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Surface modification of ZnO nano-particles with Trimetoxyvinyl Silane and Oleic Acid and studying their dispersion in organic media

ABSTRACT

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Zinc oxide nano-particles with the average diameter of about 25 nm were modified with different mole ratios of trimetoxyvinyl silane (TMVS) and oleic acid, as coupling agents, in order to modify their surface properties and render them more hydrophobic. Then, dispersibility of the surface modified nano-particles was examined in some monomers with different levels of hydrophobicity, including methyl methacrylate (MMA), butylacrylate (BuA), and styrene with low, medium and high hydrophobicity, respectively. The modified ZnO nano-particles were characterized using elemental analysis (EA), X-ray diffraction (XRD), Fourier transform infrared (FT-IR) and scanning electron microscopy (SEM). Findings revealed that the surface modified ZnO nano-particles were more dispersible in the examined organic media indicating better compatibility. TMVS behaved more efficient than Oleic acid, as coupling agent, to make compatibility with MMA; Whereas, ZnO modified with oleic acid exerted more compatibility with styrene. The mixed coupling agent (50/50 Wt% TMVS/oleic acid) showed better compatibility with BuA.

Keywords: *Surface modification; ZnO Nano-particles; Trimetoxyvinyl Silane (TMVS); Oleic acid; Dispersion.*

INTRODUCTION

Nano-sized ZnO shows great potential applications in preparing solar cells [1], gas sensors [2,3], chemical absorbents [4,5] varistors [6,7], electrical and optical devices [8–10], electrostatic dissipative coatings [11], catalysts for liquid phase hydrogenation [12] and catalysts for photo-catalytic degradations [13–15]. Enhancing homogeneous dispersion of the nano-particles in organic media greatly affect their practical applications in industry. Moreover, high surface energy of the nano-particles causes their agglomeration and limits their applications.

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Surface modification of nano-particles is a practical solution to change chemical and physical properties of the particles and prevent their aggregation in organic media.

Many efforts have been carried out toward the organically modification of nano-particles through various organo-alkoxysilanes [16–18]. Posthumus *et al.* [17] modified various oxidic nano-particles using 3-Methacryloxypropyltrimethoxysilane, and showed that the compatibility of the modified particles with organic media was improved. Grasset *et al.* [18] coated commercial ZnO nano-particles with aminopropyltriethoxysilane (APTES) under different conditions and found that the coating process was controllable, the crystallite size remained almost unchanged, the grafting process didn't modify the transmittance spectra of ZnO, and the aminosilane coating could increase the photostability of the nano-particles. Wang *et al.* [19] used a novel method to provide a silica coating on ZnO particles. They found that the silica coated powders reduced the photocatalysis reaction of ZnO particles. Siddiquey *et al.* [20] reported a rapid coating method based on microwave irradiation for silica coating of ZnO nano-particles. They showed that silica coating effectively reduced the photocatalytic activity of the ZnO nano-particles without significant loss of UV shielding ability and visible light transparency compared to the non-coated sample.

Despite several protocols reported for the surface modification of ZnO nano-particles, to the best of our knowledge, there is no report on the comparison of the effectiveness of different coupling agents for ZnO nano-particles and their compatibility in various organic media. Herein, we wish to report surface modification of ZnO nano-particles with anchoring the chains of TMVS and oleic acid to the surface of ZnO nano-particles. Then, their dispersibility in three monomers including methylmetacrylate (MMA), butylacrylate (BuA), and styrene with different levels of hydrophobicity was investigated. Grafting densities of TMVS and oleic acid on the modified particles and crystalline structure of ZnO nano-particles were also determined.

EXPERIMENTAL

Materials

Commercial zinc oxide with the average particle size of about 25 nm was supplied from Nararra (Spain) TECNAN. Trimethoxyvinyl silane (TMVS), oleic acid, acetone, O-xylene methylmethacrylate (MMA), n-hexane and styrene (St) were purchased from Merck (Germany). Butyl acrylate monomer was purchase from Loba chemie Mumbai, India.

