme assembly for amplifying the electrical sensing of proteins and DNA. The composite could have potential applications in medical diagnostics.

Patolsky et al.[180] fabricated an array of aligned nanotubes on a gold surface. An amino derivative of flavine adenine dinucleotide cofactor was coupled at the free ends of the standing tubes. In a subsequent step, glucose oxidase was reconstituted on the cofactor units. The tubes acted as a nanoconnector that electrically puts in contact the active site of the enzyme and the gold electrode. In an analogous work, glucose oxidase was covalently immobilized on nanotubes via carbodiimide chemistry by forming amide linkages between their amine residues and carboxylic acid groups at the tips.[181] The catalytic reduction of hydrogen peroxide liberated by the enzymatic reaction of glucose oxidase leads to the selective detection of glucose. The biosensor effectively performs a selective electrochemical analysis of glucose in the presence of common interfering agents (e.g., acetaminophen, uric and ascorbic acids), avoiding the generation of overlapping signals due to the presence of the different molecules. Similar nanotube–redox protein conjugates have shown enhanced sensitivity in the detection of low concentrations of hydrogen peroxide.[182]

Following a similar method, CNT were linked covalently to DNA strands by diimide activation of the carboxylic moieties.[183–189] The adducts were found to have a moderate solubility in aqueous solution.[190] A multistep route for covalently linking DNA to oxidized nanotubes has been reported by independent works.[191] The authors attached a bifunctional linker at the defect sites of the tubes, and then a chemical reaction took place between the linker and the thiol-terminated DNA strands. The resulting composites were found to hybridize selectively with the complementary sequences of oligonucleotides.

Alternatively, the self-assembly of nanotubes to gold electrodes (or nanoparticles) via DNA hybridization was demonstrated by different research groups.[192] This approach consists of two steps. In the first step, a self-assembled monolayer of single stranded DNA was adsorbed onto gold contacts by reaction with thiol-terminated oligonucleotides. In the second step, oxidized SWNT modified with oligonucleotides of the complementary sequence were allowed to hybridize with the DNA located on the gold electrode.

3.3. Grafting of Polymers to Oxidized Nanotubes

The grafting of polycationic electrolytes to defect sites of CNT has been studied by the group of Sun,[193–197] who attached poly(ethyleneimine) chains to CNT. The free carboxylic acid functions on oxidized CNT were converted to acyl chlorides. The activated tubes were mixed with poly(propionylethyleneimine-*co*-ethyleneimine), and the polymer-bound nanotubes were isolated upon amidation reaction.[193] By microscopy studies, it was found that the polymer chains were attached mainly at the tips of the CNT. Using an alternative approach, direct heating of oxidized nanotubes in the polymer melt gave soluble functionalized material.[194] The diimide-activated amidation reaction for the functionalization was greatly enhanced by continuous sonication.[195] The functionalized material was found to possess interesting optical limiting properties.[196] Haddon, Parpura, and collaborators[197b] studied the feasibility of using nanotube–polymer composites as substrates for neuronal growth. Polyethyleneimine was attached to oxidized tubes, and the resulting composite was shown to promote neurite outgrowth and branching.

Several ways have been devised to attach polystyrenes to CNT. Oxidized single-walled and multi-walled CNT were functionalized with polystyrene copolymers under amidation or esterification reactions of the nanotube carboxylic acids.[198] Nucleophilic substitution reaction of living polystyrene lithium anions with the acyl chloride-CNT was reported recently.[199] The polymer-functionalized nanotubes were shown to remain well-dispersed in common organic solvents for several days.

Qin et al.[124a] attached ATRP initiators to the carboxylic groups of CNT and studied the grafting of styrene monomers to the graphitic network. Microscopy showed that the original nanotube bundles were exfoliated into very small ropes. Simultaneously, the ATRP grafting of polystyrene chains was studied by other groups.[200,201] Kong et al.[201b] constructed amphiphilic polymer brushes on the surface of multi-walled nanotubes. They attached polystyrene-*block*-poly(*tert*-butyl acrylate) chains by sequential ATRP of styrene and *tert*-butyl acrylate. This was followed by hydrolysis of the acrylate block, giving rise to the fabrication of a nanotube composite with a block copolymer of polystyrene–poly(acrylic acid).

Jin et al.[202a] showed for the first time grafting of poly(ethylene oxide) to CNT modified with acyl chloride moieties. The solubilization of oxidized CNT by attachment of amine-terminated poly(ethylene glycol) (PEG) chains was studied by several groups.[202b,c,203] The functionalization reaction was achieved via three different approaches: (1) direct thermal reaction of the reactants, (2) acylation–amidation, and 3) carbodiimide-activated coupling. Nonlinear transmission measurements on solutions of PEG-SWNT in chloroform showed a better optical limiting performance relative to that recorded for original SWNT suspended in the same solvent.[202c]

An *in situ* ring-opening polymerization strategy was employed to grow multihydroxyl dendritic macromolecules on the surfaces of multi-walled carbon tubes.[204a] CNT were oxidized, activated with thionyl chloride, and allowed to react with a diol, thus obtaining hydroxy-functionalized MWNT (MWNT-OH). Using MWNT-OH as a growth support and $BF_3 \cdot Et_2O$ as a catalyst, multihydroxy hyperbranched polyethers–treelike macromolecules were covalently grafted on the sidewalls and ends of nanotubes via *in situ* ring-opening polymerization of 3-ethyl-3-(hydroxymethyl)oxetane. TGA measurements showed that the weight ratio of the as-grown hyperbranched polymers on the MWNT surfaces lay in the range between 20 and 87%. The products were characterized by FTIR, NMR, DSC, TEM, and SEM. These nanocomposites exhibited relatively good dispersibility in polar solvents. Haddon and co-workers[204b] demonstrated a novel route to CNT–nylon composites through covalent grafting between the polymer chain and the acidic functions of the graphitic surface of the tubes. The authors used caprolactam as both a solvent and a monomer for the *in situ* ring-opening polymerization and grafting to the oxidized CNT. Results from IR, TGA, and AFM spectroscopies confirmed the covalent grafting of the polymer chains at the defect sites. The incorporation of 1.5 wt % CNT into the nylon matrix increases the Young's modulus almost 3 times.

By carbodiimide-activated esterification reaction, oxidized CNT were functionalized with poly(vinyl alcohol).[205] The adduct was found to be soluble in highly polar solvents.[206]

Chemically oxidized MWNT were incorporated into a polymer matrix by *in situ* polymerization of methyl methacrylate monomer.[207a] Using Raman and IR spectroscopies, it was found that a chemical interaction between the polymer chain and the carboxylic moieties of the graphitic network is established. Alternatively, PMMA chains terminated with hydroxyl groups were grafted to the acidic functions of MWNT by esterification reaction.[207b]

In a different approach, Qin et al.[208] synthesized ATRP initiators attached on the carboxylic acids of oxidized nanotubes and studied the grafting of *n*-butyl methacrylate monomer on the graphitic surface. The composites were found to be soluble in a variety of solvents. The same strategy was followed for the functionalization of MWNT with acrylate polymers by *in situ* ATRP.[209]

ATRP initiators were attached to the carboxylic groups of aligned CNT, and the grafting of an acrylamide monomer was studied.[210a] It was found that the composite wettability in aqueous media is temperature dependent.[210b,c] According to the authors, this composite might have applications for drug delivery or thermally responsive nanodevices.

Ruthenium-based olefin metathesis catalysts have been attached at the defect sites of acid-treated nanotubes.[211a] These catalyst-functionalized tubes were shown to be effective in the ring-opening metathesis polymerization of norbornene monomer. This resulted in rapid polymerization starting from the graphitic surface. The polymer-modified tubes exhibited improved solubility in organic solvents. By an analogous approach, the ring-opening polymerization of *p*-dioxanone to shortened CNT resulted in the fabrication of covalently grafted nanotube−polymer composites.[211b]

Sun and co-workers[212] studied the condensation reaction of oxidized nanotubes with a modified polyimide. The covalent attachment of the two components took place by thermal treatment after solution mixing. The electrical conductivity of the composite remained unaffected, even at very low nanotube loading. Similarly, polythiophene was attached at the COOH groups on the nanotube surface.[213] This nanocomposite showed higher conductivity than a simple mixture of the two components.

Oxidized CNT were incorporated into epoxy matrixes by simple mixing via the formation of covalent bonds in the course of epoxy ring-opening esterification.[214−216] The uniformly dispersed nanotubes enhanced the overall mechanical properties of the epoxy composites up to 30%. To achieve a much better dispersion of the nanotubes, the acid-shortened material was further fluorinated at the sidewalls before mixing with the polymer matrix.[214] Using mild reaction conditions, Zhang et al.[216] added a photoinitiator system to the nanotube−epoxy composite for cationic UV curing.

