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RESEARCH ARTICLE

OPTICAL AND DIELECTRIC PROPERTIES OF EPOXY RESIN FILLED WITH TITANIUM DIOXIDE PARTICLES

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Abstract

This work studies the electrical properties for pure epoxy, and epoxy with (0, 1, 1.5, 2 wt%) titanium dioxide powder composites. The effects of titanium dioxide contents on optical and dielectrically properties of the epoxy/ titanium dioxide have investigated by several techniques were used to characterize the epoxy/titanium dioxide: UV-visible spectrophotometer reveals a new absorption band in the wavelength range (350-600) nm. The findings of this investigation show that when the proportion of titanium dioxide added increases, the absorbance increases which are ascribed to interchain interaction. The absorption coefficient (α), extinction coefficient (k), refractive index (n), the real and imaginary permittivity (ε_r, ε_i), and energy gap (E_g) of epoxy/titanium dioxide samples were effectively determined from the recorded data optical transmission technique. In addition, these epoxy have been examined in the wavenumber range (4000-500 cm^{-1}) using an FT-IR spectrometer and also a dielectric constant measurement. The thickness of all the samples is 1.2 mm ± 0.1 mm.

Keywords: Optical, Dielectric Properties, Epoxy, Titanium dioxide.

Introduction

Epoxy is a polymer made up of two chemicals known as the resin and the hardener. Monomers or short chains make up the resin. Polymers having an epoxide group at the end make form a chain. Epoxy resins are usually made through a reaction between epichlorohydrin and bisphenol [1]. The chemical structures of most epoxy resins are tetraglycidyl methylene dianiline, bisphenol diglycidyl ether, and phenol formaldehyde [2]. Epoxy resin offers outstanding adhesion, mechanical characteristics, and chemical stability. It also offers benefits such as a low percentage of contraction, low cost, and ease of shape, among others. It is widely employed in dielectric materials, anticorrosion, aerospace, shape, coating materials, and other industrial domains, and has become one of the most important materials for industrial usage [3]. Many research teams from across the globe have concentrated their efforts in the last decade on polymer composites as useful materials for electrical insulation [4], and epoxy is a popular insulating material [5,6] due to its excellent electrical insulating capabilities. Epoxy resins are also chemically compatible with most substrates and tend to wet surfaces easily making them especially well-suited to the composite application [7]. In order to generate composites with improved electrical and mechanical characteristics, titanium dioxide is sometimes added to epoxy. The most significant of these substances such as titanium dioxide, which is an amorphous white powder that is exceedingly stable. It is manufactured and utilized in the workplace in a variety of particle sizes, including fine and ultrafine sizes. As a result, it is widely utilized in a variety of commercial items, including cosmetics, plastics, and paper. As a result, titanium dioxide nanospheres in experimental animals [8] and humans [9] have been deemed physiologically inactive. Studies of the dielectric properties of polymers have increased importance because it provides an understanding to movement of molecular chains and its applications in electrical and electronic engineering. The goal of this research is to see how adding titanium dioxide powder as a filler to raw epoxy (0, 1, 1.5, and 2 wt%) effects on the optical and dielectric characteristics across the wave length range of (200-1100 nm) at room temperature. From the collected data, the absorption coefficient (A), the band gap energy (Eg), the extinction coefficient (k), and the refractive index (n) were determined. Also described and addressed are the fourier transform infrared spectroscopy (FTIR) spectra of neat epoxy and epoxy composites.

2. Experimental

2.1. Materials

All chemicals used in this work were include the following: Epoxy resin commercial compound ultra clear epoxy cast resin used was a widely used bifunctional epoxy resin, diglycidyl ether of bisphenol and the hardener from (Armor Art China Co.), the TiO_2 powder was used from Loba Chemie, India with a purity of 99.9%.

2.2 Applied Analytical Methods

Absorbance *A*, transmittance *T* of the epoxy/TiO_2 samples, over the wavelength range of 200–1000 nm were investigated using UV–Vis Lambda-365 spectrophotometer (Perkin Elmer). The absorption coefficient (α), the band gap energy (E_g), the extinction coefficient (k), the real and imaginary permittivity parts (ε_r, ε_i), the refractive index (n) are calculated from the recorded data. FT-IR Spectrometer (Perkin Elmer) in range 4000-500 cm^{-1} to use for determinate of the functions group in structure. Dielectric constant measurement (NV6111) India.