Instrumental analysis

Fourier-transform infrared spectra (FT-IR) of the samples were recorded on an FT-IR 8400S, Shimadzu for which the samples were palletized with KBr powder. Wide angle X-ray diffraction (WXR) patterns were taken on a Philips PW 1800 instrument. The morphology analysis was determined using Scanning electron micrograph (SEM) analyzer (Hitachi, JAPAN. S4160). The specific surface area (SSA) of the modified ZnO nanoparticles was determined from nitrogen adsorption-desorption isotherms using the Brunauer–Emmett–Teller (BET) method. SSA of the modified ZnO nanoparticles was measured using Belsorp II (Japan) BET machine. The amount of grafting density was determined using elementary analysis on a Thermo quest EA 1100.

General procedure for the surface modification of ZnO nano-particles with TMVS and oleic acid

Different amounts of TMVS (1-9, Wt %) were used for the surface modification of ZnO nano-particles. Typically, TMVS (1.5 ml) was dissolved in 50 ml o-xylene to form a solution of 3 Wt% of TMVS relative solvent. Then, ZnO nano-particles (1 g) were added to the above solution and allowed the reaction to perform at 50 °C under stirring for 1 h. Finally, the particles were separated by centrifuge and washed three times with toluene, to remove the un-reacted coupling agents and then dried [21]. Surface modification of the ZnO nano-particles with oleic acid and with mixtures of TMVS/oleic acid, was carried out under similar conditions as described above. The weight ratio of TMVS/oleic acid was selected as 25/75, 50/50, and 75/25.

RESULTS AND DISCUSSION

Dispersibility of the modified ZnO nanoparticles

Dispersibility of the surface modified nano-particles was examined in some monomers with different levels of hydrophobicity, including methylmethacrylate (MMA), butylacrylate (BuA), and styrene (St). For this purpose, certain amount of ZnO nanoparticles was dispersed in each monomer under stirring for 15 min at room temperature. The initial weight percent of the modified zinc oxide varied from 5 to 30 Wt%. Then, ZnO loaded monomers were allowed to stand at room temperature for 24 h. The precipitated part of the nano-particles was separated and the amount of the stable particles in dispersion was determined gravimetrically. The results are shown in Table 1. As can be seen from this table, the results indicate that the majority of TMVS-ZnO nanoparticles precipitate after 24 h in styrene and BuA (column

A- Table 1); While TMVS-modified ZnO nanoparticles formed a stable dispersion in MMA. It can be seen also from the results that when oleic acid is used as coupling agent (column B- Table 1), Oleic acid-ZnO nanoparticles are stable in St, however they were settled down in MMA and BuA. Moreover, when a mixture of coupling agents was used and the ratio of TMVS/oleic acid was varied from 25/75 to 75/25 dispersibility behavior of modified ZnO nanoparticles was changed, solid content of final stable dispersions are shown in Table 2. These findings illustrated that the surface modification of ZnO nanoparticles with TMVS and oleic acid would increase the compatibility between nanoparticles with MMA and styrene, respectively. This contributes to a better dispersion of nanoparticles either in MMA or St. One can justify this fact by comparing the polarity of monomer and relevant coupling agent.

Table 1. Dispersion of ZnO nano particles modified with oleic acid (A) and TMVS (B) in some organic media

Nano-ZnO particles (%Wt)	Dispersion in Styrene (%)		Dispersion in Butyl acrylate (%)		Dispersion in methyl methacrylate (%)	
	A	B	A	B	A	B
5	0.66	4.33	2.63	2	4.4	3.45
10	5.23	8	6.23	3.6	7.66	6.55
15	5	9.33	8.3	4.2	10.2	8.23
20	6.66	12.67	9.83	5.3	13.6	11.33
30	12.66	15.3	14.3	8.33	17.66	14.3

Reaction condition is described in the experimental section 2.3.

(A) modified with TMVS, (B) modified with oleic acid.

Table 2. Dispersion of ZnO nano-particles modified with different mole ratios of mixtures of coupling agents in some organic media

ZnO %Wt	A			B			MMA	BuA	St
	MMA	BuA	St	MMA	BuA	St			
5	0.07	1.15	1.33	1	2.8	2	4	3	1
10	0.08	1.8	2	1.5	7.8	4.8	8.2	2.8	2
20	4.15	5.2	5.6	9.1	9.33	8.78	15.8	13	4.8

St: Styrene; MMA: methylmethacrylate; BuA: butylacrylate.

Reaction condition is described in the experimental section 2.3.

(A) TMVS/oleic acid= 25/75 %Wt; (B) TMVS/oleic acid= 50/50 %Wt; (C) TMVS/oleic acid= 75/25 %Wt.

