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Graphene-based UV-curable nanocomposite coatings

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UV-curable Nanocomposite Coatings

Since middle of the 20th century, composite materials have been attracted a lot of interest in science and industry. A composite is defined as a combination of a matrix and reinforcement where the composites final properties are distinguished from each individual component, including high strength, low weight, good fatigue resistance, corrosion resistance and superior optical properties [1, 2].

Coating as a layer for protection or decoration is used to improve substrate adhesion, wettability and corrosion resistance. Using nanocomposites as a low cost and simply processed procedure than multi-layer coatings have satisfied the industry requirements [3-5].

In general, composite coatings are classified in two main categories: nanocomposites and microcomposites [6, 7]. Another classification is based on the preparation method, namely; composite with fillers directly incorporated inside them and composites formed *in-situ* through the production incorporation of fillers.

Amongst the nanocomposite preparation techniques, photochemical curing is desirable since being a solvent free process; requiring little energy, being a low temperature process, having high curing speeds, and being environmental friendly [8-10]. UV-curing induces fast formation of liquid monomers into solids including a wide gamete of favourable mechanical, physical and chemical properties [11].

Composites have gained much interest in wide application areas, such as biosensors, energy storage and electronic devices [12-16]. Here, loaded nanocomposite adopt augmented forms of the properties of each single component used, the size and shape of the reinforcing filler, the morphology of the system and the nature of interface between the components, all of which have advantageous properties such as high stiffness, strength and dimensional stability, proliferated impact strength and toughness, expanded distortion temperature, modified mechanical damping, increased permeability to gases and liquids, superior electrical properties alongside reduced manufacturing costs and reduced environmental impacts [17-37]

In UV-curable composite, one should consider UV refraction, reflection and absorption associated with the filler. This may prevent correct photo-initiator function and hence suitable curing. To overcome this problem, different strategies can be used such as increasing the intensity of the UV exposure, prolonging curing times and modifying the chemical formulation [38,96]. Hence, the unique properties of UV-curable nanocomposites, such as high speed production, chemical stability and better physical-mechanical properties of the final cured products, make it a desirable curing technique.

Graphene Nano Platelet-based UV-curable Nanocomposite Coatings

Nanofillers as reinforcing particles are used to give nanocomposites very unique properties [6]. Among the nanofillers, graphene platelets (GNPs) and it's various derivatives have attracted attention due to their impressive tensile modulus, strength, electrical conductivity, thermal conductivity, thermal and chemical stability, large surface areas and high aspect ratios [8, 10, 11, 39-41]. Graphene based nanocomposites afford thermal conductivities well suited for using in electronic devices, sensors, anti-static coatings, microwave absorbers, energy storagee devices, super-capacitors, optoelectrical properties including high transparency, applied in the touchscreens, organic light emitting diodes, liquid crystal displays and enhanced piezoelectric

properties [42-54, 61, 97]. These properties make this class of nanocomposites proper candidates for fuel cells, sensors, microwave absorbers, photovoltaics, photodetectors and pulsed lasers [55-63]. Graphene-based UV-curable nanocomposites exploit both the properties of UV cured polymers and Graphene. [6, 61]

The production of graphene nanoplatelets was first reported in 1970 [64, 65], but the first isolated free-standing single-layer graphene was reported in 2004 [66, 67], driven from the separation of graphite by micromechanical cleavage [68, 69], as reported elsewhere in this book.

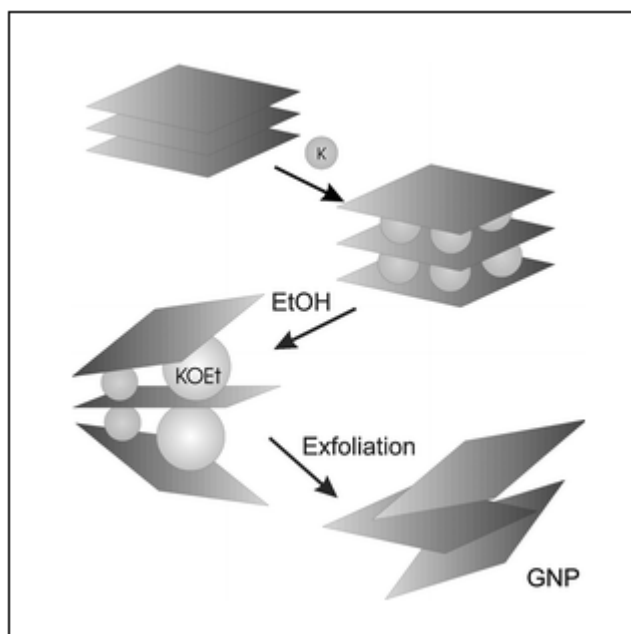
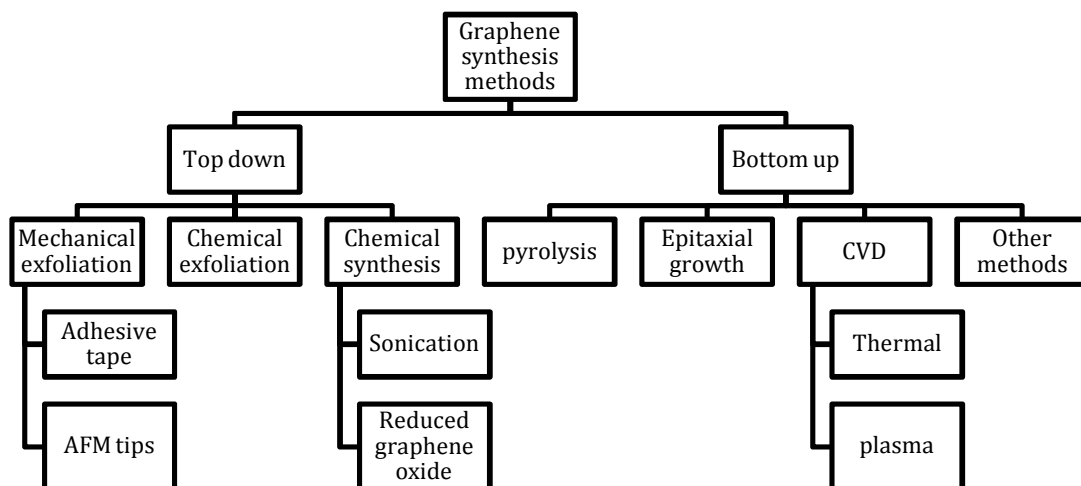


FIGURE 8.1

Schematic diagram of the production of GNP (by intercalation and exfoliation process). Graphite is intercalated with potassium metal to form the first stage compound KC8. Exfoliation in ethanol produces potassium ethoxide and hydrogen gas which aid in separating the graphitic sheets to form graphene nanoplatelets [70]

Graphene is a two-dimensional single-atom thick layer of carbon atoms which have adopted a honeycomb lattice [66,10,71]. Various routes were reported for producing graphene, however but only a few have resulted in the synthesis of high-quality graphene [72, 73]. These methods include mechanical exfoliation, thermal exfoliation and liquid-phase exfoliation of bulk graphite by sonication, as well as epitaxial growth, chemical reduction of graphite oxide, quenching, micromechanical cleavage of graphite using scotch tape, the Solvothermal synthesis and perhaps the most widely employed; Chemical Vapor Deposition (CVD) [4, 11, 59, 74].

