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Synthesis and Characterization of Well-dispersed Zinc Oxide Quantum Dots in Epoxy Resin Using Epoxy Siloxane Surface Modifier

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ABSTRACT

Zinc oxide quantum dots were synthesized using poly (dimethyl siloxane) diglycidyl ether terminated, as surface modifiers (SAs) in different concentrations by precipitation method. The epoxy siloxane modifier was chosen in order to improve the compatibility with the polymeric matrix and gain better dispersion. ZnO QDs with size of about 3 nm with optimum properties were synthesized. Structural characteristics and optical properties of synthesized ZnO quantum dots were investigated using Fourier Transform Infrared spectroscopy (FTIR), X-ray diffraction (XRD), photoluminescence (PL) and UV-Vis spectroscopy. They were powdered, purified and finally dispersed in 3 treatment levels in epoxy resin Matrix and they were distributed uniformly in the epoxy resin. The effect of these nanoparticles on the curing process of epoxy resin and 1,3-bis (aminomethyl) cyclohexane (1,3BAC) hardener was investigated using differential scanning calorimetry (DSC). The nanocomposites containing 0.05, 0.1 and 0.15 % ZnO nanoparticles presented respectively 3.6, 15.05, and 12.76 % lower heat flows than the epoxy resin which confirms the barrier effect of these nanoparticles on the activity of epoxy and amine. Prog. Color Colorants Coat. 16 (2023), 399-408© Institute for Color Science and Technology.

1. Introduction

In recent years, semiconductor nanocrystals, also named quantum dots, have attracted a lot of attention due to their unusual optical behavior [1-8]. By controlling the size and structure of quantum dots, their bandgap and emission wavelength can be changed in the infrared to ultraviolet range and they can have a bright and stable emission [8]. Zinc oxide with a wide band gap of 3.37 ev and a critical Bohr radius of about 1.4-3.5 nm is used in many applications including solar cells, sensors, medical applications, and optical diodes [1, 2, 9, 10]. Especially because it is non-toxic and environmentally friendly, widely used in biology and optical imaging [8, 11] and can be used to enhance the resistance of polymers to UV radiation [12-15] However, there are still many challenges regarding their synthesis and cheapening and industrialization, which is why they are used in a limited way in industry [8]. Another major challenge of quantum dots is that they do not have colloidal stability and after a short period of time after synthesis, they undergo the Ostwald ripening and precipitate in reaction medium [11]. Extensive applications require specific nanostructure and in fact needs functionalization and modification of their surface [2, 11, 16-18]. Quantum dots with uniform

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particle size distribution and strong emission can be produced using appropriate surface modifiers during the synthesis process [12, 19]. Stabilization of nanoparticles via organic functionalization, commercially with PMMA, PEG, PVA and Silanes, can be a good strategy to control the agglomeration and surface defects of the nanoparticles [19-21]. However, purified and powdered particles cannot be well dispersed and uniformly distributed in polymer, which limits their use [12]. Zhang et al. used four different materials including 3-mercaptopropyl tri-methoxysilane, polyvinylpyrrolidon, aminopropyl trimethoxysilane and tetraethyl orthosilicate as capping agents to modify the surface of ZnO quantum dots to control their size and proved by TEM that all sizes were below 10 nm [8, 22]. In another work, Liu, used diethylenetriamine (DETA) as ligands to modify the surface of ZnO QDs, it not only passivates the surface defects of ZnO but also suppress the overwhelming electron injection in the QLED [23]. Huang et al. used g-(2,3-epoxypropoxy)-propytrimethoxysilane surface modifier in the synthesis of ZnO QDs. Their synthesized nanoparticles showed very uniform dispersion in epoxy resin and strong luminescence in both dissolved and dry conditions [12]. Wang successfully used Holmium acetylacetonate to create compatibility between zinc oxide quantum dots and epoxy and achieved a uniform dispersion of these nanoparticles with a size of 3 nm in the epoxy substrate and excellent optical properties [24]. In our last work, we investigated the influence of the presence of ZnO QDs synthesized with epoxy siloxane surface modifier on the structural and morphological properties as well as curing behavior of Epoxy Siloxane [25].

