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Characterization and Physical Properties of PolyvinylChloride/Silica Nanocomposite Films

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ABSTRACT

Using polyvinyl chloride (PVC) and SiO_2 NPs in Tetrahydrofuran (THF). The casting method produced thin films of (PVC) with various amounts of SiO_2 nanoparticles (0.001, 0.002, 0.003, 0.004, and 0.005) g. XRD results showed that the PVC films and SiO_2 NPs had an amorphous structure. The FESEM of PVC/SiO_2 nanocomposite film shows excellent SiO_2 dispersion on the PVC film surface. The FTIR spectrum indicated that SiO_2 NPs did not influence polymer structure. UV-Vis spectroscopy has been used to study the effects of SiO_2 NPs on PVC optical characteristics, including absorption spectrum, transmission spectrum, energy band gap, absorption coefficient, extinction coefficient, refractive index, and real and imaginary dielectric constants. It was found that with increasing SiO_2 NPs amount, the optical energy gap of PVC/SiO_2 nanocomposite films was reduced from 4.36 to 3.39 eV. The study shows that an increase in SiO_2 NPs results in a change in all of these parameters.

KEYWORDS: Physical Properties, Nanocomposite, PVC/ SiO2 Nanoparticle, XRD, FESEM, FTIR, UV-Visible

الخلاصة

تم استخدام اغشية رقيقة من بولي فينيل كلوريد (PVC) و SiO_2 NPs في محلول تتراهيدروفوران (THF)) لتكوين بوليمرات جديدة قابلة للذوبان في .THF بأستخدام طريقة الصب أنتجت أغشية رقيقة من (PVC) التي تحتوي على كميات مختلفة من جزيئات SiO_2 النانوية (٠,٠٠١، ٠,٠٠٢، ٠,٠٠٣، ٠,٠٠٤، ٠,٠٠٥) غرام. أظهرت نتائج XRD أن أغشية PVC و SiO_2 NPs لها هيكل غير متبلور. يُظهر FESEM لفيلم PVC/SiO_2 النانوي تشتت SiO_2 ممتاز على سطح فيلم PVC. أشار طيف FTIR إلى أن SiO_2 NPs لم تؤثر على بنية البوليمر. تم استخدام التحليل الطيفي للأشعة المرئية وفوق البنفسجية لدراسة تأثيرات SiO2 NPs على الخصائص البصرية PVC، بما في ذلك طيف الامتصاص وطيف الإرسال وفجوة نطاق الطاقة ومعامل الامتصاص ومعامل الانقراض ومعامل الانكسار وثوابت العزل الحقيقية والخيالية. وجد أنه مع زيادة كمية SiO_2 NPs، تقل فجوة الطاقة الضوئية لفيلم المركب النانوي PVC/SiO_2 من ٤,٣٦ إلى ٣,٣٩ فولت. أظهرت الدراسة أن الزيادة في SiO_2 NPs تؤدي إلى تغيير في جميع هذه المعلمات.

INTRODUCTION

Many applications for polymeric materials pique the interest of technology and science specialists. It is useful for developing novel medicinal, industrial, and electrical applications because of its high resilience, lightweight design, and excellent optical properties [1][2]. PVC polymer has recently emerged as one of the most versatile and practical materials on the market. They have been used as composites, blends, and copolymers in several applications [3]. In terms of manufactured resin volume, it is ranked second in the world [4]. PVC membranes have numerous applications due to their chemical and heat resistance, mechanical qualities, and physical properties [5]. Silica (SiO_2) is an amorphous silicon and oxygen combination. Due to its physical properties and low cost, it is one of the most applied insulators in solid-state physics. This is a fine, white powder that is non-toxic and has several properties, including high thermal stability, improved mechanical properties, and a large specific surface area [6]. It can also be filled with polymers that contain nan-pores to create nanocomposite materials that can be used as optoelectronic components [7]. Numerous studies have been conducted to study the optical properties of PVC polymers by introducing nanoparticles like SiO_2NPs to enhance their structure. M. Abdul Nabi et al (2014) [8], in this study, the optical characteristics of PVC films doped with various

ZnO concentrations are investigated. ZnO concentration increases influence PVC optical characteristics, with the absorption spectrum rising and the transmission spectrum decreasing. The extinction coefficient, refractive index, real and imaginary parts of dielectric constants, and infinitely high-frequency dielectric constants all increased as impurity percentages increased. When ZnO concentrations rise, Urbach energy values decline. T. Abdel-Baset et al (2016) [9]. The researchers studied how silica nanoparticles added to the PVC matrix affected nanocomposite films optical and dielectric properties. The synthesized silica nanoparticles and the films produced from the PVC-SiO_2 nanocomposites were both amorphous, according to XRD examination. An SEM examination of PVC films revealed excellent SiO_2 NPs dispersion. (FTIR) spectra of nanocomposite films show that SiO_2 NPs significantly alter the intensity of functional group peaks. With the addition of SiO_2NPs, the optical band gap was reduced while the refractive index was raised. Nanocomposite films could be used as optical devices according to these results. T. A. Taha (2018) [10], studied the optical characteristics of PVC/Al_2O_3 nanocomposite films made by solution blending and casting at room temperature. An XRD analysis confirms that Al_2O_3 NPs have a rhombohedral crystal structure, while PVC is partly crystallized. The PVC film surface is well coated with Al_2O_3 NPs as seen in SEM images. Increased Al_2O_3 concentration decreased the direct optical energy gap from 5.05 to 3.60 eV and increased the Urbach energy. The optical parameters increased with increasing amounts of Al_2O_3. This study examines the optical characteristics of PVC films doped with nano-SiO_2 in various amounts.