Haddon, Parpura, and collaborators[217a,b] studied the feasibility of using nanotube−polymer composites as substrates for neuronal growth. Poly(*m*-aminobenzenesulfonic acid) was attached to oxidized tubes, and this allowed control of the branching pattern of the neuronal process by manipulating the charge carried by the modified nanotubes. In a subsequent work, the same authors showed that the composite exhibits improved sensor performance for detection of ammonia.[217c] Compared to purified nanotubes, electrodes fabricated with the composite have higher variations of resistance upon exposure of the analyte vapors.

Sano et al.[218] treated CNT bearing acid chloride moieties with a polyamine starburst dendrimer of tenth generation. AFM images revealed star-shaped nanotube structures resulting from the chemical interaction of the reactants. Green and co-workers[16] introduced starburst polyamideamine (PAMAM) dendrimers to the tube surface via carbodiimide coupling. Dendrimers are of particular interest since they hold promise for drug delivery or slow release of therapeutic molecules.

4. Noncovalent Interactions

Due to the formation of big bundles held strongly together, CNT are very difficult to disperse homogeneously in solution. One of the approaches that have been widely used to exfoliate bundles and prepare individual CNT is the noncovalent wrapping of the tubular surface by various species of polymers,[4,9] polynuclear aromatic compounds,[219] surfactants,[220] and biomolecules.[19a] Noncovalent functionalization of CNT is particularly attractive because it offers the possibility of attaching chemical handles without affecting the electronic network of the tubes. The noncovalent interaction is based on van der Waals forces or $\pi-\pi$ stacking, and it is controlled by thermodynamics.

Stacking interactions between nanotubes and polynuclear species have been reported to aid the controlled placement of the carbon structures onto various surfaces and nanoparticles. Pyrene-modified oxide surfaces have been employed for the patterned assembly of single-walled carbon nanomaterials.[221a,b] The method relies on distinct molecular recognition properties of pyrene functional groups toward the carbon graphitic structure. The initial surface modification consisted of the reaction between bifunctional molecules (with amino and silane groups) and the hydroxyl groups on an oxide substrate, generating an amine-covered surface. This was followed by a coupling step where molecules with pyrene groups were allowed to react with amines. With the area covered with pyrenyl groups, the patterned assembly of a single layer of SWNT could be achieved through $\pi-\pi$ stacking. Georgakilas et al.[221c] have attached alkyl-modified iron oxide nanoparticles onto CNT by using a pyrenecarboxylic acid derivative as a chemical cross-linker. The authors reported that the resulting material had an increased solubility in organic media due to the chemical functions of the inorganic nanoparticles.

Surfactants were initially involved in the purification protocols of raw carbon material as dispersing agents.[222] Then, surfactant-stabilized dispersions of individual CNT were prepared for spectroscopic characterization,[223,224] for optical limiting properties studies,[196a] and for compatibility enhancement of the one-dimensional structures in the fabrication of composite materials.[225] CNT composites with a variety of noncovalent wrapping agents are reviewed extensively in the following sections.

4.1. Polymer Composites

CNT are considered ideal materials for reinforcing fibers due to their exceptional mechanical properties. Therefore, nanotube−polymer composites have potential applications in aerospace science, where lightweight robust materials are needed.[226] It is widely recognized that the fabrication of high performance nanotube−polymer composites depends on the efficient load transfer from the host matrix to the tubes. The load transfer requires homogeneous dispersion of the filler and strong interfacial bonding between the two components.[227] To address these issues, several strategies for the synthesis of such composites have been developed. Currently, these strategies involve physical mixing in solution, *in situ* polymerization of monomers in the presence of nanotubes, surfactant-assisted processing of composites, and chemical functionalization of the incorporated tubes.

4.1.1. Epoxy Composites

Nanotube−epoxy composites have been widely studied. Aligned arrays of MWNT within an epoxy resin matrix were prepared by Ajayan et al.[228] The CNT material was produced by the arc-discharge technique and was dispersed in the resin by mechanical mixing. The orientation of the nanotubes was observed after cutting the composite into thin slices (thickness < 200 nm).

A method to fabricate epoxy-based composites with mechanically aligned CNT was reported by Jin et al.[229a] The composites were prepared by casting a suspension of CNT in a solution of a thermoplastic polymer in chloroform. They were uniaxially stretched at 100 °C and were found to remain elongated after removal of the load at room temperature. The orientation and the degree of alignment were determined by X-ray diffraction and TEM. The same group studied the buckling of the strained nanotubes in epoxy blends by TEM.[229b] The deformation was found to be reversible at moderate strains.

The mechanical behavior of the nanotube-based composites has been the subject of study of many research groups.[230−236] Multi-walled nanotubes ultrasonically dispersed in epoxy matrix were studied in both tension and compression by Raman spectroscopy.[230] Cooper et al.[230c,d] studied the stress transfer between the nanotubes and the epoxy matrix by detecting a shift of the Raman 2600 cm^{-1} band to a lower wavenumber. The shift indicates that there is stress transfer and hence reinforcement by the nanotubes. In other investigations,[230a,b] the authors suggest that their nearly constant value of the Raman peak in tension is related to tube sliding within the bundles and hence poor interfacial load transfer between the nanotubes.

For improved dispersion and interfacial bonding of CNT with an epoxy matrix, a surfactant-assisted processing of tubes has been studied thoroughly.[225,231a] This resulted in a 30% increase of the elastic modulus of the composite with addition of 1% nanotubes.[225] Strano and co-workers[231d] have studied the dispersion of individual SWNT into an epoxy matrix by the decoration of a nanotube surface with the protein concanavalin A. Regions of aggregation within the composite could be monitored by fluorescence spectroscopy, since they have no emission.

Cooper et al.[232f] investigated the adhesion of CNT to an epoxy matrix by pulling out a single tube with the tip of a scanning probe microscope. In most cases, the nanotube ropes underwent fracture.[232] The effect of oxidation of CNT on the mechanical durability of epoxy blends has been studied, and it was found that this treatment resulted in mechanical improvement of the composite.[236]

The thermal conductivity was studied extensively. Johnson and collaborators[237a,b] fabricated nanotube−epoxy composites and measured a thermal conductivity enhancement greater than 125% at 1% nanotube loading. In similar studies, it was found that the incorporation of nanotubes into an epoxy matrix affects the cure reaction and that the thermal degradation of the composite increases with increasing the filler concentration.[237c,d,e]

Many groups have studied the electric conductivity of dispersed CNT into epoxy polymers.[238,239] The value of the conductivity was found to be proportional to the nanotube content in the composite.

To improve the interaction of oxidized CNT with epoxy matrixes, Gojny et al.[240] attached an amino derivative to the carboxylic groups through ionic functionalization. The resulting composite showed that the bundling of the tubes was clearly reduced. Similarly, fluorinated CNT have been dispersed through sonication in an epoxy matrix, giving reinforced composite material.[241]

4.1.2. Acrylates

CNT and PMMA were mixed together in solution using ultrasonication.[242,243] A combination of solvent casting and melt mixing gave composite films with exceptional mechanical and electrical properties.[243a] Alternatively, the coagulation method was used to produce nanotube−PMMA composites.[243b] After mixing the components, precipitation took place so that the polymer chains entrapped the nanotubes and prevented them from rebundling. Raman studies of these composite materials showed modifications of the bands assigned to the nanotubes.[242]

Using the solution mixing protocol, pyrene-containing poly(acrylates) were successfully immobilized on the surface of multi-walled nanotubes due to $\pi-\pi$ stacking.[244] The modified carbon material could be easily dispersed in organic solvents and characterized by thermogravimetric analysis, TEM, and AFM.

Melt blending was used to fabricate thermoplastic polymer composites. MWNT were dispersed in a PMMA matrix, while their mechanical behavior was investigated thoroughly.[245a,b] In an analogous work, prior to the melt blending process, the nanotube material was made more compatible by mixing with poly(vinylidene fluoride). This treatment led to improved mechanical properties of the blend.[245c] Block copolymers have been extensively used to increase compatibility and dispersibility in carbon nanotube composites. Velasco-Santos et al.[246] prepared composites of nanotubes and methyl-ethyl methacrylate copolymer, modified with nonionic surfactant to improve the dispersion and manipulation of the mixture. Similarly, for dispersing high concentrations of individual CNT in organic solvents, raw material was sonicated in the presence of a synthetic block copolymer of *tert*-butyl acrylate and styrene.[247] Electron microscopy indicated that the solvent could be evaporated without provoking bundling of nanotubes, while the composite could be redispersed in ethanol solution. These samples were found to be permanently dispersed for a period of at least two months.

Sabba et al.[248] reported an exfoliation method for dispersing nanotubes in solution before mixing with poly(methyl methacrylate). They treated CNT with a solution of hydroxylamine hydrochloric acid salt, which induced an electric charge on the surface of the tubes. Therefore, the electrostatic repulsion reduced the overall forces that hold the tubes together in the form of bundles, resulting in a homogeneous polymer composite. An alternative approach for preparing composites with oriented tubes was based on a dry powder mixing method for the two components followed by a polymer extrusion technique.[249] The fracture toughness of the mixture was significantly improved by even small amounts of filler.