In this study, we synthesized zinc oxide quantum dots in the presence of poly (dimethyl siloxane) diglycidylether terminated as surface modifier in four different concentrations via precipitation method. It is assumed that active epoxide groups in the structure of this modifier can create strong physical or chemical bonds with inorganic groups on the surface of ZnO and their compatibility with Matrix leads to good dispersion of QDs in epoxy [20]. Characterization of the synthesized nanoparticles was performed using FTIR, X-ray diffraction, PL and UV-Vis. On the other hand, the effect of these quantum dots on the curing process of epoxy and 1,3-BAC hardener was investigated using DSC. The effect of these nanoparticles on the optical properties of nanocomposite was also investigated. A fairly uniform distribution of these nanoparticles in the epoxy was obtained due to the presence of epoxy groups in the structure of the surface modifier, which made it compatible with the matrix [12].

2. Experimental

2.1. Materials

Zinc acetate dehydrate ($Zn(Ac)_2.2H_2O$,0.99 %), NaOH (99 %,), ethanol (absolute) and n-heptane were supplied from Merck (Germany). Poly (dimethyl siloxane), diglycidyl ether terminated resin (epoxy-siloxane resin) with an average Mn Mw of 800 g/mol and EEW of 490 g/mol was purchased from Sigma-Aldrich (Germany) and used as a surface modifier in the synthesis process of nanoparticles. Bisphenol A based liquid epoxy resin with Epoxide Equivalent Weight (EEW) of 185-192 g/eq was provided by Khuzestan Petrochemical Co., (Iran) and used as a matrix resin in the preparation of nanocomposites. The curing agent was 1,3-Bis (amino methyl) cyclohexane (1,3-BAC) from Sigma-Aldrich (Germany) with Mw of 142.2 g/mol and Amine value of 789 mg KOH/g.

2.2. Synthesis of ZnO quantum dots

ZnO solutions containing 0.02 M NaOH and epoxy-siloxane as surface modifier (SA) at four different amounts (0.1, 0.5, 1 and 2 g) in 50 mL ethanol were heated at 60 °C for 30 min. 0.002 mol (0.37 g) $Zn(Ac)_2.2H_2O$ was directly added to the solution and then heated at 70 °C for 1 h [1]. After that, ZnO QDs were precipitated by n-heptane and the obtained white powder was washed in deionized water and ethanol five times in order to purify the ZnO QDs. Then, they were dried in vacuum for 24 h [1, 2]. The amount of SA was set at 0.1, 0.5, 1 and 2 g which were named as ZQ01, ZQ05, ZQ1 and ZQ2 samples.

2.3. Synthesis of nanocomposites

One of the synthesized ZnO QDs with best performance was selected and added to liquid epoxy resin in four different concentrations, i.e. 0, 0.05, 0.1 and 0.15 % and mixed using sonication method. 1,3-bis (amino methyl) cyclohexane (1,3-BAC) in 1:2 molar ratio used as curing agent. DSC measurements were done using NETZSCH DSC 214 instrument with 4 different hitting rates.

3. Results and Discussion

3.1. Characterization of ZnO quantum dots

3.1.1. Structural studies

X-ray diffraction patterns of ZnO QDs synthesized with different concentrations of SA are shown in Figure 1. There are differences between the patterns that help to choose a sample for making the QDs composite. The main phase of all samples is zinc oxide with hexagonal wurtzite structure (JCPDS No. 01-079-0207). However, some samples show the minor peaks of zinc hydroxide (JCPDS No. 00-001-0360) and event zinc acetate (JCPDS No. 00-001-089). Because of the minimum amounts of secondary phases, the optimum condition is seen in ZQ05. In ZQ05, the purest zinc oxide structure is seen, indicate that all precursors have been completely consumed and no by-products are formed. The concentration of siloxane SA has significant influence on the intensity and full width of half maximum (FWHM) of diffraction peaks. Broadening in diffraction peaks in ZQ01, ZQ05 and ZQ1 can be the result of size reduction effects due to organic functionalization and crystallinity reduction [1, 3-7]. The optimum concentration of SA is needed to help the formation of pure zinc oxide. Excess (ZQ1 and ZQ2 samples) or less (ZQ01) amounts of SA leads to formation of zinc acetate and zinc hydroxide beside the zinc oxide structure. In the other words, less amounts of SA leads to lack of coverage of functional groups of SA and excess amounts of that moves the system towards micelle formation as a rival to quantum dots.