MATERIALS AND METHODS

Theoretical Part

"Absorbance" refers to the ratio between the intensity of absorbed light (I_A) and the intensity of incident light (I_O) of a material [11].

$$A = \frac{I_A}{I_0} \quad (1)$$

Equation (2) can be used to calculate the absorption coefficient (α) using the optical absorption spectrum [12].

$$\alpha = 2.303\frac{A}{d} \quad (2)$$

A: is the absorbance, and d: is the thickness of the film.

The extinction coefficient (k) can be computed using Equation (3) [13].

$$k = \frac{\lambda}{4} \quad (3)$$

λ is the wavelength of the incident light.

According to Equation (4), direct and indirect transitions have the same absorption edge [14].

$$h = B(h\upsilon - E_g)^r \quad (4)$$

Where hυ: It is the energy of the photon, B: a constant whose value depends on the conduction and valence band properties, E_g: direct or indirect transition's energy gap, and r: constant whose value depends on the type of transition. r is equal to 1/2, 3/2, 2, and 3 for allowing direct, forbidden direct, allow indirect, and forbidden indirect transition respectively.

By Equation (5), can determine the reflectivity (R) using the transmittance (T) and absorbance (A) [15].

$$R = 1 - (A + T) \quad (5)$$

Equation (6) can be used to determine the refractive index (n) [16].

$$n = \frac{1 + R^{1/2}}{1 - R^{1/2}} \quad (6)$$

Where R: is the reflectance

The following equation can be used to define the dielectric constant, which clarifies a material's capacity for polarization [17].

$$\varepsilon = \varepsilon_r - \varepsilon_i \quad (7)$$

equations (8&9) can be used to determine real and imaginary portions of the dielectric constant [17].

$$\varepsilon_r = n^2 - k^2 \quad (8)$$

$$\varepsilon_i = 2nk \quad (9)$$

Experimental Work

In this work, pure PVC and PVC / SiO_2 nanocomposite films were prepared by a casting technique. Sabic company supplies polyvinyl chloride (PVC) powder. It has a molecular weight of 6000 g mol^{-1} and a chemical formula $(C_2H_2Cl)_n$. Tetrahydrofuran (THF) with a purity of 99.8% from (LOBA CHEMIE) was used as a solvent. Silica (SiO_2) NPs ((20-30) nm, particle size)

supplied from China. To make pure PVC film, 0.3g of polymer was dissolved in 15 ml of THF. Using a magnetic stirrer, the solution was thoroughly stirred to produce a homogeneous mixture. After allowing them to dissolve completely, pour this mixture onto previously cleaned and dried glass Petri dishes (diameter 5 cm) and leave to dry for 24 hrs. Composite films are formed by evaporating the solvent at room temperature. These films were peeled from Petri dishes. The films produced were semi-transparent. The amounts of SiO_2 NPs added to obtain SiO_2NPs/PVC films were (0.001, 0.002, 0.003, 0.004, and 0.005) g. UV-Visible spectrophotometers ((T 70/T 80) series UV/Visible spectrophotometer) is used for determining absorption and transmission spectra in a wavelength range (200 nm-900 nm). Fourier transformation Infrared (FTIR) spectroscopy was conducted for all films using (Bruker-Tensor 27 with an ATR unit). The structure of PVC polymer and PVC/ SiO_2 the nanocomposite film was investigated using completely computerized XRD (XRD; X'Pert PRO, PANalytical, Netherlands). High-resolution scanning electron microscopy (FEI Company, Dutch of origin) characterized the composition of the surface of pure PVC, SiO_2 NPs powder, and PVC/SiO_2 nanocomposite films.

Results and Discussion

XRD Analysis

The X-ray diffraction (XRD) technique was utilized to characterize the SiO_2 powder, the pure PVC film, and the PVC/SiO_2 nanocomposite films with an amount of SiO_2 NPs. Figure 1a shows an XRD analysis of SiO_2NPs powder to investigate its crystalline structure. An analysis of the XRD results indicates that silica has an amorphous structure since there are no diffraction peaks associated with any crystallization pattern. Amorphous nanoscale silica is characterized by a broad peak at 21.66 degrees. The literature on silica nanoparticles shows similar patterns in previous literatures [18][19]. Figure 1b shows an amorphous structure that is consistent with many studies [20][21]. Figure 1c the XRD spectra showed that the diffraction peak reduced with increasing amounts of SiO_2 NPs. Both the polymer microstructure and the nanoparticles' atomic dispersion in the film were altered by nanoparticle addition [20].