2.3 Preparation of Epoxy/ Titanium Dioxide

The epoxy resin and hardener are mixed together in a molar ratio (3:1). Epoxy/TiO_2 were prepared using the magnetic stir 1000 rpm at temperature 75 ℃ mixing method to disperse TiO_2 fillers in epoxy resin as described in previous work [10] to obtained epoxy/TiO_2 samples with 0, 1, 1.5 and 2 wt % TiO_2 contents, have thicknesses about 1.2 mm.

3. Results and Discussion

3.1. Infrared Spectroscopy (FTIR)

The FTIR spectra of sample generally a characteristic peak of functional groups at specified wavenumber is observed by the method of transmission. This method supplies sufficient data by forming relevant peak to ensure about the functional groups that formed during reaction. The Table 1 shows the IR transmittance spectrum as a function of the wavenumber of the epoxy the results showed agreement of this spectrum with the spectrum of pure epoxy recorded in the sources (4000-500 cm^{-1}). The explanation of the spectrum is due to the interaction of light with the functional groups in the epoxy compound and the occurrence of stretching of the O-H, -C-H bonds, which vary in response to the frequencies of red and infrared light according to the results shown in the Table 1 presents the principal FTIR vibrations for the epoxy resin and is divided by section according to the chemical structure of the epoxy resin [11,12]. The IR spectra appearance small shift in spectrum revealed the interference between epoxy and titanium dioxide [13,14].

Table 1: Wavenumbers of FTIR peaks and the corresponding functional groups in neat epoxy.

Wave number (cm^{-1})	Corresponding functional group
~3423	O-H stretching
~3057	Stretching of C-H of the oxirane ring
~2965	Corresponds to asymmetrical C-H stretch of -CH_3 group
~833	Corresponds to –CH out of plane deformation in aromatic
~1036	Corresponds to asymmetrical aromatic C-O
~1618 ~1509 ~1461	Corresponds to C-C stretching vibration in aromatic
~915	Corresponds to epoxide ring vibrations

3.2 UV/Visible Radiation Analysis

The typical normalized UV-Vis spectra of epoxy with filler TiO_2 content of 0, 1, 1.5, and 2 wt % composites are shown in figures 1-11. It is shown that the adding of the filler to the polymer lead to increase the intensity of peak. Critical analysis of UV-Vis spectra of epoxy/TiO_2 composites shows that the highest shift in absorption wavelength is in the range 350-600 nm.

3.2.1 Absorbance Spectrum

Figure 1 illustrates the absorption spectra of epoxy composites with filler TiO_2 content of 0, 1, 1.5, and 2 wt%. It is clearly shown that the absorption increases with increasing percentage of TiO_2 added to the epoxy and shift peak of wavelength up from 320 nm in pure epoxy to 600 nm after add 2 wt% TiO_2, which has been assigned to the first excitation transition occurring in the epoxy/titanium dioxide composite [15]. This is due to the electronic transitions in the frame of the energy bands produced by TiO_2 because the titanium dioxide is a semiconductor material that absorbs visible light [16]. Yang et al. [17] carried out a similar investigation. They investigated how TiO_2 filling affected the optical characteristics of epoxy composites and found that the addition of TiO_2 caused the absorption peak of the composite shift toward the red and increased its absorbance. According to the studies' findings, the optical characteristics of epoxy composites are significantly altered when TiO_2 is added. The electronic transitions in the TiO_2-produced energy band frames are what caused the composite's absorbance to increase and its absorption peak to shift [17].

