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One-Step Synthesis of Oval Shaped Silica/Epoxy Nanocomposite: Process, Formation Mechanism and Properties

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1. Introduction

Silica/epoxy nanocomposites have been widely employed as high strength material for aerospace, automobile, electronic and sporting equipment industries (Deng et al., 2007; Preghenella et al., 2005; Hsiue et al., 2001; Fu et al., 2008). The thermal mechanical properties of nanocomposites were reported to be influenced by the shape and size of nano-filler, volume fraction, quality of dispersion, and the interaction between filler and matrix (Ragosta et al., 2005; Kwon et al., 2008; Adachi et al., 2008; Zhang et al., 2006). These parameters determine the molecular mobility of matrix and the amount of energy dissipation at crack initiation and propagation as the stress transfer from matrix can be promoted by these high specific surface area particles.

To obtain silica/epoxy nanocomposites with good properties, many approaches have been devised to improving the dispersion of silica and the interaction between silica and epoxy (Fu et al., 2008; Kwon et al., 2008; Zhang et al., 2006; Chen et al., 2008; Wang et al., 2005; Zhang et al., 2008). The solution blending process has been reported as a method providing good silica dispersion and silica-epoxy bonding (Hsiue et al., 2001; Zhang et al., 2008; Deng et al., 2007; Liu et al., 2003; Mascia et al., 2006; Huang et al., 2005; Araki et al., 2008). However, the multiple steps are involved in this process. The spherical shaped silica synthesized by sol-gel process is normally used as a silica source due to its availability in solvents. The further step to functionalize silica surfaces by amine-terminated coupling agents is required to improve the dispersion of silica nanoparticles and their adhesion with matrix (Mascia et al., 2006). Then, the high pressure and temperature mixing with epoxy compositions is required after the above process. As the solvents are involved in this process, the steps to remove, recycle and dispose solvents are needed. This poses the environmental, health and safety issues, in addition to the additional costs involved in solvent removal/disposal. Moreover, large amounts of silica at 5-30 % by weight of total composite composition are required to obtain nanocomposite with good mechanical and thermal properties (Deng et al., 2007; Preghenella et al., 2005; Zhang et al., 2008). The high percentages of silica significantly increase the viscosity of compositions and influence many

intrinsic properties of epoxy matrix such as weight, ductility, processability and transparency. Therefore, there is a need to develop an effective, convenient and low-cost process to prepare high performance silica/epoxy nanocomposite that exhibits uniform dispersion of silica with great silica- epoxy adhesion, to target a wide range of applications.

In the present work, a "Solvent-Free One-Pot Synthesis" method was developed for the preparation of high performance nanocomposites with uniform dispersion of oval shaped silica in epoxy and strong silica-epoxy bonding. The silica formation, surface functionalization and dispersion in epoxy compositions are combined into one step at 50 °C under mechanical stirring. In this process, the solvent was not involved in the nanocomposite preparation. Thus, it is friendly to the environment and benefits to the formation of oval shaped silica because high shear rate was applied for the mixing of viscous mixture. The details on the synthesis process, the chemical composition and morphology, the thermal mechanical properties of silanized silica/epoxy nanocomposites were studied. The properties of prepared nanocomposite were compared with those of neat epoxy, non-functionalized silica/epoxy and commercial available silica/epoxy systems.

2. Experimental

2.1 Materials

Diglycidyl ether of bisphenol A (DGEBA, D.E.R.™ 332) was supplied by Dow Chemicals. Diethyltoluenediamine (Ethacure 100-LC) was obtained from Albemarle. Tetraethylorthosilicate (TEOS, ≥ 99%) and (3-aminopropyl)trimethoxysilane (APTMS, 97%) were purchased from Sigma-Aldrich. Ammonia solution (25 wt-%) was supplied by Merck. The commercial available silica/epoxy (Nanopox F400) was supplied by Nanoresins AG.

2.2 Preparation of silanized silica/epoxy nanocomposite

A mixture of epoxy, Ethacure 100-LC, TEOS and APTMS was stirred vigorously at 50 °C. The weight ratio of epoxy: Ethacure 100-LC was fixed at 3.8:1. The amount of TEOS was varied to obtain 1-4 wt-% silica in epoxy composition. The amount of APTMS was fixed at 10 wt-% APTMS to silica amount. An ammonia solution with NH_3:TEOS molar ratio of 2.3:1 was injected into the above solution and aged for 60 min. The mixture was degassed under vacuum at 75 °C and poured into a mold coated with releasing agent. The sample was then cured in an air purged oven at 130 °C for 1 h, 160 °C for 2 h and 270 °C for 4 h.

