



Review Article

**Techniques on Dispersion of Nanoparticles in Polymer Matrices:
A Review**

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ABSTRACT

Dispersibility of nanoparticles is the key problem in nanotechnology industries, and thus warrants attention on the techniques of dispersion. This review paper presents dispersibility of treated nanoparticles in polymer resin. Dispersibility of nanoparticles in polymer media is crucial in order to enhance the mechanical and thermal properties of nanocomposite. This paper concentrates on several preparations on how to incorporate nanoparticles in polymer to overcome the problem described in this review. A few techniques are discussed in this paper such as by using ultra sonication or even directly mixing nanoparticles into polymer matrix.

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INTRODUCTION

Over the years, there has been a dramatic increase in the manufacturing of polymer composites for many applications such as aerospace (Mangalgiri, 1999), automobile (Tseng & Kuo, 2011; Hung et al., 2011), electronics packaging (Davidovits, 2002), food packaging (Mallakpour & Madani, 2015), medical (Ahn et al., 2013), etc. Hussain et al. (2006) claimed that the use of organic

and inorganic fillers has become pervasive in polymeric systems. The purpose of adding synthetic or natural inorganic fillers in polymer matrix is to improve composite properties and subsequently reduce production costs (Pavlidou & Papaspyrides, 2008). In addition, the presence of nanoparticles that act as reinforcing fillers can affect changes in polymer matrix in term of flow ability, viscosity, colour, density and subsequently it also tend to improve the optical, electrical, catalytic, magnetic and thermal properties of the composite (More et al., 2015).

Transfer of nanocomposite technology requires the development of more fundamental understanding of reinforcement mechanisms due to the difficulty in characterising the state or nature of nanostructured materials in a polymer. Interestingly, nanomaterials have good properties even without being use as reinforcing materials in polymer composite. However, inclusion of nanostructured fillers without good preparation tends to produce composite with poor nanofiller dispersion. It is crucial to understand that nanofillers have high attraction among the neighbouring particles as they have a high tendency to agglomerate or clump during mixing process. Thus, the mixing process of attractive nanofillers in polymer matrix is a problematic issue. Therefore, investigations or studies on the techniques of dispersibility of nanoparticles are important to produce nanocomposites with good interfacial bonding between nanofillers and polymer matrix and isolate single nanoparticle in the polymer media.

There have been many techniques studied and applied in producing good dispersion of nanoparticles in polymer matrix. It is also important to know the original nature of nanoparticle surface (Chen et al., 2010; Chen et al., 2009). For example, if the origin surface is hydrophilic, while the polymer media is nonpolar, changing the nanoparticle surface into hydrophobic is therefore important to improve the dispersibility of nanoparticles in the non-polar media. This paper focuses on different types of surface treatment which have been done on nanoparticles including the effects of silanisation on dispersibility of the treated nanoparticles in polymer matrix and their morphology structure after the treatment process.

Techniques Used to Disperse Nanoparticles

Transferring nanocomposite technology requires development of more fundamental understanding of the reinforcement mechanisms as nanostructured materials are quite difficult to characterise. With the emergence of nanotechnology, researchers have become more interested in studying the unique properties of nanoscale materials (More et al., 2015). In addition, the dispersibility of nanoparticles in polymer medium is one of the main concerns. This is due to the high interaction among nanoparticles as their compatibility with polymer matrix which can affect the performance of the final composite produced. Interestingly, nanoparticles have a very fine particle size. Due to the high surface area, it is favourable to be embedded in a polymer matrix so as to enhance the properties of polymer (Fan et al., 2013; Fan et al., 2006; Gorga et al., 2004; Luo & Daniel, 2003). However, difficulties in mixing nanoparticles make them easier to mix in the matrix although the process of dispersing the nanoparticles in matrix has to be perfectly incorporated. Thus, the methods to ensure that the nanoparticles are evenly distributed must be comprehensive. The recommended methods for solid thermosetting reactive

prepolymers or thermoplastic polymers with solid nanoparticles are solution intercalation, melt intercalation and roll milling (Koo, 2006).