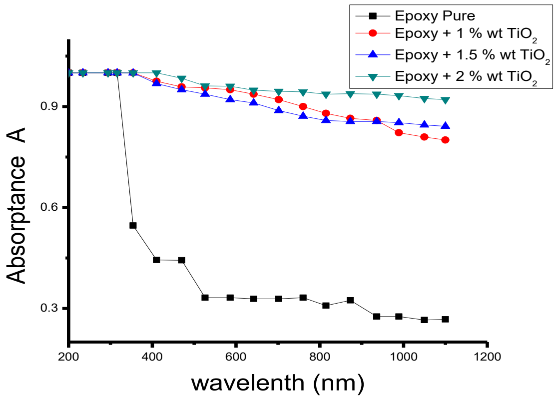

Fig. 1: The absorption spectra of epoxy with filler TiO_2

3.2.2 Transmittance Spectrum

Figure 2 shows the UV-visible transmittance spectrum of epoxy filler with TiO_2, as a function of wavelength. For pure epoxy, showed high transmittance in the range greater than 350 nm, while the rest of the grafted compounds showed a decrease in transmittance for wavelengths less than 600 nm, while wavelengths increases in the period greater than 600 nm with increasing TiO_2 concentrations [16]. In a study conducted by Yang et al. [17], they found that the addition of TiO_2 to epoxy composites resulted in a decrease in the transmittance of the composite. The decrease in the transmittance was attributed to the scattering of light by the TiO_2 particles [17].

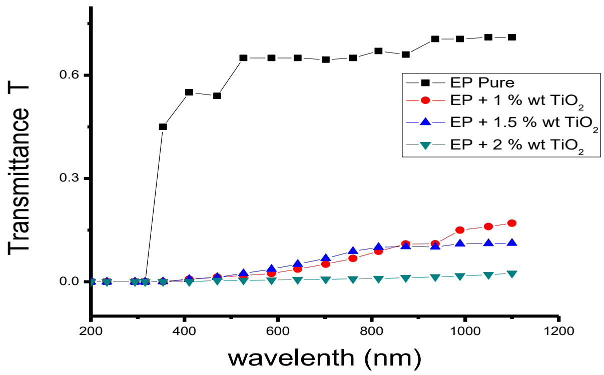

Fig. 2: Transmittance spectrum of epoxy/ TiO_2 as a function of wavelength.

3.2.3 Refractive Index

The refractive index as a function of the wavelength of epoxy grafted with different percentages of titanium oxide Figure 3. The results showed that the reflectivity, depends on the UV/Vis spectrum, the reflectivity increases with the increase of TiO_2 concentrations in the region 500-900 nm, which is the transmittance region for pure epoxy, a decrease in the reflectivity values is also observed in the region less than 400 nm, which is the region where the absorption limit of pure epoxy is located due to the reflecting a mixture of visible wavelengths. In earlier research done by both El-Khodary et al. [18] and Abdel-Aziz et al. [19], the researchers discovered that the amount of TiO_2 improved the epoxy resin's refractive index. This is due to the fact that TiO_2, a highly refractive substance, improved the material's overall refractive index when it was combined with epoxy resin [18,19].

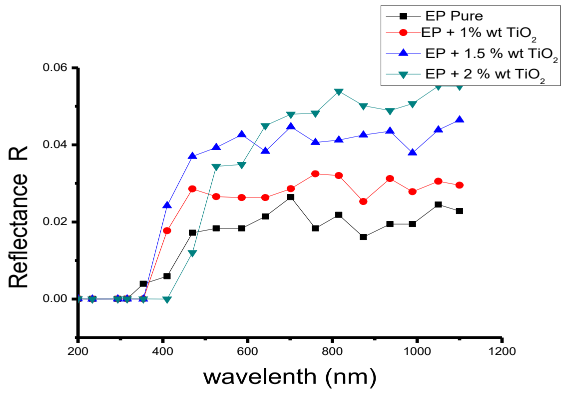

Fig. 3: Refractive index as a function of the wavelength for epoxy/TiO_2

3.2.4 Absorption Coefficient

Figure 4 shows the relationship between the absorption coefficient as a function of the UV and visible wavelength of epoxy filled with TiO_2, and the results showed a shift of the absorption limit of pure epoxy from the ultraviolet region to the visible region, with an increase in TiO_2 concentrations [16,20]. Das et al. [21] earlier study is referred to the optical and thermal properties of the resultant material were examined in this work in relation to the addition of titanium dioxide nanoparticles to epoxy resin. The researchers discovered that the amount of TiO_2 nanoparticles had an impact on the epoxy resin's absorption coefficient. The absorption limit of the epoxy resin was moved from the UV area to the Vis region as a result of the increase in the absorption coefficient of the epoxy resin with the concentration of TiO_2.