2.3 Preparation of comparative samples

The neat epoxy sample was prepared by mixing an epoxy resin with Ethacure 100-LC at weight ratio of 3.8:1. A commercial available silica/epoxy sample (Nanopox F-400, 2 wt-% silica in epoxy composition) was formed by mixing epoxy, Ethacure 100-LC and Nanopox F-400 at the weight ratio of 3.6:1:0.24. The non-functionalized silica/epoxy (2 wt-% silica in epoxy composition) was prepared following the process shown in 2.2 without adding APTMS. These mixtures were degassed under vacuum at 75 °C before transferred into a mold and cured following the same curing process as that for silanized silica/epoxy nanocomposite.

2.4 Material characterization

2.4.1 Morphology and chemical composition

The morphology, size and dispersion of silica were investigated using transmission electron microscope (TEM) that was conducted in high resolution mode using a JEOL 2100F instrument and operated at 200 kV. The samples were cut using a Leica Ultracut UCT ultramicrotome and placed on 200 mesh copper grids. The chemical compositions of nanocomposites were analyzed using an energy dispersive X-ray spectroscopy in a transmission electron microscope (EDX-equipped TEM). The chemical state of elements in nanocomposite was determined by X-ray photoelectron spectroscopy (XPS) that was conducted using a VG Escalab 220i instrument with monochromatic Al radiation and spot size of 700 μm.

2.4.2 Thermal properties

Single-cantilever mode of the dynamic mechanical analyzer (DMA Q800, TA Instruments) was used to measure the dynamic modulus (E′) and glass transition temperature (T_g) of materials by heating the samples from 25 to 250 °C with a ramping rate of 3 °C/min, frequency of 1 Hz and oscillation amplitude of 20 μm. Thermogravimetric analysis (TGA) was performed with a TA instrument Q500 thermogravimetric analyzer. The degradation temperature (T_d) of materials was measured under nitrogen atmosphere by heating the samples to 800 °C at a ramping rate of 5 °C/min. The temperature at the middle of thermal transition of composites was defined as the degradation temperature (T_d).