From Table 2 can be seen that ZnO nano-particles modified with 25/75 Wt% ratios of TMVS/oleic acid coupling agent mixtures showed more efficient dispersion in styrene (column A-Table 2); Whereas, the nano-particles modified with 50/50 Wt% ratios of TMVS/oleic acid were dispersed effectively in BuA.

To gain a better insight to compatibility of modified nanoparticles with monomer, one may consider hydrophobicity of monomer and CA. It is known that the solubility of styrene in water is less than 1 gr/L [22]. The solubility of MMA in water is 15 gr/L [23]. Therefore, ZnO nanoparticles modified with oleic acid (B) are more compatible with styrene because of the long chains of oleic acid which make nanoparticles more hydrophobe.

In order to confirm above results and study the dispersibility of modified ZnO nanoparticles, they were stabilized in a wide range of polar to non-polar solvents, such as water, ethanol, acetone, acrylic acid, 2-ethy hexyl acrylate, and n-hexane. This finding proved that pristine ZnO nanoparticles should have polar characteristics, since they were stably suspended in water as a polar solvent. Whereas, ZnO nanoparticles modified with oleic acid or/and TMVS were not dispersed and easily floated on the surface of water (Figure 1), similar results were obtained in the case of ethanol, acetone (Figure 2), acrylic acid (Figure 3) and Methyl methacrylate (Figure 4), with significant amount of precipitated nanoparticles in bottom of vessels. These results are in accordance with those obtained by Hong et al. [21] In contrary, modified nanoparticles were easily dispersed in 2-ethy hexyl acrylate and n-hexane (Figure 5), and form stable dispersions. This observation indicates that the polarity of nano-sized ZnO nanoparticles was decreased and they behave as a non-polar material after the surface modification by a mono-molecular layer of oleic acid and/or TMVS. As a result, grafting of coupling agents onto ZnO nanoparticles clearly prevented their aggregating and coarsening in non-polar solvents.



Fig. 1. ZnO nano-particles dispersed in water after 24 h under aerobic conditions. Modified with oleic acid (a), with TMVS (b) and unmodified ZnO nanoparticles (c).

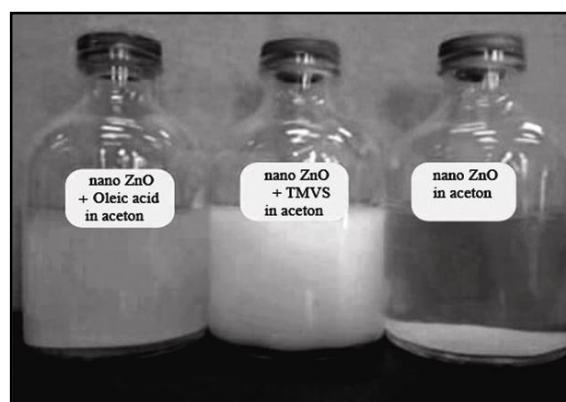


Fig. 2. ZnO nano-particles dispersed in Acetone after 24 h under aerobic conditions. Modified with oleic acid (a), with TMVS (b) and unmodified ZnO nanoparticles (c).

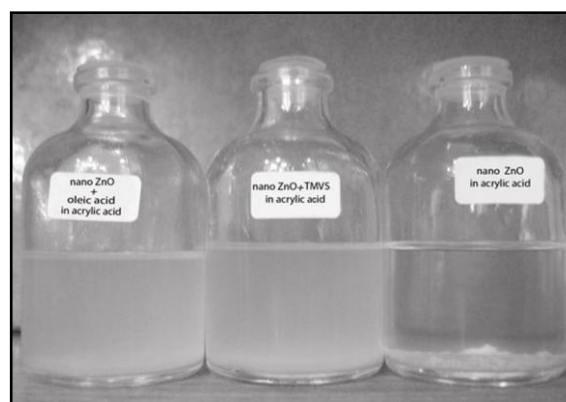


Fig. 3. ZnO nano-particles dispersed in acrylic acid after 24 h under aerobic conditions. Modified with oleic acid (a), with TMVS (b) and unmodified ZnO nanoparticles (c).



Fig. 4. ZnO nano-particles dispersed in Methylmethacrylate after 24 h under aerobic conditions. Modified with oleic acid (a), with TMVS (b) and unmodified ZnO nanoparticles (c).



