



Dispersion characteristics of dry and colloidal nano-titania into epoxy resin

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ABSTRACT

There are well known problems with the dispersion of inorganic nanoadditives into organic medium as they tend to aggregate and agglomerate, therefore the particles act like micron sized particles rather than nanoparticle scale. In this paper, we investigated the differences between dispersing dry and colloidal TiO₂ nanoparticles into epoxy resin. The dry TiO₂ was commercially obtained P25 and freeze dried TiO₂ from continuous hydrothermal synthesis (CHS). The colloidal TiO₂ was produced from CHS but without a drying stage. Specific parameters including loading level (1–30 wt.%), mechanical mixing (2–60 min), polymer heating (25–80 °C), and dry and wet addition were investigated. The nanoparticle dispersion was investigated with SEM and quantified by using Labview image analysis (National Instruments), in order to quantify the characteristic aggregate dimensions such as perimeter, maximum feret diameter, and size. TiO₂ nanoparticles were formulated and surface modified with dodecyl succinic anhydride (DDSA) by using CHS technique. Online phase transfer was used to produce colloidal DDSA–titania dispersed in toluene which could then be directly integrated into epoxy resin. This approach showed enhanced nanoparticle dispersion (nanoscale dispersion) with minimum aggregation compared with the dispersion of dry nanoparticles. Furthermore, this approach enabled the elimination of nanoparticle freeze drying and the redispersion of aggregated dry nanoparticles into polymeric matrix.

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1. Introduction

The successful achievement of polymer nanocomposites (PNs) requires the dispersion of the nanoadditives at the molecular level with minimum aggregation [1]. The key challenge when attempting to produce a nanocomposite, is avoiding aggregation of nanomaterials that need to be evenly dispersed throughout the polymer matrix. Dispersing nanomaterials into polymers is not straightforward, not least because polymers tend to be hydrophobic and im-miscible with inorganic nanoadditives [2]; the intrinsic high surface energy causes extensive aggregation into the dispersing medium [3,4].

Regardless of the nature of the nanoparticle manufacturing process, once the nanoparticles are dried they form aggregates that are difficult to redisperse. There are major advantages to any technique that is able to produce and maintain the nanoparticle in dispersion. One such technique would be CHS [5–7]. In this technique, particles are formed as supercritical water is mixed with a dissolved metal salt in an aqueous solution [5–7]. CHS offers a pretty simple route which is intrinsically scalable and chemically much more benign than many other nano-

production technologies [7]. Furthermore, CHS facilitates the online surface modification of the nanomaterial during their production; it is possible to alter the nanoparticle surface properties from hydrophilic to hydrophobic via surface modification [2,8,9]. Specific organic ligands are able to stabilize the colloidal nanoparticles and prevent coagulation by introducing a thick adsorbed organic layer which constitutes a steric barrier [10]. Anchoring groups that can be used to attach organic ligands to oxide surface are amine [11], ammonium [12], carboxylic [13,14], sulphonic, and phosphoric acid groups [15]. Carboxylate ligands are often being used for nano-oxide surface modification [15]. Fig. 1 shows the adsorption mechanisms of carboxylate ligands to TiO₂ surface, where hydrogen bonding could be a part of the adsorption mechanism [16].

To date, there are relatively few papers that describe how and why nano-oxides could be effectively dispersed into polymeric matrix. This paper investigated how dry and colloidal TiO₂ phases can be effectively dispersed into epoxy resin and how the effective level of dispersion can be quantified by using image analysis techniques. This is the first time image analysis has been used to quantify nanoparticle dispersion.

It was possible to sterically stabilize colloidal TiO₂ nanoparticles via surface modification with DDSA. Keeping the nanomaterials in dispersion was found to be an important way of avoiding poor dispersion characteristics.

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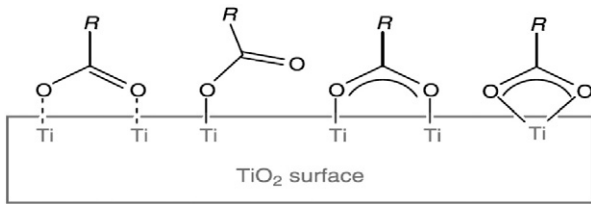


Fig. 1. Adsorption forms of carboxylate ligands on titania nanoparticles [15].

2. Experimental

2.1. Epoxy

Epoxy resin, one of the most commonly used polymer in engineering applications, was used as the dispersing medium. The employed epoxy resin was bisphenol-A (epichlorohydrin) with number average molecular weight ≤ 700 g/mol, commercially known as Epofix resin, produced by Struers. The hardener was triethylene tetramine also produced by Struers.