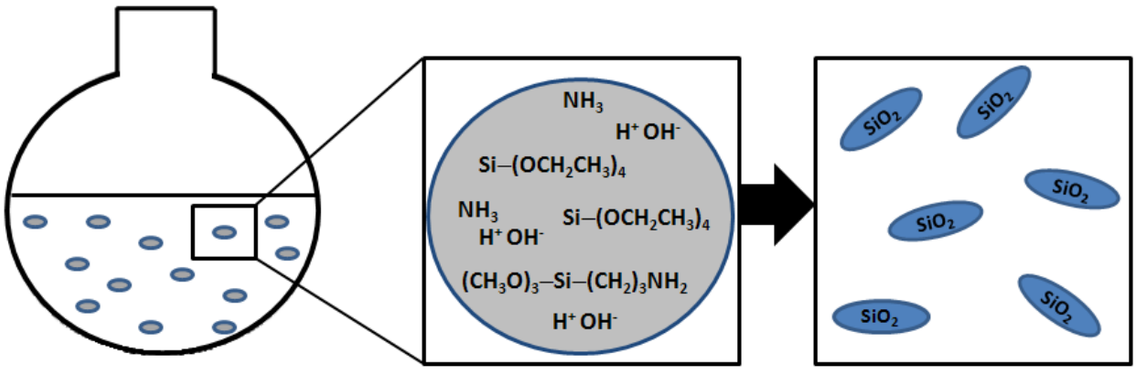
2.4.3 Mechanical properties

The flexural strength and modulus of nanocomposites were determined by 3-point bending test according to the ASTM Standard D 790-96, with specimens of 55 × 13 × 2.2 mm^3. The tests were conducted with crosshead speed of 1 mm/min, at a span length of 40 mm. The tensile tests were carried out according to the ASTM Standard D 638-03 using an Instron 5569 testing machine at tensile speed of 1 mm/min. The specimens were cut into dog-bone shape with dimension of 55 × 3 × 2.2 mm^3. The fracture toughness was measured using the Single-Edge-Notch 3-Point-Bend (SEN-3PB) Tests. The Mode-I critical stress intensity factor (K_{Ic}) was measured using SEN-3PB geometry (span = 50.8 mm) and single-edge-notched (SEN) specimens of 60 × 12.7 × 3.0 mm^3, which meets the plane strain condition requirements. A sharp notch was introduced by pressing afresh razor blade at the bottom of a saw-slot in the middle of the rectangular bar with the Instron 5569 at a crosshead speed of 0.5 mm/min. The tests were conducted on the same Instron 5569 at a crosshead speed of 1 mm/min.

3. Results and discussion

3.1 Synthesis of silica/epoxy nanocomposite

In “Solvent-Free One-Pot Synthesis” method, silanized silica nanoparticles were synthesized through the sol-gel process of TEOS, APTMS and ammonia solution (25 wt-% ammonia in water) in epoxy resin (Scheme 1).



Scheme 1. The formation of silanized silica/epoxy nanocomposite prepared using the "Solvent-Free One-Pot Synthesis" method.

In this process, the water molecules conduct the hydrolysis of TEOS and APTMS, while NH_3 is a basic catalyst that accelerates the reaction (reaction a and b, Scheme 2). The condensation of hydrolyzed TEOS was subsequently occurred to form silica nanoparticles (reaction c, Scheme 2). The hydrolyzed APTMS was functionalized onto the silica surfaces through the reaction between silanol groups of silica and hydroxyl groups of hydrolyzed APTMS to form silanized silica with $-NH_2$ functional groups those can be reacted with epoxy resin to form strong silica-epoxy bonding (reaction d and e, Scheme 2).

The formation of silanized silica as proposed mechanism was confirmed by the transmission electron microscope (TEM) with an energy dispersive X-ray spectroscopy (EDX) and the X-ray photoelectron spectroscopy (XPS). From the TEM image of 4 wt-% silanized silica/epoxy nanocomposite (Fig. 1a), the oval-shaped nanoparticles with diameter of 65-140 nm were uniformly dispersed inside epoxy resin. The preferable oval-shape of these particles (aspect ratio > 1) is possibly occurred through a high shear rate of viscous epoxy\TEOS\APTMS\$NH_3$\$H_2O$ mixture under vigorous stirring. These oval shaped silica nanoparticles could provide added benefit to the mechanical properties of the resulting nanocomposite system as it has higher aspect ratio than those spherical shaped silica. The chemical composition of dispersed particles was confirmed by EDX analysis to be Si, C and O atoms (Fig. 1b).

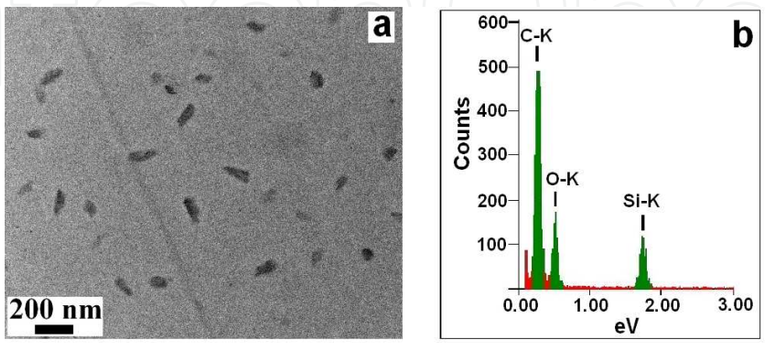
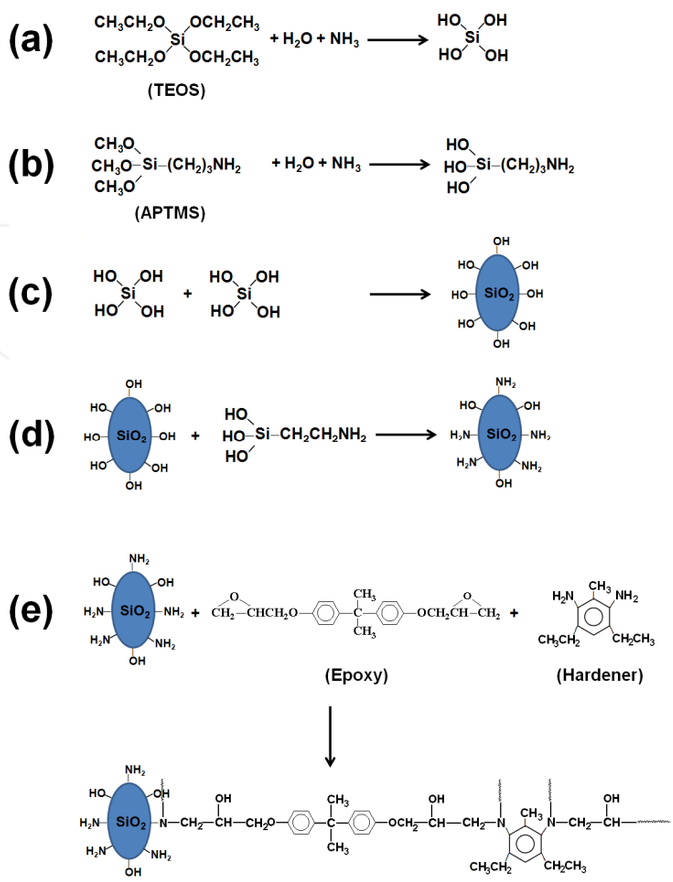