3.1.2. Chemical composition

Fourier Transform infrared spectrum (FTIR) of ZnO QDs prepared with different concentrations of siloxane SA showed a peak at 450 cm^{-1} attributed to the vibration of Zn-O bonds. Strong bonds at 817 and 1200 cm^{-1} correspond to epoxide groups and Si-O-Si, respectively, in all the samples [1, 7, 12]. The intensity of these peaks increased with increasing the SA concentration (Figure 2). A broad peak at ~ 3500 cm^{-1} and a shoulder at 1570 cm^{-1} corresponds to OH stretching and bending vibrations, respectively. The peaks at 3500 cm^{-1} can also belonged to the $Zn(OH)_2$.

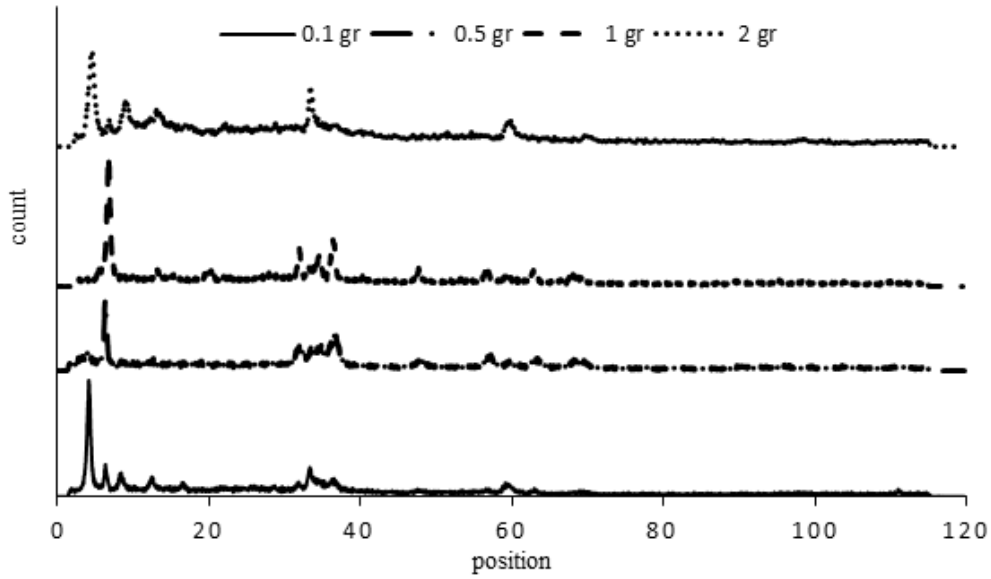

Figure 1: X-ray diffraction patterns of the synthesized ZnO particles in the presence of different amounts of SA.

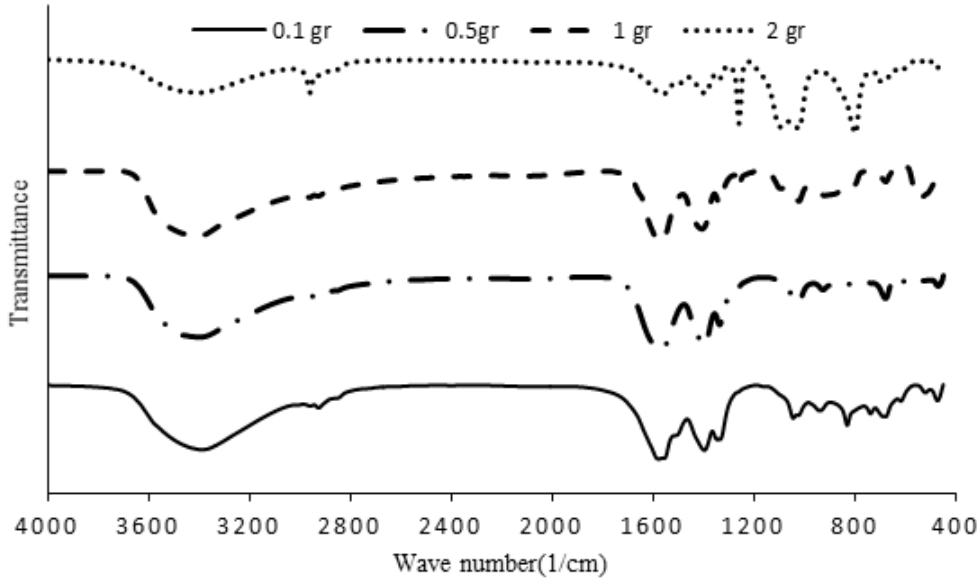

Figure 2: FTIR of the synthesized ZnO particles in the presence of different amounts of SA.

