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Effect of $MgCl_2$ dopant on optical properties of tamarind gum based biopolymer electrolytes

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Abstract

Recent research on biopolymers based on magnesium salts is scarce in the existing literature. In the current research study, solution-casting technique was used for the preparation of solid biopolymer electrolytes, using Tamarind Seed Polysaccharide (TSP) as the host biopolymer, which was doped with several varying concentrations of magnesium chloride ($MgCl_2$). UV-visible spectroscopic method was used to investigate the thermal and optical parameters in the wavelength range of 200 nm to 800 nm. From the study of optical absorption parameter, the values of optical transmission, optical refractive index, optical absorption coefficient and optical extinction coefficient were calculated. These parameters revealed optimum values for the film of composition TSP:$MgCl_2$ (70:30 wt.%).

Key findings

- The film TSP:$MgCl_2$ (70:30 wt.%) exhibited the lowest absorbed photon energy value.
- The materials developed and evaluated possess the capability to absorb certain wavelengths of light, rendering them appropriate for application in optical sensors.
- The lower values of extinction coefficient of the TSP biopolymer films suggests that the phenomenon of light scattering is minimum.

Accompanying information

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1. Introduction

Over the past three decades, the study on the application of biopolymer electrolytes for energy storage devices gained potential, leaving the researchers to investigate further potential applications. Among the various kinds of storage devices, electrochemical energy storage devices were found to be promising due to their enhanced ability to store energy. Though energy production and storage are feasible with the conventional synthetic polymers, the generation and fabrication of clean-energy storage devices for energy storage is the key factor. Thus, researchers focus on eco-friendly biodegradable solid biopolymer materials, owing to their several advantages such as low cost, bio-degradable and eco-friendly nature. So far, the studies in this field have been directed at the fabrication of new class of polymer electrolyte materials, known as biopolymer solid polymer electrolytes, owing to their great application in components such as double layer supercapacitors, batteries and solar cells.

Typically, polymer electrolytes are complexes with trapped salt ions, dissolved in the polymeric matrix system [1]. Over the past few years, polymer electrolytes have been widely used in electrochemical, electronic and electro-chromic devices, Electrical Double Layer Capacitors (EDLC), fuel cells and dye-sensitized solar cells (DSSC).

Thus, the synthetic and biopolymer-based electrolytes were often studied for their optical and electrical properties, due to their utilization in batteries, fuel cells and capacitors. The interaction of light with these classes of materials was seen to be of great interest to the researchers, especially while transiting from the conventional solar cells to LEDs or light emitting devices, which exhibited unique optical properties. This promoted the researchers to use these biopolymers for the applications in optical devices. In order to change the optical properties of these biopolymers, it was suggested to tune them using dopant materials, commonly using ionic dopant salts.

One primary application of the polymer electrolytes in optics requires assessing their optical absorption spectra [2]. The optical absorption spectra generally reveal the information about the conjugation length and the presence of functional groups within the polymer matrix. The absorbance peaks of these materials indicate specific electronic transitions, helping to elucidate the influence of the polymer structure on its optoelectronic properties. For instance, shifts in the absorption peak positions can suggest changes in the degree of crystallinity or polymer morphology, which directly affect ionic conductivity and overall performance [3]. In addition, UV-Visible spectrophotometry plays a key role in arriving at the optical bandgap values of polymer electrolytes. Through the application of Tauc's equation, the energy needed for the transition of electron from the valence band to the conduction band is estimated [4]. This is essential not only for understanding the charge transport mechanisms that occurs within the polymer matrix, but also for estimating the compatibility of the polymer with different components in an electrochemical cell. Through the analysis of how dopants and plasticizers alter the optical parameters of absorbance, transmittance, etc., the studies were carried out to optimize the electrolyte formation for improving the ionic conductivity along with stability [5]. The two important factors which decide the identity of polymer complexes are the kind of dopants and their concentrations. For example, through the doping of metal particles, semiconductor, ionic salts and metal complexes, researchers found methods to correlate both optical and electrical properties [6]. In several works, optical properties such as absorbance, transmittance, absorption edge, reflectance and extinction coefficient (K) were measured.