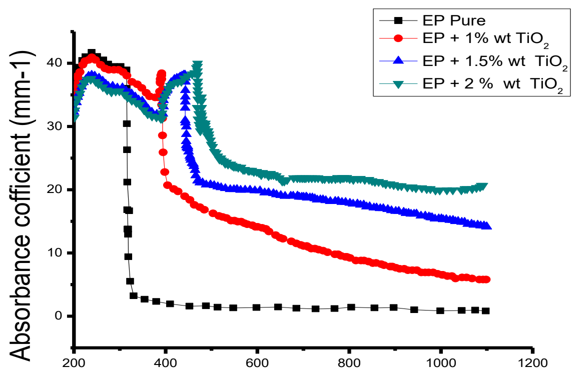

Fig. 4: Absorption coefficient as a function of the wavelength for epoxy with TiO_2

3.2.5 Extinction Coefficient

Figure 5 shows the relationship between the extinction coefficient of epoxy grafted with different percentages of titanium oxide as a function of the wavelength, where the extinction coefficient increases with the increase in the percentage of additions from oxide to epoxy, shift peak extinction coefficient from 320 nm for pure epoxy to 500 nm in epoxy with oxide. Then the extinction coefficient of saturation gradually increases with the increase in the wavelength as a result of the decrease in the distance between the particles of the mixture [20,22]. El-Khodary et al. [19] and Das et al. [21] are two other researches that looked at the impact of TiO_2 on the optical characteristics of epoxy resin. The researchers who conducted these investigations discovered that the extinction coefficient of epoxy resin increased as the amount of TiO_2 particles increased. But the authors also discovered that the sort of TiO_2 utilized had an impact on the epoxy resin's extinction coefficient. When TiO_2 nanoparticles were employed instead of TiO_2 microparticles, the epoxy resin's extinction coefficient was greater.

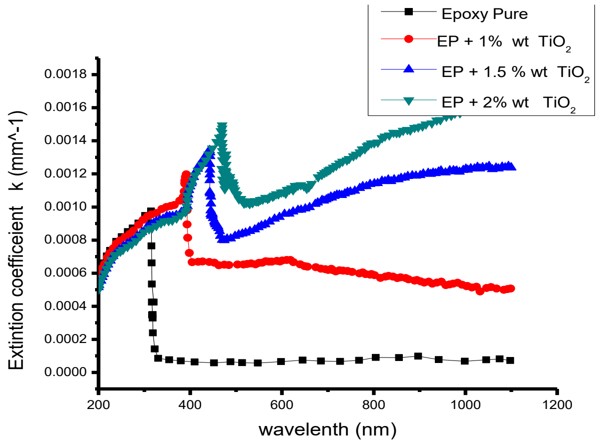

Fig. 5: Relationship between the extinction coefficient as a function of the wavelength

3.2.6 Optical Energy Gap

Ultraviolet-visible (UV/Vis) spectroscopy has become an important tool to estimate the value of optical gap energy (E_g) in epoxy. The optical absorption edge can be correlated to optical gap energy (E_g) using equation 1. To study the changes in the optical energy gap (E_g) of epoxy compounds grafted with different concentrations of TiO_2, the relationship 1 was analyzed and the value of r recorded for all compounds used the differential function when more than one method of electronic transitions is expected, using computer program (Origin lab), where r determines the absorption behavior resulting from the nature of the electronic transitions between the energy bundles of the random structure [16], and it was found that it approaches the direct transitions where the value of r approaches 0.5 for all the prepared compounds, and by substituting the value of r in the relationship 1, and its graph is $(\alpha E)^{1/r}$ as a function of E [16,23,24].

$$\alpha\ (\omega) = \frac{B\ (\hbar\omega - E_{opt})^r}{\hbar\omega} \dots\dots\dots\dots\dots\dots\dots\dots (1)$$

Where: b = constant depending on the band structure, $\hbar\omega$ = photon energy, E_{opt} = optical energy gap for random material, r = constant depends on the nature of electronic translations, α = absorption coefficient.

