

Research Article

Effects of SiO₂ Nanoparticles on Polyvinyl Alcohol/Carboxymethyl Cellulose Polymer Blend Films' Structural, Wettability, Surface Roughness, and Optical Characteristics

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The blend matrix composed of polyvinyl alcohol and carboxymethylcellulose (PVA/CMC) was prepared via the casting method. SiO₂ nanoparticles were added as reinforcement in different amounts (SiO₂ = 1, 2, 3, and 4 wt.%). The study utilized FTIR to examine the alterations in composition and the interplay between the blend matrix and the inclusion of SiO₂. Also, for the first time, the surface roughness and surface wettability of the PVA/CMC blend matrix were investigated with the addition of SiO₂ using measurements of contact angle and surface roughness parameters. The surface roughness and wettability of the blend matrix increased as the SiO₂ content increased. In addition, the blend matrix optical features were determined by the UV-visible spectrophotometer. Based on the analysis using Tauc's relation, it was found that the energy bandgap decreases from 5.52 to 5.17 eV (direct transition) and from 4.79 to 4.32 eV (indirect transition) for the PVA/CMC and PVA/CMC/4%SiO₂ blend films, respectively. The refractive index increases from 2.009 to about 2.144 for the PVA/CMC and PVA/CMC/4%SiO₂ blend films, respectively. Furthermore, optical conductivity and dielectric constants were improved for the PVA/CMC blend film after the addition of SiO₂ nanoparticles.

1. Introduction

Blended polymers are a hot topic in the scientific community due to their remarkable physicochemical properties [1, 2]. The optical properties of blended polymers make them a compelling and promising area of research. Researchers have been eagerly exploring their possible uses in various fields, like electronics, energy, and medicine [3]. These mixed films have consistently proven to be superior in terms of performance, durability, and versatility. Mixed films are the clear choice for developing cutting-edge electronics or simply seeking to improve existing technology [1, 4–7]. Blended polymers have the unique ability to modify their structure and composition to customize their optical behavior, which makes them very promising for creating innovative technologies.

Polyvinyl alcohol and sodium carboxymethylcellulose (PVA and CMC) are compatible polymers that form highly transparent blend films. PVA and CMC share characteristics like water solubility, nontoxic, abundant, cross-linked, and biodegradable polymers [8]. CMC is a pH-sensitive ionic polyelectrolyte with carboxyl groups [9]. The combination of CMC and PVA presents an opportunity to achieve the benefits of both materials. In addition, the presence of the hydroxyl (OH) groups in the backbone of PVA and CMC may lead to the formation of inter- and intra-hydrogen bonds [5, 10].

Metal oxide-polymer composites have recently gained significant research interest for their applications in catalysis, electronics, photonics, and optoelectronics [11]. Additives of nanosized inorganic particles in the polymeric matrix create nanocomposites with unique properties distinct from conventional materials [11–17]. Recently, ferrite nanoparticles

were used as additives in a PVA/CMC blend matrix to tune the optical parameters to be used in various optical applications [18, 19]. SnS [20–22], $\text{Co}_{0.9}\text{Cu}_{0.1}\text{S}$ [23], and Ag/CuS [24] nanoparticles were added to the PVA/polyvinyl propylene (PVP) blend film to adjust its optical parameters and make it suitable for optoelectronic applications. PVA/PVP blend films reinforced with SiO_2 have previously been discussed in terms of their structural and optical properties [25, 26]. The use of SiO_2 as a nanofiller for PVA/CMC blend films was rarely discussed. The influence of $\text{SnO}_2/\text{SiO}_2$ and $\text{Cr}_2\text{O}_3/\text{SiO}_2$ nanocomposites on the optical parameters and structural features of the PVA/CMC matrix have been discussed [27, 28]. The integration of SiO_2 nanoparticles has played a pivotal role in revolutionizing the enhancement of polymer surfaces' morphological structure and optical characteristics. Previously, the PVA matrix doped with various content of SiO_2 nanoparticles showed an increase in the hydrophilicity, as the water contact angle with the film surface was found to decrease with increasing the SiO_2 content in the PVA matrix [29]. While, SiO_2 nanoparticles were observed to enhance the hydrophobicity and raise the roughness of the PVDF-HFP films [30]. Also, the optical properties and wettability of the polystyrene films were affected by the inclusion of SiO_2 in the matrix [31]. SiO_2 was found to play a vital role in changing the wettability of polymer films [29, 31].

Herein, novel PVA/CMC films reinforced with different amounts of SiO_2 are fabricated. In addition, the wettability and surface roughness properties of the blend matrix are investigated for the first time, as is the influence of various concentrations of silica on it. To characterize the functional groups of the prepared films, Fourier transform infrared spectroscopy was used. Moreover, the surface roughness and the water contact angle were investigated. Utilizing the absorption and transmission data from UV–visible spectroscopy, one can accurately estimate the optical parameters of PVA/CMC/ SiO_2 films. This approach provides a reliable and efficient way to analyze these films, providing valuable insights to both academics and business experts.

