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MUSTANSIRIYAH JOURNAL OF PURE AND APPLIED SCIENCES

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RESEARCH ARTICLE – PHYSICS

Preparation and characterization of Epoxy/TiO_2 thin films for Anti-UVB coating

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Article Info.

Article history:

Received
11 March 2024

Accepted
21 April 2024

Publishing
30 September 2024

Abstract

Thin films of pure epoxy and Epoxy/TiO_2 were successfully prepared using the spin coating method. The epoxy was doped with different weight ratios of TiO_2 nanoparticles for the purpose of studying the effect of increasing its percentage on the properties of epoxy thin films. The surface topography of the prepared films was studied using an atomic force microscope (AFM), which showed that the surfaces of the samples became rougher with an increase in the doping of TiO_2 nanoparticles. The functional groups were measured using a Fourier infrared spectrometer (FTIR), which showed the presence of TiO_2 bonds in the epoxy matrix at wave numbers 400 and 862 cm^{-1}. Optical measurements for absorbance, transmittance, and reflectivity showed a clear effect of TiO_2 nanoparticles on the optical properties of the prepared samples through high absorbance for ultraviolet ray's B (UVB) and low absorbance for visible light. Reflectivity decreases at the wavelengths of UVB and increases at the wavelengths of visible light with an increasing doping ratio. This makes these thin films candidates for use in anti-UVB coatings.

The official journal published by the College of Education at Mustansiriya University

Keywords: Epoxy/TiO_2, Spin coting, Anti-UVB.

1. Introduction

All polymer compounds containing epoxy groups in their molecular structure are collectively called epoxy resins. Epoxy resin has unique physical and chemical qualities, including exceptional adhesion to both metal and non-metal surfaces, favorable dielectric characteristics, little shrinkage upon setting, reliable dimensional stability in products, elevated hardness, and flexibility [1], [2]. It has favorable characteristics and is resistant to alkali and the majority of solvents, so it is widely used in various sectors of impregnation, laminates, adhesives, coatings, etc. Many researchers have been conducting research on epoxy resin since 1958 and have put it into industrial production at a very fast speed; it has been booming all over the world [3]. Epoxy is often used in optical applications for encapsulating components and providing protective coatings. When choosing epoxy adhesives, spectral transmission and reflection are crucial qualities for two main reasons. Light signals in electro-optical circuits may need to traverse the epoxy joint contact. Understanding the spectrum transmission of a substrate is crucial for choosing the optimal UV-curing epoxy [4]. Epoxy materials have good transparency and chemical properties. With the advantages of stable performance, adjustable mechanical properties, and easy processing and molding, it is becoming more popular and used in optical devices [5]. Research has shown that doping epoxy with nanomaterials improves many of its properties, and thus the demand for it has increased in practical applications. Oxides nanoparticles are dispersed and encapsulated in specific epoxy materials. The prepared epoxy/oxides nanoparticle's composite material combines the functions of nanoparticles properties with the ease of processing epoxy [6] [7]. Epoxy doped with TiO_2 nanoparticles can enhance anti-UVB properties due to the high absorbance of the coating film in ultraviolet region, especially in UVB range (280 to 320 nm). The addition of TiO_2 nanoparticles can improve the UVB reflectance of the coating in the ultraviolet spectrum [8]. The refractive index of epoxy resin can be increased by incorporating TiO_2 nanoparticles, leading to improved optical properties. Size-dependent reflective properties of TiO_2 nanoparticles play a crucial role in the development of reflectors. The surface coating of TiO_2 nanoparticles with polymerizable chelating agents can further enhance their properties. Epoxy mixed with TiO_2 nanoparticles displays customizable features in see-through coatings. Adding TiO_2 nanoparticles allows for adjusting the index of the epoxy material, enhancing its qualities. This adjustability provides the ability to manage the characteristics of the substance, making it suitable for scenarios requiring optical standards to be fulfilled [9] [10] [11]. There are many techniques which epoxy thin films can be prepared, such as: spin coating, dip coating, spray coating and doctor blade coating [12]. The study focuses on producing coatings of epoxy doped with TiO_2 nanoparticles for UVB protection; the properties of thin films were also studied.

