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# Carbon Nanotube-Based UV-Curable Nanocomposite Coatings

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Additional information is available at the end of the chapter

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## Abstract

This chapter covers the preparation and properties of ultraviolet (UV)-curable nanocomposite containing carbon nanotubes as fillers. UV-curing technology is of particular interest due to its unique properties such as rapid curing process and solvent-free formulation. Alongside with the advantages of utilizing this curing method, carbon nanotubes undergo benefits including high aspect ratio, high transparency, and good mechanical properties. Carbon nanotubes (CNTs) are hollow cylindrical shaped configuration; consist of one, two, or more walls with an interlayer of non-covalent van der Waals force acting among the carbon atoms of various walls. Besides influencing the UV curing process, the CNTs loaded UV-curable nanocomposites sustain modified surface, thermal, mechanical, physical, and conductive properties which are discussed in this chapter. The health and safety concerns of using these classes of nanocomposite are further discussed.

**Keywords:** Carbon Nanotubes, UV-Curable Coatings, Nanocomposites, Physical and Mechanical Properties, Ultraviolet Radiation

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## 1. UV-curable nanocomposite coatings

In recent years, polymer nanocomposite coatings have found many applications due to their high strength, light weight, good fatigue, corrosion resistance, and superior optical properties and appearance, controlled anisotropic properties and low costs [1–5].

The addition of inorganic materials to polymers is an important method to produce materials benefiting each individual component properties. These composite materials are determined by the components behavior, the degree of dispersion and the interfacial properties [6]. This multi-functionality is important because polymer coatings should not only propose decorative

or protective functions, but also should provide other demands such as electronic discharge, superior mechanical properties, large operating temperature range, and good chemical resistance [7–9].

Among nanocomposite preparation methods, ultraviolet (UV)-curing technique is largely employed to produce hybrid materials, especially in form of films, developing polymeric thermoset matrices. UV light allows a fast transformation of liquid monomer to solid films with tailored physicochemical and mechanical properties. This is known as a fast, environmental friendly method. Unlike traditional thermal curing, the substrate should not be heated for curing, so it can be finished at room temperature, no solvent is used and the complete conversion is obtained in seconds [6, 10–17].

Generally, nanocomposites are divided into two main categories: nanocomposites with filler directly incorporated inside, and in-situ production of fillers.

The properties of final UV-cured nanocomposites are dependent on factors such as properties of each component, size and shape of the fillers, morphology of system, and the nature of interface between components. Their properties are as follows: high stiffness, high strength, and high dimensional stability, increased strength and toughness, high distortion temperature, good mechanical damping, increased permeability to gasses and liquids, excellent electrical properties, and low manufacturing costs.

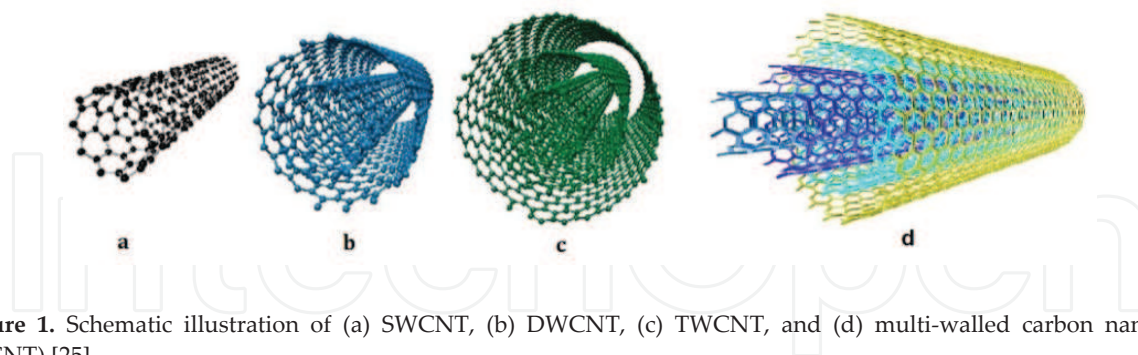
In UV-curable nanocomposites, one should consider the absorption, refraction, and reflection of UV light by the reinforcing fillers for not reducing the curing rate and conversion [18].

## 2. CNT-based UV-curable nanocomposites coatings

Among reinforcing fillers, carbon nanotubes (CNTs), appears as an interesting candidate [11, 12]. CNTs are gaining scientific and industrial interests due to their outstanding characteristics such as high tensile strength and modulus, diffusion and transport properties, antivibration and damping properties, low intensity and excellent electrical and thermal properties [19–23].

CNTs can be considered as graphene sheets rolled to form a tube. Depending on synthesis route and reaction parameters, single-walled carbon nanotubes (SWCNTs), double-walled CNTs (DWCNTs), triple-walled CNTs (TWCNTs), and multi-walled CNTs (MWCNTs) can be formed (**Figure 1**). MWCNTs are grown of several independent tubes in concentric circles. The electronic properties are depending on the number of graphene walls [22, 24, 25].

Various methods have been reported for CNTs syntheses, arc discharge [26], laser ablation [27–29], and chemical vapor deposition [30–32]. Arc discharge is the arc evaporation of graphite in the presence of inert gas, so the CNT is formed on electrode during quenching. The second method, laser ablation, vaporization of graphite is targeted by laser, then, CNTs are formed on receiver during quenching. The chemical deposition method which is the most used consists of decomposition of hydrocarbons over transition metal catalyst to form CNT [33].



**Figure 1.** Schematic illustration of (a) SWCNT, (b) DWCNT, (c) TWCNT, and (d) multi-walled carbon nanotube (MCNT) [25].