3.1.3. Photoluminescence properties

Figure 3 shows PL spectra of ZnO QDs with different SA concentrations. Some authors believe that PL radiation is associated with surface defects, it is quite clear that changing in the ligand graft density (ZnO: SA ratio) will change the size of the clusters [10, 24]. Generally, by reducing the QDs particle size, the quantum confinement switches the energy of the conduction and valance bands, and leads to a blue shift [4, 13]. Except ZQ2 sample, all specimens in the visible photoluminescence region contain a strong cyan emission peak. The sample ZQ2 showed a blue peak in the visible region, which is the edge emission values of ZnO bulk. It seems that the ZQ2 sample is not in the quantum size region (its size is rather than Bohr radius). The ZQ05 sample exhibits the highest emission intensity which can be originated from the formation of the purest zinc oxide structure [13, 21]. In the other samples, zinc hydroxide or zinc acetate minor phases (Figure 1) act as a competitor for electron – hole recombination. On the other hand, the electron excited by the high energy photons migrates to the zinc hydroxide or acetate instead of radiative transition to relax to the ground state. So, the reduction of luminescence efficiency can be related to the non-radiative transition of electron towards zinc hydroxide or acetate and the conversion to thermal energy [26].

3.1.4. Morphological studies

Morphology of the synthesized nanoparticles and the samples synthesized with different concentrations of surface modifier is evaluated using SEM, as shown in Figure 4. According to the Figure, ZQ01 and ZQ05 have particles less than 10 nm in size, which cannot be accurately measured due to the resolution limitations of the SEM images. On the other hand, the ZQ1 and ZQ2 samples have larger agglomerated particles. In order to specify the shape and size of ZnO QDs nanoparticles, TEM image of the ZQ05 (which was the best candidate for making nanocomposite) is shown in Figure 5. As seen, the particles are relatively separate and are mostly under Bohr radius (2.34 nm) [13]. Because of the existence of a kind of polymeric coverage from SA, QDs seem to be spherical and are partially agglomerated. ZnO QDs are mono-dispersed with an average particle size of less than 3 nm that is in the range of Bohr radius of ZnO and is in good agreement with other zinc oxide quantum dots synthesized with different surface modifiers by other researchers, which are in the approximate range of about 2 to 8 nm [10, 15, 24].

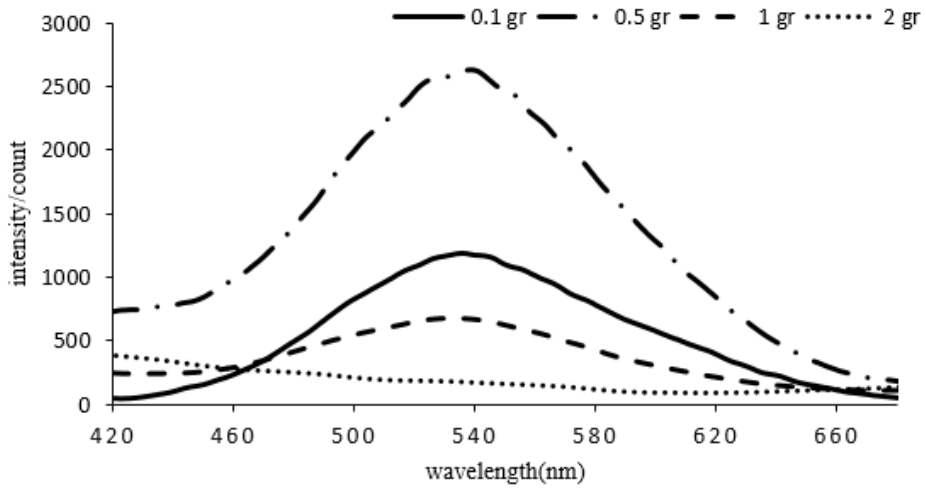

Figure 3: PL spectra of the synthesized ZnO particles in the presence of different amounts of SA.