However, the disadvantage of melt intercalation is related to the low thermal stability of the organic modifiers used because the melt intercalation process usually takes up in 180°C to 200°C (Tjong, 2006). Whereas, for liquid thermosetting reactive prepolymers or thermoplastic polymers with solid nanoparticles, the recommended processing methods are in-situ polymerisation, emulsion polymerisation and high shear mixing (Koo, 2006) (Hai et al., 2014) (Herrera & Gonzalez, 2005). A research done by Canché-Escamilla et al. (2014) mixed silica or hybrid nanoparticles manually with resin until the powder was homogeneously mixed with the resin, and the produced paste was smeared over a glass surface with a spatula to yield a semi-transparent film and ensure optimal particle dispersion (Canché-Escamilla et al., 2014).

In another research by El Saeed et al. (2015), ZnO polyurethane nanocomposite (ZPN) coating films were prepared by dispersing the ZnO nanoparticles by sonicated in xylene solvent and ultrasonic waves using sonicator model, and the dispersed ZnO NPs were directly mixed with polyurethane base through manual stirring (El Saeed et al., 2015).

Phoo-ngernkham et al. (2014) used 1 to 3 wt. % of nano-SiO₂ and nano-Al₂O₃ to be embedded in inorganic polymer matrix. Initially, they combined the dry material (fly ash) and nanoparticles (nano-SiO₂ and nano-Al₂O₃) together until the mixture became homogeneous. This was followed by adding liquid alkaline solution into the dry mixture, while stirring was continued until the paste become homogeneous. The dispersibility of the nanoparticles in inorganic polymer matrix tends to improve the compressive strength of composite up to approximately 56 MPa. However, the authors observed that adding of 3wt% of nanoparticles was excessive due to the low amount of inorganic polymer to bind and interact with the high amount of nanoparticles. This is in comparison to the work of Rees et al. (2008) who mixed the 0.1g of Al₂O₃ nanoparticles with activation solution first before adding the dry components for the formation of inorganic polymer nanocomposite.

Another work by Nyugen et al. (2014) studied nanoparticle dispersion by ultrasonication in which Transducer Digital Sonifier Model 450 (Branson Ultrasonic Corporation, USA) was used to disperse the nanoparticles. For the ultrasonication, the maximum power input and frequency used were 400W and 20 kHz, respectively, whereas the ultrasonic horn that immersed into the suspension had a tip diameter of 13 mm and the sonication amplitude is in the range of 10–65 lm. In order to reduce the heating up of suspensions during sonication, the ultrasonic mode was set with a pulse ratio on/off 0.1/0.1 (s/s), followed by cooling the vessel of suspension using an ice-water bath. Nyugen et al. (2014) also believe that nanoparticle concentration could give impact on cluster size, as well as the viscosity of suspension.

Lee et al. (2005) claimed that introducing surfactant to the clay surface enabled a good compatibility between inorganic clay and organic polymer or monomer for good clay exfoliation, which is also a fire hazard material. However, without surface modification, natural clay can only disperse well in water-soluble polymers. Lee et al. (2005) stated that using water as nanoclays carrier could yield surfactant-free nanocomposites with a good clay dispersion in hydrophobic polymers.

Chemical agents can be used to improve nanoparticle surface and its interfacial properties with polymer matrix and also function as stabiliser against coagulation or aggregation by conserving the charge on nanoparticle surface. The Van der Waals force theory is useful to understand the interaction between nanoparticles and polymer matrix. This theory defines attractive and repulsive forces between molecules and explains that weaker forces, stronger polar and electrostatic interactions, or covalent interactions, can influence the interactions of particle-particle.

In fact, to enable well dispersal of nanoparticles, it is convenient to conduct pre-processing of the nanoparticles which includes purification steps to eliminate the impurities, deagglomeration for dispersing individual nanoparticles and chemical functionalisation so as to improve the nanoparticle-polymer interaction and enhance the properties (Peponi et al., 2014).