2. Experimental and Characterization

PVA/CMC films were prepared by casting the solution. First, PVA (molecular weight (M_n) = 1×10^6 , QualiChem's company, India) and CMC (0.7% of substitution and $M_n = 1 \times 10^5$) powders, with a ratio of 80/20, were dissolved separately in distilled water. Subsequently, the polymer solutions were blended by adding the PVA solution to the CMC solution while stirring constantly at room temperature for 2 hr, until a homogenous mixture was achieved. Then, the prepared PVA/CMC blend solution was divided into five flasks; one of them was pristine without additive, and the others were reinforced with $x\text{SiO}_2$ ($x = 0, 1, 2, 3,$ and 4 wt.%) using probe ultrasound procedure. After the distribution of SiO_2 nanoparticles in the blend matrix, the mix was poured into Petri plates and left to dry at room temperature (25°C) for several days. The prepared samples with 0, 1, 2, 3, and 4 wt.% SiO_2 in PVA/CMC blend were labeled as PCS0, PCS1, PCS2, PCS3, and PCS4, respectively. Finally, the prepared films with approximately a thickness of about $100 \mu\text{m}$

were utilized for measurements. The position and intensity of the chemical bonds of the films before and after the inclusion of various SiO_2 concentrations were recorded via Fourier transform infrared (FTIR) spectroscopy. A roughness tester (SRT-6600) was utilized to measure the surface roughness of the polymer films. This tester relies on a moving sensor on the film surface to identify a roughness profile curve and instantly record the roughness parameters. The wettability of the PVA/CMC blend films can be accurately determined by measuring the contact angles using the reliable Ossila Digital Goniometer (Model L2004A1, Sheffield, UK), with an accuracy $\pm 1^\circ$. An ultraviolet–visible spectrometer was used to measure the blend films' absorbance both before and after SiO_2 was added.

3. Results and Discussion

3.1. FTIR, Contact Angle, and Surface Roughness Analysis. Figure 1 shows PCS films' FTIR spectra. The bond positions and assignments for the pristine PVA/CMC are presented in Table 1.

The broadband in the region $3,200\text{--}3,500 \text{ cm}^{-1}$ is assigned to —OH group. The fingerprint bands in the region from $1,500$ to 400 cm^{-1} show the characteristics bond of PVA and CMC. The band observed at $1,070 \text{ cm}^{-1}$ is related to C—O bond of CMC, and the same band also was assigned for PVA. The SiO_2 impact on the vibrational modes in terms of a reduction or increase in band intensity with increasing SiO_2 content was observed due to crosslinking with the blend functional groups. For the pristine sample (PCS0), the bond at $2,166 \text{ cm}^{-1}$ was shifted to higher wavenumber at $2,167, 2,168, 2,169,$ and $2,169 \text{ cm}^{-1}$ for PCS1, PCS2, PCS3, and PCS4, respectively. The bands at 957 and 856 cm^{-1} are assigned to the stretching of CH_2 and C—C bonds, respectively. These bands are slightly shifted with increasing SiO_2 concentration in the PVA/CMC film, as shown in Figure 1(b). The band at 920 cm^{-1} is linked to the C—C stretching band of PVA was vanished with the inclusion of higher content of SiO_2 nanoparticles, which might be due to the destruction of this bond and formation of the new bond Si—O—C [37, 40]. The band at $1,095 \text{ cm}^{-1}$, which is related to both PVA and CMC [41], ascribed to the C—O bond of the PCS0 sample, was shifted to $1,079 \text{ cm}^{-1}$ after the inclusion of 4 wt.% SiO_2 . This is ascribed to the combination of the Si—O band of the silica at $1,060 \text{ cm}^{-1}$ with the C—O bond of the polymer blend matrix [40]. Besides the weak band observed at $1,234 \text{ cm}^{-1}$ which attributed to the Si—O—Si asymmetric vibrations [36, 42]. In addition, the Si—O—Si stretching mode at 800 cm^{-1} at the higher content of SiO_2 nanoparticles testified to the presence of SiO_2 in the blend matrix, as mentioned elsewhere [36, 40]. The FTIR analysis results are conclusive; they show that the PVA/CMC blend and SiO_2 have a clear and specific interaction. These findings demonstrate the potential for this blend to offer unique benefits and applications in the field.

The interaction between the liquid and solid surfaces is highly dependent on surface-free energy, and it is the main parameter that influences the wetting property. The contact