Thus, the application of UV-Visible spectrophotometry for calculating the optical parameters of the polymer electrolytes is an essential study that gives an insight to improve the efficiency of the polymer electrolytes through the bandgap evaluation [7]. It was shown that two factors, i.e., the nature of dopants and their concentrations, effect the properties of the polymer systems [8]. For example, the addition of metals, metal ion complexes, semiconductor particles and dopant salts can regulate the electrical and optical properties [9, 10].

As per the existing literature, no work has been reported on the preparation and the study of optical properties of magnesium chloride doped TSP biopolymer electrolyte films. Through this study, the feasibility and the application of these prepared biopolymer electrolyte films for the study of the optical properties was reported. The effect of these properties on the various internal mechanisms of the polymer matrix was also obtained and reported.

2. Experimental methods and techniques

TSP biopolymer was procured from TCI Chemicals (India) in powder form, and the dopant salt $MgCl_2$ was procured from Loba Chemie (India). These were used in the purest form for the preparation of the biopolymer electrolyte films. $MgCl_2$ was incorporated into the TSP biopolymer electrolyte. Through the process of solution casting technique, free-standing, transparent films of compositions TSP:$MgCl_2$ (100:0, 90:10, 80:20, 70:30 and 60:40 wt.%) were obtained. The schematic representation of the technique is represented by Scheme 1.

The thickness of these films was estimated using a screw gauge, having values around 100 μm. For studying the absorbance and other optical parameters of the prepared biopolymer electrolyte films, UV-visible spectroscopy was done at room temperature by using JASCO V-670 UV Spectrophotometer (scan speed – 400 nm/min, data interval – 1 nm and data points – 601).

3. Results and Discussion

3.1. FTIR Analysis

FTIR analysis is a powerful technique used to identify and analyse the functional groups present in a given sample based on transmission and absorption of the infrared light beam. This method gives valuable information about the molecular composition and chemical structure of any given material [11]. FTIR analysis gives a spectrum, which represents the vibrational frequencies specific functional groups in the molecules. This technique is widely used to characterize and identify different compounds in various fields, including chemistry, materials science and biology. In the present work, FTIR analysis was deployed to examine the complexation between the TSP biopolymer electrolyte and magnesium chloride ($MgCl_2$), confirming the presence of functional groups and the formation of complexes.

The FTIR spectra for pure TSP along with different weight ratio of $MgCl_2$ films for the wavenumber range 4000–500 cm^{-1} are shown in Figure 1. For pure TSP, a reduction in the OH bond wave number at 3340 cm^{-1} was observed compared to the $MgCl_2$ incorporated TSP polymer electrolyte films. The pure TSP biopolymer has a characteristic peak centered at 3340 cm^{-1}, which is attributed to (–O–H) or the hydroxyl group stretching.

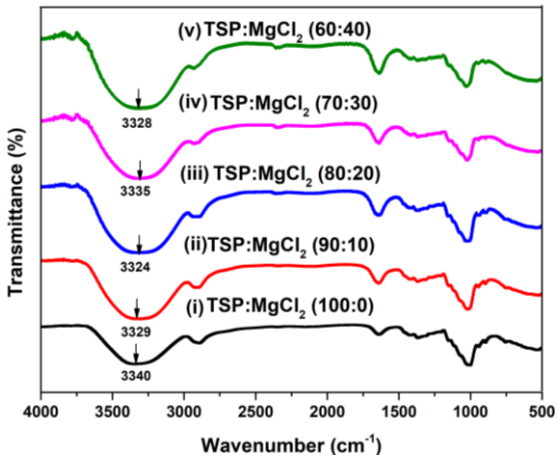

Figure 1 FTIR spectra of pure TSP with different wt.% of $MgCl_2$ (i) 100:0, (ii) 90:10, (iii) 80:20, (iv) 70:30, and (v) 60:40 films.