Mokhena (2012) conducted surface treatment on sisal nano-whiskers using alkaline solution, while Vasiliev et al. (2009) conducted a research on highly dispersible polymer-coated silver nanoparticles, whereby the silver was prewashed with deionized water and treated with piranha solution for an hour. Factually, the piranha solution is used to clean organic residues off silver.

Silane Treatment of Nanoparticles

As for the silane treatment of nanoparticles, Wang et al. (2011) used silane coupling agent to graft on the titanium dioxide (TiO_2) surface. The grafted modification of TiO_2 was performed in liquid phase, where silane coupling agent was added into deionised water before mixing it with TiO_2 . Wang et al. (2011) also suggested that the pH value of the mixture could be control by adding ammonium hydroxide ($\text{NH}_3\text{H}_2\text{O}$) and hydrochloric acid (HCL) solutions into the mixture. The slurry then underwent ultrasonic treatment for a range of time before centrifuging it at 4000 rpm for 20 minutes.

Most mixtures, in combination with nanoparticles, need some sort of forces to break the bonds within the particles so that the latter can be dispersed homogeneously into the matrix. The dispersibility of nanoparticles in organic medium is shown through the size of Lipophilic Degree (LD). The primitive TiO_2 has some hydroxide radicals on its surface, causing it to become hydrophilic and thus sinks in deionised water. On the other hand, modified TiO_2 floated

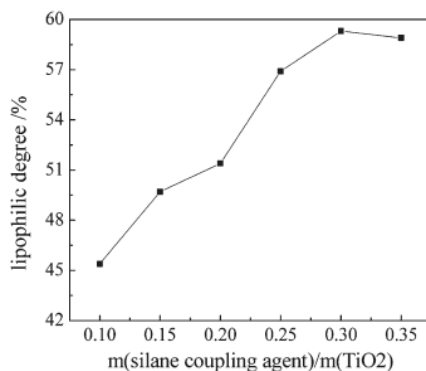


Figure 1. Effect of modifier dosage on the lipophilic degree (LD) (Wang et al., 2011)

on the surface of deionised water. This reveals that the surface of TiO_2 changed drastically after modification.

Another research by Yang et al. (2013) improved the surface properties of calcium carbonate (CaCO_3) nanoparticles to react with (styrene-butadiene rubber) SBR latex by modifying the surface of CaCO_3 nanoparticles with silane coupling agents. Meanwhile, Monfared et al. (2014) produced glass nanoparticles through wet milling process and they modified the surface of glass nanoparticles using mercaptopropyltrimethoxysilane (MPTMS). The composite was prepared by mixing these Silane-treated nanoparticles with monomers.

There are two methods used in preparing these composites. The first was by dispersion in solvent method, whereby glass nanoparticles were sonically dispersed in acetone, before adding to resin and then acetone was evaporated. The other method was done by directly adding glass nanoparticles into resin. For each method, 3 different groups of composites were produced, with inclusion of 5, 7.5 and 10 wt. % of glass particles, respectively. They noted D samples as in dispersion in solvent method and N samples as in non-dispersion in solvent method. As shown in Table 1, there is a significant pattern of results among the samples. Flexural strength in group D shows better mechanical properties in term of flexural strength and modulus compared to group N. Moreover, increasing the nanoparticle content tends to improve the mechanical properties as well for both D and N.