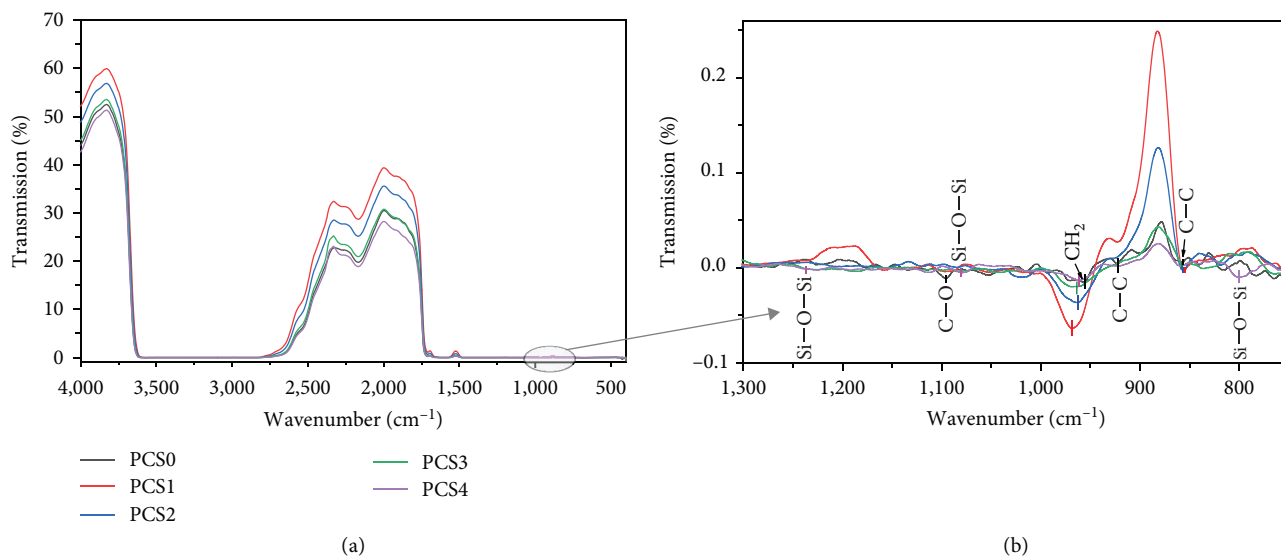
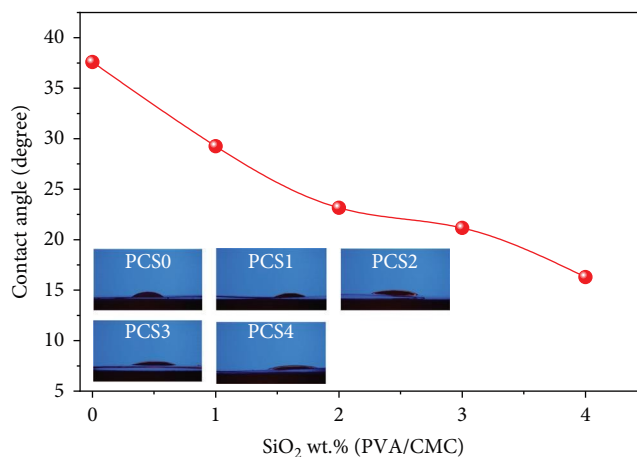
FIGURE 1: (a, b) FTIR spectra for PVA/CMC/SiO₂ films.

TABLE 1: FTIR bands assignment of PVA/CMC blend film.

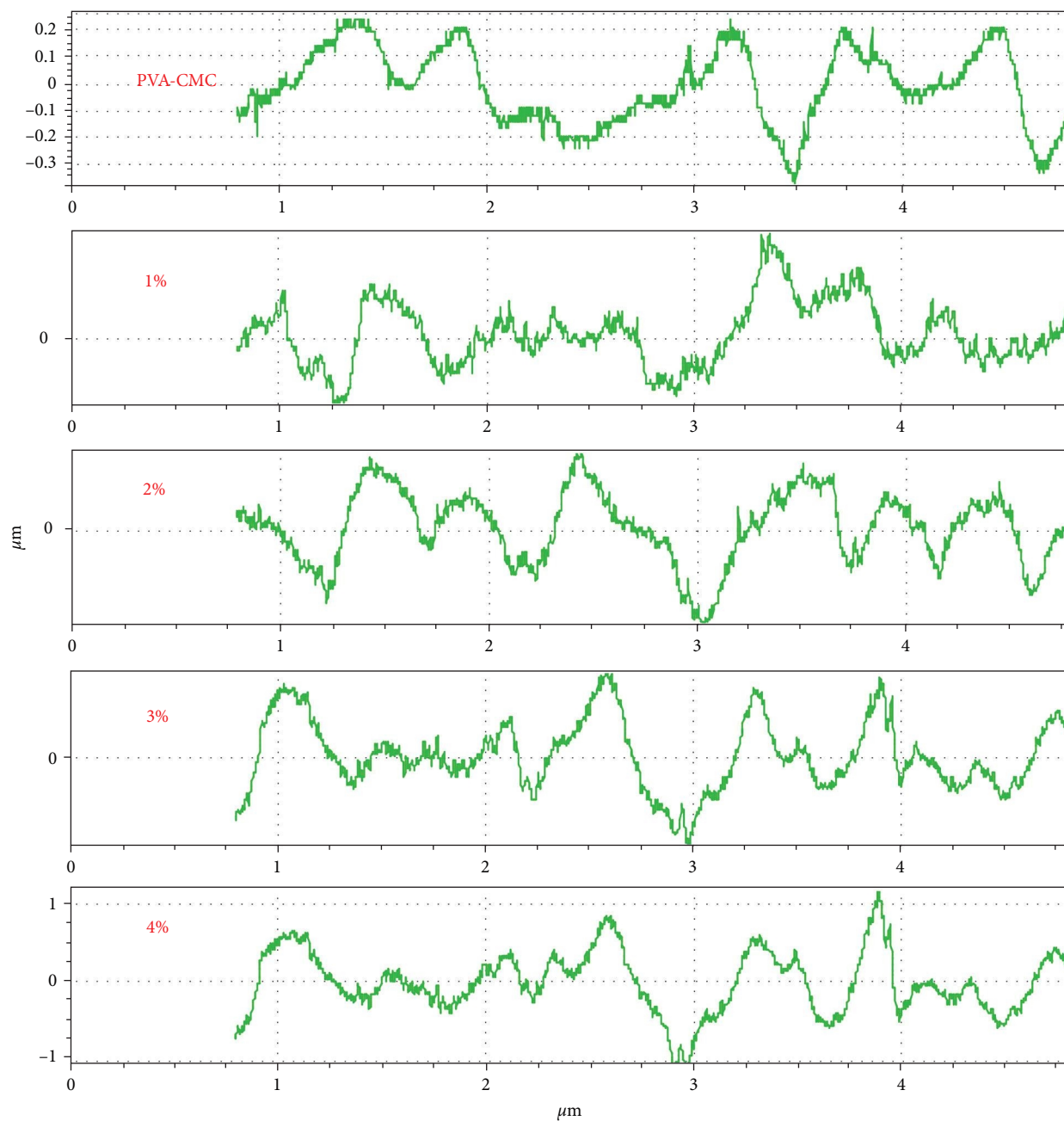
Wavenumber (cm ⁻¹)	Assignment
3,287	—OH stretching [32]
2,924	—CH ₂ asymmetric stretching [32]
1,720	C=O stretching [32]
1,662	C=C stretching [32]
1,560	C—H and C—O stretching [32, 33]
1,484	CH ₂ bending [34]
1,250	—CH ₂ bending [35]
1,234	Si—O—Si stretching [36]
1,095	C—O stretching of CMC [35]
920	C—C stretching [37]
957	Rocking vibration of CH ₂ [38]
856	C—C stretching [35, 39]
800	Si—O—Si stretching [36]