Many of the properties of the nascent, pristine graphene are reflected in graphene based nanocomposites. Graphene sheets can be well distributed in polymeric systems resulting in transparent composites with enhanced thermal and electrically conductivity [75].

**FIGURE 8.2**

Summary of graphene synthesis methods [76]

Choosing the appropriate synthesis method is technology-driven and evidently critical for the realization of functionally enhanced composites [59]. A summary of all synthesis methods is shown in Figure 8.2. There are two main approaches; “*top down*” and “*bottom up*”. The first approach involves the production of graphene starting from bulk graphite, whereas in “*bottom up*” synthesis graphene is grown on various substrates which are subsequently removed latterly.

Oxidation of graphite, to form graphene oxide (GO) is perhaps one of the most common routes to generate oxygen-based functional groups on graphene nano sheets. As the oxygen groups on the surface of carbon sheets are hydrophilic, GO can, much to the benefit and for ease of processing, be easily dispersed in water. Thus there would be difficulty dispersing these sheets in organic solvents [77-79]. One approach to overcome this problem is functionalizing the graphene by carboxylic, epoxy and hydroxyl groups [74].

Another common method is grafting various macromolecules onto graphene [76]. It improves the hosts thermal stability, prevents the aggregation of graphene and renders covalent integration of graphene into complex organic systems. There are two strategies to reach this goal, grafting to and grafting from. “*Grafting to*” pertains to link pre-synthesized macromolecules to graphene, whilst “*grafting from*”, the growing of macromolecules from the surface of graphene [74, 80].

Preparation methods

Reaching a graphene based nanocomposites with superior properties relates to achieving a good dispersion of graphene into the polymer matrix and the interface of the polymer and filler [81]. Using graphite, good exfoliation is critical in achieving truly graphene-like monolayers that can be subsequently uniformly dispersed throughout the polymer matrix [59].

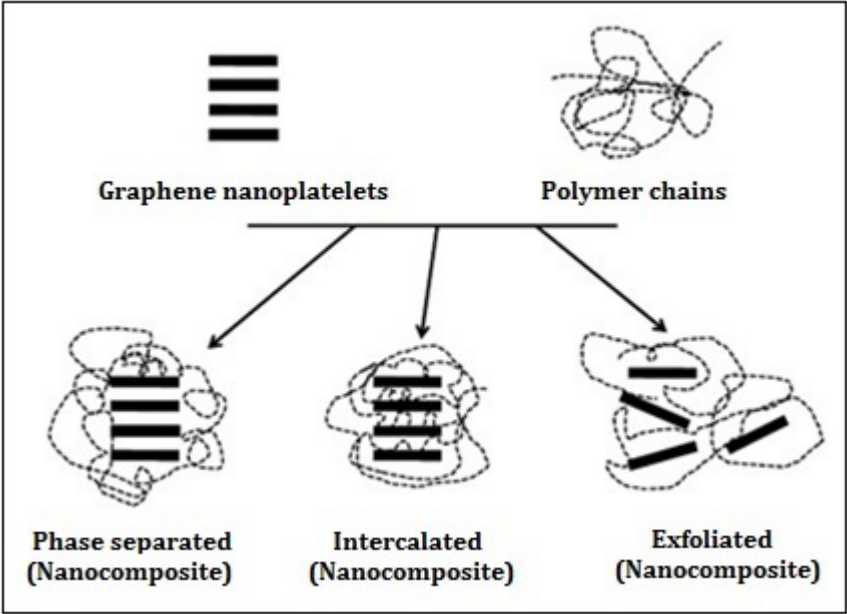


FIGURE 8.3
Three different nanocomposite based on different mechanism of graphene nanoplatelets through polymer matrices [82]

Figure 8.3 illustrates some different graphene/polymer nanocomposites. There are three commonly used methods for fabricating graphene-based nanocomposites, the first and perhaps the most widely adopted, largely due to its simplicity, is solution-based intercalation. Here the first step is dispersing bulk graphite in a media through ultrasonication and then dispersing this with the host polymer. Following this, the mixture is drop-cast, spin-coated or vacuum filtrated, to form a thin film that can be UV-cured to form the desired product [59]. Other common methods are melt intercalation and *in-situ* polymerization [83].

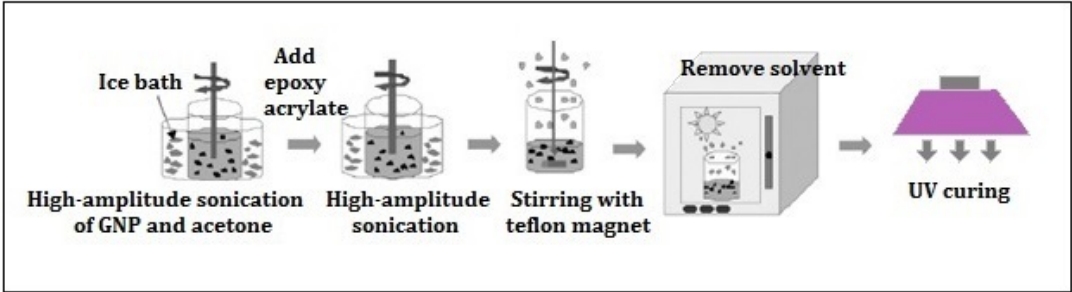


FIGURE 8.4
Fabrication process of a graphene/epoxy acrylate nanocomposite

Almost all solution-based approaches require mixing by sonication, as illustrated in Figure 8.4. As shown, after the dispersion and removing the solvent, the formulations are cured by UV exposure.