Fig. 1. Homogeneous dispersion of oval shaped silica nanoparticles in epoxy matrix prepared using the "Solvent-Free One-Pot Synthesis" method (a), the chemical composition of silanized silica/epoxy nanocomposite detected by EDX-equipped TEM (b).



Scheme 2. Proposed mechanisms for the formation of silanized silica/epoxy nanocomposite prepared using the "Solvent-Free One-Pot Synthesis" method.

The XPS of silanized silica/epoxy nanocomposite showed broad peak of Si2p corresponding to three Si species (Fig. 2a). The part at highest binding energy (B.E.) of 103.8 eV represents the silanol groups (Si-OH) on silica surfaces. The Si-O-Si structure of silica particles and Si-alkylamine of functionalized APTMS was found at 102.5 and 101.7 eV respectively. The XPS result of non-functionalized silica/epoxy nanocomposite was compared with the above sample to confirm the present of APTMS functionalized on the silica surfaces (Fig. 2b). As expected, only two Si species were obtained from the non-functionalized silica/epoxy sample at 103.1 eV and 102.1 eV, respectively. The species at high B.E. refers to the silanol groups (Si-OH) on silica surfaces, while the one at lower B.E. is Si-O-Si structure of silica particles.

In accordance with the Si2p spectra, the N1s of silanzied silica/epoxy nanocomposite showed two Ni species represented the N1s of Ethacure 100-LC at B.E. 398.9 eV and APTMS at B.E. of 400.3 eV (Fig. 2c). In Etacure 100-LC, N atoms are connected with a stronger electron donor group (benzene ring) than N atoms of APTMS, therefore, they showed a signal at lower binding energy (Liu et al., 2006). However, only one N from C-N linkage between epoxy and Etacure 100-LC at 399.9 eV was observed from the non-functionalized sample (Fig. 2d).