FIGURE 2: The correlation between the concentration of SiO₂ and the contact angle in the PVA/CMC films.

angle measurement, which indicates the wetting property's degree, can be used to explain the wetting characteristic. Surface wettability of hydrophobic material decreases with increasing contact angle, while surface wettability of hydrophilic material increases with decreasing contact angle [43]. The contact angle value of drop-distilled water on the film surface was measured for PVA/CMC blend film and PVA/CMC films doped with SiO₂. Figure 2 shows the average contact angle (θ), measured on both sides (left and right), of water droplets on the surface of the PCS blend films. The angle of contact decreases from 37.6° for PCS0 to about 29.3°, 23.2°, 21.2°, and 16.3° for PCS1, PCS2, PCS3, and PCS4, respectively, as the SiO₂ concentration increases in the blend film. This affirms the more hydrophilicity of the blend film after the inclusion of SiO₂, owing to SiO₂'s high hydrophilicity [26]. The reduction in contact angle is an indication of the increase in wettability with increasing SiO₂ concentrations. Consequently, micropores could be formed because of enhancement of the

wettability and it may cause a change in the surface roughness. The surface roughness for PVA/CMC blend films with the effect of various concentrations of SiO₂ was investigated, as shown in Figure 3. From the roughness curves, the roughness parameters were extracted, and Table 2 presents the results. It is crucial to note that R_a signifies the mean height, R_p is the maximum peak height, R_q is the root mean square profile height, R_v is the maximum valley depth, and R_z is the average maximum height [44, 45].

It is obvious that the values of R_a , R_z , R_p , R_q , and R_v all increase with increasing SiO₂ concentration in the blend film. It confirms the rise in film roughness with increasing SiO₂ concentrations. This impact of SiO₂ nanoparticles on the surface roughness is consistent with previous observations for PVDF-HFP [30, 46]. PVA and CMC are hydrophilic polymers, in contrast to PVDF, which is a hydrophobic polymer. The contact angle of the PVDF film was found to increase with increasing SiO₂ concentrations, while here the contact

FIGURE 3: Roughness curves for PVA/CMC/SiO₂ blend films.TABLE 2: Roughness parameters of PVA/CMC/SiO₂ films.

Sample ID	R_a	R_z	R_q	R_p	R_v	R_{sk}	R_{ku}	R_k
PCS0	0.123	0.521	0.146	0.241	0.28	-0.209	2.032	0.406
PCS1	0.186	0.739	0.218	0.381	0.357	-0.287	2.079	0.553
PCS2	0.232	1.028	0.276	0.526	0.502	0.115	2.017	0.665
PCS3	0.296	1.274	0.354	0.705	0.57	0.41	2.212	0.86
PCS4	0.322	1.424	0.382	0.758	0.666	0.209	2.139	1.023