Achieving uniform dispersion is critical. Poor graphene dispersion within the polymer matrices not only lowers their reinforcing efficiency, but also causes inter-plane slippage within the graphene when externally loaded [103]. This is due to entanglement of graphene by intermolecular Van der Waals force which often causes them, as well as the other nanocarbon allotropes to agglomerate aggressively during solution preparation. Figure 8.5 shows the effect of graphene content in forming nanocomposites. By increasing the graphene concentration, the viscosity can be decreased following the formation of 3D networks within samples.

UV curing of this class of nanocomposites can offer superior advantages due to their easiness of dispersing graphene into the low viscosity liquid monomers prior to the curing process [11]. Compared to thermal curing, UV curing has results in fewer aggregates due to the rapidity of the process [8, 81, 84].

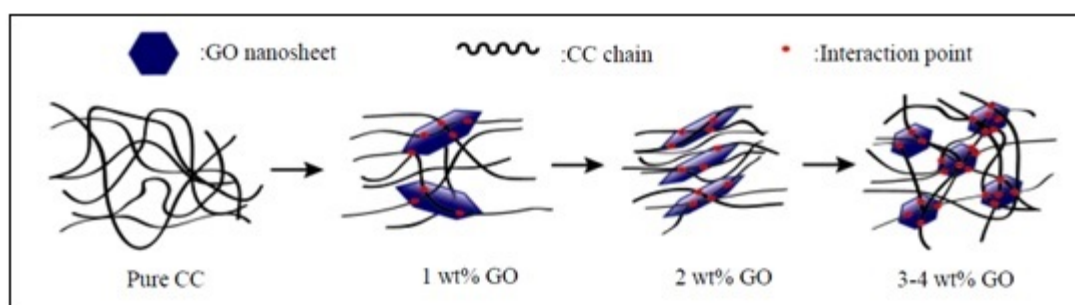


FIGURE 8.5

Schematic illustration of a cellulose carbamate (CC) solution containing different contents of graphene oxide (GO) [85]

So far, a diverse range of strategies have been reported to achieve good GNP dispersions including using surfactant and polyelectrolytes to enhance exfoliation, with most focusing on the underpinning physical or electrostatic interactions [86]. Another viable route to reach a homogenous dispersion state is by producing interfacial bonding between graphene and the matrix through chemical functionalization. Organic molecules attached to graphene surfaces may also be engineered to enhance the dispersion. One particularly effective route is the covalent attachment of a secondary polymer onto the graphene surface to prevent graphene aggregation [85].

Characterization methods

Various analytical methods have been pursued to study the dispersion, interface, curing process and properties of GNPs based UV-curable nanocomposites [75, 87, 88].

To investigate the dispersion state of graphene within the polymer matrix, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Transmission Electron Microscopy (TEM) have been widely used by Guo, Jaong and Eda et al. [8, 10, 11, 81].

To study the photopolymerization and UV-curing kinetics, photo-Differential Scanning Calorimetry (photo-DSC) have been employed, in addition to calculating the gel content value and measuring the Real-Time Fourier Transform Infrared spectroscopy (RT-FTIR) are applied [11, 75,96].

Studying the properties such as mechanical, physical, thermal and electrical properties of the cured nanocomposite are achieved by using Dynamic Mechanical Thermal Analysis (DMTA), Differential Scanning Calorimetry (DSC) and Thermo Gravimetric Analysis (TGA) [10, 11, 81, 89, 96].

Properties of GNPs- based UV-cured Nanocomposite Coatings

Dispersion state

Figure 8.6 exhibits the dispersion of graphene in UV-cured epoxy nanocomposite. As seen, nanofillers are embedded in the resin making an intercalated structure.

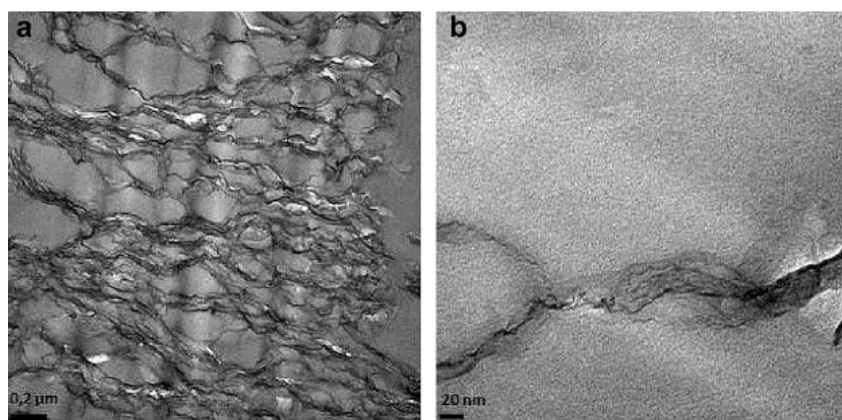


FIGURE 8.6

TEM images of graphene-epoxy nanocomposite at a 1.5 wt% filler content. Magnitude (a) 0.2 μm and (b) 20 nm [75]

Other methods to investigate the intercalated network of graphene within the polymer matrix are Raman spectroscopy and X-ray diffraction [101,102]. Raman spectroscopy is a powerful non-destructive tool to determine the graphitic quality of carbon materials [104]. The Raman G peak centred at 1581 cm^{-1} is indicative of a graphitic material. In GO, the G peak becomes broaden and shift to 1594 cm^{-1} . In addition, the D band at 1363 cm^{-1} becomes prominent as a result of extensive oxidation. An increased D/G intensity ratio is often observed upon reduction of the exfoliated GO.

UV-curing process

GNPs and all graphitic nanocarbons, absorb light in UV region [98]. The absorption of graphene in UV to visible region has been investigated by UV-Vis spectroscopy which is shown in Figure 8.7. It means existence of a competition between nanofiller and photoinitiator in absorbing UV which results in a less effective curing process. This reduction in the polymerization rate and final conversion is a function of loading fraction. Kardar et al. showed that increasing the temperature from 25°C to 85°C and enhancing UV intensity in the range of $2\text{--}20\text{ mW cm}^{-2}$ would elevate the rate of polymerization and the final conversion in loaded composites [98]. However, increasing the energy of output lamp will be useful to overcome this problem [75, 89].