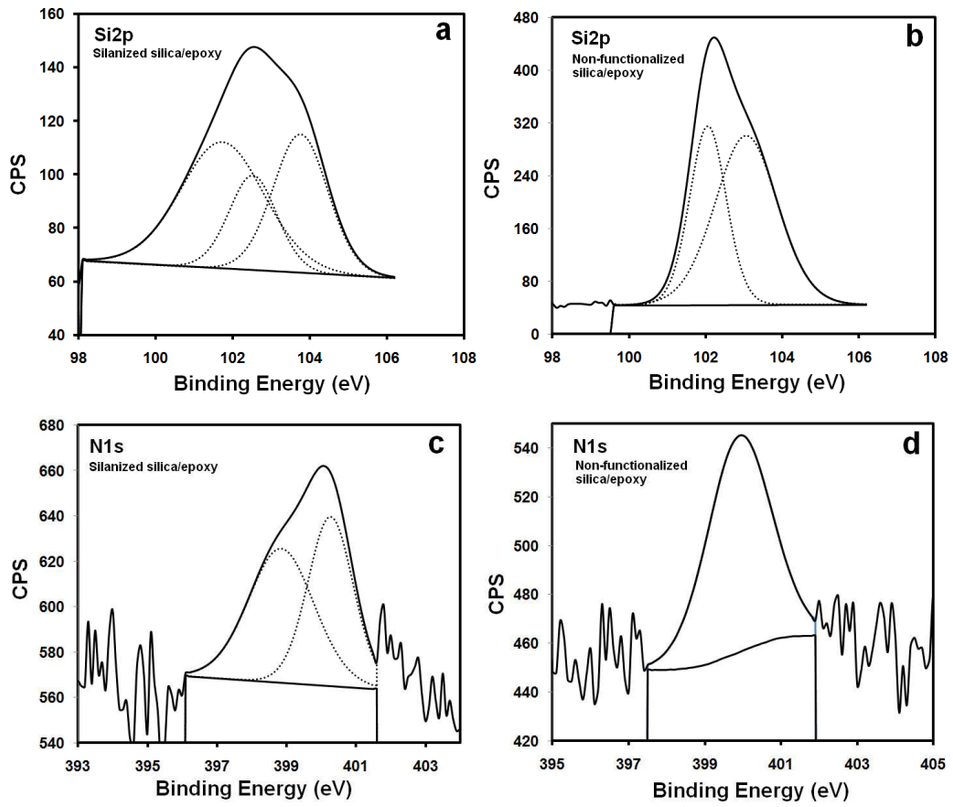


Fig. 2. XPS shows Si2p and N1s curve fit of 2 wt-% silanized silica/epoxy (a and c) and 2 wt% non-functionalized silica/epoxy nanocomposites (b and d).

Therefore, it can be concluded that the oval shaped silica nanoparticles were formed by the sol-gel process of TEOS in the presence of ammonia solution. The APTMS was functionalized on silica surfaces and acts as a linker to form a strong filler-matrix bonding during curing process at elevate temperature of 130-270 °C.

3.2 Thermal mechanical properties of silanized silica/epoxy nanocomposite

The thermal mechanical properties of silanized silica/epoxy nanocomposites were studied at various silica contents of 1-4 wt-% in epoxy composition. The thermal properties of nanocomposites were tested by DMA and TGA. The mechanical properties such as tensile modulus, flexural modulus and fracture toughness were done by tensile, 3-point bending and single-edge-notch 3-point-bend (SEN-3PB) tests.

From the DMA measurement, the glass transition temperature (T_g) of nanocomposites with 0-2 wt-% silanized silica were comparable at 205-210 °C (Fig. 3). The depletion of T_g to 185 °C and 161 °C was occurred when incorporated 3-4 wt-% silica into epoxy resin. Similar trend of result was achieved from the degradation temperature (T_d), in which the comparable T_d at 375-376 °C was shown in 0-2 wt-% silanized silica. However, the T_d of composite became lower to 374 and 360 °C at 3 and 4 wt-% silica contents. The depletion of T_g and T_d at high silica content may occurred from the retardant of cross-linked reaction of epoxy network by large amount of silica particles (Chen et al., 2008).

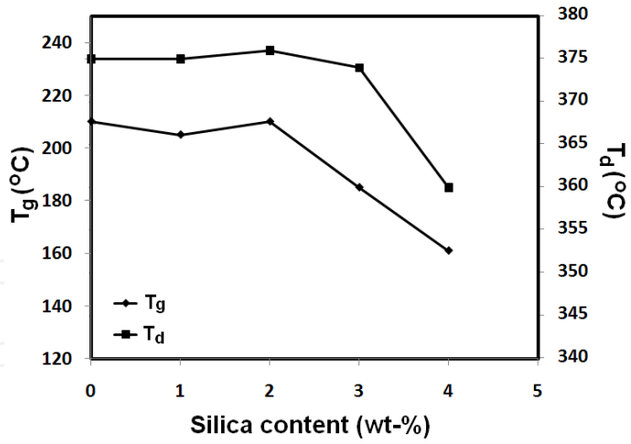


Fig. 3. T_g and T_d of neat epoxy and silanized silica/epoxy nanocomposites prepared at different silica contents of 1-4 wt-% in epoxy composition.