Table 1
Mean value of flexural strength, flexural modulus and micro hardness among the groups (Monfared et al., 2014)

Composites	D1	D2	D3	N1	N2	N3
Flexural strength (Mpa)	63.98	69.07	75.22	55.83	58.38	59.99
Flexural modulus (Mpa)	1259.53	1295.08	1388.83	1210.78	1280.49	1334.26
Microhardness (VHN)	20.73	21.35	24.56	17.22	19.47	23.23

Dantas et al. (2012) studied the effects of fibre post surface after plasma and the common treatments and also evaluated the adhesion between treated fibre posts and Rely X Unicem resin cement. They conducted six types of treatments, which are silane, hydrofluoric acid, hydrofluoric acid plus silane (these are the most common treatments used in research works), plasma polymerisation with argon and ethylenediamine plasma (EDA). In order to compare the plasma and other common treatment methods, results after silane, hydrofluoric acid and hydrofluoric acid with silane treatments were evaluated. As a result, they observed that the most hydrophilic surface was seen in the samples treated with silane, followed by the treatment with hydrofluoric acid and finally hydrofluoric acid with silane.

The Morphology of Nanoparticles Dispersion

Dispersion of the silica nanoparticles and hybrid silica/PMMA nanoparticles could be observed in the SEM images (Figure 2). Figure 2 shows the results obtained on the fractured zone of the

composites from the flexural test. Both the fractured zones have similar amount of nanoparticle content. However, it was found that the dispersion of hybrid silica/PMMA is better than that of the composite with only silica content. It is therefore likely that well dispersed hybrid nanoparticles can result in higher modulus for composite material (Canche-Escamilla et al., 2014) (Tamon et al., 1998) (Šupová et al., 2011). The low flexural strength observed from the analysis in Figure 2 above, especially for the composite filled with only silica nanoparticles, indicates a poor dispersion of nanoparticles into the polymer matrix.

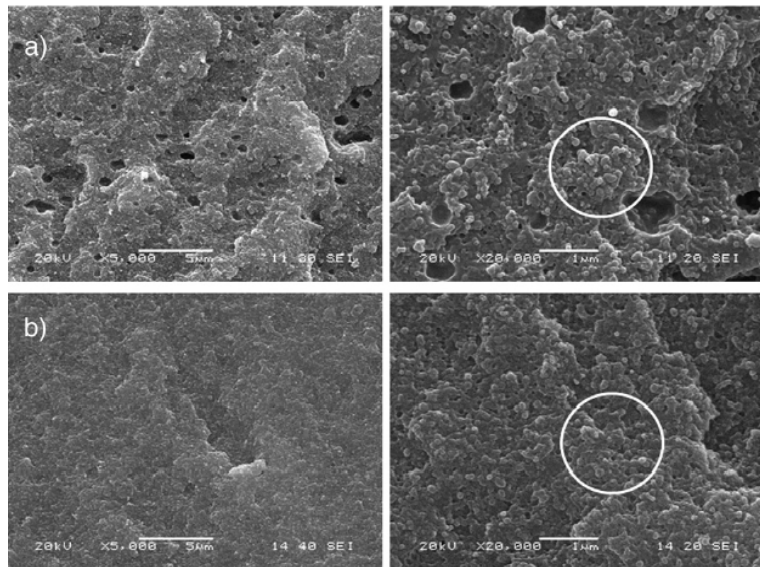


Figure 2. SEM images of the fracture zone of composites. Type of Filler used: a) silica and b) hybrid silica/PMMA (78/22) nanoparticles (Canche-Escamilla et al., 2014)

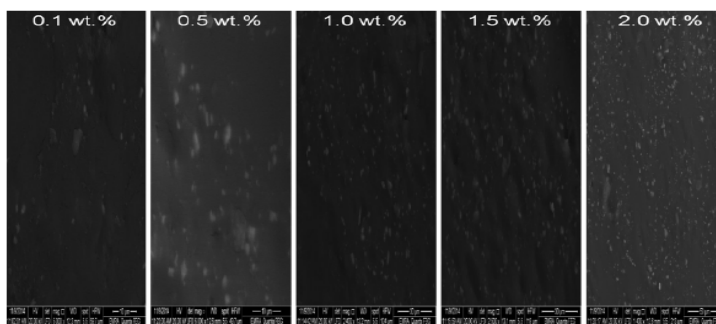


Figure 3. The SEM images of ZPN coated films containing ZnO NPs at different loading levels (El Saeed et al., 2015)