The diameter of CNTs is in the range of 5–40 nm, which is 2000 times thinner than a human hair. The length is usually about 10  $\mu\text{m}$  and their aspect ratio is in the range of hundreds to thousands and also they have strength 10–100 times higher than the strongest steel at a fraction of weight and possess electrical current transfer capacity 1000 times greater than copper wire [1, 6, 13].

Among conductive fillers, CNTs offer the highest aspect ratio lead to higher transparency and better mechanical properties than carbon black or metal nanofillers. The only consideration to make is their black color and also the need of lowest possible concentration in formulation [24]. Besides, CNTs bear self-aggregation and poor solubility in water and organic solvents [34, 35]. In order to overcome these drawbacks, several approaches such as non-covalent and covalent functionalization have been reported. Covalent functionalization is grafting macromolecules using both “*grafting to*” and “*grafting from*” methods. These approaches make the CNTs to exhibit hydrophilic, hydrophobic, and amphiphilic properties [20, 21]. Non-covalent functionalization approach includes solution mixing [36–38], melt mixing [39, 40], and in situ polymerization [22]. It should be notice that the electrical properties of CNTs are influenced by their structure and diameter of nanotubes [41].

CNTs-based nanocomposites are used in many application including strain sensors [42], damage sensors [43], gas sensors [44], electromechanical actuators [45], conducting plastics-photovoltaic devices [46], optoelectronics [47], electrostatic dissipation [48], electromagnetic interference shielding [49], optical barriers [50], cost-effective transparent electronics [46], composite mirrors [51], plastics with high thermal dissipation, and biomaterial devices [52, 53]. They also have optical properties that fall in to the following categories: photoluminescence, light emission and photonic properties, optical non-linearity, and optical limiters [54–57].

### 3. Preparation of CNT-based UV-curable nanocomposites

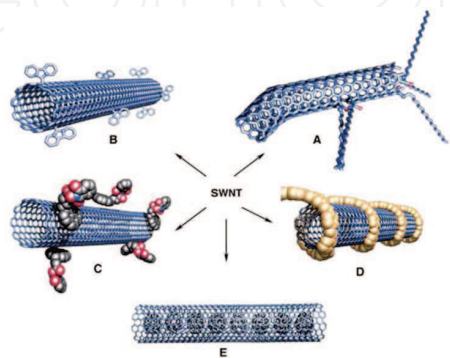
To have a desirable CNT-based nanocomposite, homogeneously dispersion of CNTs within the polymer matrix is required [58, 59]. It is also very important to stabilize the dispersion in order to prevent aggregation of CNTs [60, 61]. Sonication and mixing are known to result in a

good dispersion, but, the most proper method is surface functionalization of CNTs [62–66]. Though several mixing routes are known to disperse CNTs in polymer matrices including dry powder mixing, solution blending, melt mixing, in-situ polymerization and surfactant-assisted mixing [67–71]. **Table 1** represents the advantages and disadvantages of known CNT functionalization methods. By functionalizing CNTs, a direct bonding between the nanotubes and polymer is formed [72]. Researchers have shown that the high dispersion of these CNTs is achieved by sonication [20]. This functionalization is known to enhance the interface and load-transfer in CNTs/polymer nanocomposites. **Figure 2** reveals the functionalization of SWCNTs by two macromolecules.

Method		Principle	Possible damages to CNTs	Interaction with polymer matrix	Reagglomeration f-CNTs in matrix
Chemical method	Side wall	Hybridization of C atoms from $Sp^2$ to $sp^3$	*	Strong	*
	Defect	Defect transformation	*	Strong	*
Physical method	Polymer wrapping	Van der Waals forces, $\pi$ - $\pi$ stacking	–	Variable	–
	Surfactant adsorption	Physical adsorption	–	Weak	–
	Endohedral method	Capillary effect	–	Weak	*

**Table 1.** Advantage and disadvantage of various CNT functionalization routes [33].

Commonly, solution method, a combination of dispersing CNTs in a liquid medium by sonication, mechanical, and magnetic stirring mixing by the polymer solution, and finally evaporating solvents is suggested to prepare a CNT-based UV-curable nanocomposite [7, 19, 54].



**Figure 2.** Different functionalized SWCNTs [73].

Sonication is considerable step for breaking aggregates and entanglements of CNTs [20, 74, 75]. Homogeneously dispersed MWCNTs UV-curable resin by solution mixing method can be achieved (**Figure 3**) [6].



**Figure 3.** SEM rift image of the morphology of AG/MWCNTs UV-cured nanocomposite [6].

#### 4. Characterization methods of CNT-based UV-curable nanocomposites

To characterize a CNTs/polymer nanocomposite, both the dispersion of nanofillers and the interface between components must be studied. These two factors have massive influence of the final nanocomposite properties.

The morphology investigation by the mean of dispersion is achieved by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) [19, 21, 41].

The photo curing process is perused by photo-differential scanning calorimetry (photo-DSC) [10, 19, 21] and calculating gel content [7]. The kinetic of photopolymerization of nanocomposites is provided by real-time Fourier transform infrared spectroscopy (RT-FTIR).

Viscoelastic characterization, glass transition temperature ( $T_g$ ), Tan delta (the value of loss factor), and damping values of the cured nanocomposites are carried out by dynamic mechanical thermal analysis (DMTA) and DSC [19, 23].

Thermal stability of the cured nanocomposite and its hardness can be investigated by thermogravimetry [19] and nanoindentation [7, 19], pencil test [13], respectively.