The storage modulus of nanocomposite was increased in accordance with the amount of silica incorporated into epoxy resin (Fig. 4). The similar trend of result was obtained from the fracture toughness, represented as the mode-I critical stress intensity factor (K_{Ic}) (Fig. 5). The K_{Ic} was dramatically improved for 54 % by incorporated 4 wt-% silanized silica into epoxy. The value was increased from 0.35±0.20 MPa.m$^{1/2}$ at 0 wt-% (neat epoxy) to 0.76±0.13 MPa.m$^{1/2}$ at 4 wt-%. It means that the solid silica nanoparticles those uniformly dispersed and formed strong bond with epoxy provide extra reinforcement to the composite structure.

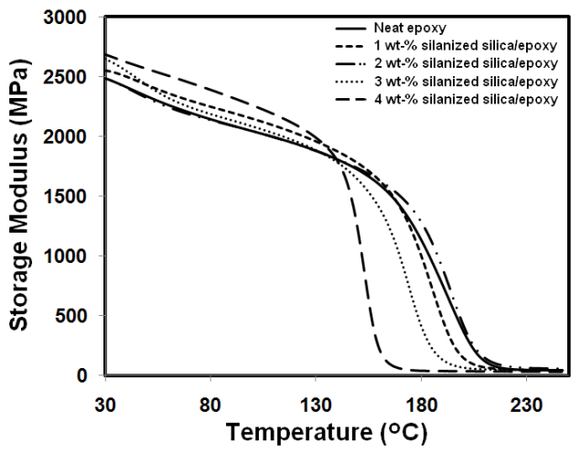


Fig. 4. Plot of storage modulus against temperature of neat epoxy and silanized silica/epoxy nanocomposites prepared at different silica contents of 1-4 wt-% in epoxy composition.

The enhancement of flexural and tensile modulus emphasizes the advantage of oval-shaped silica of the present method to the reinforcement of epoxy resin (Fig. 6). 20% improvement on the flexural and tensile modulus was achieved when introduced few percentages of oval shaped silica (2-3 wt-%) into epoxy, whereas the modulus became maintained at silica content greater than 3 wt-%. As the oval-shaped silica nanoparticles of the present method are uniformly dispersed in epoxy matrix with very low degree of aggregation between particles and form strong bond with epoxy, only few percentages of silica were required to

improve the properties of neat resin. No different on the flexural property was observed when added too small quantity of silica (1 wt-%) into epoxy because the number of reinforced silica particles is not enough to prevent the fracture of brittle matrix. At the optimum ranges where the number of silica nanoparticles is enough to reinforce epoxy structure, the improvements of properties were achieved.

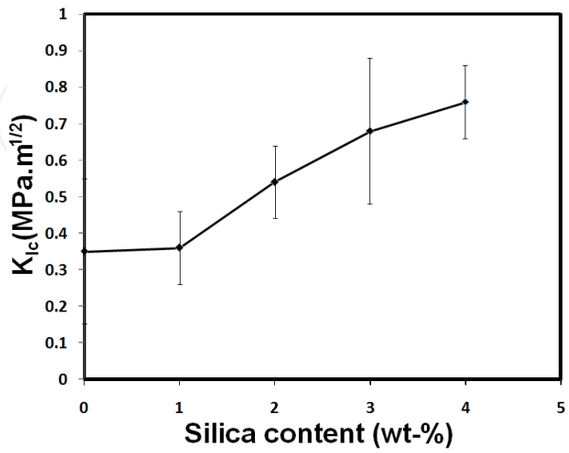


Fig. 5. Mode-I critical stress intensity factor (K_{Ic}) represents the fracture toughness of neat epoxy and silanized silica/epoxy nanocomposites prepared at different silica contents of 1-4 wt-% in epoxy composition. The K_{Ic} value is increased at higher amount of silica content in composite.