Putz et al.[250a] prepared nanotube−PMMA composites by *in situ* radical polymerization of the monomer. The spectroscopic studies showed clear evidence of cohesive interactions between the surface of nanotubes and the polymer chain. Ajayan and co-workers[250b,c] have studied the stiffness of thick-aligned MWNT−PMMA composite disks, prepared by *in situ* polymerization. Aligned arrays of tubes grown on a quartz substrate were immersed into excess monomer solution, and the resulting polymer occupied the interstitial pores of the nanotube arrays. Stiffness properties were studied using Vicker's microhardness as well as through the force curves generated by an AFM instrument.

Electrical conductivity measurements of nanotube−acrylate composites showed that small weight percentage additions of tubes dramatically increase the magnitude of the electric current permittivity, whereas, by using the method of a PMMA suspended dispersion, nanotubes could be deposited between metal electrodes for field emission applications.[251]

Aligned CNT in a polyester matrix were obtained by polymerizing the tube–monomer dispersion under the application of a constant magnetic field.[252] Magnetic susceptibility and electric conductivity measurements showed that the orientation of the nanotubes was magnetic field induced.

Enzyme-containing acrylate–nanotube composites have been explored as novel biocatalytic materials.[253] Chymotrypsin was added to a nanotube–PMMA dispersion, and the activity of the resulting mixture was found to be higher than that in a polymer–enzyme film. The authors reasoned that the incorporation of nanotubes might offer a higher surface area for interactions with the enzyme.

Harmon and co-workers[254] studied the effect of ionizing radiation on the mechanical properties of nanotube–PMMA composites. It was concluded that the radiation resistance of the polymer may be increased through the addition of small amounts of CNT. The most dramatic change observed after radiation was in the dielectric properties of the composite.

Soluble multi-walled nanotubes obtained via amidation reaction of oxidized material with long chain alkylamines were mixed in solution with an acrylate copolymer in various loadings.[255] Compared to the neat polymer, the composite had improved mechanical properties due to efficient distribution of the filler component.

4.1.3. Hydrocarbon Polymers

CNT have been dispersed in a variety of hydrocarbon polymers, such as polystyrene, polypropylene, and polyethylene. Many research groups have prepared polystyrene composites by solution or shear mixing.[9,256,257] The mechanical properties of the blends were improved compared to those of the neat matrix. Moreover, the interfacial strength between the reinforcement and the matrix has been studied through molecular mechanics simulations, and it was estimated that the shear stress of such a system is about 160 MPa, significantly higher than those for most polymer composites.[235b,258]

Barraza et al.[259a] dispersed nanotubes in a styrene monomer solution, and the mixture was subjected to polymerization under emulsion conditions. The composite exhibited solubility in organic solvents, and the electrical resistivity dropped substantially due to the incorporation of the tubes. In a recent work,[259b] double-walled CNT–polystyrene composites were synthesized by *in situ* nitroxide-mediated polymerization. In a second step, the presence of the stable nitroxide radical on the tube surface allowed reinitiation of the polymerization of different monomers.

Covalently functionalized CNT by diazonium salts have been mixed with polystyrene, giving better dispersion and compatibility, while the glass transition properties were examined in detail.[86] The maxima in the differential scanning calorimetry spectra are at slightly higher temperatures for the composite samples. Similarly, as-prepared and defect-functionalized single-walled nanotubes were admixed with polystyrene using the electrospinning technique.[260] The composite membranes showed a significant enhancement in the mechanical properties, and among the samples, the blend with the functionalized tubes gave the best results.

Amphiphilic copolymers of polystyrene were used for encapsulation of individual tubes.[261a] By using the right binary solvent system (dimethylformamide/water), the copolymers act as a common micelle and cause permanent dispersion of the nanotubes. Moreover, stable dispersions of CNT were obtained after their incubation with A–B–A block telomers, where the A block is either poly(alkylacrylamide) or glucopyranoside chains and the B block is polystyrene.[261b]

Instead of preparing composites of well dispersed nanotubes in a polymeric matrix, Coleman et al.[262] showed that polystyrene chains could be intercalated into the porous internal sites of carbon nanotube sheets by simply soaking the components in solution phase. Tensile tests on the composites showed enhanced toughness by a factor of 28, indicating that the intercalated polymer transmits the load to the tubes.

The electrical conductivity of nanotube–polystyrene composites was examined in detail, thus giving the conclusion that defective nanotubes within the polymer blend transport the electric current more efficiently.[263] CNT have also been studied as potential oxidation retarding components in polymer composites.[264] The matrixes examined were polystyrene, polyethylene, and polypropylene. Boron doping in nanotubes was found to lead to a small increase in antioxidant efficiency.

Another thermoplastic polymer that is used extensively for strong composite materials is polypropylene. The most common ways of composite fabrication are shear mixing[257c,265] or melt blending.[266–269] Grady et al.[270a] mixed soluble defect-functionalized CNT with polypropylene in solution followed by solvent evaporation. By studying the crystallization behavior of the polymer matrix, it was concluded that the presence of the nanotubes is critical for nucleating crystallinity in polypropylene.[268a,d,270] The thermal and flammability properties of polypropylene filled with multi-walled nanotubes have been investigated.[266b] Flammability properties were measured using a calorimeter and a gasification device. It was found that more than 2% weight of CNT is required to increase the ignition delay time of the composite.

Barber et al.[271] studied the interfacial strength of a glass fiber–polypropylene composite using embedded CNT as stress sensors. Previous work has shown that stresses in polymer systems can be measured using CNT and Raman spectroscopy.[272] During mechanical testing of the composite, Raman spectra of the nanotubes were recorded and the strain conditions of their environment were evaluated in real time.

In addition, CNT have been functionalized noncovalently with polyethylene by melt blending,[273a–g] controlled polymer crystallization,[273h] or *in situ* supported coordination polymerization,[273i] and with polynorbornene by *in situ* polymerization.[274] Barber et al.[275] investigated the adhesion of CNT to a polyethylene–butene matrix by pulling out a single tube with the tip of atomic force microscope. It was concluded that the polymer mechanical properties in the vicinity of the nanotube appear to show differences when compared to those of the bulk polymer behavior. The interfacial separation stress was found to be about 47 MPa.

4.1.4. Conjugated Polymers

An interesting class of polymer composites that has attracted much attention is that of conjugated polymers such as poly(phenylenevinylene) (PPV). The first polymer that was mixed with CNT was poly(phenylacetylene).[276] The composite was prepared by *in situ* polymerization of phenylacetylene in the presence of the tubes. It was found that the polymer chain wraps the nanotubes helically and this induces solubility of the blend in common organic solvents. Under harsh laser irradiation, the nanotubes exhibited a strong photostabilization effect, protecting the wrapped polymer from photodegradation.

Because of the great promise of conjugated polymer composites in photovoltaic devices, the CNT were mixed with PPV and their optical properties were investigated.[277] The quantum efficiency obtained was 1.8%,[277b] which arises mainly from the complex interpenetrating network of polymer chains with the nanotube film. The predominant electronic interaction between the two components is nonradiative energy transfer from the excited polymer to the tubes. A modified PPV, poly[2,5-dimethoxy-1,4-phenylenevinylene-2-methoxy-5(2′-ethylhexyloxy)-1,4-phenylenevinylene] (M3H-PPV), was used also for photoluminescence studies in composites with CNT.[277e,278]

A polymer that has been studied extensively in optoelectronic applications as a CNT dopant is poly(*m*-phenylenevinylene-*co*-2,5-dioctyloxy-*p*-phenylenevinylene) (PmPV).[278−283] The substitution pattern of the polymer chain leads to dihedral angles resulting in a helical structure. The coiled conformation allows the polymer to surround the surface of nanotubes by interacting with $\pi-\pi$ forces. In the seminal work of Blau and co-workers,[279a,e] it was found that, after the incorporation of CNT, the electrical conductivity of the conjugated polymer film was increased by up to 8 orders of magnitude. Because of the luminescent properties of the polymer, the composite was used in the fabrication of optoelectronic memory devices.[280] Through the special interaction between the two components, it was demonstrated that solutions of the polymer could keep the CNT suspended indefinitely.[279c] Raman and absorption studies suggested that the polymer wraps preferentially with nanotubes possessing a specific range of diameters. The same group suggested that incorporation of raw nanotube material in PmPV could lead to efficient phase separation from the main impurity, the amorphous graphitic shells.[279d,281b,282e] A nondestructive purification method for CNT was addressed using a one-step process. Amorphous carbon impurities tend to sediment out of solution, whereas the nanotubes stay in suspension. Atomistic molecular dynamics studies have elucidated the strong nature of the interaction between the polymer and the nanotubes.[281e]