2.2. TiO_2 used in the experiments

Commercially available TiO_2 known as P25 by Degussa with an average particle size of 25 nm was used as a dry nanopowder. The

impact of solid loading level and mechanical mixing on titania P25 dispersion was evaluated; titania–epoxy nanocomposites of different titania loading levels 1, 5, 10, 15, 20, 25, and 30 wt.% were prepared by mechanical mixing and vacuum cast. Different mixing times of 2, 10, 30, and 60 min were employed for each loading level. Dispersed TiO_2 was prepared by using CHS technique; Fig. 2 is a schematic of the system.

From Fig. 2, the ScW passes down an inner nozzle pipe (A) against an up flow of cold metal salt (B). Nanoparticles are formed at the interface of the two fluids (C), and the buoyancy of the heated flow causes the nanoparticle slurry to be carried upwards (downstream) for the capping point (D) where nanoparticle surface modification can be performed. The nanoparticle flow stream will be cooled down prior to collection point (E). Titanium IV bis (ammonium Lactato) di hydroxide (TIBALD), 50 wt.% solution in water, (Aldrich) was used as the metal salt for TiO_2 synthesis by CHS; dodecenyl succinic anhydride (DDSA) (Aldrich, 98%) was employed as the surface modifier.

2.3. Determination of the dry nanoparticle dispersion efficiency

The nanoparticle dispersion in epoxy resin was investigated with SEM (Quanta 600 by FEI incorporation) at a magnification of 1300 \times . The TiO_2 could be seen from the background resin as white particles and could therefore be quantified by using image analysis program Labview software (National Instruments). A script was established

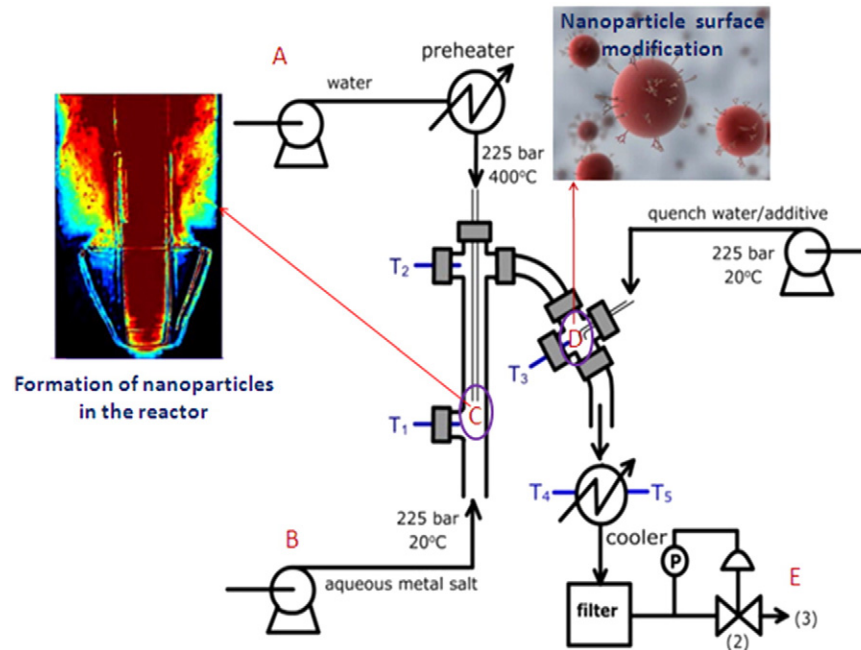


Fig. 2. Flow diagram of continuous hydrothermal synthesis system [7].

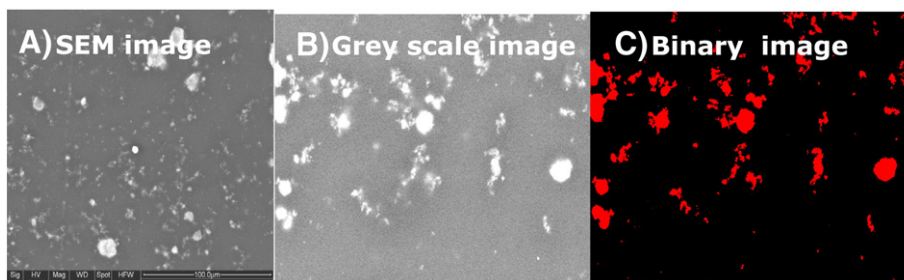


Fig. 3. Image analysis for the dispersion of 1 wt.% TiO_2 into epoxy.

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