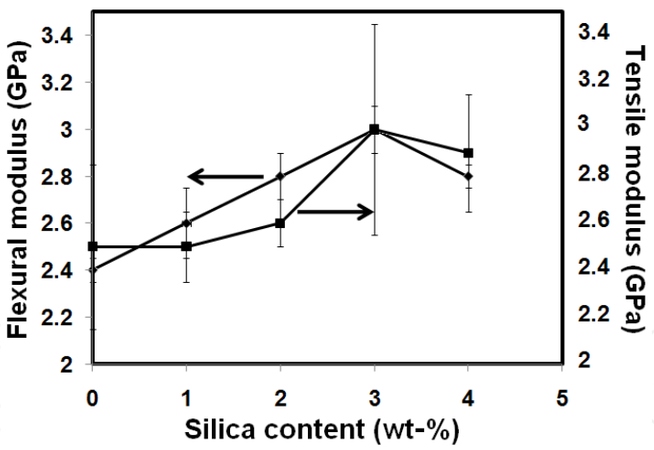


Fig. 6. Flexural and tensile modulus of neat epoxy and silanized silica/epoxy nanocomposites prepared at different silica contents of 1-4 wt-% in epoxy composition.

3.3 Comparison between the thermal mechanical properties of silanized silica/epoxy nanocomposite and comparative references

The silanized silica/epoxy nanocomposite prepared using the present method was compared with three comparative references, which are the neat epoxy resin, non-functionalized silica/epoxy and commercial silica/epoxy. The silica content of nanocomposite systems was fixed at 2 wt-% in epoxy composition.

From DMA and TGA results, it was shown that the T_g and T_d of neat epoxy resin and silanized silica/epoxy were comparable at T_g of 210 °C and T_d of 376 °C (Fig. 7). The commercial silica/epoxy showed slightly lower temperature at T_g of 209 °C and T_d of 374 °C. The lowest T_g was obtained from non-functionalized silica/epoxy nanocomposite at T_g of 207 °C and T_d of 372 °C. It was found from these results that the surface functionalization of silica particles is important to the strength of composite network, in which the thermal stability of nanocomposite structure can be promoted by strong silica-epoxy bonding.

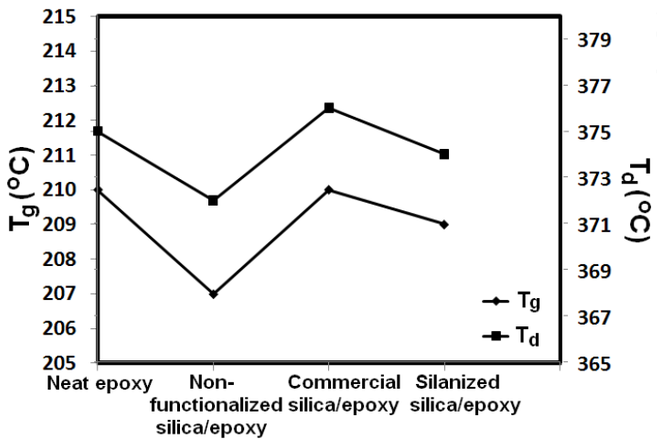


Fig. 7. Comparison between the T_g and T_d of 2 wt-% silanized silica/epoxy nanocomposites and comparative references (neat epoxy, 2 wt-% non-functionalized silica/epoxy and 2 wt-% commercial silica/epoxy).

The comparative results of storage modulus were correspondence with the T_g (Fig. 8.). The highest value was achieved from silanized silica/epoxy at 2510 MPa, while the lower modulus was obtained from non-functionalized silica/epoxy and commercial silica/epoxy nanocomposites at 2330 and 2370 MPa, respectively.

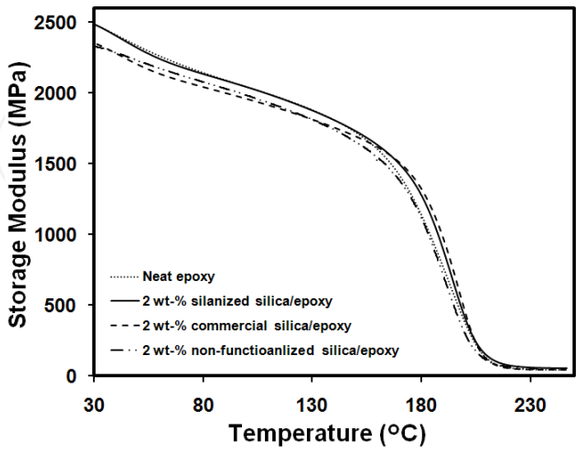


Fig. 8. Comparison between the storage modulus of 2 wt-% silanized silica/epoxy nanocomposites and comparative references (neat epoxy, 2 wt-% non-functionalized silica/epoxy and 2 wt-% commercial silica/epoxy).