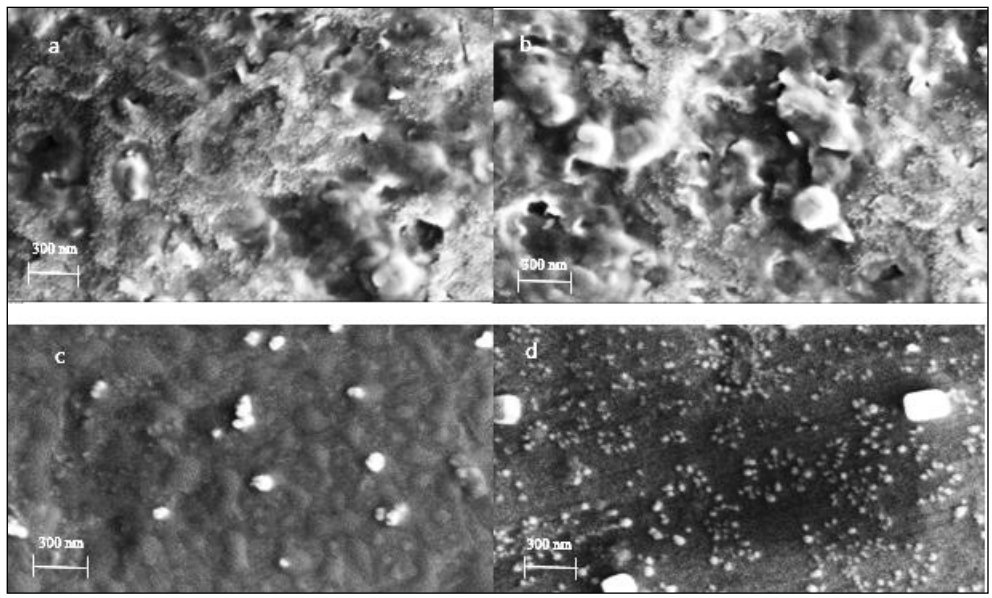

Figure 4: SEM images of the synthesized ZnO particles containing 4 different SA values a) 0.1gr (ZQ01), b) 0.5gr (ZQ05), c) 1gr (ZQ1) d) 2gr (ZQ2).

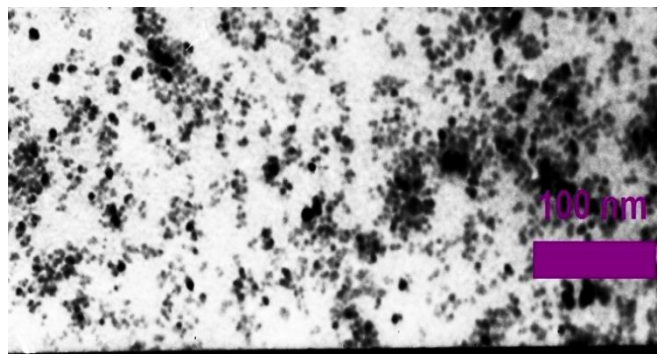

Figure 5: TEM images of the ZQ05 sample.

3.1.5. UV-Visible absorption spectra

The UV-Visible adsorption spectra of ZQ01, ZQ05, ZQ1 and ZQ2 are shown in Figure 6. As can be seen, the efficient UV emission shifts to higher energies with a sharp peak by decreasing the size of ZnO QDs [4]. The quantum dot samples have higher interaction with the incident light and show the exact position of bandgap as the significant changing the slope in the curves (shown by the number on the curves). So, the ZQ01 and ZQ05 sample are clearly in the quantum confinement region with the 4.13 and 3.87 eV bandgaps, respectively. The bandgap values are significantly wider than the bulk ZnO (3.2 eV) [13, 21, 26, 27].