For epoxy compounds grafted with concentrations of TiO_2, using the relationship 1, where the curves were obtained in Figure 6 and Table 2, it is shown that the value of E_g decreases with increasing concentrations of TiO_2. This is due to the shifting of the absorption limit from UV to the visible region with increasing TiO_2 concentrations, and the resulting change in the electronic structure of the energy bands and the emergence of localized states, which reduced the optical energy gap width of the epoxy by the influence of TiO_2 impurities [25,26].

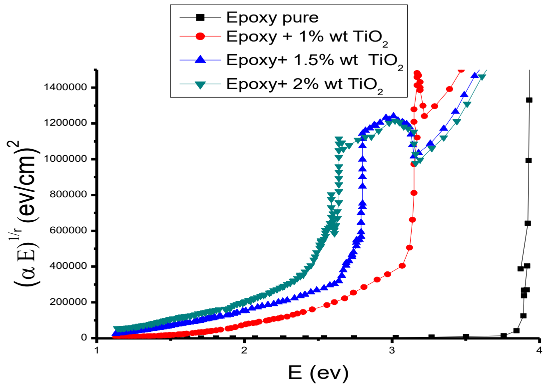

Fig. 6: $(\alpha E)^{1/r}$ as a function of optical gap energy for epoxy with TiO_2

Table 2: values optical energy gap for epoxy/TiO_2

Type of Sample	Thickness Sample (mm)	Optical Gap Energy E_g (ev)
Epoxy pure	1.2	3.8
Epoxy pure	0.25	3.8
Epoxy pure	0.8	3.8
Epoxy + 1% wt TiO_2	0.25	3.2
Epoxy +1.5% wt TiO_2	0.25	2.8
Epoxy + 2% wt TiO_2	0.25	2.5

3.2.8 Real Dielectric Constant

Figure 7 shows the relationship between the real dielectric constant calculated from the relationship 2 as a function of wavelength, and the results showed an increase in the value of the real dielectric constant, with an increase in TiO_2 concentrations in the visible region while decreasing in the spectrum region (200 -350 nm), which is the region of strong adsorption of epoxy, the value of ε_r increased from (1.5-2) for pure epoxy in the visible region to (7-8) for the compound (epoxy + 1.5%wt), the true dielectric constant is related to the refractive index In the relationship 2, and if the behavior of their curves in Figure 7 is compared, it can be said that they are similar, and both depend on the electronic polarization of the insulating compound [27,28], which increases with increasing concentrations of TiO_2.

$$\varepsilon_r = n^2 - K^2 \quad (2)$$

Where: n = refractive index, K = extinction coefficient.

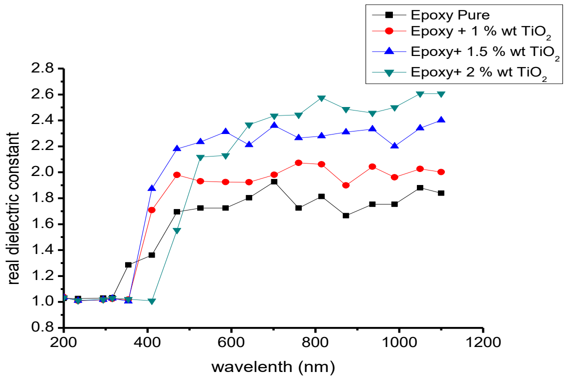

Fig. 7: Real dielectric constant as a function of the wavelength for epoxy with TiO_2

3.2.9. Imaginary Dielectric Constant

Figure 8 show the relationship between the imaginary dielectric constant and the wavelength of epoxy doped with different concentrations of titanium dioxide. The results showed the dependence of the imaginary dielectric constant (ε_i) on the UV/Vis spectrum, but with a different behavior from the real dielectric constant (ε_r), where it increases in the high absorption region (320-400 nm), and decreases in the penetration region is greater than (500 nm), meaning that the imaginary dielectric constant depends on K, and the highest value of the imaginary dielectric constant is shown in the figure 8 as an expression of the peak energy absorption from the electromagnetic field. Due to the occurrence of a match between the frequency of the applied field and the movement of particles of the polar material, the latter works to track changes in the electromagnetic field, which is consistent with the optical properties of dielectric materials [29].