Stoddart, Heath, and co-workers[283] studied composites of nanotubes with alkoxy-modified phenylene vinylene-type polymers. They characterized the composites with PmPV by UV−vis, NMR, and AFM, whereas the performance in a photovoltaic device was improved.[283a] In a subsequent work, the same researchers studied for comparison the chemical interactions of CNT with PmPV and poly(2,6-pyridinylenevinylene-*co*-2,5-dioctoxy-*p*-phenylenevinylene) (PPyPV).[283b] In both cases, they observed dispersion of the tubes in the organic media. The concept of solubilizing nanotubes by using macromolecules with well-defined cavities was studied recently. A hyperbranched polymer was synthesized and was found to suspend CNT in organic solvents.[283c] Similarly, functionalized conjugated polymers that have the capacity to form pseudorotaxanes were mixed with CNT, affording structures with potential applications in actuation and electronics.[283d]

An alternative strategy for solubilizing CNT was reported by Chen and co-workers.[284a] The authors attached noncovalently short rigid oligomers of poly(aryleneethynylene) type. The major interaction between the polymer backbone and the nanotube surface is most likely $\pi-\pi$ stacking, whereas no helical wrapping of polymer chains occurred. This allowed a 20-fold solubility enhancement for small diameter nanotubes. In a subsequent work, the authors demonstrated the homogeneous dispersion of such tubes in matrixes of polystyrene or polycarbonate.[284b] These composites show dramatic improvements in the electrical conductivity at low filler loading (percolation threshold at 0.045 wt %).

Nanotube−polypyrrole composites have been engineered by *in situ* chemical[285] or electrochemical polymerization.[73,286,287] These types of composites have been used as active electrode materials in the assembly of a supercapacitor,[288] for the selective detection of glucose,[73,289] and for selective measurement of DNA hybridization.[290] The detection approach relied on the doping of glucose oxidase and nucleic acid fragments within electropolymerized polypyrrole onto the surface of nanotubes. Recently, nanotube−polypyrrole composites have been studied as gas sensors for NO_2.[291]

Electrochemical polymerization of aniline onto CNT electrodes for the deposition of conducting polymeric films has been reported by independent works.[292] Alternative strategies involve the chemical polymerization of aniline or solution mixing of nanotubes and the conjugated polymer.[132,293a−e] The blends exhibited an order of magnitude increase in electrical conductivity over the neat polymer.[293f,g]

Liu et al.[294] have successfully assembled poly(aminobenzenesulfonic acid)-modified SWNT with polyaniline via the simple layer-by-layer (LBL) method. The obtained PANI/PABS−SWNT multilayer films were very stable and showed a high electrocatalytic ability toward the oxidation of reduced β- nicotinamide adenine dinucleotide (NADH) at a much lower potential (about +50 mV vs Ag/AgCl). In the case of six bilayers, the detection limit could go down to 1×10^{-6} M.

Blends of nanotube−poly(alkylthiophene) have been fabricated,[295] and their electrical properties were studied.[295−297] The enhanced photovoltaic behavior of the composites makes them ideal candidates as solar cells for energy conversion.[297] For improved light harvesting, organic dye molecules were incorporated into the blend and the resulting photocurrent was 2 orders of magnitude larger as compared to that of the nanotube−polymer blend device.[297c]

4.1.5. Other Nanotube−Polymer Composites

(i) Polyacrylonitrile.[298−301] For the fabrication of nanotube composites, different methods have been used like solution mixing with the aid of sonication,[298,300a] electrospinning,[299] and *in situ* polymerization of the monomer in the presence of tubes.[301c] The performance of such composites was studied in supercapacitor electrode applications,[300a] whereas the mechanical properties study showed a 100% increase in tensile modulus at room temperature, significant reduction in thermal shrinkage, and a 40% increase in glass transition temperature.[300b,301a,b]

(ii) Polycarbonates.[302] Nanotube composites were first prepared by solution mixing[302a,e] and were characterized by Raman spectroscopy.[302a] Another fabrication strategy involves melt extrusion[302b,c,d] followed by fiber spinning for well-aligned nanotubes in the matrix.[302d] The polymer sheath around the nanotube surface was studied thoroughly by SEM, giving direct evidence for tube−polymer interaction.[302e]

(iii) Aminopolymers.[303,304] By using a solution mixing approach, O'Connell et al.[303a] succeeded in solubilizing CNT in aqueous media by wrapping them with poly(vinylpyrrolidone). The process was found to be solvent-dependent, since dissociation of the tube−polymer complexes took place when tetrahydrofuran was used. By the same strategy, SWNT were directly dispersed in alcoholic solvents by sonicating the tubes in the presence of poly(vinylpyridine).[303b] Depending upon the alcohol, it was possible to disperse up to 300 mg of raw material per liter of solvent.

Single-walled nanotube polyimide composites were synthesized by *in situ* polymerization of monomers and sonication.[304a] The resulting blends showed electrical conductivity enhancement by 10 orders of magnitude at low filler loading (0.1 wt %).[304a,c] The dispersion of nanotubes in the polymer matrix was studied by magnetic force microscopy,[304b] showing also the presence of agglomerates within the polyimide.

(iv) Fluoropolymers.[305−307] The first fluoropolymer used for the successful dispersion of CNT was Nafion.[305] The components were mixed in solution, and the resulting blends were found to behave as potential actuators.[305a] By application of a voltage to the composite films, the authors observed deflections up to 4.5 mm. Wang and co-workers[305b,c] reported the ability of Nafion to solubilize nanotubes in alcoholic media. The polymer-induced solubilization permitted the modification of the electrode surfaces for amperometric sensing of hydrogen peroxide or dopamine. Similarly, Guo et al.[305d] studied the electrochemistry and the electrogenerated chemiluminescence of a ruthenium(II)−tris(bipyridine) complex after its immobilization in a nanotube−Nafion composite film. The system showed a three orders of magnitude higher sensitivity and long-term stability, compared to neat Nafion films on carbon electrodes.

Nanotube−Teflon composite electrodes were prepared by dry-state mixing for effective amperometric sensing of glucose and ethanol.[306] Poly(vinylidene fluoride) or its copolymers has also been used as a matrix for nanotube composites,[307] while electrical conductivity measurements were obtained in electrospun fibers from DMF solutions.[307a]

(v) Poly(vinyl alcohol).[281c,308−312] The first papers reported the solution mixing of CNT with the polymer matrix in aqueous media and subsequent preparation of the film by casting.[308,309] The presence of nanotubes was found to stiffen the material and retard the onset for thermal degradation. The electrical properties of the composites were measured by impedance spectroscopy, and the percolation threshold was found to lie between 5 and 10 wt % loading. Furthermore, microscopy studies suggested extremely strong interfacial bonding between the components as the presence of nanotubes nucleates the crystallization of the matrix.[309] Covalent modification of CNT with ferritin protein prior to polymer mixing was shown to increase the modulus of the polymer matrix by 110% with the addition of 1.5 wt % filler material.[310]

An alternative processing consists of dispersing the nanotubes in surfactant solutions and recondensing the material in the flow of PVA solution, forming ribbonlike structures.[311,312] These fibers were found to bend without breaking, while tensile stress measurements showed Young's modulus values up to 40 GPa. By using scanning electron microscopy, most of such fibers had diameters of about 30−40 μm.

(vi) Poly(ethylene glycol).[247,313,314] The fabrication of nanotube−PEG composites by solution mixing was first demonstrated by Goh and co-workers.[202a,313a,b] The resulting blends were found to have enhanced mechanical properties due to hydrogen bond interaction between the defect sites of the nanotubes and the oxygen atoms of the polymeric chain.[313a] Using different approaches, CNT were chemically functionalized by fluorination before mixing with PEG[313c] or were processed by an electrospinning technique.[313d] Electron microscopy showed improved uniformity of the composite, while the storage modulus increased five times in comparison to the neat polymer at 4% loading.[313c] Motivated by the applications of CNT in biology, the groups of Dai[314a,b,c] and Star[314d] investigated the nonspecific binding (NSB) of proteins to the surface of tubes. They showed that prevention of NSB of certain biomolecules on SWNT can be achieved by coating the graphitic surface with ionic surfactants and PEG.

For dispersing high concentrations of individual CNT in aqueous media, as-prepared CNT were sonicated in the presence of a synthetic block copolymer of ethylene glycol and propylene glycol.[247] Electron microscopy indicated that the composite could be dried without bundling of nanotubes and be redispersed in water solution. These samples were found to be permanently dispersed for a period of at least two months.