3.2. Characterization of nanocomposites

3.2.1. Thermal Analysis

ZQ05 (ZnO QDs with 0.5 g surface modifier) which represented better features were chosen and added to liquid epoxy resin to investigate their influence on final nanocomposite behavior. Nanocomposites with four ZQ05 concentrations, i.e. 0, 0.05, 0.1 and 0.15 %, were prepared and their curing reactions with 1,3-BAC were studied by DSC. According to the DSC results shown in Figure 7 and Table 1, incorporation of ZnO QDs into the epoxy resin exhibited a reduction in the heat released from curing reaction. The nanocomposites containing 0.05, 0.1 and 0.15 % ZnO QDs presented 3.6, 15.05, and 12.76 % lower enthalpy values than the neat resin matrix, respectively. The ZnO QDs can cause a barrier effect and decrease the probability of reaction functional groups of amine and epoxy. This phenomenon occurred with increasing the concentration of ZnO QDs up to 0.1 %, after which nanoparticles begin to agglomerate and lose their function.

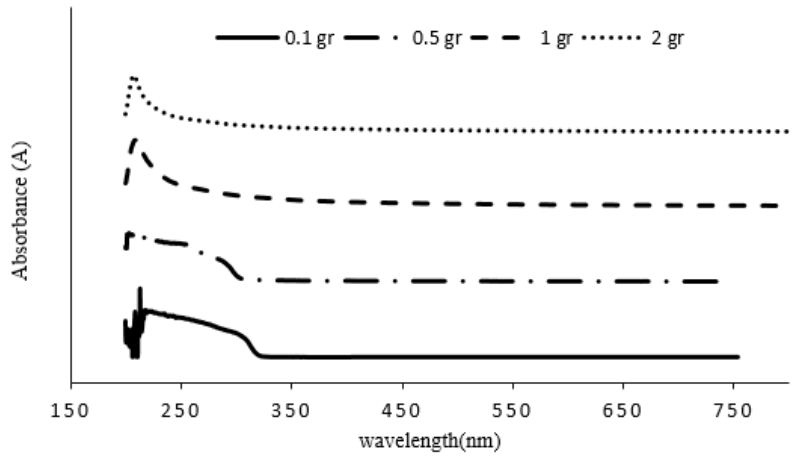

Figure 6: UV-Visible absorption spectra of the synthesized ZnO particles in the presence of different amounts of SA.

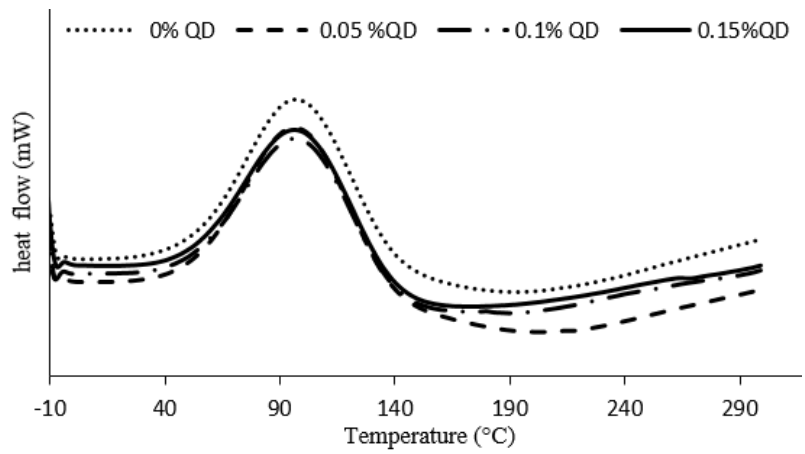

Figure 7: DSC curves of the epoxy/ZnO QD nanocomposites in the presence of different amounts of ZnO QDs.

Table 1: DSC extracted data for the epoxy/ZnO QD nanocomposite.

sample	T_o (°C)	T_m (°C)	T_e (°C)	ΔH (J/g)	r max (1/min)	T (r max) (°C)
0 % QD	54.3	95.7	137.1	174.7	1.72264	97.12144
0.05 % QD	58.2	95.0	136.5	168.4	1.76668	98.27
0.1 % QD	58.5	95.3	142.2	148.4	1.82036	97.41652
0.15 % QD	55.7	95.1	137.1	152.4	1.86957	97.11455

T_o is the onset temperature, T_e is the end-point temperature and T_m is the peak temperature.