#### 5. Properties of UV-cured nanocomposite coatings based on CNT

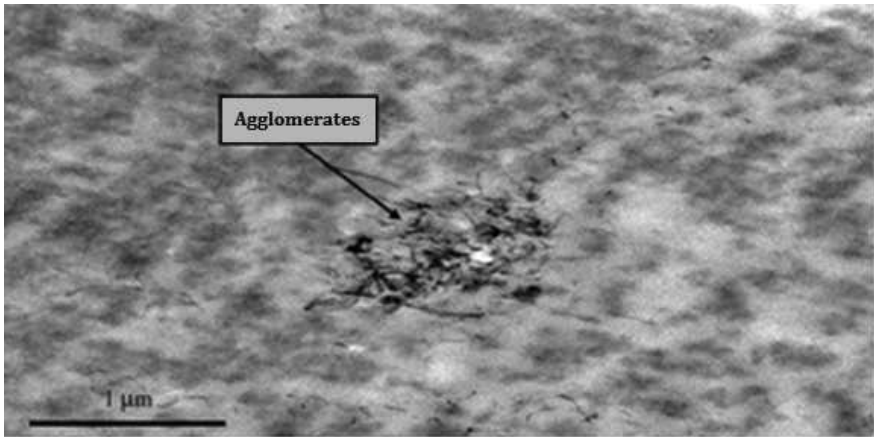
In this section, the properties of CNTs based UV-cured nanocomposites are discussed. As mentioned before, the final properties are influenced by the dispersion stated and interface between CNTs and polymer. Having a well-dispersed CNTs within the polymer matrix results



in good mechanical, physical, electrical, and thermal properties which individually will be discussed. The addition of these filler to the polymeric matrices, influence the UV-curing process, though enhance the final properties of the loaded composite such as conductivity, surface and tribological properties, thermal and physical-mechanical of the cured nanocomposite.

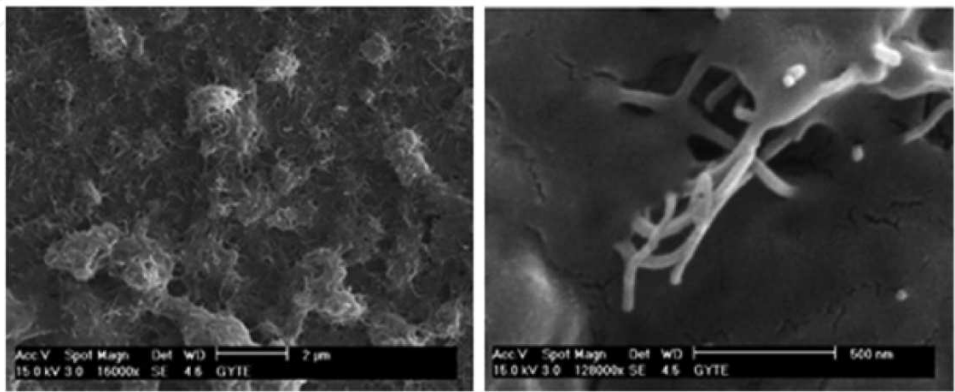
5.1. Dispersion

The homogeneity and stability of CNTs in nanocomposites plays a significant role in the final product. **Figure 4** represents the TEM images of a CNTs/polymer nanocomposite. It reveals even after sonication, some agglomerates are present. This suggests the use of wetting and dispersing additives alongside with shear forces [10, 24].



**Figure 4.** The remaining agglomerates in TEM images even after sonication [19].

A good dispersion of CNTs within the polymer matrix (**Figure 5**) increases the physical and mechanical properties of nanocomposite coatings [21].

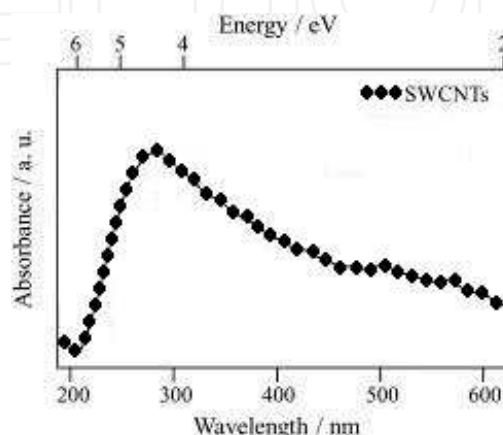


**Figure 5.** SEM images of CNTs/polymer nanocomposite indicating good dispersion and interaction between nanofiller and polymer matrix [21].

## 5.2. Curing process

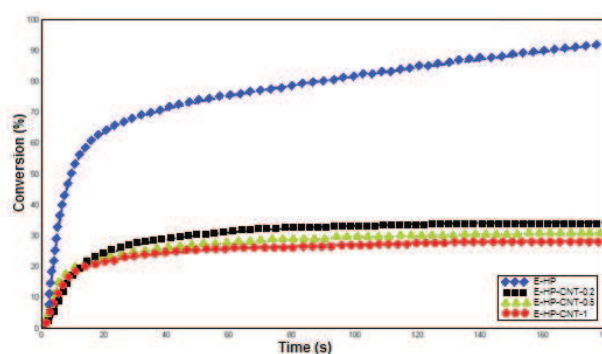
As mentioned in Section 1, the nanofiller may itself absorb the UV light, resulting to a competition between photoinitiator and reinforcing nanofiller which can led to a less effective curing process.

**Figure 6** illustrates the absorption spectroscopy results of SWCNTs. As can be seen, CNTs have light absorption in UV region.



**Figure 6.** SWCNTs absorption spectroscopy.

To overcome this drawback, one can use higher UV light intensities and longer irradiation times [7, 76]. Another approach to dominate this effect of CNTs. They used a hyper-branched polymer (HP) in their formulation which caused an enhancement in final conversion. Their results (**Figure 7**) reveal by the aid of HP in the CNTs/polymer nanocomposite, a higher conversion in shorter time can be achieved.