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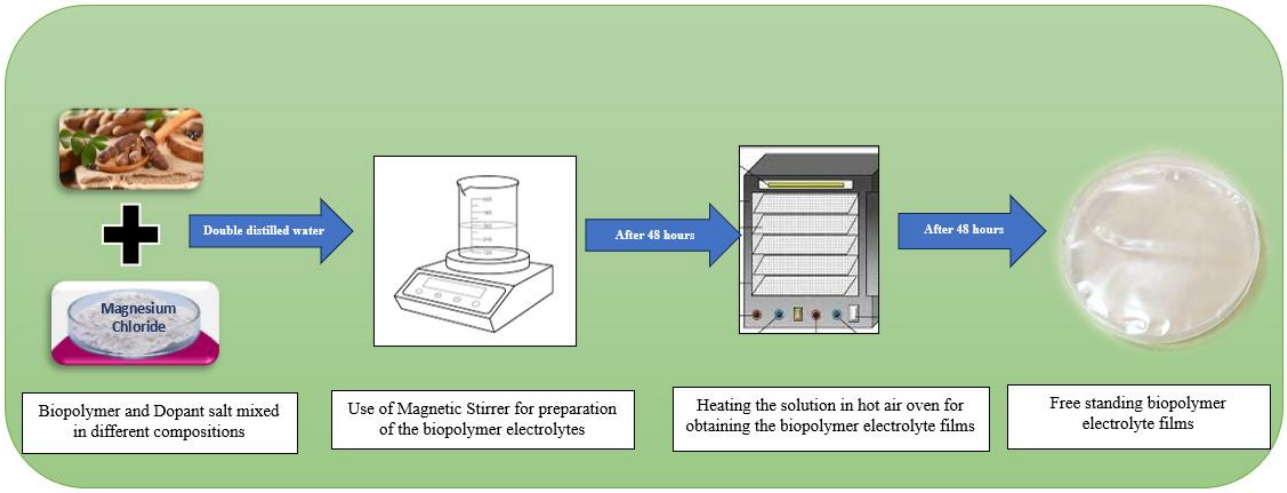


Scheme 1 Schematic representation of Solution-casting method.

The other peaks located around this wave number are at 3329 cm^{-1}, 3324 cm^{-1}, 3335 cm^{-1} and 3328 cm^{-1} for TSP: $MgCl_2$ 90:10, TSP: $MgCl_2$ 80:20, TSP: $MgCl_2$ 70:30 and TSP: $MgCl_2$ 60:40 wt.% ratios of the biopolymer electrolyte sample, respectively.

The shift observed here could definitely be due to the interaction between the TSP host biopolymer's hydroxyl group with the Mg^{2+} cations of the salt. Similarly, other peaks localized around the characteristic wavenumbers for the pure TSP biopolymer were also found to be displaced around each characteristic wavenumber. Thus, the results from FTIR analysis demonstrated that a complexation occurred between the host biopolymer and the salt. Along with these peak shifts, there are also variations visible in the intensities and peak widths, which confirm that a complexation is formed between TSP and $MgCl_2$.

The absence of crystalline peaks in the XRD pattern confirms the formation of a homogeneous mixture of TSP biopolymer and $MgCl_2$. FTIR analysis further proves the result of this complexation. The possible complexation of $MgCl_2$ with the TSP host biopolymer is depicted in Scheme 2 below.

3.2. UV visible spectroscopic measurements

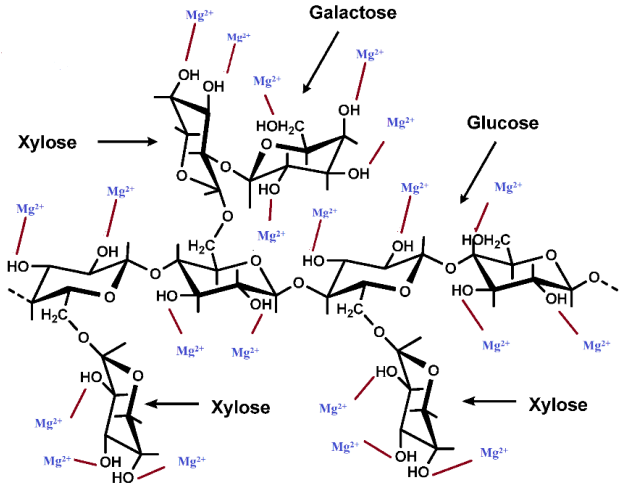
Ultraviolet Visible (UV-Visible) spectrophotometry is a technique which is used to investigate the optical parameters of polymer electrolytes. Through the analysis of parameters such as optical absorbance, optical transmittance, etc., in the UV-Visible region helps in comprehending the bandgap energies and electronic transitions in such materials.