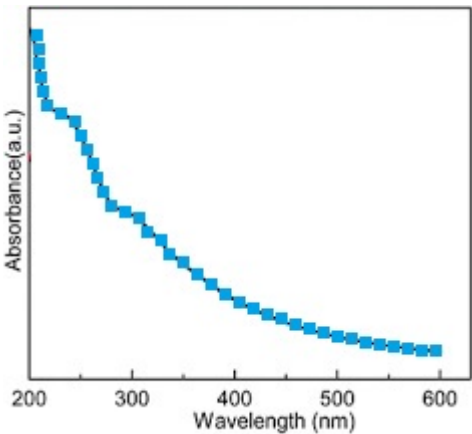


FIGURE 8.7
Absorption spectra of graphene

Researchers have reported some methods to overcome this disadvantage by increasing the energy of output lamp, extending modifications to the curing time, modifying the formulation (adding hyper-branched polymer) and using reduced graphene oxide [4, 75, 89]. A typical RT-FTIR conversion curve for a polyethyleneglycol diacrylate resin and its nanocomposites containing 0.5 and 2 wt% graphene oxide is shown in Figure 8.8. In this curve, the plateau gives the final active groups conversion and the photopolymerization rate can be derived by the slope of the curve. Figure 8.8 indicates that the addition of GO does not influence the rate while causes a reduction in final conversion [11]. Figure 8.8 illustrates in the presence of graphene filler, after 60 seconds of irradiation, the monomer to polymer conversion is 5 orders of magnitude less in comparison to the blank UV-curable resin.

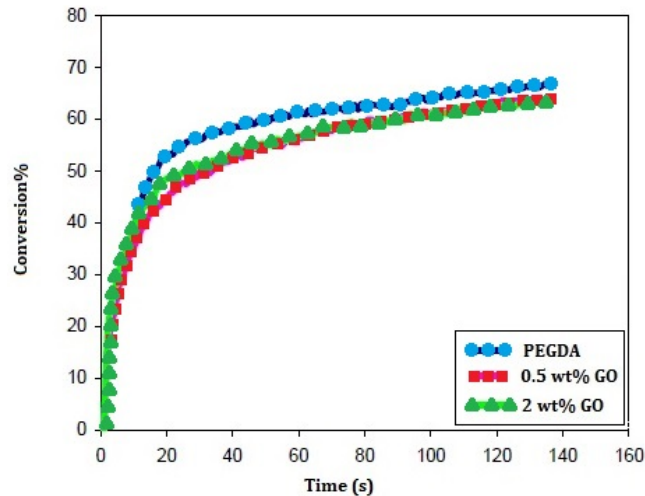


FIGURE 8.8
RT-FTIR conversion curve as a function of irradiation time for neat polyethyleneglycol diacrylate (PEGDA) resin containing 0.5 wt% and 2.0 wt% of graphene oxide (GO)-water dispersion. Radial photoinitiator concentration 2 wt% at aintensity of 30 mW/cm² with a film thickness 50 μm [11]

Calculating the gel content value by ASTMs method is another method to determine the curing process and the effect of loadings on curing. For instance, in the same GO/PEDGA nanocomposite, increasing the filler loading does not influence the gel content value. A high gel content (98%) even for the loaded resin, indicates the formation of a tight cross-linked network and the absence of extractable oligomers in the cured composite [11].

Scratch resistance

Scratch resistance is a valuable physical-mechanical property in coatings. High scratch resistance enhances the durability and appearance of the final coating in demanding, outdoor usage. Graphene is a naturally stiff material. Incorporation of graphene into polymer composites reduces scratch depth dramatically [40]. It is due to increased stiffness of final nanocomposite [10]. This effect can be observed in Figure 8.9 which illustrates a typical reduction of 5 times in scratch depth following the addition of graphene.

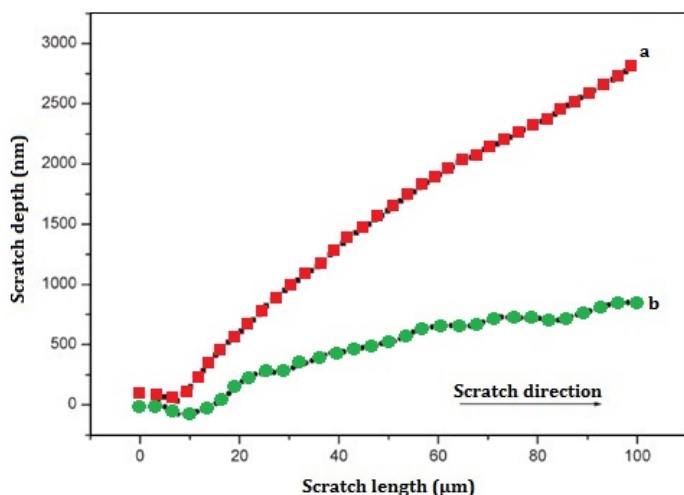


FIGURE 8.9

Studying the effect of graphene on nano-scratch depth for graphene/PU nanocomposite. (a) PU and (b) graphene/PU nanocomposite [74]

Gas permeation

GNPs incorporation can allow for the final composite to be impermeable to gases and liquids [90, 91]. Thus it is possible to use a polymeric host containing defect-free graphene sheets as a barrier membranes owning large surface area with mechanical integrity due to impermeability of graphene platelets [65]. Figure 8.10 indicates that the higher the graphene loading, the more impermeable the final nanocomposite becomes.

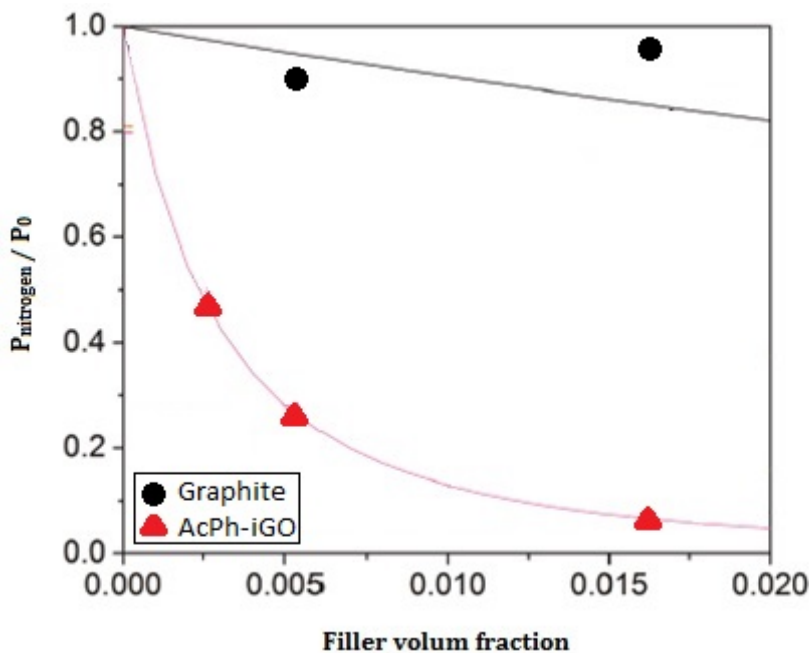


FIGURE 8.10 Nitrogen permeation of acetylphenyl isocyanate treated graphene oxide (AcPh-iGO) normalized by permeability P_0 of neat polymer. The solid curves are predicted [65]

Comparing to non- conductive clay platelets, conductive graphene has lower density and higher aspect ratio which triggered their potential application as advanced gas barrier polymer nanocomposite. An example of this effect is shown in Figure 8.11 which results from the low-wetability and dispersed graphene in the epoxy which makes the oxygen diffusion pathway increasingly difficult by reducing its diffusion length [105].