Figure 3 presents the SEM images obtained from the fractured surface of polyurethane coating containing zinc oxide nanoparticles. The dispersibility of the nanoparticles was found to be uniform throughout the coating film. From left, the bright particle indicating the nanoparticles is increasing in amount. This finding presents that even though the nanoparticles are uniformly dispersed regardless of the amounts of nanoparticles used, the low loading of nanoparticles still indicates surface roughness compared to the higher loading of nanoparticles which also improved in crack resistance. This also accords with earlier observations, which showed that evenly dispersion of the nanoparticles gave impacts on the mechanical performance for the composite (El Saeed et al., 2015).

The amount of nanoparticles can contribute to the distribution of the nanoparticles in the polymer matrix. Some of the composites can be prepared by inclusion of large volume of nanoparticles and interestingly, the nanoparticles can uniformly distribute throughout the whole matrix. However, there are some produced composites which have limitation on the amount of nanoparticles used (El Saeed et al., 2015; Canche-Escamilla et al., 2014; Khare & Burris, 2010; Nobile et al., 2015). In certain cases, as the loads of nanoparticles increase, the viscosity of the polymer resins increase, which likely turn the nanocomposite into paste (Canche-Escamilla et al., 2014; Monfared et al., 2014; Oriakhi, 1998). Therefore, selection of suitable polymer and nanoparticle needs to be thoroughly done in order to have a good filler-polymer interactions and composition.

In a research by Rangari et al. (2009), the high resolution TEM micrographs showed that tungsten trioxide (WO_3) nanoparticles are porous and well dispersed in the epoxy resin (Figure 4).

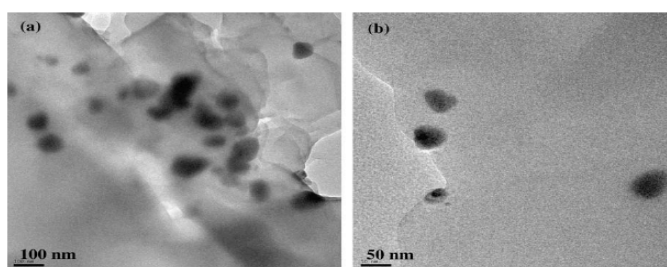


Figure 4. The TEM micrograph of (a) 1 wt. % WO_3 /SC-15 epoxy at 100 nm scales and (b) 1 wt. % WO_3 /SC-15 epoxy at 50 nm scale (Rangari et al., 2009)

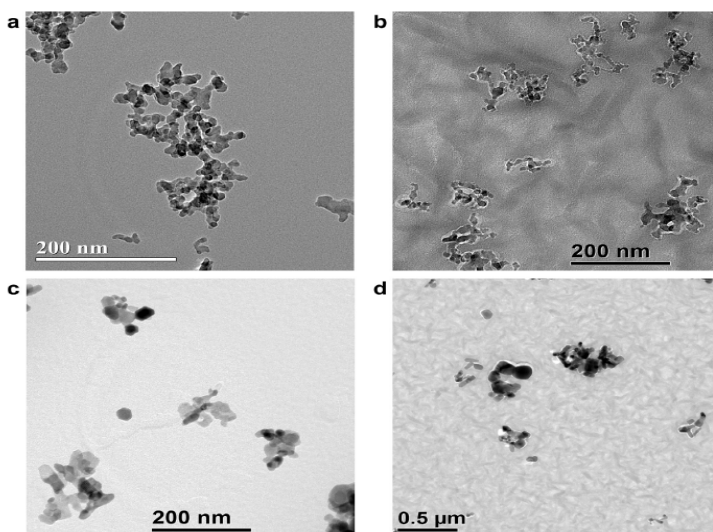


Figure 5. The TEM images of Al₂O₃/P18Et2/MEK 1 mg/1 mg/ml (a), nanocomposites of P (VDF-TrFE)/Al₂O₃ 10 wt. % (b), ZnO/P18Et2/MEK 1 mg/1 mg/ml (c) and nanocomposites of P (VDF-TrFE)/ZnO 10 wt. % (d) (Nyugen et al., 2014)