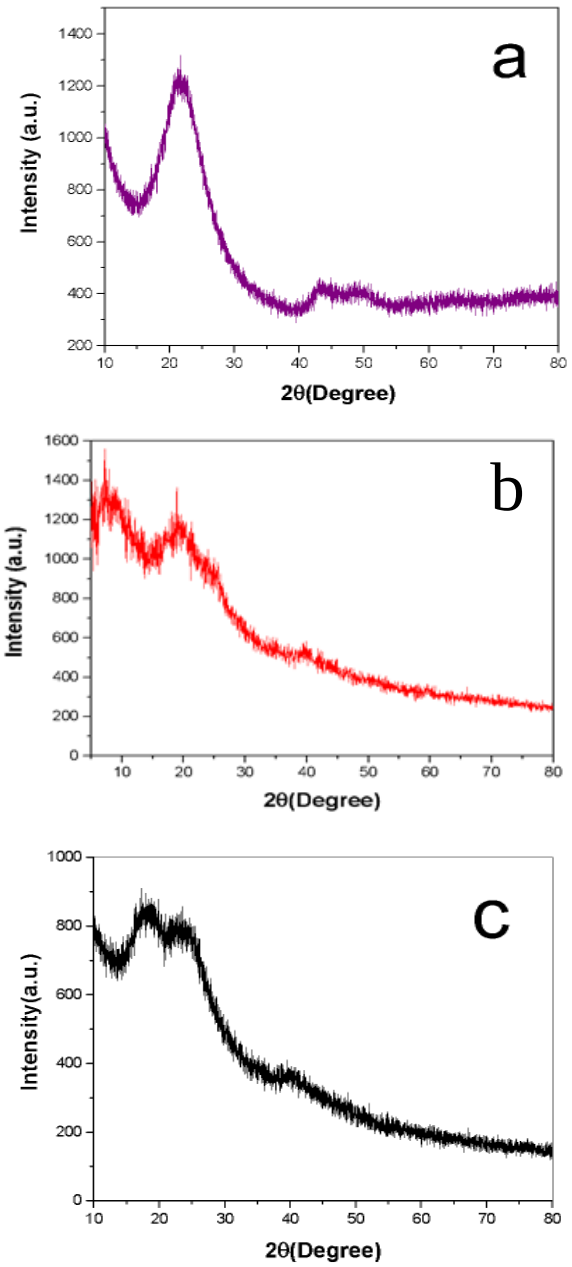


Figure 1. The XRD patterns of a) SiO_2 NPs powder, b) pure PVC film, c) PVC/ SiO_2 nanocomposite film.

FESEM Analysis

Field emission scanning electron microscopy is a technology that, like SEM, generates a broad range of information from sample surfaces, Figure 2-a, b, c shows images of a polymer PVC, a nano-powder of pure SiO_2, and nanocomposite films made of PVC/SiO_2. Figure 2a shows the morphology of pure PVC film. The surface is smooth and without pits or pores. The results were similar to previous research [22]. Figure 2b depicts SiO_2 NPs morphology. As a result, the particles are spherical and granular. These results matched with research [23] and with research [9]. So, Figure 2c shows the

morphology of PVC/SiO_2 nanocomposite films. It is showing an excellent SiO_2 dispersion on the surface of the PVC film. This is in agreement with research [9].

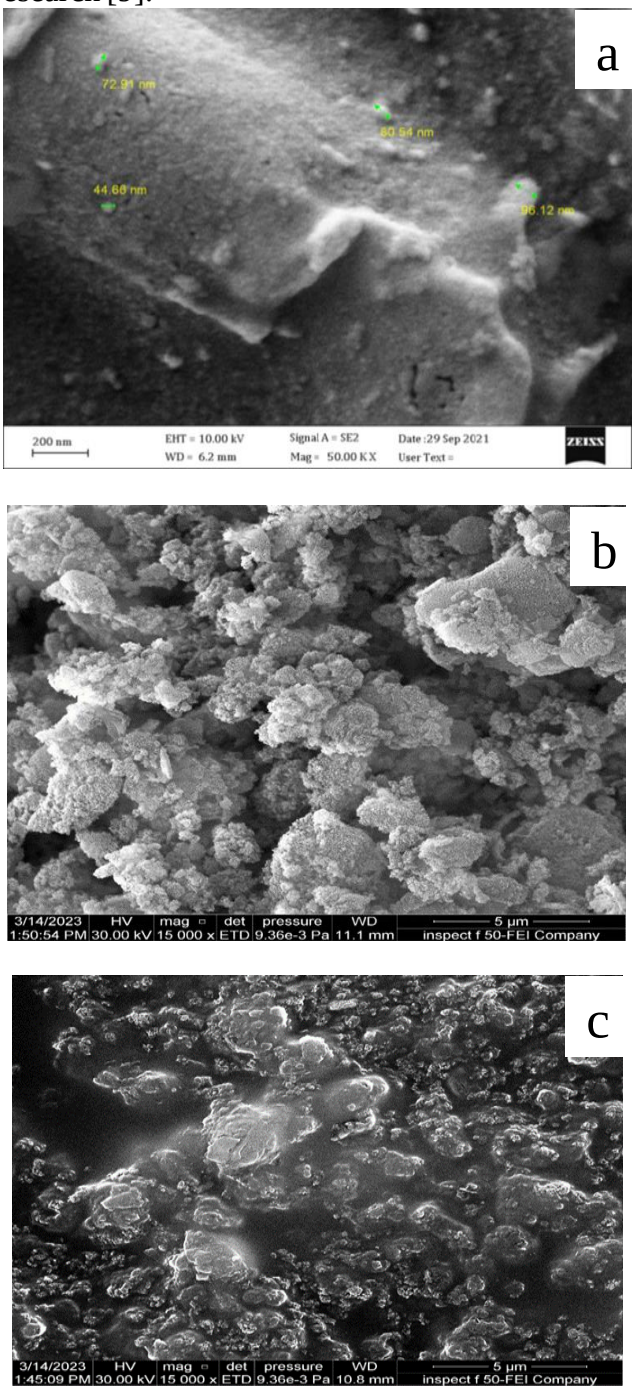
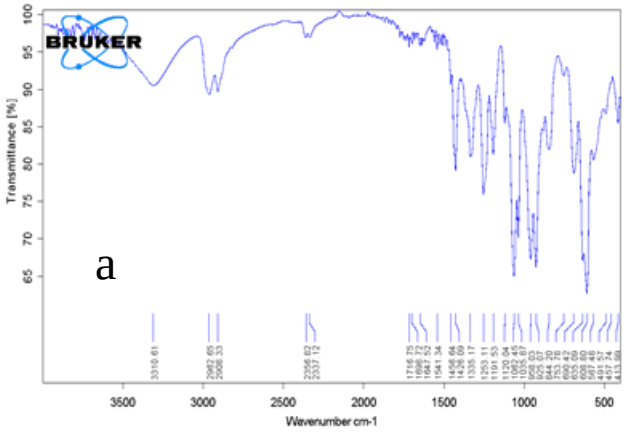