2. Experimental procedure

Thin films of pure epoxy and epoxy doped with various weight ratios of TiO_2 nanoparticles were prepared using the spin coating process. Both epoxy and hardener supplied by Easy Pour Epoxy Co., were used, the hardener was (Amine-based hardener) type. The TiO_2 nanoparticles powder supplied by MKnano Co, 99% purity and 50 nm particles size was used. The pure epoxy thin film prepared by the epoxy solution was mixed with the hardener in a ratio of 10:1 and dropped on the glass substrate installed in the spin coating device at a speed of 1500 rpm for 1 minute, then left to dry and harden at room temperature for a day. TiO_2 nanoparticles were added in weight ratios (2, 4, and 6% wt) to the epoxy solution, and it was placed under continuous stirring and then transferred to the ultrasonic bath. After that, the hardener was added to it, and the same previous steps were repeated to prepare Epoxy/TiO_2 thin films. Structural measurements were carried out by an atomic force microscopy type (type TT-2 AFM) and a Fourier infrared spectrometer (FTIR) type Shimadzu FTIR-8400S that was used to measure functional groups of the sample. Photographed the samples using an optical microscope. The absorption and transmittance spectra were measured with an optical spectrometer type (SHIMADZU UV-1650) spectrophotometer. The thickness of the prepared thin films was 450+25 nm, and the optical interferometer method with He – Ne Laser 632 nm was used to calculate it.

3. Results and discussions

The atomic force microscope was used for imaging for the purpose of studying the topography of the pure epoxy and doped with TiO_2 nanoparticles thin films. Figure 1 displays atomic force microscope images of the epoxy and Epoxy/TiO_2 thin films. The images demonstrate that the pure samples' surface is uniform and mostly devoid of spherical protrusions and raised areas. The spin coating approach in creating epoxy thin films is effective. The uniformity starts to shift slowly with the introduction of TiO_2 nanoparticles. As many balls and ridges appear on the surface of the prepared sample after doping and the average grain size decreases, from 66.88 nm to 38.62 nm, this is a clear indication of the effect of doping on the topographic properties of the Epoxy/TiO_2 thin films. Also shows a clear change in the values of both roughness and root mean square after the doping process, from 36.47 nm and 40.00 nm to 14.77 nm and 12.81 nm respectively (see Table 1). Figure 2 represents optical microscope images of pure epoxy and Epoxy/TiO_2 thin films at ×1000 magnification. From these images, it was noted that the pure epoxy thin films are free of defects and agglomerations and have very high homogeneity. The images of the doped thin films showed the appearance of simple agglomerations spread regularly within the epoxy. These agglomerations increase with the increase in the weight ratios of TiO_2 in epoxy thin film; this is due to the agglomerations of TiO_2 nanoparticles.

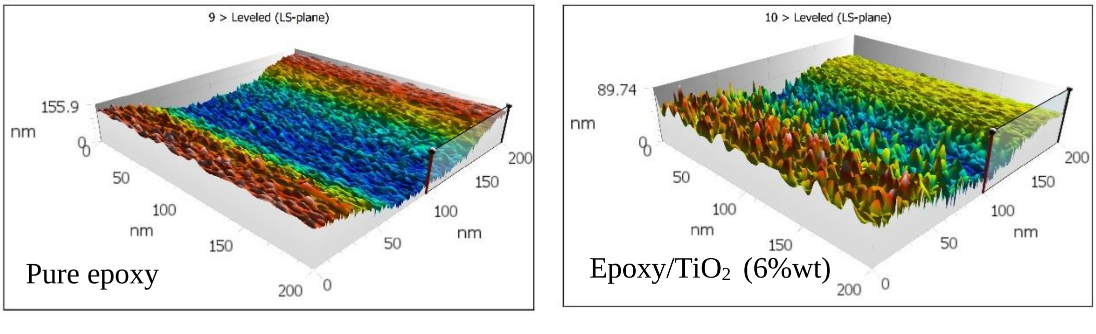

Figure 1. Atomic force microscopy images of pure epoxy and Epoxy/TiO_2 thin films.

Table 1. Topography parameters of pure epoxy and Epoxy/TiO_2 thin films.

TiO_2 weight ratios	Grain size (nm)	RMS (nm)	RS (nm)

0	66.88	40.00	36.47
6%wt	38.62	12.81	14.77

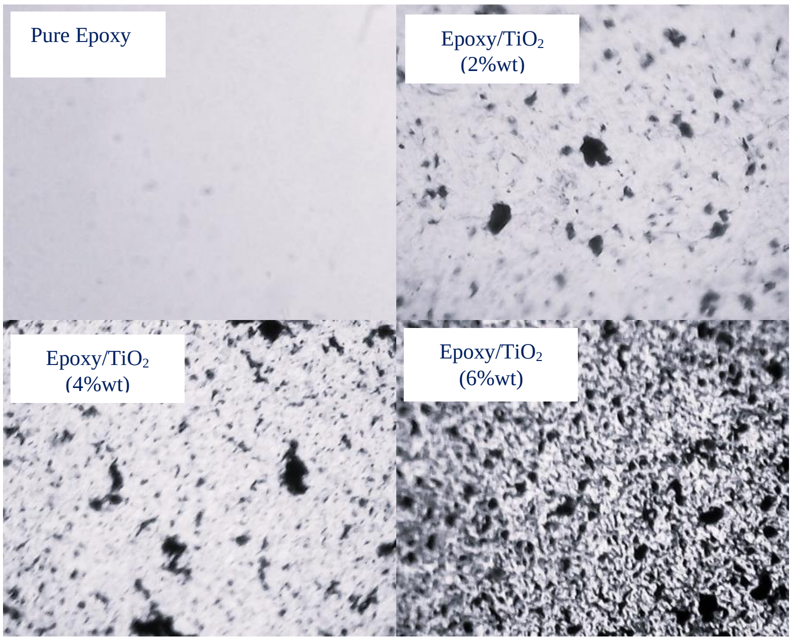

Figure 2. Optical microscope images of pure epoxy and Epoxy/TiO_2 thin films.