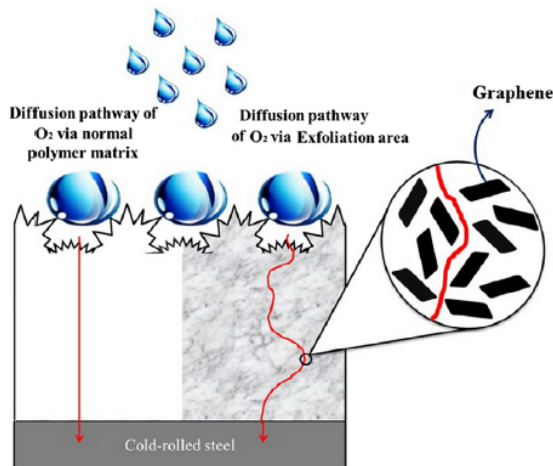


FIGURE 8.11 Schematic representation of oxygen diffusion pathway within an unfilled (left) and filled (right) epoxy. Adapted from [105]

The reduction in gas permeation is attributed to the barrier properties of the graphene layers dispersed within the polymer matrix.

Electrical properties

One way to increase the electrical conductivity of nanocomposites is using conductive fillers such as graphene, carbon nanotubes, carbon black, metal nanowires and metal powders [65]. Graphene incorporation is a proven mean of enhancing the electrical conductivity of nanocomposites [81]. Using such fillers, a percolation network is formed leading to a rise in the electrical conductivity (Figure 8.12). Hence, they would be eliminated to migrate into the polymer matrix segregated and would localize on the surface of polymer particles which results in the coverage of whole particle. It would be the main reason of the low percolation in these groups of composites. The percolation threshold (defined as the critical filler loading fraction to which the composite becomes conductive past a defined threshold) of graphene in GNPs nanocomposite is very low (below 2 wt.%), allowing the formation of uninterrupted conducting network for the electrons within the polymer matrix [92]. This low percolation threshold means a low amount of graphene, as the conductive elements can induce filler contact to form an effective conducting path which results in the formation of the conductive composite [99].

Graphene-based polymer composite have extraordinarily low electrical percolation threshold (0.1 vol %- noted that it varies from polymer to polymer) compared to other nanomaterials. It is primarily due to the large conductivity and aspect ratio of graphene sheets [100]. In semiconductive graphene-based coatings, there are two main factors, low percolation threshold and high electrical conductivity [100]. This low percolation is needed in order to have lower the costs and increase the transparency if required. The percolation threshold itself depends the aspect ratio of graphene and free space settlement of graphene nanofillers. Thus, the conductivity is bounded by two factors, elemental conductivity of the constituent graphenes and the electron loss at the conductive pathway formed by them in polymeric matrixes. Note the low percolation threshold of the nanocomposite due to large conductivity and aspect ratio of GNPs [11]. This low percolation threshold of graphene offers the benefits of both the cost and easy processing of the high conductive composite.

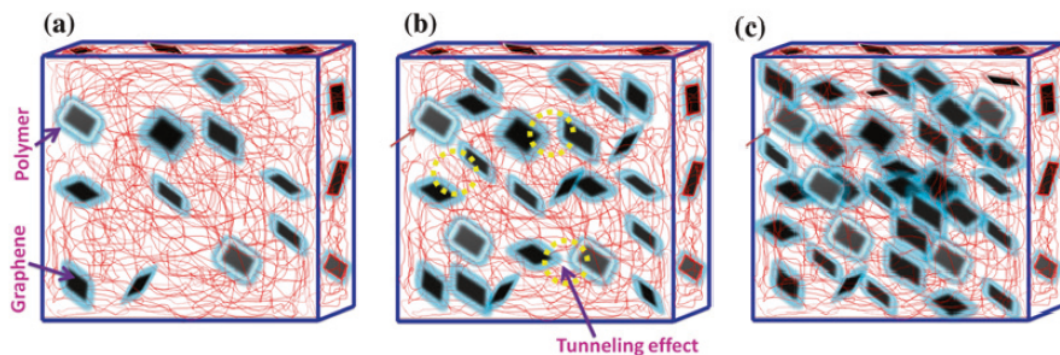


FIGURE 8.12

Percolation process in GNPs-based nanocomposites [99]

Recently, Galego et al. [89] have further enhanced the electrical conductivity of graphene based UV curable nanocomposites by using gold-functionalized graphene sheets. In this research, GNPs were

used as the substrate with gold latterly deposited. Here, polymer chains easily penetrated into the junctions and increased the electron loss transferring through graphene networks. This is the reason for increased conductivity of these nanocomposites [74]. This can be attributed to a charge transfer mechanism which is strongly enhanced as a result of gold NPs.

Mechanical properties

The presence of graphene in nanocomposites will increase the stiffness and tensile strength of the polymer matrix because defect-free graphene is the stiffest material in nature and it's intrinsic strength alongside with the interactions between particles and the host (Figure 8.13) [65]. The presence of these inorganic fillers within the soft polymer enhance the final mechanical properties of the composite. In UV-curable nanocomposites, graphene sheets limits the polymeric chain mobility. It can increase the glass transition temperature (T_g) [89]. In Figure 8.13, the effect of graphene on the tensile modulus is highlighted. Figure 8.12 illustrates the tensile strength in a UV-cured composite in different carbon-based fillers by way of comparison. As a result of addition of more GNPs, the tensile increased.