3.2.2. Optical properties

Optical transmittance spectra of crystalline ZnO QDs in the vast region from 200 to 1100 nm are shown in Figure 8. For accurate exploration, three regions, i.e. UV, Visible, and NIR regions, were evaluated separately shown in Figures 9-12 and Tables 2 and 3. In UV region (300-380 nm), nanocomposite containing 0.15 % ZnO QDs showed the highest absorption. ZnO QDs are UV absorbent and their higher concentration leads to less transmission of UV irradiation. In visible region, different concentrations of QDs have no effect on the amount of light transmission. And finally in NIR region, the presence of ZnO QDs results in the reduction of infrared irradiation transmission. In other words, ZnO QDs increase heat absorption. The results are quite consistent with DSC results. ZnO QDs which absorb heat will reduce the total released heat during curing reaction. At the same time, sample containing 0.1 % ZnO QDs that has the highest heat absorption, showed the lowest amount of heat released from curing reaction.

Table 2: IR transmittance of the epoxy/ZnO QD nanocomposite.

Nanocomposite	Transmittance (%)
0 % QD	88
0.05 %QD	86
0.1 % QD	84
0.15 %QD	86

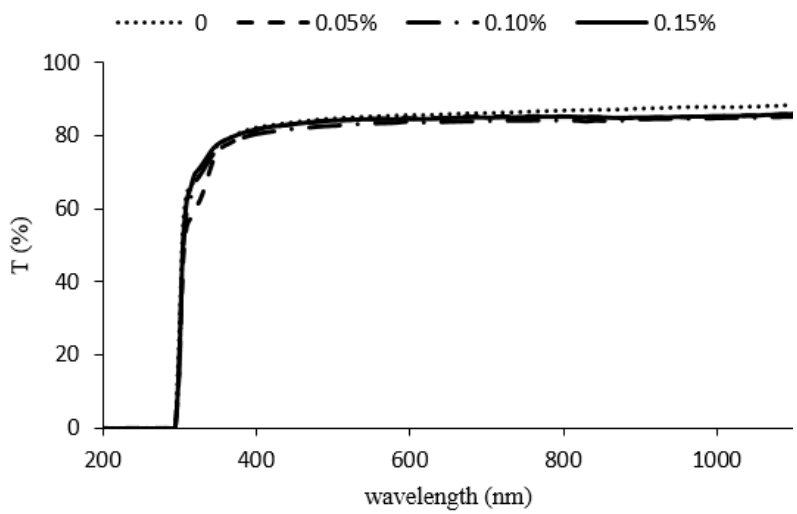

Figure 8: Optical transmittance spectra of the epoxy/ZnO QD nanocomposites in the presence of different amounts of ZnO QDs.

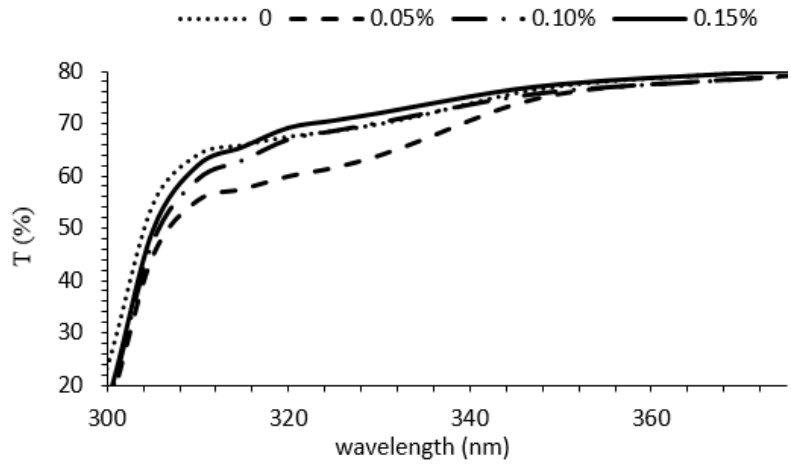

Figure 9: UV transmittance spectra of the epoxy/ZnO QD nanocomposite in the presence of different amounts of ZnO QDs.