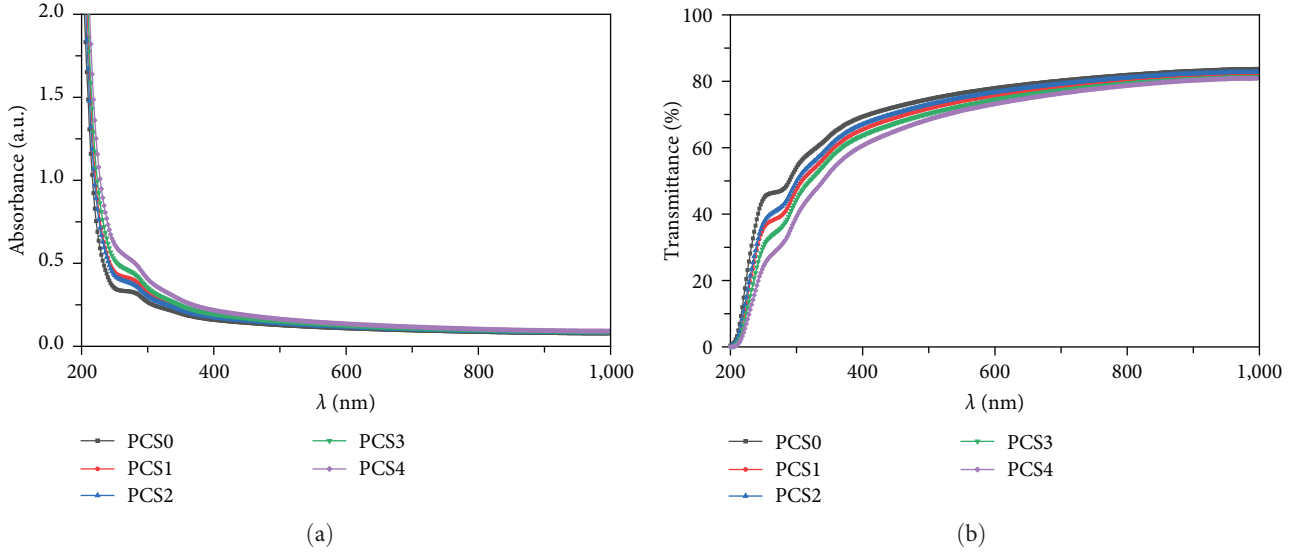


FIGURE 4: Plot of (a) absorbance and (b) transmittance vs. (λ) for PVA/CMC/SiO₂ films.

angle was found to decrease with increasing SiO₂ concentrations. The skewness parameter (R_{sk}) indicates the degree of surface unevenness of a polymer film. A negative value of R_{sk} indicates a flat surface, while a positive value indicates the presence of peaks and deep scratches. As shown in Table 2, the R_{sk} values for PCS0 and PCS1 samples are negative, revealing low roughness of the film surface [44]. The R_{sk} values for the others, PCS2, PCS3, and PCS4, are positive and show an increase with increasing SiO₂ concentrations, which reveals the increase in the roughness of the film's surface.

3.2. Optical Properties. The spectra (transmittance and absorption) of PVA/CMC films as a function of the incident light wavelength (λ) are displayed in Figure 4. Obviously, the absorbance of the blend films increases gradually with increasing the SiO₂ concentrations, as shown in Figure 4(a). In addition to the shift in the absorption edge toward the higher wavelength region, indicating to the decrease in the bandgap of the blend films. The hump observed at about 276 nm is related to the π - π^* transition [45].

With a rise in SiO₂ concentrations, the transmittance of blended films goes down, as shown in Figure 4(b). This highlights the importance of controlling the concentration of SiO₂ to ensure maximum transmittance and, ultimately, optimal performance of the blended films. In the visible-region around 500 nm, the transparency of the blend films decreases from 75% for PCS0 to about 73%, 72%, 70%, and 68% for PCS1, PCS2, PCS3, and PCS4, respectively. The addition of SiO₂ to the blend matrix causes an increase in the number of absorbed atoms and, of course, a decrease in the transmittance of light. The observed influence of SiO₂ on optical transmittance aligns with previous findings documented in the literature [31]. The film absorption coefficient (α) is determined using the absorbance (A) and the film thickness (t) according to the following relation [47]:

$$\alpha = \frac{2.303 \times A}{t}. \quad (1)$$

According to this, the optical parameters, which include E_{gd} (direct-bandgap), E_{gi} (indirect-bandgap), E_U (tail-band-width, or Urbach energy), n (refractive-index), k (extinction coefficient), ϵ' (optical dielectric constant), ϵ'' (optical dielectric loss), and σ_{opt} (optical conductivity), can be calculated using the following relations [33]:

$$(\alpha h\nu)^2 = B_1 (h\nu - E_{gd}), \quad (2)$$

$$(\alpha h\nu)^{1/2} = B_2 (h\nu - E_{gi}), \quad (3)$$

$$\ln \alpha = \ln \alpha_0 + \frac{h\nu}{E_U}, \quad (4)$$

$$n = \left(\frac{1+R}{1-R} \right) + \sqrt{\frac{4R}{(1-R)^2} - k^2}, \quad (5)$$

$$k = \frac{\alpha \lambda}{4\pi}, \quad (6)$$

$$\epsilon_r = n^2 - k^2, \quad (7)$$

$$\epsilon_i = 2nk, \quad (8)$$

$$\sigma_{opt} = \frac{\alpha n C}{4\pi}, \quad (9)$$

where B_1 and B_2 are constants, and C is the speed of light (3×10^8 m/s).

As illustrated in Figure 5, the E_{gd} and E_{gi} are derived from the relation between $(\alpha h\nu)^2$ and $(\alpha h\nu)^{1/2}$ versus $(h\nu)$,