(vii) Silicon Polymers.[247a,315] Modification of CNT by silicon-based polymers was found to activate the fluorescence of the tubular structures for better observation and manipulation.[315a] Frogley et al.[315b] performed mechanical studies in nanotube−silicon elastomer composites showing a stiffness increase of about 200% at 1% loading. Block copolymers of poly(dimethylsiloxane) have been used recently for the dispersion of CNT in organic solvents.[247a]

(viii) Polyelectrolytes.[303a,316−318] One of the most studied polymers for nanotube doping is poly(ethyleneimine). This amine-rich polymer was found to adsorb irreversibly on tubular surfaces after solution phase treatment, while the potential application of the composite in field effect transistor devices[316a,b] or selective detection of gas traces[316c] was demonstrated by conductance measurements. For the fabrication of super strong nanotube−poly(ethyleneimine) composites, many groups have developed the stepwise adsorption of nanotubes and polymer thin films onto a substrate via electrostatic interactions and/or chemical linking.[316d,e] Microscopy studies confirmed the structural homogeneity of the prepared composites, which displayed an ultimate tensile strength of 150 MPa.[316e] In addition, it was found that the morphology of the nanotubes can induce differences in the mechanical performance. The replacement of hollow tubes with bamboo-type nanotubes significantly improved the strength of the composite. In a similar work, Guldi et al.[316f] studied the organization of CNT into films with poly(ethyleneimine) by AFM. It was found that perfect ring structures form spontaneously after electrostatic interactions between the oxidized tubes and the polyelectrolyte. The electrical conductivity of such composite films was studied extensively by Kovtyukhova et al.[316g] Due to the presence of CNT in the plane of the thin films, the electrical properties could be enhanced by several orders of magnitude.

By the LBL assembly, nanotube−poly(diallyldimethylammonium chloride) composites can be formed via electrostatic interactions onto substrates.[317a−d] The protocol for composite fabrication involved the alternate immersion of flat glass surfaces into solutions of nanotubes and polymer. The Coulomb nature of the interactions between the carboxylic groups of the oxidized nanotube surface and the positive charges of the polyelectrolyte was confirmed by rheological studies in solution.[317e] By similar approach, Pavoor et al.[317f] fabricated multilayer composites of nanotubes and poly(allylamine hydrochloride).

Alternatively, polyelectrolyte LBL assemblies on CNT have been fabricated by initially modifying the nanotube surface with an ionic pyrene derivative followed by electrostatic deposition of polystyrene sulfonate and poly(diallyldimethylammonium chloride).[318a] Microscopy data confirm the formation of polymeric shells around the tubular surfaces of the carbon materials.

Instead of immersing the glass substrates into the solutions, Carrillo et al.[318b] carried out the deposition of hydrolyzed poly(styrene-*alt*-maleic anhydride) on the nanotube surface using a flow cell reactor. The authors reasoned that such polymers would adsorb noncovalently via hydrophobic interactions. The attached polymer layer contains carboxylic groups that can be used to graft a second polyelectrolyte of opposite charge. These depositions can be repeated to build a multilayered film of polycations and polyanions. In a subsequent step, gold nanoparticles could be attached to the polymer-coated nanotubes via ionic interactions.[318b,c]

O'Connell et al.[303a] have studied the solubilization of nanotubes, by mixing them with polystyrenesulfonate in aqueous media. The surfactant-like polymer is supposed to disrupt the hydrophobic interface with the solvent molecules and cause partial exfoliation of the bundles. The nanotubes were found to unwrap by changing the solvent medium, as precipitation was observed. In a similar approach, Kotov and co-workers[318d] showed that poly(vinylpyridinium bromide) chains formed exceptionally stable CNT dispersions in aqueous media.

(ix) Polyesters.[319] CNT were dispersed in a poly(vinyl acetate) emulsion-based matrix, and the electrical properties were investigated as a function of filler loading.[319a] A very low percolation threshold was achieved (below 0.1%) as a result of segregated networks. To achieve low percolation thresholds (about 0.2%), Nogales et al.[319b] studied the fabrication of polyterephthalates composites by using an *in situ* polycondensation reaction. The authors dispersed CNT in butanediol and subsequently added the phthalate reagent for starting the polymerization. The agglomeration effect of the tubes seems to lead to the formation of conducting networks within the insulating matrix.

By using melt blending under high stirring, Peeterbroeck et al.[319c] prepared composites of CNT–poly(vinyl acetate) copolymer, as well as ternary systems with organo-modified clays. Both thermal and mechanical properties of the composites were enhanced by the presence of the nanofiller. A synergistic effect was observed when clays and nanotubes were added simultaneously.

Shape memory polymers can recover their original shape when heated above some critical temperature. Instead of trying thermal actuation, Cho et al.[319d] have studied the potential of MWNT–polyurethane composites as electroactive actuators. When an electric field of 40 V was applied at room temperature, the composite recovered the shape that it should have above the transition temperature within 10 s. The energy conversion efficiency was estimated to be almost 10%.

(x) Polyamides.[320] Nylon nanocomposites have been prepared by *in situ* polycondensation of the appropriate diamines and acyl chlorides in the presence of nanotubes. The first reports described improvements of the mechanical properties below 20%.[320a,b] More recently, nanotube–nylon blends have been fabricated by melt mixing. Upon incorporation of 1% MWNT, the elastic modulus improved by about 115% and the tensile strength by about 124%.[130a,320c,d]

(xi) Poly(vinylcarbazole).[310,321] Using either purified MWNT or alkylamine-modified MWNT, Dai and collaborators prepared PVK composites by solution mixing.[321a] Fluorescence quenching of the polymer by the modified tubes showed that the latter could act as electron acceptors in the ground or excited state. In contrast, purified tubes did not improve the photoconductivity of the polymer matrix due to miscibility problems. Potential use of these composites in the fabrication of light emitting devices was envisaged.[321b]

(xii) Poly(*p*-phenylene benzobisoxazole).[322] This polymer has been synthesized in the presence of CNT under polycondensation conditions. The tensile strength of the composite containing 10% of filler material was about 50% higher than that of the neat matrix, whereas the presence of the nanotubes was evidenced by Raman spectroscopy.

(xiii) Phenoxy Resin.[323] Goh and co-workers reported the fabrication of *in situ* modified nanotube–phenoxy composites by melt mixing. During the thermal treatment of the components, imidazole groups were covalently attached to the defect sites of the nanotube surfaces. It was suggested that the functionality helps the dispersion of hydrophobic tubes within the hydrophilic matrix via hydrogen bond interaction.

(xiv) Natural Rubber.[324] The effects of incorporation of nanotubes on the mechanical properties of an elastomer matrix have been described. Dynamic mechanical analysis showed a strong interaction between the components, whereas the vulcanization reaction of rubber was accelerated in the presence of nanotubes.

(xv) Petroleum Pitch.[325] SWNT were dispersed in a petroleum pitch matrix to form composites with enhanced properties. The tensile strength, modulus, and electrical conductivity improved by 90%, 150%, and 340%, respectively, as compared to those of unmodified pitches.

4.2. Interactions with Biomolecules and Cells

CNT can interact with many biomolecules without forming a covalent conjugate. The electronic properties of CNT coupled with the specific recognition properties of the immobilized biosystems would therefore generate a miniaturized biosensor.[326] An important class of substrates having high affinity with the graphitic network are proteins. They tend to adsorb strongly on the external sides of nanotube walls and can be visualized clearly by microscopy techniques. In the seminal work of Tsang and co-workers,[327] metallothionein proteins were found to adsorb onto the surface of multi-walled CNT, as evidenced by high-resolution TEM.

Streptavidin was found to adsorb on nanotubes presumably via interactions between the graphitic surface and the hydrophobic domains of the biomolecule[328a] or even via charge-transfer interactions.[328b] The immobilization of streptavidin on CNT has been reported as the key approach for the controlled deposition of carbon wires on specific surfaces. Keren et al.[329] showed that the protein-coated nanotubes could be assembled on a DNA scaffold through recognition schemes based on biotin–streptavidin specific interactions. This approach allowed the precise localization of CNT in field-effect transistor devices.

To prevent the nonspecific adsorption of streptavidin, CNT have been decorated noncovalently by a surfactant/polymer mixture.[314a] The authors showed that specific binding of the protein can be achieved by cofunctionalization of the CNT

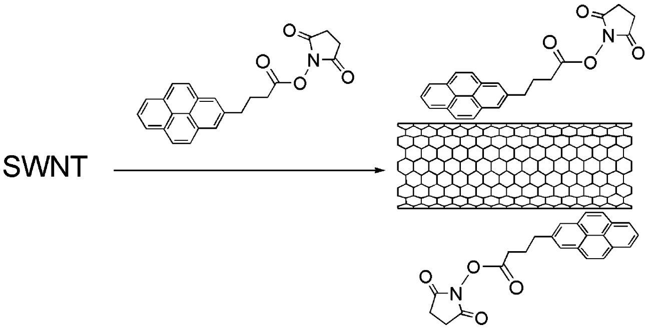


Figure 19. Interactions of nanotubes with pyrene derivatives.

with biotin, a molecule which exhibits extremely high affinity to streptavidin.