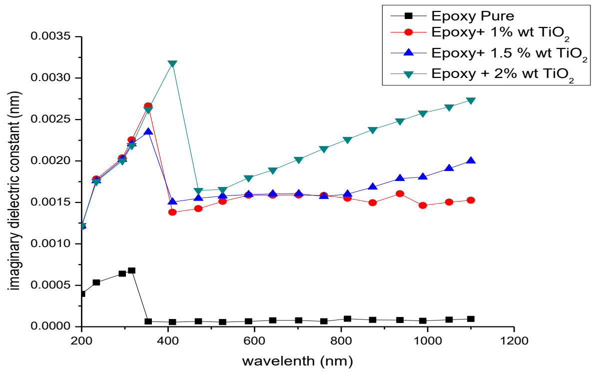

Fig. 8: Imaginary dielectric constant as a function of the wavelength for epoxy with TiO_2

3.2.10 Results to Dielectric Properties

The dielectric properties of epoxy compounds doped with TiO_2, which were studied, are capacitance (C), dielectric constant (ε_r), and capacitive reactance (X_c, at room temperature, 50 Hz) by instrument of a Dielectric Constant Measurement shown in Figure 9-11, to know the effect of each type of additive on the insulating properties and these compounds are: (epoxy pure) and (epoxy + 4%wt TiO_2). Figure 9 shows a diagram of the dielectric constant for epoxy compounds grafted with TiO_2 individually and collectively. The results showed that the dielectric constant values reached 2.3 and 6.09 Table 3 for pure epoxy, and epoxy with 4%wt TiO_2, respectively. The higher capacitance of epoxy/TiO_2 composites makes them useful for a variety of applications, such as capacitors, sensors, and antennas.

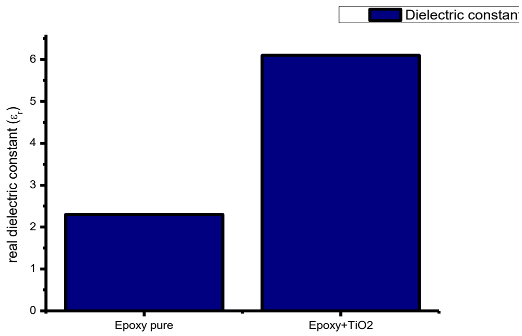

Fig. 9: dielectric constant for epoxy pure and epoxy/TiO_2

This is probably attributed to an increase in the ratio of interfacial electric polarization by the effect of TiO_2, which belongs to the polar insulators, and can be inferred by its solubility in the polar methanol solvent. Table 3 shows the dielectric constant with the additions, individually or collectively and by the effect of adding TiO_2, probably due to the increase in the amount of interpolarization of the dipoles of the TiO_2 and epoxy,

Table 3: results of dielectric properties for epoxy pure and epoxy with TiO_2

Compound	d(mm)	Area (mm^2)	C (pF)	ε_r at 50 Hz	$X_c \times 10^7$ (Ω)
epoxy	3.7	134 X 62	45	2.301	7.07714084
epoxy + 4 % wt TiO_2	3.7	134 X 62	113	6.092	2.81833044

However, even though the relative permittivity of epoxy/TiO_2 composites is lower than that of pure epoxy, the capacitance of the composites can be higher. This is because the TiO_2 particles increase the surface area of the composite, which allows more charge to be stored. The relative permittivity of epoxy/TiO_2 composites is lower than that of pure epoxy because the TiO_2 particles disrupt the regular structure of the epoxy [28].

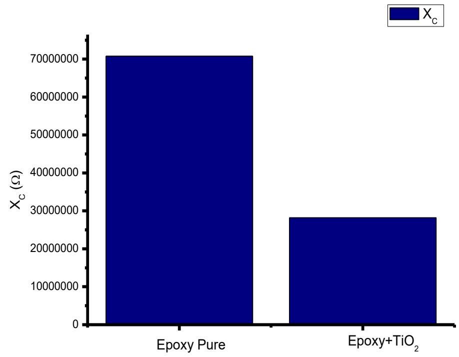

Fig. 10: capacitive reactance for epoxy pure and epoxy/TiO_2

The dielectric constant for these compounds at 50 Hz is called the static dielectric constant; Because it does not depend on the frequency change in the low frequencies range, and in comparison, which is consistent with previous studies in this regard [16,28].