The flexural and tensile modulus of neat epoxy resin were found to significantly be enhanced by incorporating silanized silica of the present method (Fig. 9.). In contrast, the poorest properties were achieved when mixed commercial silica with epoxy resin. This may occurred through the aggregation of high concentrated masterbatch of commercial silica (40 wt-%). The size and shape of silica also influence the mechanical properties of nanocomposite. As the silanized silica is oval in shape and its size is suitable for good composite reinforcement, only few percentages of silica are required for the property enhancement. Therefore, the special mixing process may require for the dispersion process of commercial silica in epoxy. Larger amount of commercial silica is also required to achieve a similar range of properties as our silanized silica/epoxy system. Therefore, it can clearly be seen from the comparison that the silanized silica/epoxy nanocomposite prepared by the present method is a competitive method where good thermal mechanical properties of epoxy can be enhanced through one-pot, solvent-free process and required only few percentages of silica.

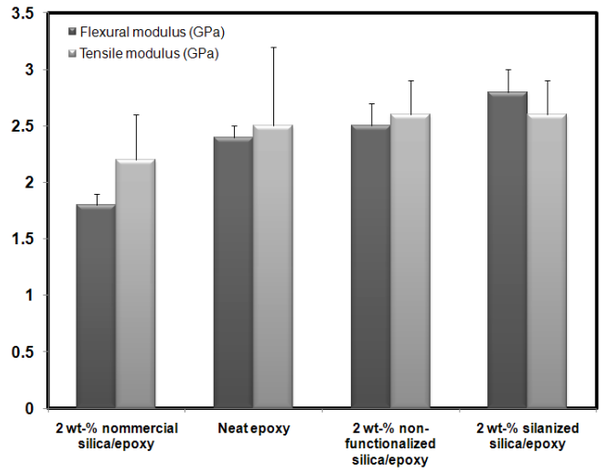


Fig. 9. Comparison between the flexural and tensile modulus of 2 wt-% silanized silica/epoxy nanocomposite and comparative references (neat epoxy, 2 wt-% non-functionalized silica/epoxy and 2 wt-% commercial silica/epoxy).

4. Conclusion

Oval shaped silica/epoxy nanocomposites with uniform silica dispersion and strong silica-epoxy adhesion were effectively and conveniently synthesized via the "Solvent-Free One-Pot Synthesis" method from TEOS, APTMS, ammonia solution, and epoxy compositions at 50°C. Small amount of silica incorporation in the nanocomposite (1-4 wt-% silica in epoxy composition) could enhance the property of silica/epoxy composite significantly. The lower T_g at higher silica loading is due to the decrease of the cross-linked density of epoxy network. The high performance silica/epoxy nanocomposites prepared using the present method exhibit better mechanical properties over neat epoxy (20% and 17% improvements on the flexural and tensile modulus) and commercial available silica/epoxy nanocomposite systems (36% improvement on the flexural modulus).

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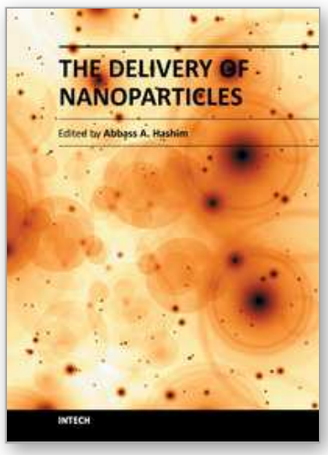

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Nanoparticle is a general challenge for today's technology and the near future observations of science. Nanoparticles cover mostly all types of sciences and manufacturing technologies. The properties of this particle are flying over today scientific barriers and have passed the limitations of conventional sciences. This is the reason why nanoparticles have been evaluated for the use in many fields. InTech publisher and the contributing authors of this book in nanoparticles are all overconfident to invite all scientists to read this new book. The book's potential was held until it was approached by the art of exploring the most advanced research in the field of nano-scale particles, preparation techniques and the way of reaching their destination. 25 reputable chapters were framed in this book and there were alienated into four altered sections; Toxic Nanoparticles, Drug Nanoparticles, Biological Activities and Nano-Technology.

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