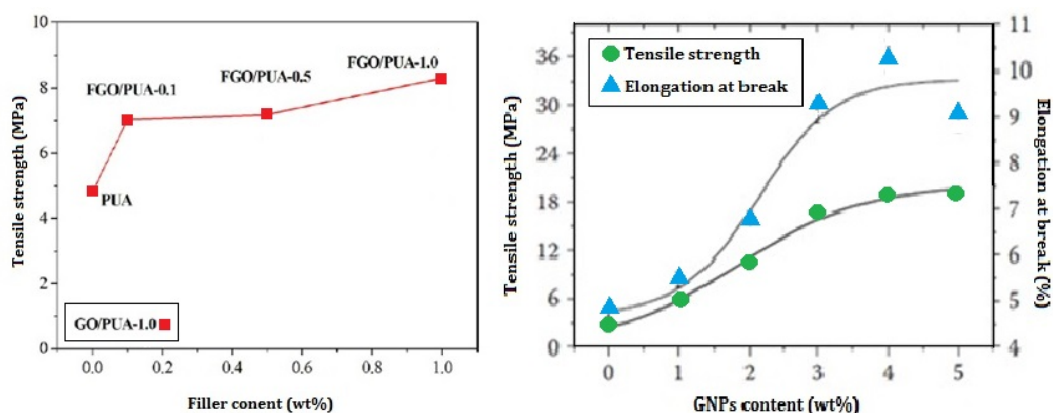


FIGURE 8.13

(a) Tensile strength as a function of filler content and (b) Tensile strength and elongation at break of epoxy nanocomposite as a function of GNPs content [10,59]

As it can be seen from Figure 8.14, the addition of graphene to the UV-curable systems led to an increase in storage modulus and viscoelastic properties [75]. These enhancements are known to be related to graphene's high specific surface area which increases the interaction between the polymeric chain and the graphene sheets.

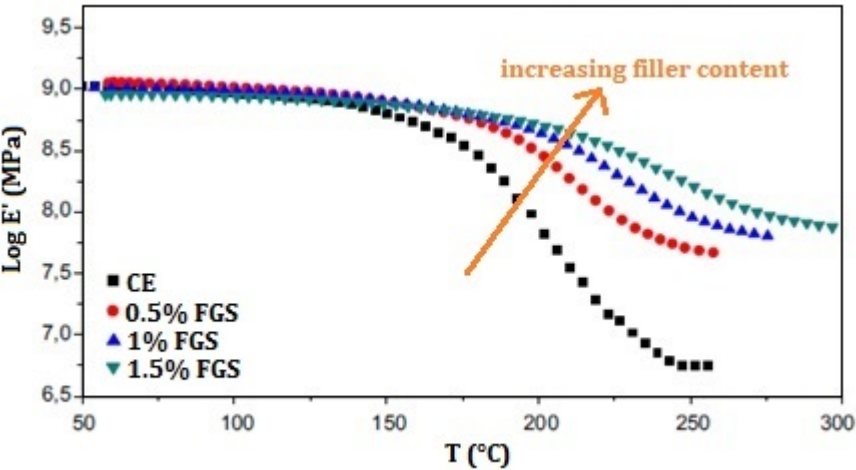


FIGURE 8.14 DMTA results shows the largest Storage modulus is gained for 1.5% functionalization of graphene sheet incorporated to an epoxy UV-cured system [75]

As can be seen in Figure 8.15, the incorporation of graphene into the matrix, the modulus is increased in glassy region [96].

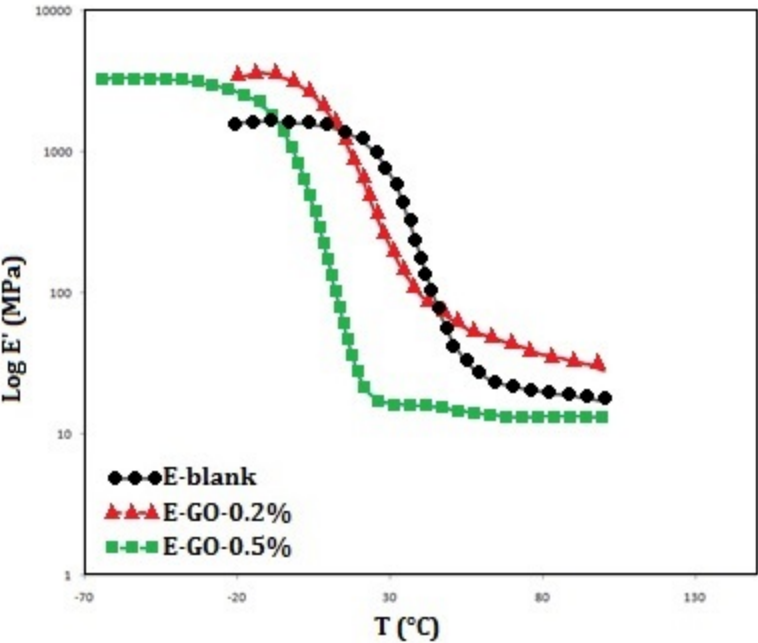


FIGURE 8.15 The storage modulus (E'), as a function of time in graphene-based UV- curable nanocomposites loading with 0.2% and 0.5% graphene oxide [96]

The $\tan\delta$ (E''/E') curve indicates a reduction in T_g temperature by addition of GNPs due to the reduction in curing conversion (Figure 8.16). Though the $\tan\delta$ is enhanced by 0.2% graphene oxide, it should be noted that the increased width illustrates the homogeneity of graphene within the polymer matrices [96].

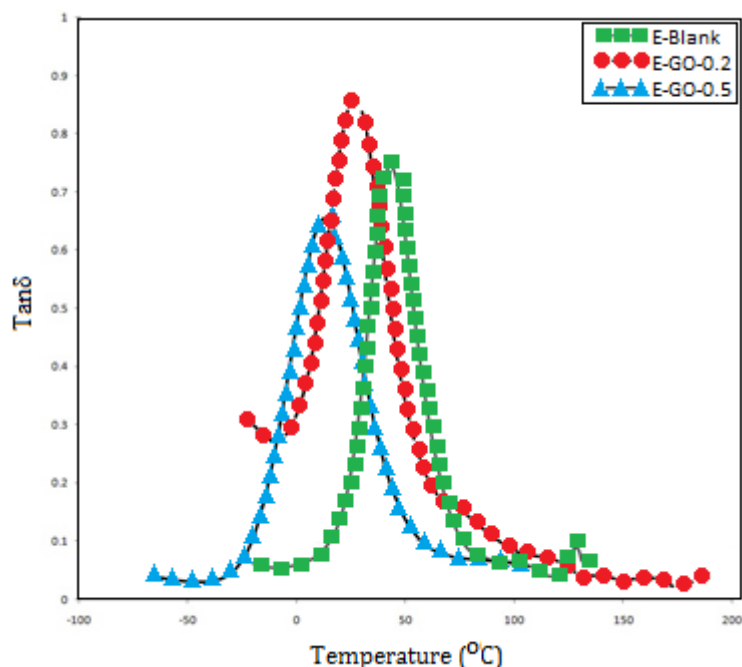


FIGURE 8.16

Tan δ curve for UV-cured nanocomposites in different graphene oxide (GO) loadings [96]

Thermal properties

Thermal stability enhancement is an advantage of using graphene based reinforcements. One way is the Changing of thermal exfoliation methods from conventional one to hydrogen arc discharge exfoliationh resulting in production of graphene with higher quality and a flat like structure [104]. The mobility of polymeric chains near the graphene sheets in the nanocomposite is slower which makes the nanocomposite to have a higher thermal degradation temperature [65].