Azamian et al.[330] prepared several nanotube−protein composites and characterized them by AFM. Concerning biosensor technology, glucose oxidase, an enzyme which catalyzes the oxidation of glucose, has been immobilized onto the surface of CNT,[330,331] and it is extensively used in clinical tests. The nanotube−enzyme conjugate was integrated on a carbon electrode for voltammetric detection of glucose, resulting in an increase of the catalytic response of more than 10 times due to the presence of conducting CNT. Other examples of such electrochemical biosensors concern the hemoglobin system[332] for hydrogen peroxide detection, the myoglobin composite for nitric oxide[333a,b] or hydrogen peroxide[333c] detection, the hemin conjugate for oxygen gas sensing,[334a] the microperoxidase-11 system for oxygen reduction,[334b] the cholesterol esterase system for blood analysis,[335a] and the horseradish peroxidase system for hydrogen peroxide reduction.[335b] Karajanagi et al.[336] have investigated the secondary structure and activity of enzymes adsorbed on CNT by FT-IR spectroscopy and AFM imaging. The authors concluded that certain protein substrates retain their catalytic activity, while others experience structural perturbation on the surface of the tubes. The reason for these differences still remains unclear.

Similarly, monoclonal fullerene-specific antibodies have been shown to specifically bind to the surface of nanotubes.[337] The binding cavity of the antibody consists of a cluster of hydrophobic amino acids. An analogous nanotube−antibody conjugate was found to function as immunosensor for *Staphylococcus aureus*.[338] Wang et al.[339] observed that peptide sequences rich in histidine and tryptophan residues can be isolated from peptide phage-display libraries by specific binding to CNT. The peptides presented a certain degree of flexibility, which allowed them to adopt the appropriate folding to wrap around the tubes. The hydrophobic parts of the peptide chain were suggested to act as symmetric detergents.

A different approach for the noncovalent modification of CNT with biomolecules involves the use of bifunctional linkers, based on a pyrene moiety (Figure 19).[340]

The anchor molecule can adsorb irreversibly onto graphitic surfaces due to van der Waals interactions. In a subsequent step, enzymes can be covalently attached to the activated pyrene by nucleophilic attack of the basic amino acid residues. Using this binding approach, Dekker and coworkers[340b] studied the effect of immobilized glucose oxidase on the electrical conductance of CNT. They observed that the presence of the attached enzyme decreases the electrical conductance. Upon adding trace quantities of glucose molecules, an increase in conductance takes place, suggesting the use of the composite as a sensor for enzymatic activity. At the same time, several groups have studied the change of the electric properties (sensitivity) of the CNT in the presence of various biomolecules.[314b,c,d,341] In general, the results show that carbon tubes are excellent biosensors with potential applications in medicine and nanobiotechnology.

Synthetic peptides were designed not only for nanotube coating but also for the solubilization of the carbon material.[342] Amphiphilic helical peptides were found to fold around the graphitic surface of the nanotubes and to disperse them in aqueous solutions by noncovalent interactions. Most importantly, the size and morphology of the coated fibers can be controlled by peptide−peptide interactions, affording highly ordered structures.

Another example of assembly on the carbon nanotube surface involves the synthetic single-chain lipids.[343] Regular striations could be seen on the entire nanotube network by microscopy studies.[343a] Moreover, the polar part of the lipids could participate in the selective immobilization of histidine-tagged protein through metal ion chelates. In a different approach, Artyukhin et al.[343b] deposited alternating layers of cationic and anionic polyelectrolytes on templated carbon nanotubes. The authors demonstated the occurrence of spontaneous self-assembly of common phospholipid bilayers around the hydrophilic polymer coating CNT. The lipid membrane was found to maintain its fluidity, and the mobility of lipid molecules can still be described by a simple diffusion model.

Noncovalent interactions between DNA and CNT, as well as certain organization properties of such systems, have been reported.[188,327,344−353] Techniques used to study DNA−nanotube systems include TEM,[344] UV/IR spectroscopy,[345,346] and flow linear dichroism.[347] Clear evidence of binding between the components was observed in each case.

Several groups have reported that DNA strands interact strongly with CNT to form stable hybrids that can be effectively dispersed in aqueous solutions.[311d,348,349] Moreover, by wrapping the nanotubes with a DNA sequence of alternating guanine and thymine bases, it was possible not only to separate metallic from semiconducting tubes but also to perform a diameter-dependent separation via ion exchange chromatography.[350] Further supporting information about the nature of each eluted fraction was confirmed by fluorescence and Raman spectroscopic characterization.[351]

Xin et al.[352] fabricated nanotube−DNA composites by using the pyrene methylammonium compound as the chemical linker. The ammonium groups interact electrostatically with the phosphate moieties of the DNA backbone, whereas the pyrenyl moiety is adsorbed onto the graphitic surface by van der Waals forces. Through AFM imaging, it was concluded that two-thirds of the tubes were anchored with DNA strands. The latter were used as templates for the direct positioning of CNT on a Si surface. A similar modification strategy involves the attachment of pyrene-modified oligonucleotides to the sidewalls of the nanotubes. In this case, Taft et al.[188] introduced the polynuclear aromatic compound onto the 5′-end of a DNA by covalent binding. To visualize the immobilized strands, complementary sequences were thiolated and attached to gold nanoparticles. This strategy allowed analysis of the DNA−CNT conjugates by scanning electron microscopy.

The electrostatic assembly of DNA on nanotube-modified gold electrodes via the cationic polyelectrolyte poly(diallyl dimethylammonium chloride) (PDDA) has been evaluated.[353]

The piezoelectric quartz crystal impedance technique and electrochemical impedance spectroscopy were used to characterize the system. PDDA plays a key role in the attachment of DNA to MWNT acting as a bridge.

The presence of CNT in a polymerase chain reactor was also found to increase the amount of products at nanotube concentrations below 3 mg/mL.[354]

The preparation of carbon nanotube electrodes for improved detection of purines, nucleic acids, and DNA hybridization was reported.[355] The graphitic surface was found to facilitate the adsorptive accumulation of the guanine bases and eventually to enhance their oxidation signal. In a recent work,[355d] the change in the electrochemical response of guanine in leukemia K562 cells was detected by using a MWNT-modified carbon electrode. The voltammetric responses of the cells were found to decrease significantly, whereas the cytotoxicity curves were in good agreement with conventional tests such as ELISA.

To make CNT soluble in aqueous media, many groups explored the possibility of decorating the graphitic surface with carbohydrate macromolecules. In the work of Regev and co-workers,[356] it was shown that CNT can be dispersed in an aqueous solution of Arabic Gum by nonspecific physical adsorption. Arabic Gum is a highly branched arabinogalactan polysaccharide, which seems to cause efficient unbundling of the nanotube ropes. This was supported by TEM imaging and X-ray scattering spectroscopy.

Star et al.[357a] studied the complexation of nanotubes with starch and, in particular, its linear component amylose. This polysaccharide consists of glucopyranose units and adopts a helical conformation in water, forming inclusion complexes with various substances. The initial experiments revealed that CNT are not soluble in an aqueous solution of starch but, rather, are soluble in a solution of a starch−iodine complex. The authors suggested that the preorganization of amylose in a helical conformation through complexation with iodine is critical for a single tube to enter the cavity of the helix. In a subsequent work, the enzymatic degradation of starch in its water-soluble composites with CNT was studied by direct microscopy imaging and electronic measurements.[357b] It was observed that CNT precipitated after hydrolysis of the polysaccharide chains.

Using dimethyl sulfoxide/water mixtures, Kim et al.[358] reported the solubilization of nanotubes with amylose. In these media, the polysaccharide adopts an interrupted loose helix structure. The authors claimed that the helical state of amylose is not a prerequisite for nanotube encapsulation. In addition, the same group studied the dispersion capability of other amylose homologues, pullulan and carboxymethyl amylose. These substances could solubilize CNT but to a lesser extent than amylose. Several other examples of helical wrapping of linear or branched polysaccharides around the surface of CNT have appeared since.[359]

The complexation of nanotubes with cyclodextrins, macrocyclic analogues of amylose, was studied thoroughly. The first composite was prepared by a simple grinding procedure, which has been reported to cut HipCO tubes.[360] Alternatively, both components have been mixed in refluxing water and the resulting conjugate was fully characterized by UV−vis, Raman, and DSC spectroscopies.[360b] The results showed clear evidence of strong intermolecular interaction between the nanotubes and the cyclodextrins.