FTIR spectroscopy identifies the functional groups present in epoxy. FTIR spectrum can detect epoxide groups (-C-O-C-) present in epoxy, also FTIR can monitor the curing process of epoxy resins. Changes in the FTIR spectrum during curing can provide information about the crosslinking reactions and the formation of new chemical bonds. FTIR can be used to characterize additives, fillers, or modifiers in epoxy formulations. Characteristic absorption bands in the FTIR spectrum reveal the presence of these components. Figure 3 presents the spectra for the pure epoxy and doped with different weight ratios of TiO_2 nanoparticles. The pure epoxy spectrum shows absorption bands at (720, 818, 1040, 1221, and 1662 cm^{-1}), which refer to C-H groups, the C-O-C ring, C-O-C ethers, C-O stretching, and the C=C aromatic ring, which indicate the epoxy strucaral bands [13] [14] [15]. The spectra of Epoxy/TiO_2 show new peaks at 400 and 862 cm^{-1}, which refer to the Ti-O and Ti-O-Ti bands [16]

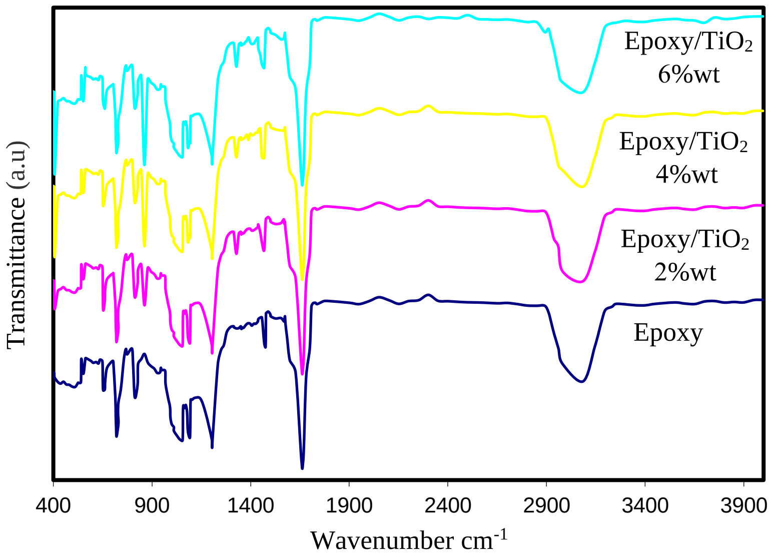

Figure 3. The FTIR spectra of samples.

Absorption, transmittance, and reflectivity spectra of anti-UVB thin films are important aspects of the study and analysis of these thin films. These properties depend on the interaction of light rays with thin films and provide a deep understanding of their optical properties. Figures (4, 5, and 6) indicate the absorption, transmittance, and reflectivity spectra of pure epoxy and Epoxy/TiO_2 thin films in different weight ratios of TiO_2 NPs. Thin films absorb energy at different wavelengths, as reflected in their absorption spectra. These spectra can provide information about the materials that make up the thin films and how they interact with light in a specific wavelength range. The absorption spectrum in Figure 4 shows that the samples exhibit high absorption at wavelengths shorter than 350 nm; beyond this point, the films maintain a stable and low absorption level. On the other side, see the absorption spectrum increasing as a function of increasing the TiO_2 nanoparticle weight ratios, this is due to the uniform dispersion of TiO_2 in the epoxy, thus increasing the effective surface through which the light passes. On the other hand, increasing the optical path that light takes during its passage through the thin films, and thus increases the absorbance [17] [18]. Transmittance describes thin films' ability to pass light through them without scattering or absorbing it. The composition and thickness of thin films affect transmittance and play a critical role in determining the efficiency of thin films in reducing glare and increasing reflectance. Figure 5 shows the transmittance spectrum as a function of wavelength for pure and doped samples. It was noted that the behavior of the transmittance spectrum is opposite to the behavior of the absorption spectrum. Reflectivity expresses the ability of thin films to reflect light rather than pass it. Reflectivity can affect the efficiency of films in reducing glare and improving visibility when used in anti-reflective applications. The reflectivity spectrum was calculated based on the equation below [19] [20].