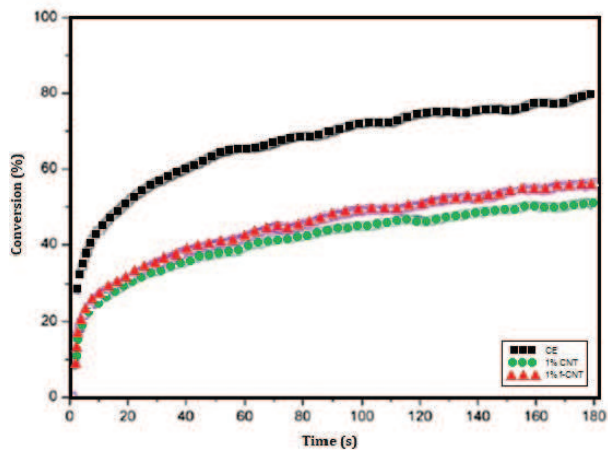


**Figure 7.** The effect of hyper-branches polymer in a CNT-based UV-curable nanocomposite [77].

Commonly used method for investigating curing process, kinetic of photopolymerization, and curing behavior of UV-curable polymer is by photo-DSC, RT-FTIR and measuring the gel content. In photo-DSC analysis, the heat of the photo-initiated polymerization reaction is measured [4, 21, 77], In RT-FTIR method, the conversion of active groups is followed by



monitoring the decrease in of those groups peaks in the conversion curves as a function of radiation time (**Figure 8**) [7]. In this curve, the rate of polymerization (slope of the initial linear segment) and the final value of conversion can be driven. It is perceived by using CNTs, both the polymerization rate and the final conversion are decreased, but utilization of functionalized CNTs, rises the  $T_g$  of the system due to the formation of crosslinked polymer network, hampering the mobility of active groups while the reaction progresses result in higher value of conversion. This effect is more pronounced in UV-cured nanocomposite containing oxidized CNTs.



**Figure 8.** FTIR conversion curves for the neat cationically photocurable epoxy resin (CE) and it’s composites at 1 wt% of multi-walled CNTs (1% CNT) and 1 wt% oxidized of multi-walled CNTs (1% f-CNT) content [7].

The gel content values of the cured coatings can be obtained by the weight loss after being adrift in solvent.

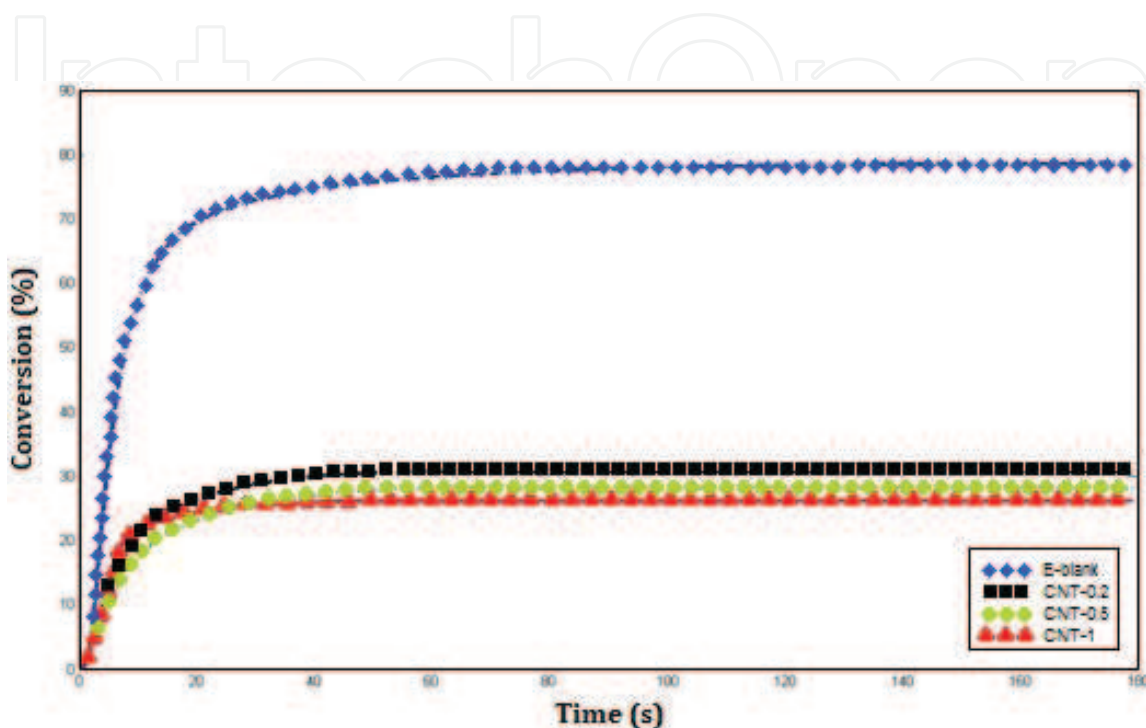
**Table 2** represents the polymerization rate and gel contents of the neat polymer and the loaded ones. The results suggest that the nanosize filler have great absorption of the UV-light, leading to a strong shielding effect. It is revealed that these fillers reduce the generation of reactive types at the initial of UV-induced reaction [7].

Sample	Polymerization rate <sup>a</sup> (s <sup>-1</sup> )	Conversion <sup>b</sup> (%)	Conversion <sup>c</sup> (%)	Gel content (%)
CE	9.6	80	100	100
1% CNT	5.5	51	98	99
1% f-CNT	5.6	51	88	100

<sup>a</sup>Values of the slope at the initial stages of the conversion-time curves.  
<sup>b</sup>Value of the plateau in the conversion curves with a UV-light intensity of 35 mW/cm<sup>2</sup> and an irradiation time of 3 min. Film thickness of 25 μm.  
<sup>c</sup>Determined by single spectra taken before and after 5 min of irradiation at 55 mW/cm<sup>2</sup>. Film thickness of 25 μm.