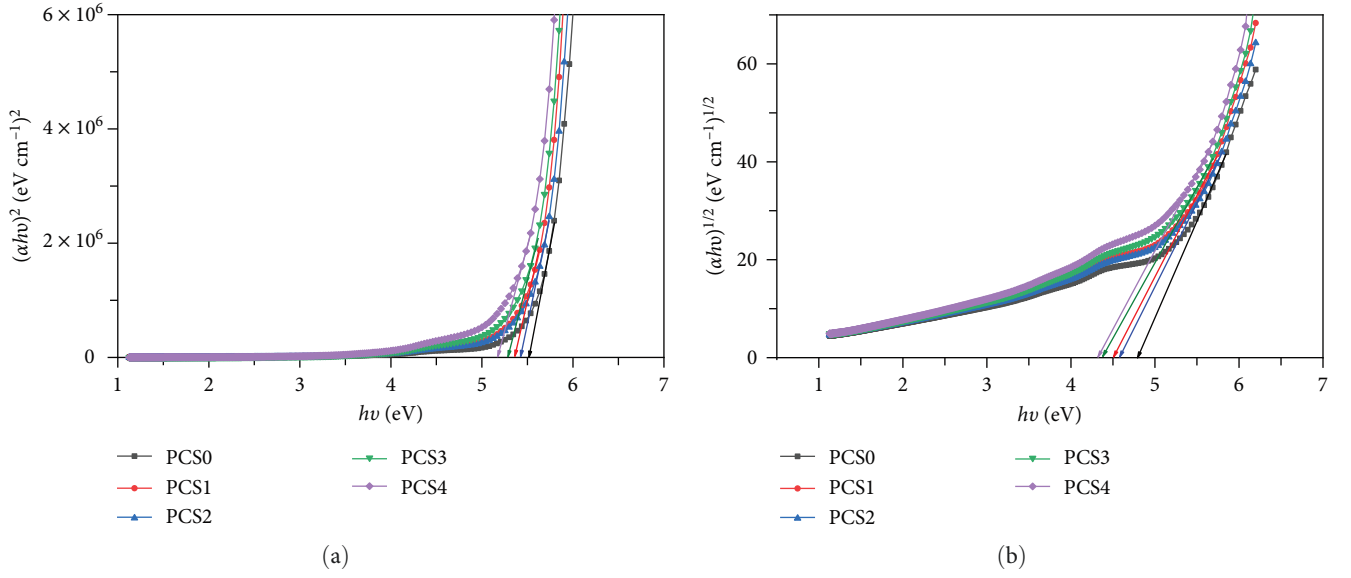


FIGURE 5: The dependences of $(\alpha h\nu)^2$ (a) and $(\alpha h\nu)^{1/2}$ (b) on $(h\nu)$ for PVA/CMC/SiO₂ films.

TABLE 3: Optical parameters for PVA/CMC/SiO₂ films.

Film ID	E_{gd} (eV)	E_{gi} (eV)	E_U (eV)	n ($\lambda = 600$ nm)
PCS0	5.52	4.79	0.84	2.009
PCS1	5.36	4.51	0.83	2.074
PCS2	5.42	4.57	0.86	2.047
PCS3	5.28	4.38	0.87	2.106
PCS4	5.17	4.32	0.91	2.144

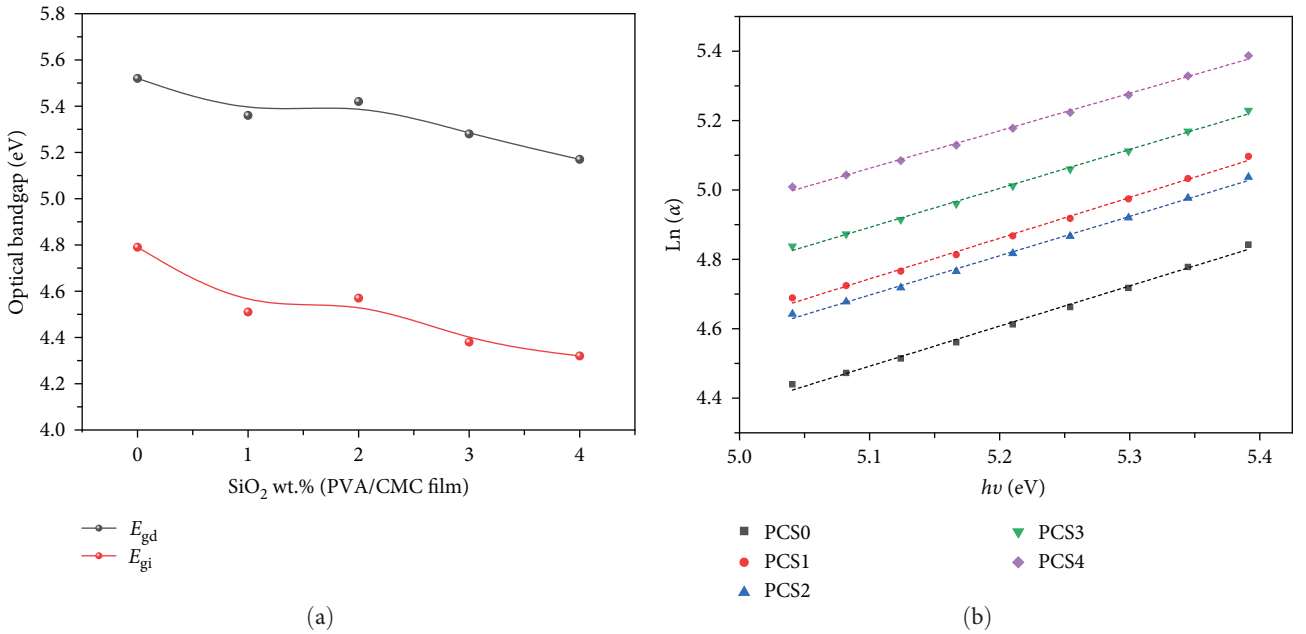


FIGURE 6: (a) Optical bandgap dependence on SiO₂ content in PVA/CMC film, and (b) $\ln(\alpha)$ vs. $h\nu$ for PVA/CMC/SiO₂ films.