3.2.1. Analysis of optical absorbance

Optical absorbance is a measure of the amount of photon energy or the light energy that the sample absorbs. This provides important details about the composition of a material. This is stated as the variation in the intensity of the light radiation after passing through an optical medium. Absorbance spectroscopy is used in the fields of chemistry and material sciences in order to assess and arrive at the optical properties of the prepared samples.

Shown in Figure 2 is the plot of wavelength (range 200 nm – 800 nm) vs. optical absorbance (at 303 K) for pure TSP and different concentrations of $MgCl_2$.

As observed from this plot, as the salt concentration rises, there is a gradual rise in the optical absorption [12].



Scheme 2 Representation of possible interaction of $MgCl_2$ with TSP biopolymer.

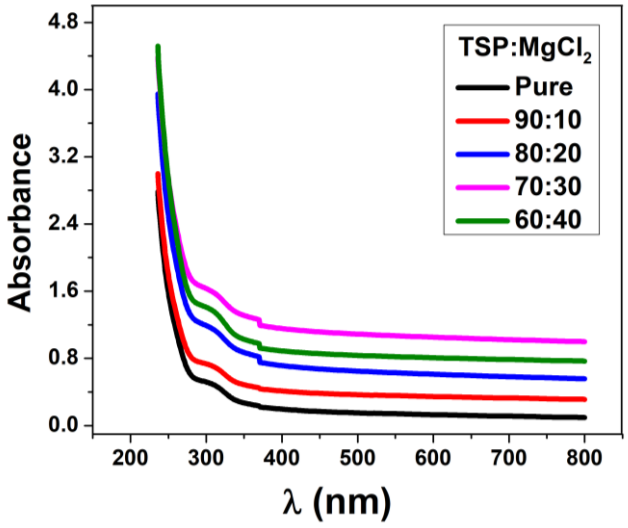


Figure 2 Optical absorbance for $MgCl_2$ doped TSP films.

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The highest absorption was observed for the film TSP:$MgCl_2$ (70:30 wt.%), and, as the concentration of the salt increased, absorbance reduced for the combination having 40 wt.% salt. Thus, it indicates that the film TSP:$MgCl_2$ (70:30 wt.%) has maximum absorbance, showing the application in light-blocking material [13]. In general, the absorbance spectra show a high value of absorption for the threshold wavelength range at the beginning of absorption [14].

Through the doping of salt to polymer electrolytes, the dissociation of salt into anions and cations occurs, which interact with the polymer segments through electrostatic interactions. All such possible interactions lead to the formation of transfer of charges between the salt ions and the polymer, which results in a change in the polymer's electronic structure [15]. This is explained through the molecular orbital theory.

According to this theory, all electronic transitions that are solely responsible for the phenomenon of optical absorbance of polymer electrolytes typically occur from the lowest unoccupied molecular orbital (LUMO) to the highest occupied molecular orbital (HOMO) of the system.

The presence of the dopant salt ions modifies these energy levels in the orbitals, which eventually leads to a peak shift in the spectrum of optical absorption of the complex [16]. In the current scenario, the property of optical absorbance of magnesium salt-doped TSP biopolymer electrolyte is stated due to the development of new electronic regions inside the band gap of the material. These new states possibly arise from the interaction of the magnesium ions and biopolymer chains that alter the electronic properties, thereby resulting in the change of the optical absorbance. Research studies by Zhang et al. have shown that optical parameters of salt-doped polymer electrolytes can be tuned through variations in the concentration of salt ions in the system [17]. In the study done by Abdullah et al., similar results were observed: the absorption was maximum in the UV region and minimum in the visible region [16].