Fig. 5. ZnO nano-particles dispersed in n-hexane after 24 h under aerobic conditions. Modified with oleic acid (a), with TMVS (b) and unmodified ZnO nanoparticles (c).

Effect of coupling agent concentration

The influence of coupling agent (CA) concentration on the dispersion of ZnO nanoparticles was studied by dispersing 5 Wt% of surface modified nanoparticles in acetone as an organic solvent.

The concentration of coupling agent was varied from 1 to 9 weight percent. The results are illustrated in Figure 6. It can be seen from this figure that both TMVS and oleic acid behave similarly showing better dispersion of ZnO nanoparticles in acetone firstly while CA concentration increases till 5%, then by further addition of CA, the extend of dispersion decreases. This finding indicated that coupling agents reaches to a saturation level probably after single-molecular-layer adsorption of CA close to the concentration of 5% and higher concentration of

coupling agents had no effect on the improvement of dispersion. When the coupling agent concentration was increased further, the long chains of oleic acid entangled with each other, thus hindered the carboxyl group ($-\text{COOH}$) of the oleic acid to react with the hydroxide groups on the surface of ZnO nano-particles [21].

FT-IR spectroscopy

Figure 7(a and b) shows the FT-IR absorption spectra of (a)TMVS-capped ZnO nanoparticles and (b) oleic acid-capped ZnOnanoparticle..Figure 7(a), describes the infrared spectrum of the coupled TMVS-ZnO nanoparticles. The exhibited absorptions bands at 1650 , 1100 , and 941 cm^{-1} would be ascribed to the characteristic peaks of $\text{C}=\text{C}$, OCH_3 , and $\text{Zn}-\text{O}-\text{Si}$ stretching vibrations, respectively [24]. Interaction of TMVS with ZnO nano-particles (Figure 8) would be carried out according to the following reaction.

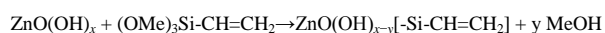
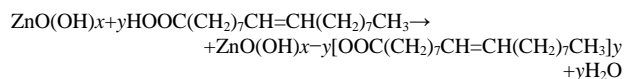


Figure 7b shows the FT-IR spectrum of oleic acid-capped with ZnO nano-particles. The peaks at $\sim 1577\text{ cm}^{-1}$ are devoted to the stretching vibrations of $\text{COO}-\text{Zn}$, implying that the $-\text{COOH}$ group of oleic acid and the $-\text{OH}$ groups on the surface of ZnO nano-particles have conducted the following reaction [21]:



The peaks at 1700 cm^{-1} are assigned to the stretching vibrations of the $\text{C}=\text{O}$ group of the free oleic acid. These observations suggested that a mono-molecular layer of oleic acid would be formed on the surface of nano-sized ZnO [21].

Studying XRD patterns

To investigate the influence of coupling agent on the crystalline structure of ZnO nanoparticles, X-ray diffraction patterns of the particles before and after the modification process were recorded as described in Figure 9. It is clear that the spectra of nanoparticles bearing coupling agents were almost the same as those of the un-modified particles, implying that the modification had not altered the crystalline structure of the ZnO nanoparticles. The obtained XRD patterns illustrated

that after modification, the characteristic peaks have been broadened, however, were still in accordance with the bare ZnO XRD pattern. These findings revealed that the grafted silane coupling agent (TMVS) did not influence the crystalline structure.

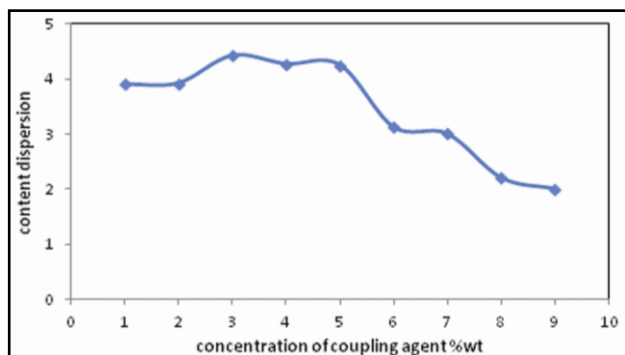


Fig. 6. Dispersion of ZnO nano-particles modified with various concentrations of coupling agents (oleic acid and TMVS) in Acetone.