Figure 2. The FESEM patterns. (a) pure PVC film, (b) SiO_2 NPs powder, and (c) PVC/ SiO_2 nanocomposite film.

FTIR Analysis

Fourier-transform infrared spectroscopy is important because it can reveal the key characteristics of the peaks of pure PVC, SiO_2 NPs, and PVC/ SiO_2. The FT-IR spectrum of pure PVC polymer is depicted in Figure 3a. A large number of bands are also tabulated in Table 1. Peaks between (625-970) cm^{-1} indicate C-H bending of the plane, and peaks between (600-800) cm^{-1} indicate (C-Cl) stretching overlaps on the C are (C-H) stretching bands at (635.09 cm^{-1}). C-O stretching vibration is indicated by peaks between (1015 and 1300) cm^{-1}, CH_2 bending vibration is shown by peaks between (1300 – 1380) cm^{-1}, and C=C vibrating stretching is indicated by peaks between (1430 – 1600) cm^{-1}. Carbonyl stretching vibration (C=O) is shown by the peaks that developed between (1550-1780) cm^{-1}. The two peaks appeared at (2908.33) cm^{-1}in addition to (2962.65) cm^{-1} indicating C-H stretch aliphatic vibration respectively. The FTIR spectrum of pure SiO_2 NPs powder is shown in Figure 3b; infrared tests were conducted to determine the nature and purity of metal NPs. O-Si-O bonds exhibit bending vibrations at a peak near 462cm^{-1}. The Si-O-Si symmetric stretching vibration mode of bridging oxygen between tetrahedral structures is associated with a band between 804.08 cm^{-1} and 853.08 cm^{-1} [24]. The peak around 1072.65 cm^{-1} was corresponding to Si–O–Si asymmetric stretching of bridging oxygen between tetrahedral bridging oxygen within the tetrahedral [25]. The hydroxyl group peak for H_2O at cm^{-1} and 1630 cm^{-1}, as well as the peak of the (Si-OH) group at 894.27 cm^{-1}, were not seen. To prove probable intermolecular interaction between the components of nanocomposites, FTIR spectra were studied for (PVC/ SiO_2) nanocomposites and for the different amounts (0.001, 0.002, 0.003, 0.004, 0.005) g as shown in Figure 3(c, d, e, f, and g) to clarify the positions of the peaks that appeared with types of bonds may be seen in Table 1. It observed that there are no interactions between the nanoparticle and the polymer. The transmittance decreases marginally with an increase in the amount SiO_2NPs because of the increase in the density of the nanocomposites with the increase in the number of nanoparticles [26].