Complexation of SWNT with 12-membered cyclodextrins by simple solution mixing was found to enable not only their solubilization in water but also their partial separation with respect to diameters and the determination of the number of nanotube types on the basis of NMR spectra.[361a] Purified SWNT and cyclodextrins mixed by a mechanochemical high-speed vibration milling technique were also solubilized in an aqueous medium due to the formation of noncovalent-type complexes and debundling of tubes.[361b]

Another class of molecules that have been immobilized onto CNT is light harvesting species, such as phthalocyanines,[158,362] porphyrins,[128,363] and dyes of phenazine and thionine type.[364] The decoration of the graphitic surface resulted from $\pi-\pi$ interactions with the conjugated molecules or from chemisorption at the carboxylic defect sites of the nanotubes. The phthalocyanine composites exhibited an enhanced photosensitivity, which was ascribed to the photoinduced charge transfer from the dye molecule to the carbon tubes. Researchers have reported the dissolution of CNT in organic solvents[363a,b,d,f] or aqueous media[363c,e,g] via noncovalent adsorption of porphyrins. The interaction of the components was evident by detecting the fluorescence quenching of the porphyrin molecule due to energy transfer to the tubes. Sun and co-workers[363b] reported that porphyrin derivatives adsorb selectively onto semiconducting nanotubes in a solubilized sample, according to Raman, near-IR absorption, and bulk conductivity characterizations. The authors proposed this procedure as a convenient method for the separation of semiconducting and metallic CNT. Recently, Satake et al.[363d] have synthesized stable CNT−porphyrin composites by condensation of tetraformylporphyrins and diaminopyrenes on the nanotube surface, whereas Guldi and co-workers[363e−i] have applied two different approaches. In the first work,[363e,f] the authors immobilized either oligo-anionic or oligo-cationic porphyrin derivatives onto modified CNT via electrostatic interactions. A cationic or anionic derivative of pyrene was used as an electrostatic anchor for binding the porphyrin chromophores, respectively. In a similar work, the supramolecular association of pristine CNT with poly(porphyrin) chains was studied thoroughly.[363g] In these novel donor−acceptor ensembles, quenching of photoexcited porphyrins by CNT results in the creation of long-lived radical ion pairs. Chichak et al.[363j] discovered that a porphyrin derivative carrying two pyridine ligands enters into a self-assembly process with a palladium(II) complex and can simultaneously solubilize SWNT in aqueous solutions. The combination of both complexes is suggested to form charged acyclic and/or cyclic adducts on or around the sidewalls of CNT. The potential application of this approach is that the nanotubes might be sorted out according to diameter.

Basiuk et al.[365a] studied the possibility of reversible modification of CNT sidewalls with metal complexes, such as Ni− and Cu−tetramethyl tetraazaannulene (TMTAA), by taking advantage of the stacking process. Despite the aromatic nature of the ligand, its geometry is distorted from the plane because of the presence of four methyl substituents interfering with the benzene rings. As a result, the molecule adopts a saddle-shaped conformation, with the CH_3 groups and benzene rings turned to opposite sides of the MN_4 coordination plane. This geometry was especially attractive, since it roughly matches the curvature of small-diameter tubes. By the same $\pi-\pi$ stacking mechanism, electroactive complex Prussian blue was found to interact effectively with the graphitic network of CNT.[365b]

Studies of the interactions between CNT and biological samples are still limited. The group of Dai demonstrated that oxidized CNT were able to complex proteins by electrostatic interactions and could act as molecular transporters. Proteins were internalized into the cells via the endocytosis mechanism, and they exerted their biological activity once released from the endosomes.[174b] Mattson et al.[366a] reported the feasibility of using CNT as a substrate for neuronal growth. Neurites could grow on and extend from unmodified multi-walled CNT. More elaborate neurites and branching were formed when neurons were grown on MWNT coated by physisorption of 4-hydroxynonenal. This work suggested the biocompatibility of CNT as a substrate for neurons. One extension of this study is the use of CNT for the potential preparation of neural prosthesis. CNT are not biodegradable, and they could be used as implants where long-term extracellular molecular cues for neurite outgrowth are necessary, such as in regeneration after spinal cord or brain injury. In a different approach to the same issue, functionalized CNT were deposited onto glass coverslips. The functional groups were removed by heating, after which neurons were deposited on the regenerated, pure CNT. It was found that postsynaptic currents and the firing activity of the neurons grown on CNT were strongly increased as compared to the case of a pure glass substrate.[366b]

Supronowicz et al.[367] reported the application of nanocomposites consisting of blends of polylactic acid and CNT that can be used to expose cells to electrical stimulation. The current delivered through these novel current-conducting polymer−nanophase composites was shown to promote osteoblast functions that are responsible for the chemical compositions of the organic and inorganic phases of bones. By using the above polymer as matrix, Khan et al.[368] performed a study to evaluate the feasibility of CNT-based composites for cartilage regeneration and *in vitro* cell proliferation of chondrocytes.

It was also shown that multi-walled nanotubes can be used as scaffolds in tissue engineering.[369a] Their potential application in this field was confirmed by extensive growth, spreading, and adhesion of the mouse fibroblast cell line L929. Weisman and co-workers[369b] have studied the growth of mouse cells in the presence of nanotubes. It was shown that significant quantities of SWNT could be ingested by macrophages without any toxic effects. Moreover, the ingested tubes remained fluorescent and were imaged at wavelengths above 1100 nm.

5. Endohedral Filling

Among the wide number of studies on CNT, the ability to fill their inner cavities with different elements[370] was extensively investigated for producing nanowires or for efficient storage of liquid fuels. Research was first devoted to filling arc-produced multi-walled nanotubes.[371] It was predicted that any liquid having a surface tension below $\sim$180 mN·m^{-1} should be able to wet the inner cavity of tubes through an open end in atmospheric pressure.[371c] In the case of high surface tension, a highly pressurized liquid must be used to force it to enter inside the cavity.

Attempts were made to fill MWNT *in situ*, by subliming metal-containing compounds during the growth process.[372] In the following section, the various examples of filling CNT will be discussed in detail.

5.1. Encapsulation of Fullerene Derivatives and Inorganic Species

In this section, only SWNT have been considered. The groups that first observed the filling of SWNT[373] worked with C_{60}[374] and inorganics[375,376] as encapsulated species. Concerning the fullerene case, the pioneering study[374a,b,c] showed that the so-called peapods formed spontaneously as byproducts during the purification of raw nanotube material using the pulsed laser vaporization (PLV) method. Other groups have observed fullerene peapods in as-prepared tubes formed by catalyzed carbon arc evaporation.[374d,e]

The controlled synthesis of high amounts of peapod-like structures was achieved starting from oxidized SWNT in the presence of added fullerenes under vacuum at high temperature (400−600 °C), giving yields in the range 50−100%.[377] The rather low sublimation temperature of fullerenes and their thermal stability make the above method suitable for C_{60} peapod fabrication.

The fullerene-filled nanotubes have been characterized spectroscopically,[378a,b] and their electronic properties were studied in detail.[378c] During electron beam irradiation within an electron microscope, peapods underwent remarkable transformations, such as dimerization, coalescence, and diffusion of C_{60} molecules.[374,379] Iijima and co-workers[379b] studied the thermal behavior of fullerene peapods at temperatures approaching 1200 °C. The authors observed full coalescence of the fullerene molecules within the tube cavity, leading to formation of double-walled CNT. The resulting assembly was fully characterized with Raman spectroscopy,[379d,e,f] while the structural transformation was followed by X-ray diffraction analysis.[379g] The intertube spacing between the two graphitic layers was found to be about 0.36 nm.

Concerning the fabrication of fullerene peapods with alternative strategies, researchers have succeeded in encapsulating fullerenes into single-walled tubes by using alkali-fullerene plasma irradiation.[380] High filling of CNT with fullerenes in solution phase at 70 °C was reported by the groups of Iijima[381a] and Kuzmany.[381b] Exohedrally functionalized fullerenes were instead inserted into SWNT in a solution of supercritical carbon dioxide (sc-CO_2).[382] The authors demonstrated the formation of peapod structures by doping nanotubes with a methanofullerene $C_{61}(COOEt)_2$[382a,b] or fullereneoxide $C_{60}O$[382c] in sc-CO_2 at 50 °C under a pressure of 150 bar.