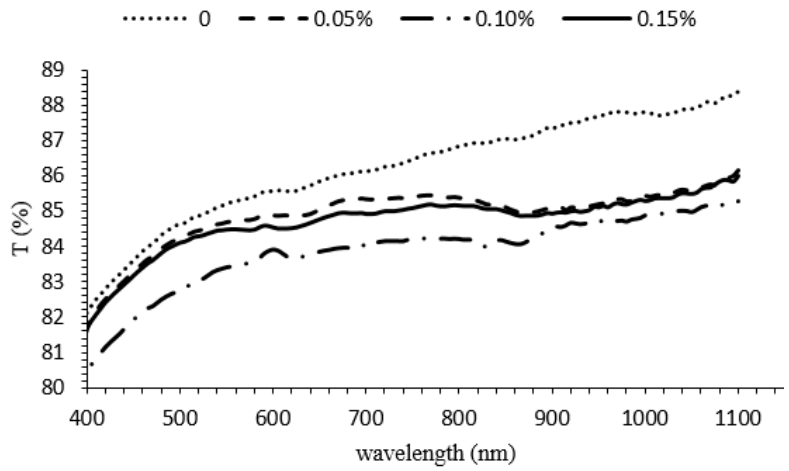

Figure 10: IR transmittance spectra of the epoxy/ZnO QD nanocomposite in the presence of different amounts of ZnO QDs.

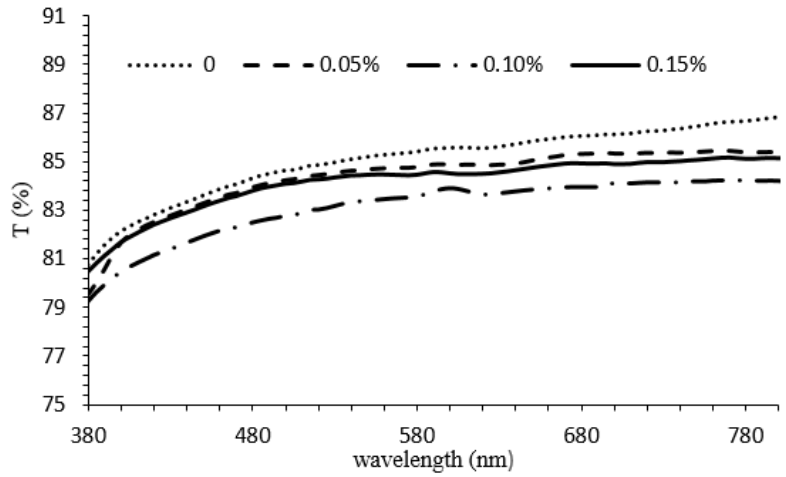

Figure 11: Light absorbance spectra of the epoxy/ZnO QD nanocomposite in the presence of different amounts of ZnO QDs.

Figure 12: The image of transparency of the epoxy/ZnO QD nanocomposite films.

Table 3: Type and characterization of some surface modifiers used in the ZnO QD preparation.

Ref	Surface modifier	Size (nm)	Other effect
23	diethylenetriamine (DETA)	Lower than 20	The PL spectra of QDs on different substrates all present a redshift and decreased
11	different hydroxylated polymers	3.8 to 5	Fluorescence enhancement occurred when ZnO QDs were synthesized in the presence of the hydroxylated polymers
18	siloxane	4.1 ± 0.5	The broad-band visible PL, which originates from surface or defect states, becomes much weaker and undergoes a red-shift of 10 nm and a gradual enlargement compared to starting ZnO@AEPTE QDs
24	Ho(AcAc)3	3.0 ± 0.5	Good dispersion in epoxy and the emission intensity and the UV absorptivity increased.
Our Work	poly (dimethyl siloxane) diglycidyl ether terminated,	About 3	Good dispersion in epoxy and increased the absorbance of UV radiation.

4. Conclusions

In the past decades, the use of quantum dots has expanded considerably, given their desirable propertie. The main problem that limits their consumption is the fact that they cannot be uniformly dispersed in polymeric matrices. Application of suitable surface modifier to enhance the compatibility with polymeric matrix can be a good idea. Epoxy-siloxane surface modifiers led to the synthesis of stable QDs, but their concentration is a key point. In order to investigate the effect of surface modifier concentration on the final properties, four different amounts of QDs were used. ZnO QDs synthesized by 0.0125 M epoxy-siloxane are about 3 nm in size and have blue light emission. These QDs were distributed uniformly in epoxy resin and enhanced its properties. They showed no effect on the transparency of the epoxy film but increased the absorbance of UV radiation. They also played a catalytic role in curing reaction and increased its rate, leading to the incomplete curing of the epoxy.

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