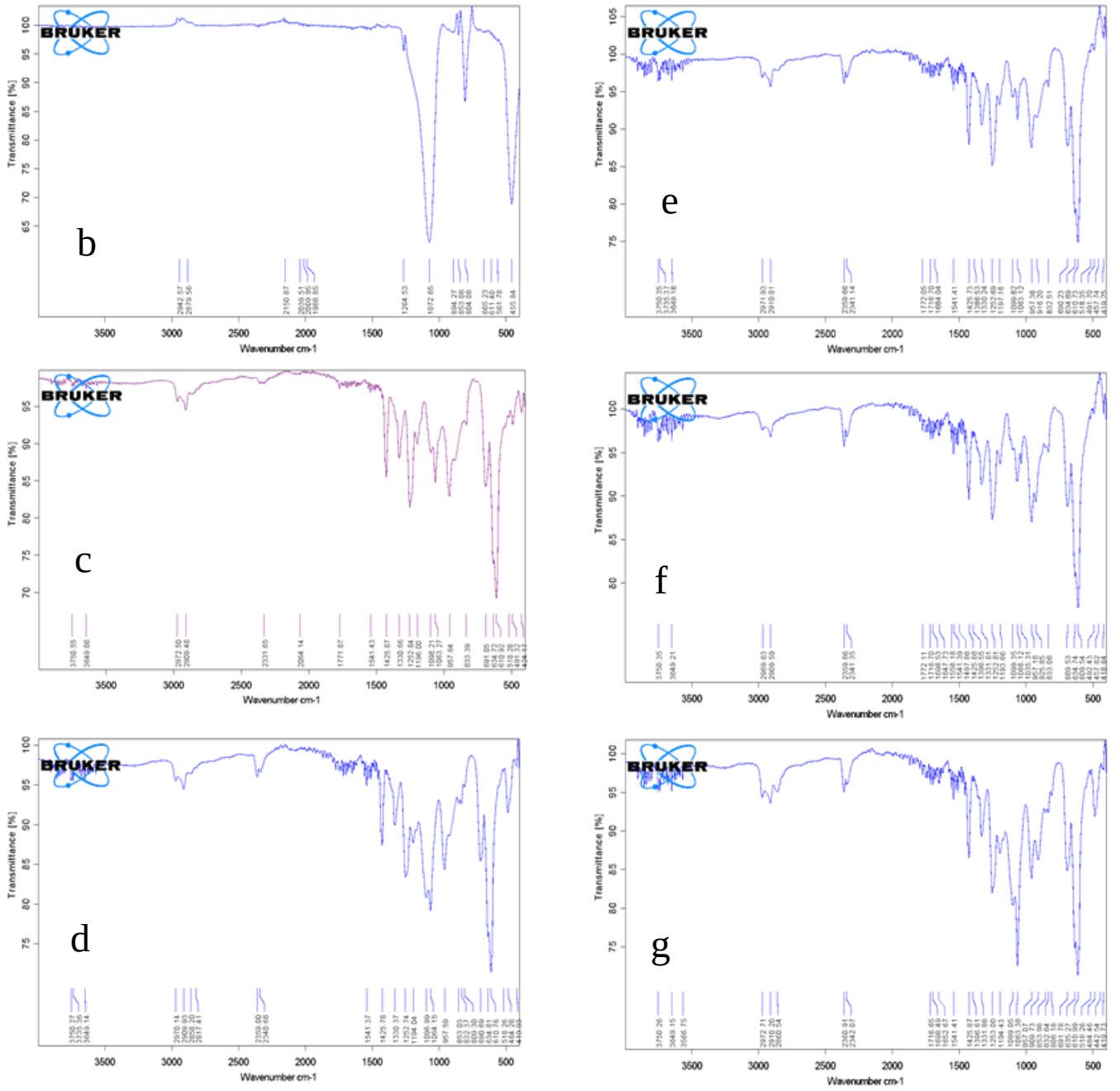


Figure 3. FTIR spectrum for a) PVC pure, b) SiO_2 nanoparticles, c) PVC/(0.001) SiO_2 nanocomposite film, d) PVC/(0.002) SiO_2 nanocomposite film,e) PVC/(0.003) SiO_2 nanocomposite film ,f) PVC/(0.004) SiO_2 nanocomposite film, g) PVC/(0.005) SiO_2 nanocomposite film.

Table 1. FTIR-characteristics of PVC film and PVC/ SiO_2 nanocomposite films.

Bands	PVC	PVC/ SiO_2NPs (0.001g)	PVC/ SiO_2NPs (0.002g)	PVC/ SiO_2NP$_S$ (0.003g)	PVC/ SiO_2NPs (0.004g)	PVC/ SiO_2NPs (0.005g)
C-CL (600-800)) cm^{-1}	608.80 635.09 690.42 753.78	610.92 634.72 691.05 ……	610.76 634.81 690.69 ……	610.73 634.69 690.23 ……	609.54 634.74 689.53 ……	610.99 635.27 691.78 ……
C-H Out phase bend (625-970)	844.20 925.07 958.03	833.39 957.64 ……	809.30 832.37 853.03	832.51 916.20 957.38	833.08 925.85 957.18	808.18 832.64 853.98

cm^{-1}	.....	.....	957.59	.....	.....	909.73 957.07
C-O Stretch (1015-1300) cm^{-1}	1035.87 1062.45 1120.04 1191.53 1253.11	1063.27 1098.21 1196.00 1252.84	1064.15 1096.99 1194.04 1252.74	1063.12 1099.82 1197.16 1252.69	1035.31 1066.12 1099.35 1193.66 1252.81	1063.38 1099.05 1194.43 1253.00 .....
CH_2 bending (1300-1380) cm^{-1}	1335.17	1330.66	1330.37	1330.24	1331.61	1331.88
CH_2 wagging C=C stretch (1430-1600) cm^{-1}	1456.46 1541.34	1541.43	1541.37	1541.41	1497.86 1541.39 1558.18	1541.41 ....
C=O Stretch (1550-1780) cm^{-1}	1647.52 1698.72 1716.75	1771.87	....	1684.04 1716.70 1772.05	1647.73 1698.53 1716.70 1772.11	1652.67 1698.49 1716.65
C-H Stretch Aliphatic (2800-3000) cm^{-1}	2908.33 2962.65	2909.48 2972.50	22817.1 2858.20 2909.93 2970.14	2910.01 2971.93	2909.59 2969.63	2860.54 2910.20 2972.71 .....