respectively. The E_{gd} for the pure PVA/CMC blend film is about 5.52 eV, which is consistent with previous work [18, 19]. From Table 3, the E_{gd} and E_{gi} values slightly decreased with increasing the SiO₂ concentrations in the

PVA/CMC blend film. Figure 6(a) displays the variation of the optical bandgap (E_{gd} and E_{gi}) values with the SiO₂ content in the PVA/CMC blend film. It shows a decline in the bandgap value as the SiO₂ content increases in the blend

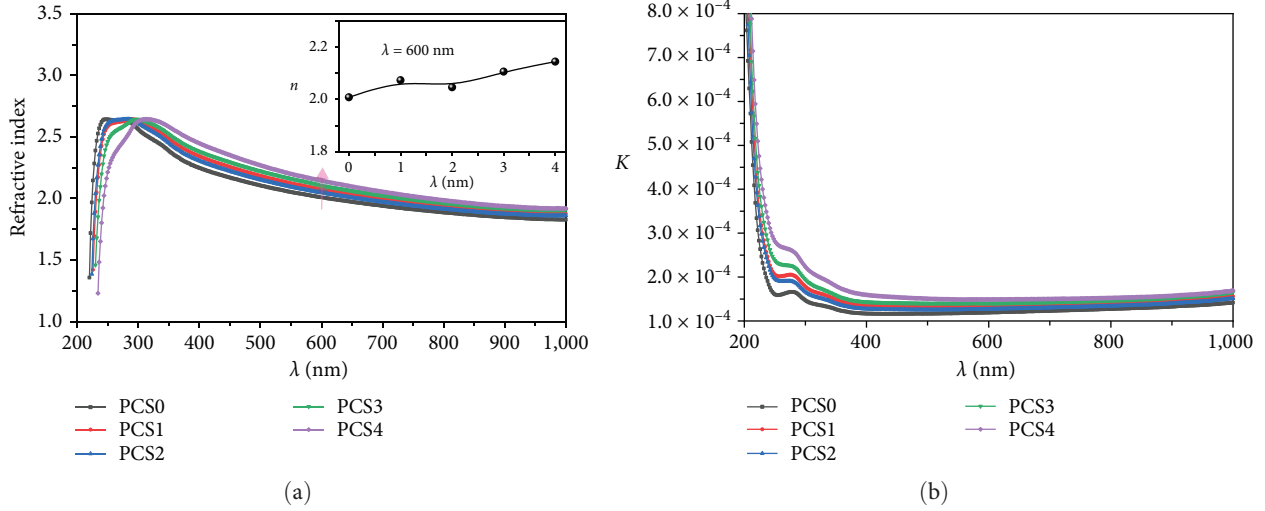


FIGURE 7: The wavelength-dependent n (a) and k (b) for PVA/CMC/SiO₂ films.

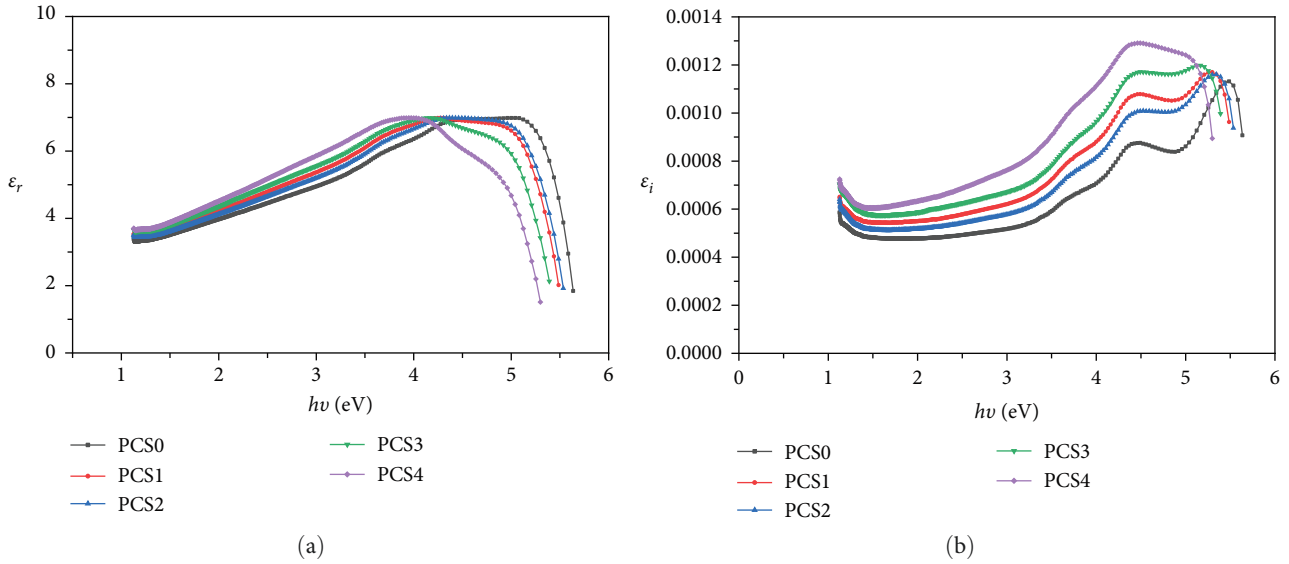


FIGURE 8: The dependences of ϵ_r (a) and ϵ_i (b) vs. $(h\nu)$ for PVA/CMC/SiO₂ films.