**Table 2.** Conversion values and gel content of the nanocomposites with 1 wt% loading fraction [7].

Furthermore, the addition in content of CNTs, influence the curing behavior of the nanocomposites. The more loading, leads to less curing conversion. This can be attributed to the competition of UV absorption between the fillers and photoinitiators [77]. This reduction in final conversion is shown in **Figure 9**.



**Figure 9.** Curing behavior of photocurable epoxy acrylate with different CNTs content [77].

### 5.3. Surface properties

Surface properties of a UV-cured nanocomposite is divided into surface hardness and its surface chemistry.

CNTs are unsaturated systems owning highly mobile electrons, depending on the polymer chemistry, there would be distinct interactions [24]. Likewise, the functionalization type and method is very important parameter to be controlled.

### 5.4. Tribological properties

One indication of good dynamic mechanical property of nanocomposite can be the elevated surface hardness [7, 19]. Surface hardness can be measured by nano-/micro-indentation and pencil test [20]. CNTs nanofillers are known to increase this property. These results are shown in **Figure 10**.

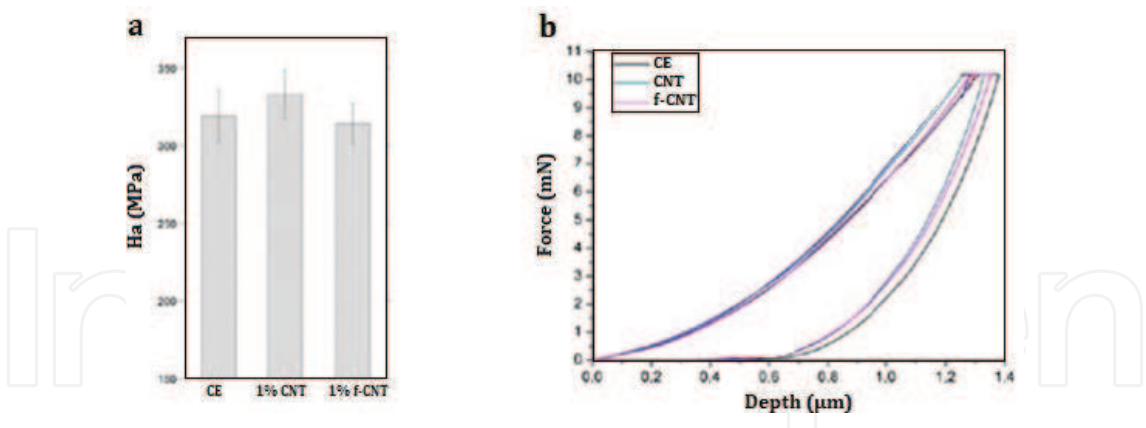


Figure 10. (a) Surface hardness values and (b) load vs depth curve [7].

CNTs have self-lubricating properties. In higher CNTs content in a nanocomposite, agglomerates are higher which increase the friction coefficient. Therefore, the tendency for matrix fragmentation is enhanced, producing loose wear debris at the contact interface [19]. **Figure 11** represents the friction coefficient of a UV-cured nanocomposite as a function of different loadings. It is observed that wear rate is raised by the increasing of CNTs content.

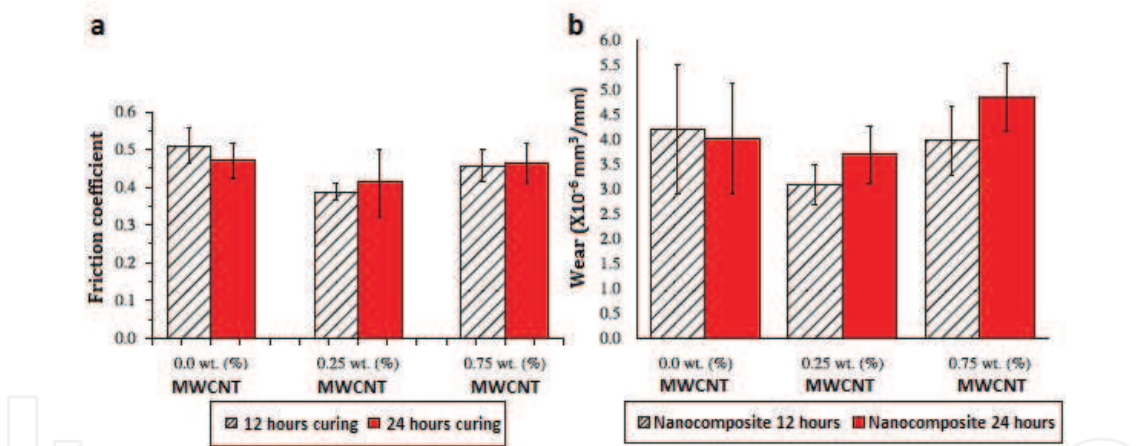


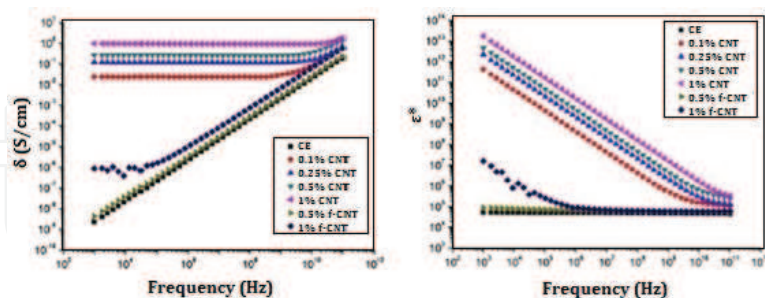
Figure 11. Friction coefficient (a) and wear rate (b) for CNT/polymer nanocomposite as a function of curing time by UV-A irradiation cycles 12 and 24 h [19].