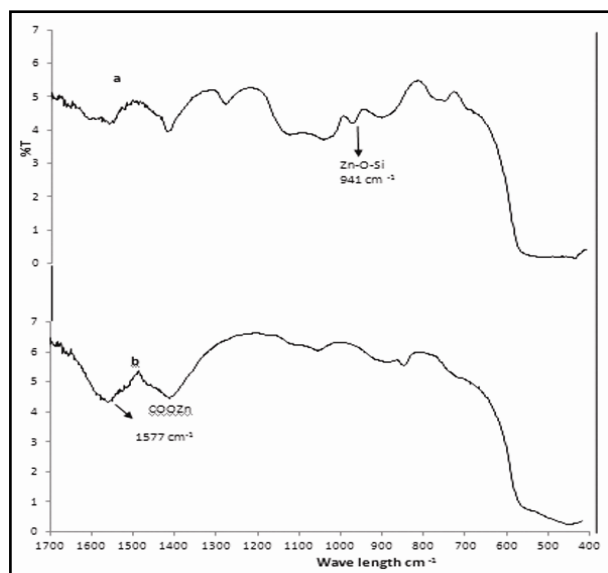


Fig. 7. FT-IR spectra of (a) TMVS-capped ZnO nano-particles (b) oleic acid-capped ZnO nano-particles.

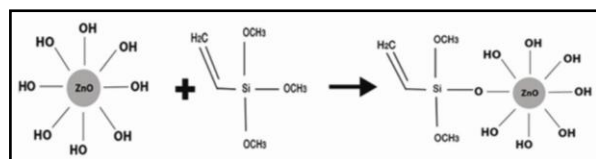


Fig. 8. Chemical modification of ZnO nano-particles with TMVS.

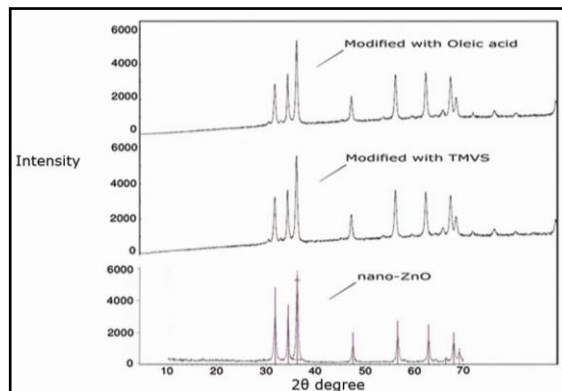


Fig. 9. X-ray diffraction patterns of ZnO nano-particles before and after modification.

Studying SEM images

Figure 10 shows the scanning electron micrographs (SEM) of the bare and the modified ZnO nano-particles. Figure 10a demonstrated that most of the bare ZnO nano-particles have been quasi-spherical and their average diameter was ~25 nm. The SEM images of the ZnO nano-particles and the oleic acid-capped ZnO nano-particles are shown in Figure 10b.

The SEM images confirmed that the morphology of the oleic acid-capped ZnO nano-particles has been close to the un-modified ZnO nano-particles [21]. The SEM images of the ZnO nano-particles before and after modification with TMVS are also shown in Figure 10c. It is confirmed that the morphology of the nano-particles after modification were similar to the un-modified ZnO nano-particles.

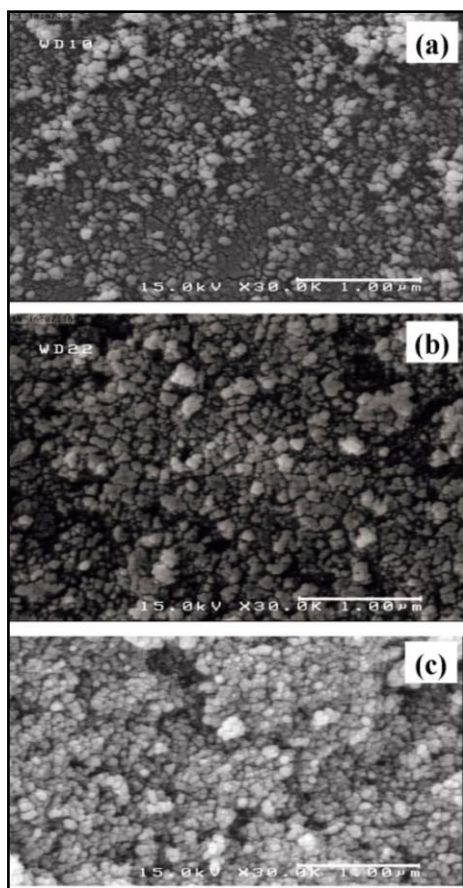


Fig.10.SEM images of (a) ZnO nano-particles (b) Oleic acid-capped ZnO nano-particles (c) ZnO nano-particles modified with TMVS