Optical Properties

The absorbance spectra of pure PVC and PVC/ SiO_2 nanocomposite films are given in Figure 4. UV-visible absorption spectrum for PVC/SiO_2 nanocomposite films. The absorbance increases as the amount of SiO_2 nanoparticles in the PVC/ SiO_2 nano combination increase [27]. As well, pure PVC did not have a peak, but when SiO_2 was added a peak appeared as shown in Table 2.

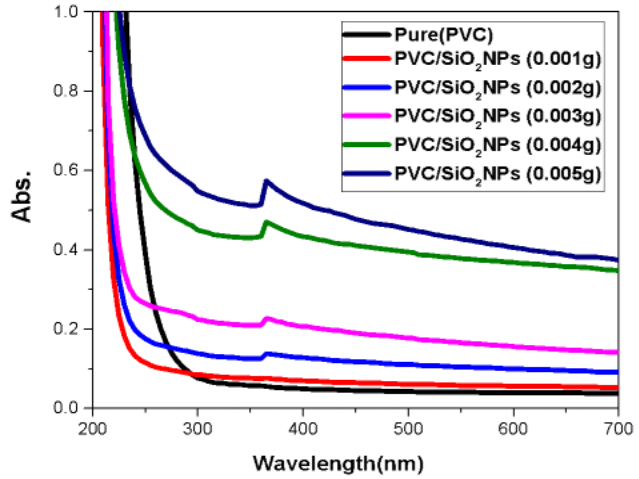


Figure 4. The absorption spectrum of pure PVC and PVC/ SiO_2 nanocomposite films.

Table 2. The highest peaks of the absorption spectrum of PVC film and PVC/ SiO_2 nanocomposite films

Samples	Wavelength (nm)	Absorbance
Pure PVC	----------	----------
PVC/SiO_2NPs(0.001g)	365	0.076
PVC/SiO_2NPs(0.002g)	365	0.139
PVC/SiO_2NPs(0.003g)	365	0.228
PVC/SiO_2NPs(0.004g)	365	0.471
PVC/SiO_2NPs(0.005g)	365	0.575

The PVC is transparent to visible light, as is well known. This is shown in Figure 5, which represents the transmitted spectra of PVC/ SiO_2 nanocomposite materials and pure PVC polymer. Pure PVC polymer has a higher transmittance than PVC/ SiO_2 nanocomposites. The transmittance of pure polymers and nanocomposites is lower in the UV field than in the visible range. They have a very low UV transmission, which makes the addition of SiO_2 nanoparticles clearly shows its effect. Transmittance reduced as the amount of SiO_2 increased, as predicted by [28].

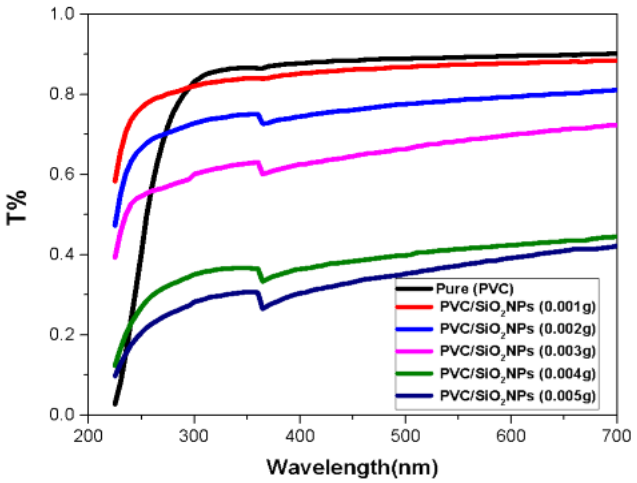


Figure 5. The transmittance spectra of pure PVC and PVC/ SiO_2 nanocomposite films.

Each absorber molecule or ion has an attribute called the absorption coefficient (α). It is described as the substance's capacity to absorb light with a specific range of wavelengths per unit length. Equation (2) could be used to compute the absorption coefficient. The relationship between the absorbance coefficient and wavelength for all samples with different concentrations of SiO_2 NPs is shown in Figure 6. Direct electronic transitions are expected when high absorption coefficient values ($\alpha > 10^4$ cm^{-1}) are at higher energies, and the electron and photon energy-momentum is conserved. The indirect electronic transitions were inferred from the low absorption coefficients ($\alpha > 10^4$ cm^{-1}) at low energies. This work calculates absorption coefficient values for low energies and indirect electronic transitions allowed.