### 5.5. Conductivity

CNTs embedded in polymer matrix, are protruded from the surface, offer permanent electrical, surface and volume conductivity [24]. In a CNT/polymer nanocomposite, the critical filler content is called the percolation threshold. By formation of a CNTs interconnected tridimensional network, a low-resistance path for the moving charge carriers is provided [54].

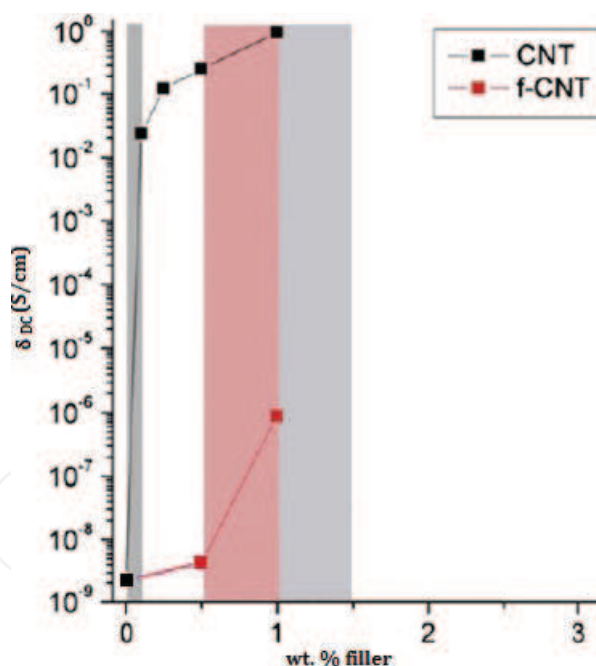
The geometry of nanofillers plays an important role in the formation of conductive pathways. Using CNTs would increase both electrical conductivity and dielectric permittivity of the cured

nanocomposite. This enhancement (**Figure 12**) is a proof of the formation of a conductive network in polymer matrix [7].



**Figure 12.** AC conductivity and permittivity vs frequency for nanocomposites at different loading [7].

By extrapolating the broadband AC conductivity, it is possible to obtain DC conductivity values (**Figure 13**). In this figure, the filled areas represent the electrical percolation region for each of nanocomposite differing in filler. It reveals that the most efficient nanofiller is MWCNT [7].

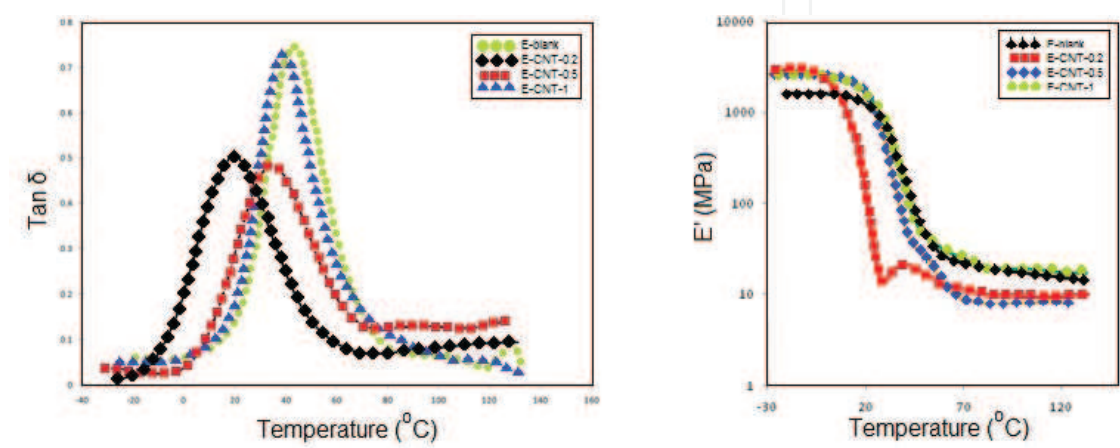


**Figure 13.** Values of the DC electrical conductivity of nanocomposite as a function of different nanofillers (CNT and oxidized-CNT) [7].

Besides electrical conductivity, industry is interested in advantages of thermally conductive polymer nanocomposite. These nanocomposites are light weight, corrosion resistant and easily processed, which make them better candidate than metal parts. Using CNTs with its superior efficient, thermal conductivity will overcome this demand [54].