Specific surface area studies

Specific surface area was determined by N₂ adsorption method using a Belsorp (Japan) BET machine. Samples were degassed at 150 °C for 4 h and N₂ adsorption isotherms were determined until 77 K. The results are shown in Table 3, from which can be seen that specific surface area of the unmodified nanoparticles reduced from 23.716 to 22.57 and 17.03 for TMVS and oleic acid modified nano-particles, respectively.

Table 3. Specific surface area of non-grafted and grafted zinc oxide nano-particles

Compounds	A BET (m ² /g)
ZnO(U.M)	23.716
ZnO(M.T)	22.57
ZnO(M.O)	17.034

U.M: un-modified, M.T: modified with TMVS, M.O: modified with oleic acid. As determined by the BET method, degassed at 150 °C for 4 h.

Elemental analysis (EA)

The amount of coupling agent grafted onto ZnO nano-particles was also quantified by elemental analysis. To obtain the grafting density of coupling agent, the number of silane/ oleic acid molecules chemically bonded to the surfaces of the ZnO nanoparticles was determined by elemental analysis from the difference in carbon content (ΔC, wt %) before and after grafting using the following equation (1) (Berendsen equation) [25].

$$\text{Grafting density } (\mu\text{mol.m}^{-2}) = \frac{10^6 \times \Delta C}{[100 \times M_c \times N_c - \Delta C (M_{CA} - 1)] \times S} \tag{1}$$

Where, ΔC is the difference of carbon content (wt %) between the sample and the unreacted zinc oxide. M_c and N_c are the atomic mass (M_c=12 g.mol⁻¹) and the number of the carbon atoms of the coupling agent, respectively. M_{CA} is the molecular weight of the coupling agent and S is the specific area of the ZnO nano-particles, (SSA= 50 m².g⁻¹). Table 4 shows the weight percent of carbon and the grafting densities of coupling agents on ZnO nanoparticles.

Table 4. Elemental analysis of TMVS and oleic acid grafted ZnO nano-particles

Modifier	[Coupling agent] _x μmol.m ⁻²	Elemental analysis		
		Carbon content (%)	Grafting density μmol.m ⁻²	Grafting yield (%)
TMVS	194	1.6181	5.6	2.88
Oleic acid	94.4	4.1943	70.82	75

Reaction condition is described in the experimental section 2.3

Moreover, the grafting yield, which corresponds to the fraction of coupling agent grafted onto ZnO nano particles, can be calculated using following equation (2) where [coupling agent]_x (imol.m⁻²) is the initial concentration of coupling agent, which is not hydrolyzed yet.

$$\text{Grafting yield (\%)} = \frac{\text{Grafting density}}{[\text{Coupling agent}]_x} \times 100$$

(2)

Grafting density and grafting yield of TMVS is within acceptable range and in accordance with other works [26].

Grafting density is higher for the oleic acid capped ZnO nanoparticles than for the TMVS-ZnO nano particles. This is likely due to different chemical structure and carbon number of among two coupling agents.

CONCLUSIONS

The results showed that it is possible to modify the surface of ZnO nano-particles both with oleic acid and TMVS. The surface modified nano-particles are able to be dispersed in different organic media such as acrylic monomers. Surface modification of ZnO nano-particles render their surface more hydrophobic and make them compatible with organic media. In comparison between TMVS and oleic acid, as coupling agent, the first was more efficient to modify the surface of nano-particles and made them more compatible with organic media. Combination of these two coupling agents allows controlling the hydrophobicity and dispersibility of the ZnO nano-particles in the solvents with various polarity. ZnO nano-particles modified with 25/75 Wt% ratios of TMVS/oleic acid coupling agent mixtures showed more efficient dispersion in styrene; Whereas, the nano-particles modified with 50/50 Wt% ratios of TMVS/oleic acid were dispersed effectively in BuA and ZnO nano particles modified with 75/25 Wt% ratio of TMVS/Oleic acid more efficient dispersion in MMA. Grafting density of TMVS and oleic acid are within acceptable range.

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