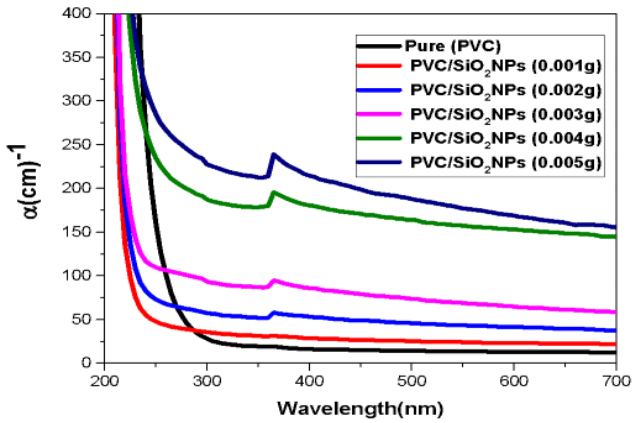


Figure 6. The absorption coefficient of pure PVC and PVC/ SiO_2 nanocomposite films.

To describe magnetic wave propagation in a medium, it is necessary to study the refractive index. Figure 7 shows how the refractive index of PVC/SiO_2 nanocomposite films varies with wavelength [28]. The refractive index of the PVC polymer increases along with the amount of SiO_2 NPs added, increasing the density of the nanocomposites. This case is characterized by a rise in the refractive index caused by an increase in the incident photon' dispersion.

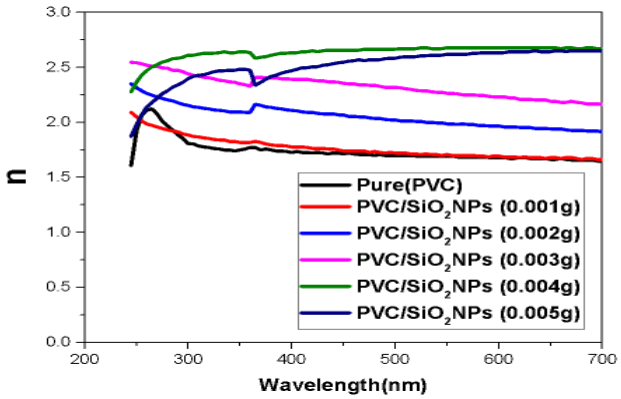


Figure 7. The refractive index of pure PVC and PVC/ SiO_2 nanocomposite films.

Figure 8 shows a relationship between photon energy and absorption for PVC/ SiO_2 nanocomposite films. The energy gap values for all PVC/SiO_2 nanocomposites films samples are described in Table 3. It is clear that when the amount of SiO_2 increases, the energy gap values decrease. A reduction in energy gap values is caused by an increase in localized levels of forbidden energy band gaps. Initially, PVC had an energy band gap of 4.36 eV, but as the amount of SiO_2 NPs increased, it decreased to 3.39 eV. [29][30].

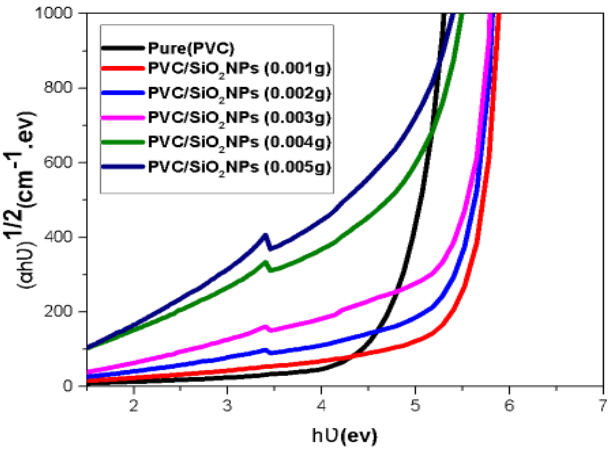


Figure 8. The optical energy gap of pure PVC and PVC/ SiO_2 nanocomposite films.

Table 3. The energy gap (E_g) of pure PVC and PVC/ SiO_2 nanocomposite films.

Samples	Energy gap(eV)
Pure PVC	4.36
PVC/SiO_2NPs(0.001g)	4.5
PVC/SiO_2NPs(0.002g)	4.34
PVC/SiO_2NPs(0.003g)	4.25
PVC/SiO_2NPs(0.004g)	4.06
PVC/SiO_2NPs(0.005g)	3.39

The variation of the PVC polymer's extinction coefficient can be seen in Figure 9 when SiO_2 nanoparticles are added in different amounts. The extinction coefficient increases as the ratio of SiO_2 NPs increases.

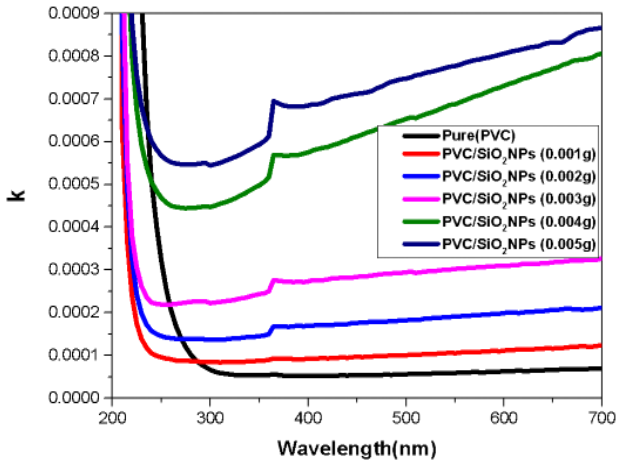


Figure 9. The extinction Coefficient of pure PVC and PVC/ SiO_2 nanocomposite films.