Not only has C_{60} been inserted into the cavity of nanotubes, but also some higher order carbon spheres, such as C_{70},[383] C_{78}, C_{80}, C_{82}, and C_{84}.[383a] X-ray diffraction measurements indicate 72% filling with C_{70} molecules as a total yield. Using TEM, the encapsulation of an endohedral metallofullerene $La_2@C_{80}$ was demonstrated by Smith et al.[384a] Other examples of metallofullerenes inside nanotubes include $Gd@C_{82}$,[377b,c] $Sm@C_{82}$,[384b] $Dy@C_{82}$,[384c] $Ti_2@C_{80}$,[384d] $Gd_2@C_{92}$,[384e] $La@C_{82}$,[384f] $Sc_2@C_{84}$,[384g] $Ca@C_{82}$,[384h] and $Ce@C_{82}$.[384i] Atoms inside fullerenes can be clearly seen as dark spots in microscopy images, whereas the metallofullerene itself exhibits an unusual type of rotational motion inside the confined space. Raman spectroscopy of such peapods gave evidence of polymerization of the encapsulated species, while the upshift in nanotube bands implies that a charge transfer between the host and the guest might occur.[385] By using a low-temperature STM, Shinohara and co-workers[386] proved that the endothermic insertion of metallofullerenes into the cavity of nanotubes modulates spatially the nanotube electronic band gap. Using this approach, an array of quantum dots was fabricated, with potential applications in nanoelectronics, such as solid-state quantum computers.[387]

Besides fullerenes, other materials introduced into the nanotube cavity include pure elements, inserted most often in a two-step process. A metallic salt was first inserted endohedrally, by using a suitable solvent, or in its molten state, and it was subsequently transformed into its reduced form (metal) by heat treatment in a hydrogen atmosphere or by photolytic reduction. The main advantage of this approach is that the heat treatment of nanotubes and the salt is close to room temperature. By these strategies, nanowires of CNT doped with Ru,[375] Bi,[388] Ag,[389] Au,[389c] Pt,[389c] Pd,[389c] Co,[390a] and Ni[390b] have been fabricated. The goal was to produce nanowires which could be used in applications for electric current transport. The only surprising result comes from a work of Zhang et al.,[389b] where the authors claim that silver nanowires can be obtained after heat treatment of silver nitrate peapods in air atmosphere, though, under these conditions, silver oxide might be produced. An alternative way to insert metals into nanotube cavities is by a plasma ion irradiation method. Through this approach, Cs atoms have been intercalated and evidenced by microscopy techniques.[391] Incorporation of iodine atoms in the form of helical chains inside single-walled nanotubes has been reported by Fan et al.[392] The authors immersed CNT in molten iodine and observed the peapod structures by TEM. One of the most exotic applications of CNT filled with a molten metal was the preparation of miniaturized thermometers. The group of Bando described how the height of a column of liquid gallium inside nanotubes varies linearly and reproducibly in the temperature range 50−500 °C.[393]

Beside doping with pure elements, CNT can also be filled with metallocenes, such as ferrocene, chromocene, vanadocene, cobaltocene, and ruthenocene.[394] The insertion occurs from the vapor phase of the sandwich-type species with formation of linear metallocene chains inside the tubes.[394a] The cobaltocene is observed to fill only nanotubes of one specific diameter, whereas the metal ion seems to interact with the nanotube surface through electron transfer.[394b] Similarly, Kataura et al.[383b] reported the fabrication of Zn-diphenylporphyrin peapods within CNT cavities. Optical absorption and Raman spectra suggested that the encapsulated molecules were deformed by interaction within the CNT.

Concerning encapsulation of inorganic salts, carborane molecules[395a] and K/Cs hydroxides[395b] were imaged inside CNT both as discrete species and as monodimensional chains of zigzag type. By treating the peapod structure of the K/Cs hydroxide with water, it was observed that the filling is removed and the resulting tubes can be refilled by other salts. Another class of compounds encapsulated are metal halides such as $(KCl)_x(UCl_4)_y$ and $AgCl_xBr_y$,[389a] CdI_2 and $ThCl_4$,[396] $CdCl_2$,[396,397a] $TbCl_3$,[397a] TiCl and PbI_2,[397b] CoI_2,[398] $LaCl_3$ and LaI_3,[399] KI,[389d,397a,400] $ZrCl_4$,[401] $AgCl_xI_{1-x}$,[402] BaI_2,[403] and $MoCl_5$ and $FeCl_3$.[404] In most cases, these fillers were admixed with CNT in their molten state within a sealed ampule or they were sublimed. Electron beam irradiation of such peapod structures induced cluster formation within the filling material, due to sequential elimination of the anions.[401]

In an alternative one-step approach, nanotube opening/filling took place by photolyzing a suspension of raw material in chloroform, in the presence of various metal chlorides.[404] After the irradiation, dark short wires were observed in the microscope images, assigned as fillings in the tube cavities. The structural changes of inorganic nanocrystals within the confined space of tube cavities have been thoroughly analyzed.[405]

CNT have also been studied as potential electrolyte transport channels in biological systems.[406] Molecular dynamics simulations showed that ion occupancy inside uncapped nanotubes is very low. When partial charges were placed on the rim atoms of the tube and an external electric field was applied, it was found that an aqueous solution of potassium chloride electrolyte could occupy the space inside the nanotube channel. In a subsequent experimental work, researchers have demonstrated the transport of Ru ions in aqueous medium through the channels of a thickness-aligned CNT membrane embedded in a polymer matrix.[407] The flux of Ru ions passing through the membrane was determined by cyclic voltammetry. Molecular transport through CNT cores could be gated by modifying the open nanotube tips with certain biomolecular complexes such as streptavidin−biotin.

Various metal oxides have also been inserted inside the cavities of CNT, including CrO_3[389e,408] and Sb_2O_3.[409] In the case of chromium oxide, a solution approach was adapted, in which the filling material interacts with the acid medium at room temperature. The tips of the nanotubes were opened by oxidation, and the oxide was inserted in the cavity of the tubes, though there was great uncertainty about the oxidation state of the chromium in the peapod structure.

Reaction of SWNT with organic molecules having large electron affinity and small ionization energy was found to result in p- and n-type doping, respectively.[410] Optical characterization revealed that charge transfer between SWNT and molecules starts at certain critical energies. X-ray diffraction experiments revealed that molecules are predominantly encapsulated inside the tubes, resulting in an improved stability in air atmosphere.

5.2. Encapsulation of Biomolecules

Open-ended multi-walled nanotubes provide internal cavities (2−10 nm in diameter) that are capable of accommodating biomolecules of suitable size. It has been shown that small proteins, such as lactamase, can be inserted into the internal cavities of tubes.[411] Comparison of the catalytic activities of immobilized enzyme with those of the free species in the hydrolysis of penicillin showed that a significant amount of the inserted lactamase remained catalytically active, implying that no drastic conformational change had taken place. DNA could also enter into the CNT cavities, and DNA transport has been directly followed by fluorescence spectroscopy.[412] Molecular dynamics simulations showed that a DNA oligonucleotide consisting of eight bases could be encapsulated into CNT in aqueous medium.[413] Both van der Waals and hydrophobic forces were found to be important for the dynamic interaction of the components.

Yeh et al.[414] have studied the electrophoretic transport of single-stranded RNA molecules through the 1.5 nm wide pores of CNT membranes by molecular dynamics simulations. Without an electric field, RNA remains hydrophobically trapped in the membrane despite the large entropic and energetic penalties for confining charged polymers inside nonpolar pores. Differences in RNA conformational flexibility and hydrophobicity result in sequence-dependent rates of translocation, a prerequisite for nanoscale separation devices.

5.3. Encapsulation of Liquids

A particular area of interest is the use of carbon tubes in nanofluidics applications. Nanofluidics is envisioned as a key technology for designing biomedical devices, in which the dominant transport process is carried out by natural and forced convection. As a starting point, the interaction of water with the interior cavities of CNT has been studied. A fundamental issue is the ability of a solvent to wet the hydrophobic channels, as this would facilitate solution chemistry inside the tubes.[415] The behavior of water molecules encapsulated into CNT has been studied by molecular dynamics simulations. The effects of confinement on the hydrogen bond structure were modeled, and the results indicated that the average number of hydrogen bonds decreases by comparing with bulk water.[416] In the very narrow tubes, the bond network was found to suffer a dramatic destruction, and in some cases, water molecules formed long linear chains.[416c]

Another parameter that was used in the simulation studies was pressure. It was found that, by applying axial pressures from 50 to 500 Mpa, water can exhibit phase transition into new ice formulations inside a tube.[417] At the same time, Hummer and co-workers[418] reported the spontaneous and continuous filling of a 0.8 nm diameter cavity by a one-dimensionally ordered chain of water molecules, using a molecular dynamics simulations approach. The authors suggested that CNT might be exploited as unique molecular channels for water. In other theoretical papers,[419] it was proposed that a single-water chain within CNT can be formed only in narrow diameter cavities (less than 0.811 nm) under physiological conditions. In the wider nanotubes, water appears to be arranged as a stacked column of cyclic hexamers.[419b]

Experimental observation of encapsulated aqueous fluid inside hydrothermally synthesized CNT was reported by Gogotsi et al.[420] By electron irradiation heating, the liquid inclusion was shrunk, due to evaporation inside the tubes. By applying parallel molecular dynamics simulations, Werder et al.[421] studied the behavior of water droplets confined in CNT. Contrary to the wetting behavior observed experimentally,[420] the results of the study indicated that no wetting of the pristine nanotubes occurred at room temperature.

6. Concluding Remarks

The chemistry of CNT is a current subject of intense research, which produces continuous advances and novel materials. However, the controlled functionalization of CNT has not yet been fully achieved. Solubility continues to be an issue, and new purification and characterization techniques are still needed. It is hoped that, with the effort carried out in many laboratories, we will be able to witness full control of size and shape, with new interesting applications in composites and electronics.

7. Acknowledgments

Part of the work reviewed here was carried out with partial support from the EU (RTN network “WONDERFULL” and “FUNCARS”), MIUR (PRIN 2004, prot. 2004035502), the University of Trieste and CNRS.

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CR050569O