Enhancing thermal stability of polymers can be achieved by using nanofillers. The fact is that nanofillers act as barriers and limits the propagation of heat generated by the external environment inside the polymer matrix. Results have shown that by further functionalization of graphene, the onset decomposition temperature would increase, which is useful for high temperature applications (Figure 8.17) [74].

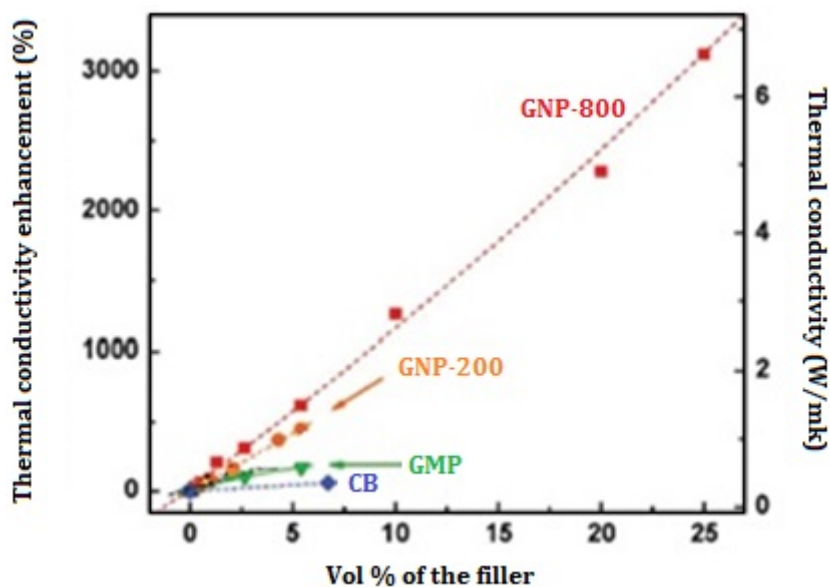


FIGURE 8.17 Thermal conductivity enhancement of epoxy-based nanocomposite using different fillers: grafitic microparticles (GMP), graphene nanoplatelets (GNPs) exfoliated at 200° C (GNP-200) and 800° C (GNP-800), carbon black (CB) [24]

The enhancement of Thermal conductivity and heat flow in the composite by increasing the filler content is a result of GNPs presence in the form of plates which facilitates the conductivity of the final cured composites (Figure 8.18).

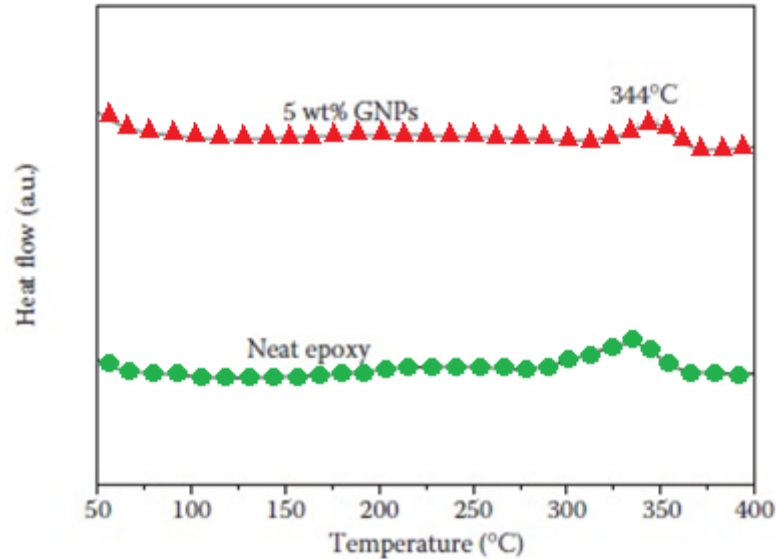


FIGURE 8.18 Incorporating of GNPs into epoxy matrices increases the heat flow of the final nanocomposite [59]

Table 8.1 shows the properties of an UV-cured nanocomposites. As it can be seen, the enhancement trend in T_g is in correlation with nanocomposite cured by different methods.

TABLE 8.1

Properties of UV-cured graphene/epoxy nanocomposites [75]

Sample	Conv.[%] ^a	Conv.[%] ^b	Gel content [%] ^c	T_g [°C] ^d	T_g [°C] ^e
CE	80	100	100	155	206
0.5% FGS	65	100	100	167	230
1% FGS	59	95	100	181	230
1.5% FGS	50	98	99	195	243

(a) Plateau values of the RT-FTIR conversion curves as a function of irradiation time (UV intensity of 35 mW/cm²).

(b) Determined by the single spectra taken before and after 5 min of UV irradiation (UV intensity of 55 mW/cm²).

(c) Determined gravimetrically ASTM D2765-84.

(d) Determined by DSC analysis, scan-rate 20 °C/min.

(e) Determined by DMTA analysis, tensile configuration, 1 Hz frequency.

Furthermore, In Figure 8.19, the thermal stability of an epoxy nanocomposite containing different wt% of GNPs is observed. As it can be seen, increasing the GNPs content leads to more stable nanocomposite.