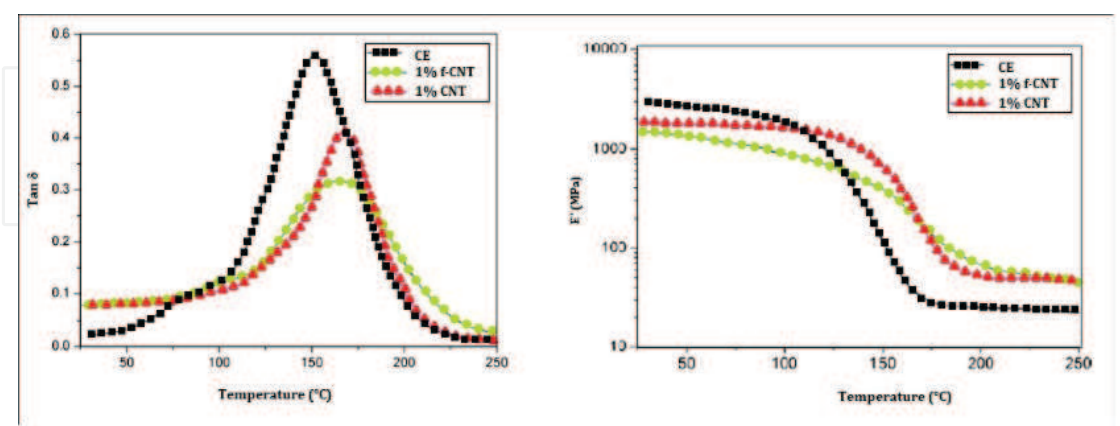
5.6. Dynamic mechanical properties

In analogy to neat polymers, filled nanocomposites are known to have higher  $T_g$ . **Figure 14** demonstrates the tan delta curves as a function of temperature for prepared nanocomposites. The increased  $T_g$  is followed by a shift in the maximum value of tan delta and a reduction in damping. It can be revealed that CNTs would withhold the polymer chain movements led to damping decrement and higher  $T_g$ . By increasing the CNTs content, a stronger nanofiller/polymer is attained, resulting in higher maximum value of tan delta and greater damping effect due to the covalent banding between the components [77].



**Figure 14.** Tan delta and E' curve obtained by DMTA analysis for nanocomposites containing CNTs and functionalized CNTs (f-CNT) [77].

Meanwhile, the changes in the modulus values driven by DMTA analysis are not significant (**Figure 15**), although a slight increase in rubbery region is detected [7].



**Figure 15.** Modulus obtained by DMTA analysis for different nanocomposites containing CNTs and f-CNTs [7].

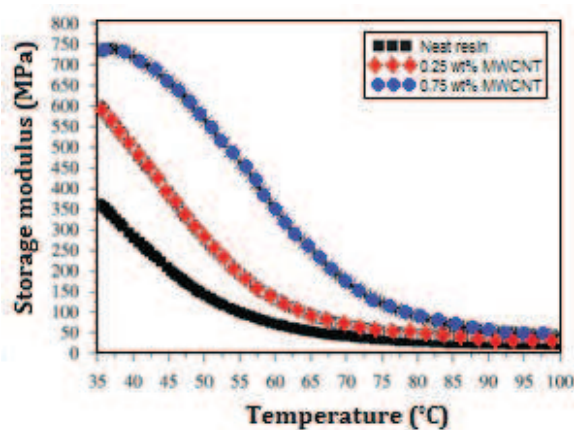
It is well known that the addition of CNTs will raise the storage modulus and stiffness of the nanocomposite (**Table 3 Figure 16**) [14, 19].



Samples	Storage modulus <sup>a</sup> (MPa)	Loss modulus <sup>a</sup> (MPa)	T <sub>g</sub> (°C)
Neat 12 h	428	141	55
0.25 wt% MWCNT 12 h	570	166	58
0.75 wt% MWCNT 12 h	551	154	63
Neat 24 h	288	110	53
0.25 wt% MWCNT 24 h	507	135	58
0.75 wt% MWCNT 24 h	732	152	68

<sup>a</sup>Data taken at 40°C.

**Table 3.** DMA results for neat polymer and nanocomposites [19].



**Figure 16.** Storage modulus for neat resin and its nanocomposite at different loadings [19].

In nanocomposites, CNTs frequently turn in their direction within the polymer matrix. This could improve the mechanical properties, likewise fatigue resistance or bending strength. Even in the case of no improving in mechanical properties, the good dispersion of CNTs would prevent any negative impression on it [24].

## 6. Health and safety concerns

Using nanocomposites carrying nanofillers, some consideration must be count from fabrication of CNTs to the final application of the nanocomposite. The consideration for health and safety of humans and environment.

The concern about CNTs is using its powder form and dusts. To prevail this issue, dispersions of polymeric wetting and additives of high molecular weight is suggested [24].

When embedded in polymer matrix, the concerns are about releasing of CNTs to the environment after the polymer degradation (**Figure 17**) [78].