$$R=1-T-A$$

Shown in figure 6 as a function of wavelength for pure and doped samples. It was found that the reflectance of epoxy thin films was higher in the UV spectrum, specifically in the ranging from 280 to 320 nm (UVB range), while there was a clear decrease in reflectance in the visible region. From another hand shown in this figure, the reflectance decreases in the UVB region and increases in the visible region with increasing weight ratios of TiO_2 nanoparticles, and the maximum reflectance value in the end of UVB region 0.21 at 318 nm, and it shifts very little forward the visible region with doped. From the results of the absorption, transmittance, and reflectivity analyses can be comprehensively understood for the properties of pure epoxy and Epoxy/TiO_2 thin films, and based on the results above, we can use these thin films in anti-UVB coatings in fields such as optics, electronics, and energy applications.

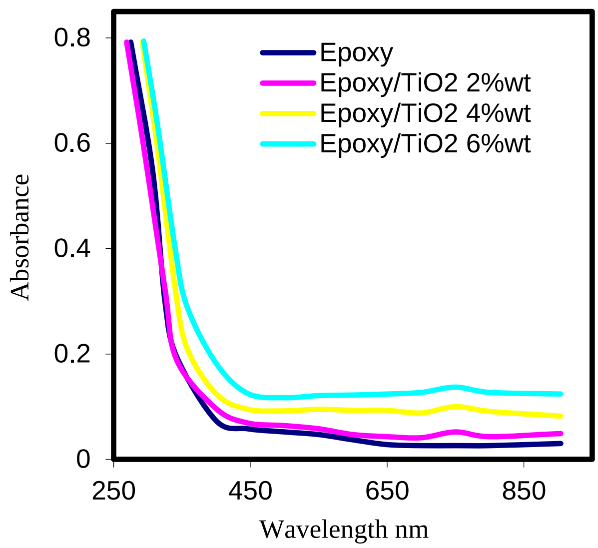

Figure 4. Absorbance spectra of epoxy and Epoxy/TiO_2 thin films.

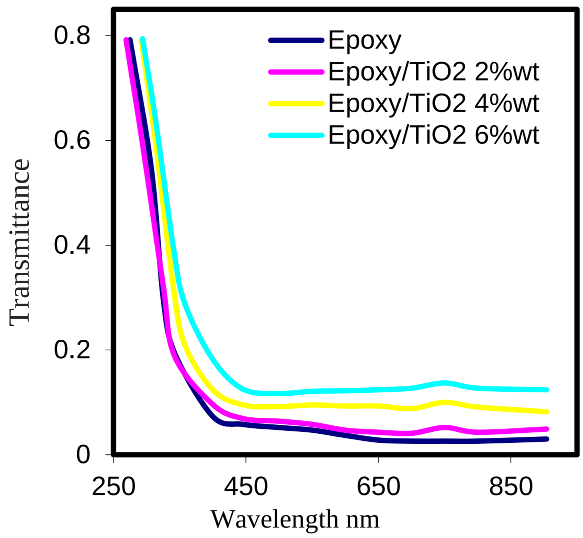

Figure 5. Transmittance spectra of epoxy and Epoxy/TiO_2 thin films.

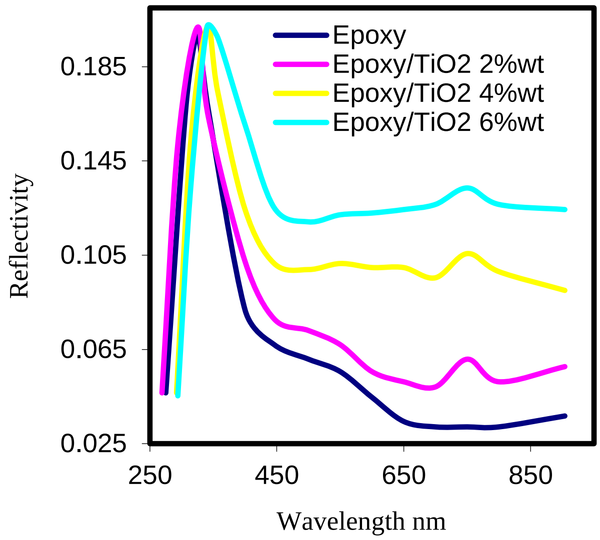

Figure 6. Reflectivity spectra of epoxy and Epoxy/TiO_2 thin films.

4. Conclusions

From the above results, it is clear that pure epoxy and Epoxy/TiO_2 thin films can be successfully prepared using the spin coating method. The addition of TiO_2 NPs to epoxy clearly affects the topography of these thin films, which spreads well through the epoxy matrix. It was also shown through UV-Visible examinations that it is possible to use these thin films in anti-UVB coatings, which is considered one of the rays that pose health risks to humans, while at the same time the thin films almost maintain their permeability to visible light.

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