According to Equation (5), Figure 10 shows the variation in PVC polymer reflectance with different amounts of SiO_2 NPs. A nanocomposite's reflection spectrum changes with wavelength. The reflection spectrum is increased due to the addition of SiO_2NPs.

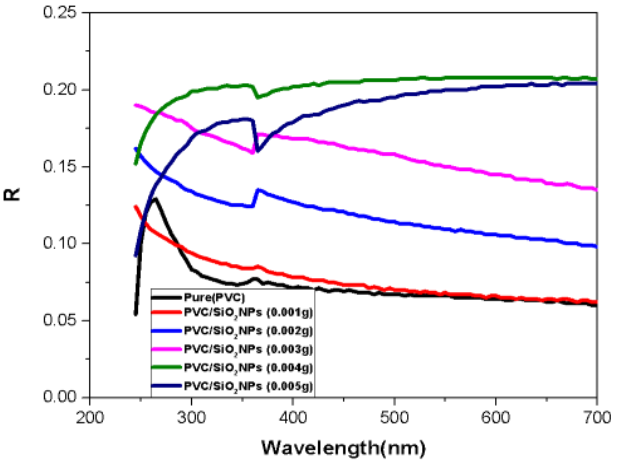


Figure 10. The reflection spectrum of pure PVC and PVC/ SiO_2 nanocomposite films.

For PVC/ SiO_2 nanocomposites, the imaginary and real dielectric constants (ε_r&ε_i) were calculated using the equation (8 & 9). Figures 11 & 12 show the real and imaginary components of the PVC/SiO_2 nanocomposite dielectric constants. The variation of (ε_r) is assumed to be most impacted by (n^2) because of small values of (k^2), whereas the variation of (ε_i) is used for (k) values because of the difference in absorption coefficients. The real dielectric constant has high values when compared to the imaginary dielectric constant.

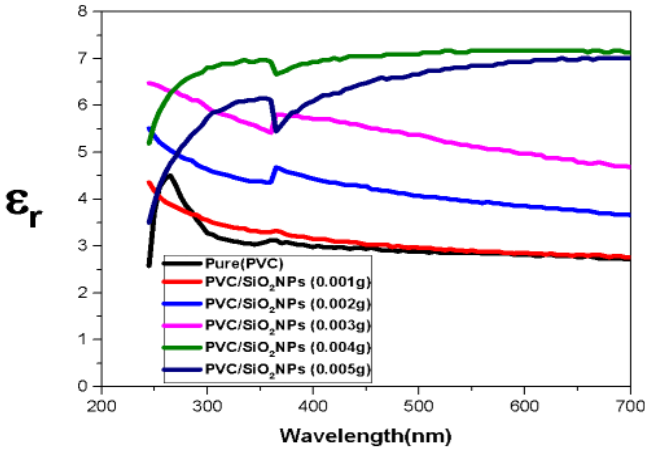


Figure 11. The real part of the dielectric constant for pure PVC and PVC/ SiO_2 nanocomposite films

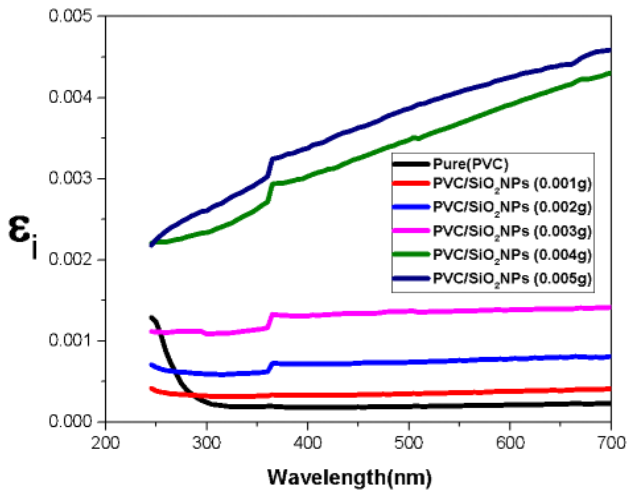


Figure 12. The imaginary part of the dielectric constant for pure PVC and PVC/ SiO_2 nanocomposite films.

CONCLUSIONS

The casting method has been successfully used to prepare nanocomposite films made from PVC and SiO_2. By XRD study, the evaluation of SiO_2NPs indicates that the structure is amorphous. FTIR demonstrated that NPs have no detrimental effect on polymer structure from FESEM images of PVC/SiO_2 nanocomposite membranes, the nanocomposite membrane shows spherical clusters of particles distributed over its surface. Optical properties can be enhanced by doping PVC with SiO_2 nanoparticles. Absorption increases and permeability decreases with increasing SiO_2. Increasing the filler amount decreases the optical energy gap from 4.36 to 3.39 eV.

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Disclosure and Conflict of Interest: The authors declare that they have no conflicts of interest.

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