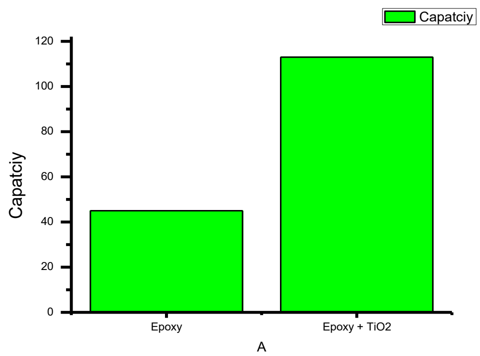

Fig. 11: Capacitance for epoxy pure and epoxy/TiO_2

Conclusion

Through this study, the researchers reached many conclusions, the most important of which are:

- The dependence of the optical properties of all compounds under study on the UV/Vis spectrum, as these properties change with the frequency of the spectrum, and in general, it was found in all compounds that the values of transmittance, reflectivity, and refractive index increased, the real dielectric constant is in the visible spectrum region, while those values are lower in the short wave spectrum (200-350 nm) in the strong absorption region.
- Increasing the values of the optical properties (absorption, reflectivity, absorption coefficient, refractive index, real dielectric constant, imaginary dielectric constant) of epoxy resin with increasing concentration of TiO_2 additives.
- Decrease in the values of optical properties (transmittance, optical energy gap) for epoxy resin by increasing the concentration of TiO_2 additives.
- Oscillating decrease in the optical properties (absorption, reflectivity, absorption coefficient, extension coefficient, refractive index, real dielectric constant, imaginary dielectric constant) of TiO_2 in all regions of the spectrum.
- The addition of TiO_2 to epoxy composites can be used in many applications, including: Electronics (circuit boards and solar cells), coatings, adhesives, rubber, plastics and fibers..etc..

Data Availability

The original data are available upon request from the corresponding author.

Competing Interests

The authors declare that they have no conflict of interest.

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مقالة بحثية

الخواص البصرية والعزلية للإيبوكسي النقي والإيبوكسي المحشو بمسحوق ثاني أكسيد التيتانيوم

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المُلخّص

تضمنت هذه الدراسة قياس الخواص البصرية والعزلية للإيبوكسي النقي والإيبوكسي المحشو بمسحوق ثاني أكسيد التيتانيوم بحسب النسب التالية: (0، 1، 1.5، 2% بالوزن). تم فحص تأثيرات محتويات ثاني أكسيد التيتانيوم على الخواص البصرية والعزلية للإيبوكسي/ثاني أكسيد التيتانيوم من خلال عدة تقنيات: مقياس الطيف الضوئي المرئي والأشعة فوق البنفسجية حيث بينت هذه القياسات عن وجود نطاقات امتصاصية جديدة في مدى الطول الموجي (350-600) نانومتر. اظهرت النتائج أنه عندما تزداد نسبة ثاني أكسيد التيتانيوم المضافة، تزداد الامتصاصية التي تُعزى إلى التفاعل بين الايبوكسي وثاني أكسيد التيتانيوم وتم تحديد: (معامل الامتصاص α، معامل الخمود k، معامل الانكسار n، ثابت العزل الحقيقي ε_r، ثابت العزل الخيالي ε_i، و طاقة الفجوة E_g) لعينات الايبوكسي/ثاني أكسيد التيتانيوم. بالإضافة إلى ذلك تم فحص هذه العينات باستخدام مطيافية الاشعة تحت الحمراءFT-IR في مدى العدد الموجي (4000-500 سم-1) وأيضًا تم قياس ثابت العزل لجميع العينات بسمك (1.2 مم ± 0.1 مم).

الكلمات المفتاحية: الخصائص البصرية، الخواص العازلة، الايبوكسي، ثاني أكسيد التيتانيوم.

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