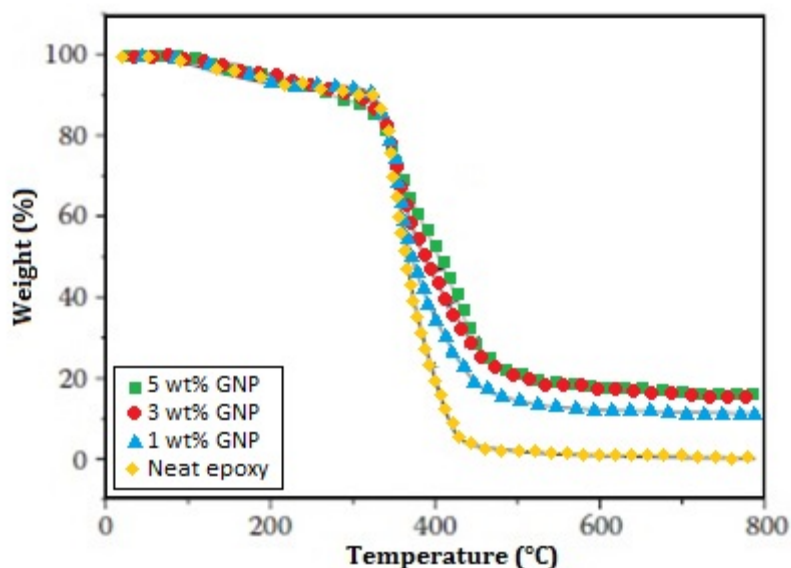


FIGURE 8.19

TGA results for GNPs/epoxy nanocomposite for different loading of GNPs [59]

Health and Safety Concerns

From the fabrication of GNPs to be used as nanofiller in nanocomposite, process of UV-curing to the degradation of nanocomposite, one should consider the safety concerns. As a new technology,

there must be concerns about degradation of this class of nanocomposite, particularly in outdoor usage. Figure 8.20 illustrates an example of releasing of nanoparticles to the environment [93]. Commonly, UV-Cured coatings, owing to their cross-linked structure, are not easily recycled. In the best condition, they can be fractionally degraded and removed from the surface. Much research has focussed on the breakdown of UV curable resins, and subsequent release of nanomaterials of thermoset network by thermal decomposition or hydrolysis [94].

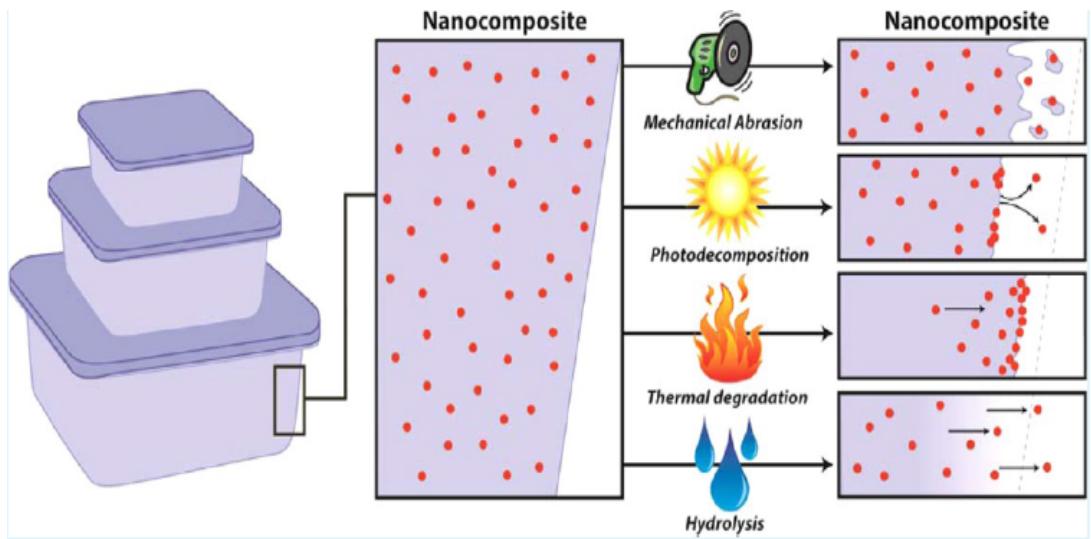


FIGURE 8.20
Releasing of the nanoparticles after degradation of nanaocomposite [93]

There are significant concerns about the broader uses of graphene [95]. The releasing time of graphene from the nanocomposite to the environment after degradation of polymer matrix by UV exposure has been also studied [95]. Furthermore, the graphene within the cross linked network can enhance the degradation temperature of the polymer from 299 °c to 316 °c [10].

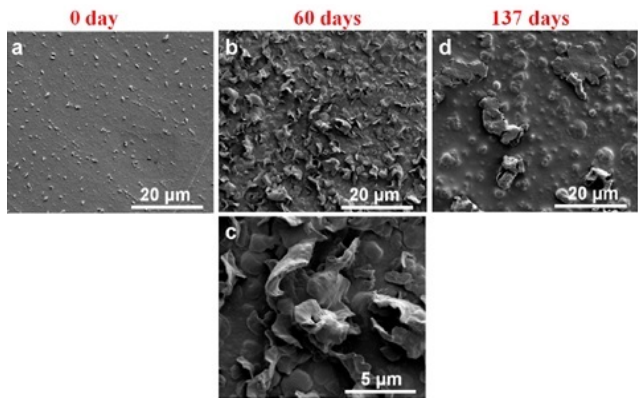


FIGURE 8.21
FE-SEM images of graphene based composites exposed to UV. (a) before exposure, (b) after 60 days exposure, (c) 60 days at higher magnification and (d) 137 days of exposure [95]

It is widely debated, however there is on-going fear that any released nanoparticles may pose a severe risk to human health and the wider environment. After 137 days of UV exposure, graphene particles were clearly agglomerated on the surface, as shown in Figure 8.21. A clear benefit here is that nano-scale graphene is not released into the environment. Nevertheless, such work is on-going and the under-pinning mechanisms dictating the release of nano-fillers into the environment, and the associated environmental consequences, remain timely challenges to be studied.

Conclusion

Graphene-based UV-curable nanocomposites have attracted much attention due to their superior properties of both graphene nanofillers and UV-curing process. As a graphene filled nanocomposite, they own high tensile modulus, high strength, and high electrical and thermal conductivity. As a UV-cured nanocomposite, they are cost effective, fast to produce in masse, and environmental friendly. Combining the advantageous properties of both the host matrix and graphene requires a fine dispersion of GNPs. The ease in which graphene can be dispersed in solution, low viscosity monomers before curing, is the unique advantage of graphene-based UV-Curable nanocomposites. The functionalization type and methods of graphene platelets, filler content and their dispersion state influence the final physical, mechanical, thermal and electrical properties of the UV-cured nanocomposite. Although UV curing process is known to be environmental friendly, consideration must be taken due to the presence of nanofiller inside the composite, which might pose human and environment safety concerns, after degradation of polymer matrix. Further research is needed for other polymer matrices and to ascertain whether any of these graphenes has released to the environment during exposure and as to whether such composites can find a role on the global commercial stage.

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