The dispersion of Al₂O₃ and ZnO nanoparticles at different concentrations in organic solvent and polymer solutions is shown in Figure 5 (Nyugen et al., 2014). The findings revealed that with optimum ultrasonic parameters, the stabilised nanoparticles exhibited the same final cluster size in aqueous, organic and polymer suspensions. Over the tested range, the solid concentration had very low effects on the cluster size. This finding further supports the idea that the dispersion results in low concentration suspensions could be transferred to highly concentrated suspensions or even to a polymer solutions (Nyugen et al., 2014; Rouxel & Vincent, 2014). The SEM photo in Figure 6 shows that the agglomeration of glass particles was removed by silanization process.

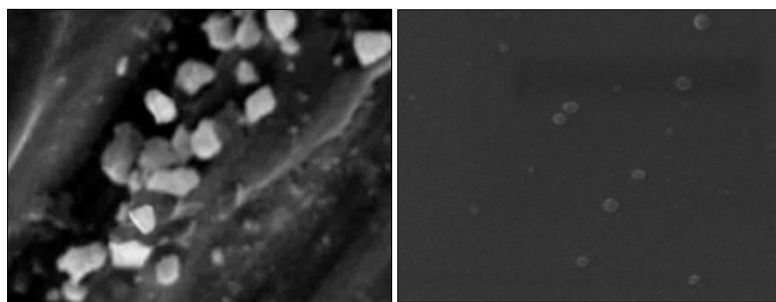


Figure 6. The SEM photos of (left) milled glass particles and (right) silanised glass particles (Monfared et al., 2014)

The research by Yang et al. (2013), showed that CaCO_3 was dispersed evenly in ethanol using the ultrasonication process. As a result, Transmission Electron Microscopy (TEM) images of CaCO_3 in Figure 7 reveal that before the modification, CaCO_3 nanoparticles were aggregated in aqueous solution with irregular shape due to the high surface energy and surface polarity. After the modification, it could be seen that CaCO_3 dispersed notably. This might be caused by the reduction of surface free energy and the increase of steric hindrance effects due to grafting of macromolecular chains of silane coupling agent onto the surface of CaCO_3 nanoparticles. Figure 7 shows the TEM images of CaCO_3 nanoparticles before and after the modification.

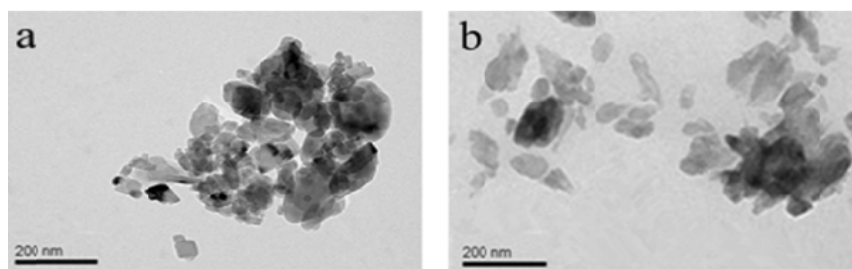


Figure 7. The TEM images of CaCO_3 nanoparticles (a) unmodified and (b) modified with 5wt% amount of silane coupling agent (Yang et al., 2013)

CONCLUSION

The trend of using of nanoparticles is based on their unique properties, which meet a wide range of applications and market needs. The selected nanoparticles need to be compatible with the polymer matrix in order to obtain the desired results, while the dispersion technique should be selected properly to uniformly disperse and distribute nanoparticles within the polymer matrix. Surface treatment or functionalisation on nanoparticles tends to improve the dispersibility of nanomaterials in the polymer matrix. The improved properties of the nanoparticles not only depend on the distribution of the nanoparticles but also their size, shape, concentration of nanoparticles used, type of nanoparticles and compatibility with the polymer matrix.

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