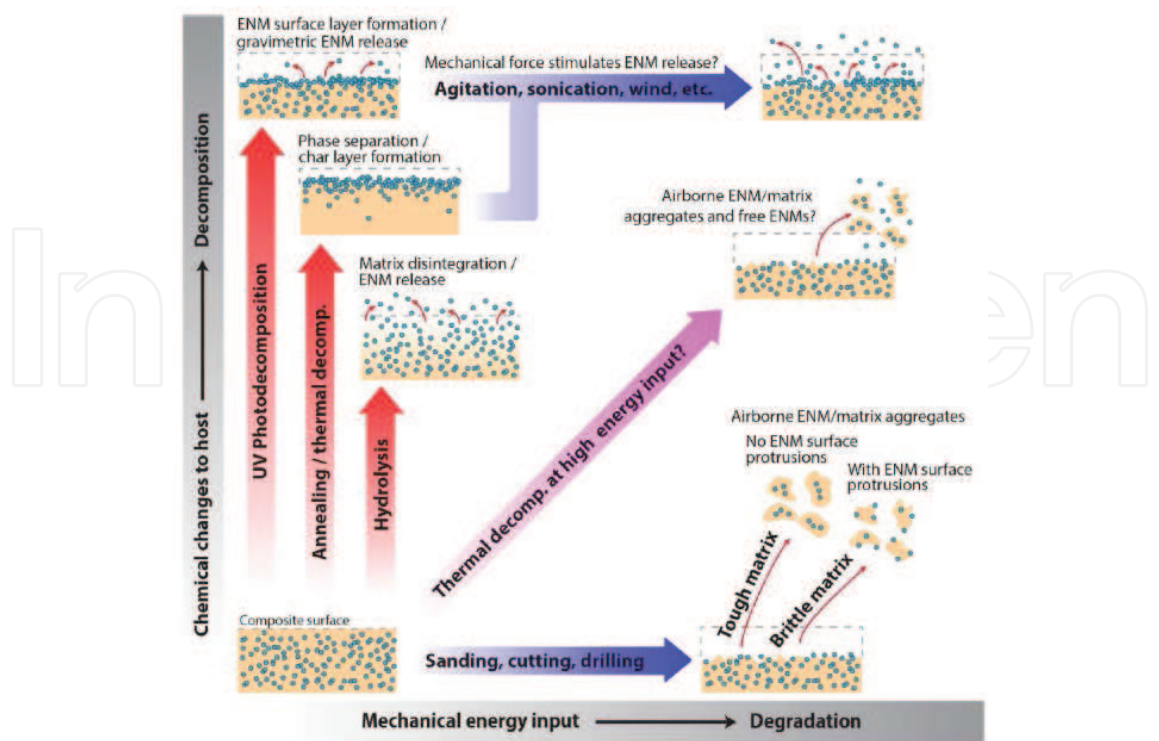


Figure 17. Schematic of releasing engineered nanomaterials (ENM) pathways from nanocomposite due to mechanical degradation or chemical decomposition of the host materials [79].

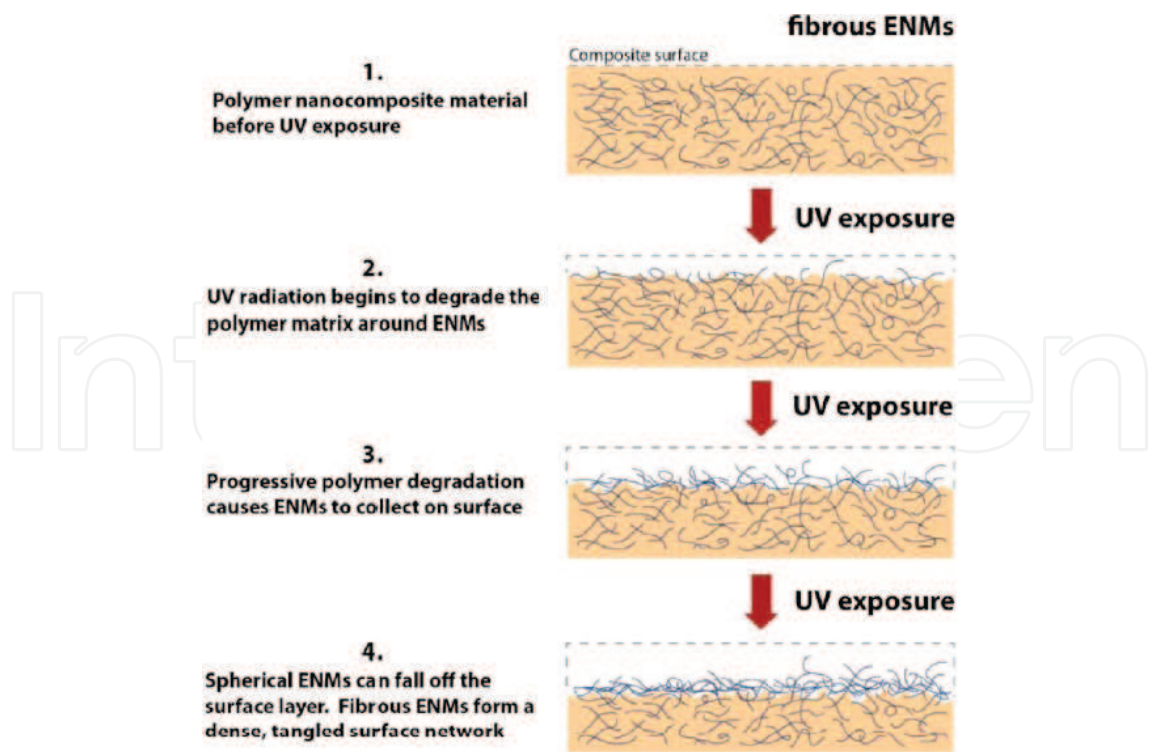
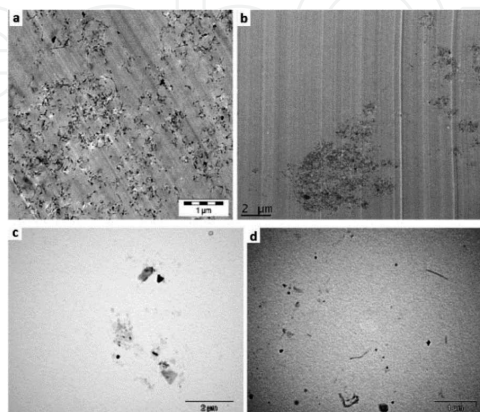


Figure 18. Conceptual model of engineered nanomaterial (ENM) released from UV-degraded polymer composite [79].

**Figure 18** represents the schematic of the UV exposure of nanocomposite filled CNTs and its releasing procedure.

Researchers showed a good dispersion of CNTs within the polymer matrix may decrease the releasing of nanofiller to the environment (**Figure 19**). It can be seen a large amount of fragments are released from the bad dispersed material.



**Figure 19.** Electron microscope images of CNT/polymer composite with (a) good dispersion and (b) bad dispersion. (c) and (d) show particles released from these material [79].

## 7. Conclusion

Filled UV-curable nanocomposites have gained attentions due to their low cost, improved properties and being a fast and green process. Among nanofillers, carbon nanotube with their unique properties as a nanofiller have been widely used. A challenge in these classes of nanocomposite is the dispersion of the filler within the polymer matrix. Dispersion and the interface between components play an important role in the final properties of the cured nanocomposite. A well-dispersed system led to better curing, enhanced mechanical properties, improved thermal, and electrical behavior. Finally, the degradation of these nanocomposites should take into account for the health of human and environment.

## Author details

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