matrix. This behavior is related to the defects created as the SiO₂ is added to the blend matrix, and this result is in good accordance with the literature [20, 48]. The phenomenon could be associated with the interactions between the molecules of the blend matrix and the SiO₂ atoms. These interactions may form a hydrogen bond that creates localized states within the forbidden energy zone and reduces the distance between the valence and conduction bands. This disorder and defects created were affirmed by investigation of the tail width, Urbach energy (E_U), of the blend films using Equation (4) [49]. The linear portion from the relation between $\ln(\alpha)$ and $(h\nu)$, Figure 6(b), is the reciprocal of E_U . It is clear from Table 3 that the E_U value increases from 0.84 eV for PCS0 to about 0.91 eV for PCS4, confirming the blend matrix's increasing disordering and bandgap decreasing.

According to Equations (5) and (6), n and k values are computed regarding λ , as shown in Figure 7. Evidently, n and k both rose as SiO₂ concentrations increased and fell as incident light wavelengths increased.

As the SiO₂ concentration increases in the blend matrix, the number of atoms colliding with incident light increases and the reflectivity of the films increases as well. As a result, the refractive index rises along with the slowing of light velocity inside the blend matrix. On the other hand, the k value represents the loss of energy caused by light scattered or absorbed by the material [50]. As shown in Figure 7(b), it showed a normal behavior in the UV region, as it decreases sharply as it is related to the bandgap region, which has high energy sufficient to be absorbed and the loss caused by scattered or absorbed light will be less.

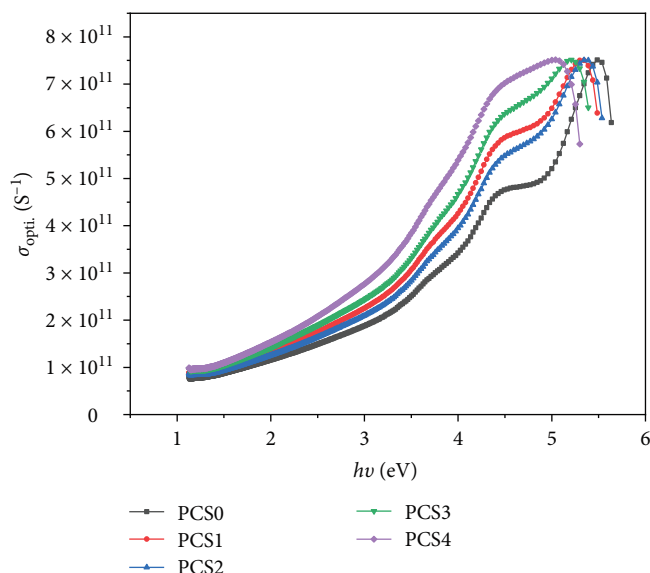


FIGURE 9: The $\sigma_{\text{opt.}}$ vs. (hv) for PVA/CMC/SiO₂ films.

The ϵ_r and ϵ_i as a function of the $h\nu$ are illustrated in Figure 8. Both optical parameters are increased with increasing the SiO₂ concentrations in the PVA/CMC blend films. An increase in ϵ_r value is directly proportional to the higher density of states within the bandgap, resulting from the increased concentration of SiO₂ [51, 52]. In addition, the ϵ_i value increased with increasing SiO₂ concentration, and the absorption band at the higher photon energy region shifted toward the lower photon energy. This is explained by the fact that as SiO₂ concentration rises, the number of free charge carriers in the matrix also rises [51].

The optical conductivity ($\sigma_{\text{opt.}}$) dependence on photon energy is displayed in Figure 9. It shows an increase in $\sigma_{\text{opt.}}$ with increasing SiO₂ concentrations, which could be due to the rise in charge carriers in the blend films.

The SiO₂ impact on the $\sigma_{\text{opt.}}$ of PVA/CMC films results from the transfer of charge between the blend's molecules and SiO₂ [53]. The interstitial spaces between the matrix chains are filled by SiO₂ and form a segregated network [54]. Hence, the optical conductivity increases as well.

4. Conclusion

Probe sonication and casting solution techniques were utilized for preparing the blend matrix of PVA/CMC with various amounts of SiO₂. The FTIR analysis revealed the interaction between the blend matrix molecules and SiO₂ nanoparticles. The contact angle and surface roughness parameter investigations confirm the modification of the PVA/CMC surface after the inclusion of SiO₂, where the surface roughness and surface wettability of the PVA/CMC matrix were increased. This is related to the hydrophilic of the SiO₂ nanoparticles. The optical analysis showed a slight decrease in the blend film transparency with increasing the SiO₂ amount, and a redshift was observed in the absorption edge, which confirms the reduction of the optical bandgap energy. The decrease in transparency and increase in film absorption raise their possibility of being

used in UV-shielding applications. The optical bandgap decreases from 5.52 eV for pure PVA/CMC to 5.17 eV for the PVA/CMC/4 wt.% SiO₂. This drop was related to the defects in the materials, and the refractive index rises due to increasing matrix density. The prepared current matrix, PVA/CMC/SiO₂, is considered a potential candidate for optical applications.

Data Availability

All the data are available in the manuscript.

Conflicts of Interest

The author declares that there are no conflicts of interest.

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