Thus, the optical absorbance of salt-doped polymer electrolytes is to be understood in terms of interactions of the polymer chains and the ions of the salt, which eventually lead to a change of electronic structure of the material through the formation of new electronic states in the optical band-gap of the material. Referring to the insights from molecular orbital theory gives a theoretical structure to understand and give an estimate of all the optical properties of these materials [18]. Similar results were reported by Saha et al. [19] and Kiran et al. [2] in their studies on optical properties of sodium acetate doped TSP biopolymer electrolytes and sodium nitrate doped PVA polymer electrolytes, respectively.

3.2.2. Analysis of optical transmittance

Optical Transmittance (denoted by T) is explained as the ability of the incident light of a desirable wavelength to pass through the sample. Typically, the value lies between 0 (corresponding to no transmission state) and 1 (corresponding to complete transmission state). It is important to note that optical transmittance is inversely proportional to the optical absorbance. As the magnitude of absorbance increases, the magnitude of the optical transmittance reduces. In other words, when there is complete absorption of photon energy by the material, it generally transmits lesser energy. Optical transmittance is also defined as the ratio of transmitted power (P) by the sample to the incident power (P_0):

$$T = \frac{P}{P_0} = \frac{I}{I_0} (\text{since } P \propto I). \quad (1)$$

In addition, whenever light energy with intensity (I) falls normally on any substance, a ratio of it (I_0) entering the cubicle with thickness t is absorbed, and this is described through the relation given by Beer–Lambert law [20]:

$$I = I_0 e^{-\alpha t}, \quad (2)$$

where I denotes the intensity of the beam incident normally (at 90°) on the substance, I_0 denotes the light intensity entering the cubicle, α denotes the optical absorption coefficient and t denotes the thickness of the film.

From equations (2) and (3), the value of transmittance T is calculated by [7]

$$T = e^{-\alpha t}, \quad (3)$$

where α denotes the optical absorption coefficient and t is the sample thickness. Thus, using this equation, the optical transmission data was obtained from the parameters of optical absorbance and alpha.

The variation of transmittance with wavelength is denoted as transmission spectra.

The spectra of transmittance for pure TSP and various concentrations of $MgCl_2$ incorporated TSP for the range 200–800 nm are depicted in Figure 3. As shown in the figure, pure TSP has the maximum value of optical transmittance, which shows a reduction of intensity with a rise in the $MgCl_2$ salt concentration. As observed, optical transmittance for the film TSP: $MgCl_2$ (70:30 wt.%) concentration has the lowest value. This small value of the optical transmittance confirms and correlates to the results from the absorbance spectra. Since optical transmission inversely proportional to optical absorbance, this film shows maximum value of optical absorbance in the ultraviolet region, while the values of transmission are minimal. Similar results have been reported by Prasanna et al. [18] in their study on optical properties of $NaNO_3$ doped PVA polymer electrolytes.

3.2.3. Analysis of optical absorption coefficient

The optical absorption coefficient (which is denoted by α) quantifies the rate of exponential decay in the intensity of light as it propagates through an optical material. It is represented as the combined effect of absorption cross-sections of all the absorbing species per unit volume of the material [21].

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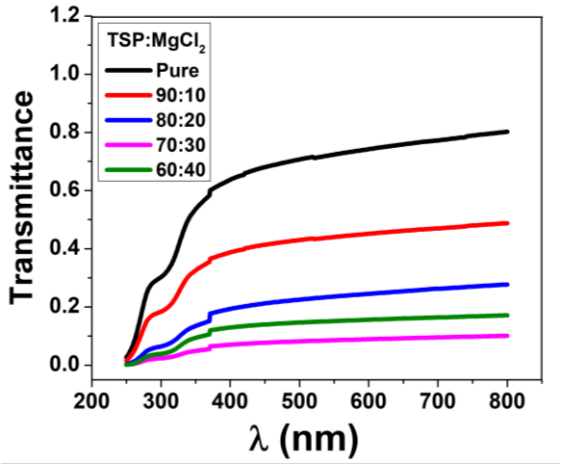

Figure 3 Optical absorbance for $MgCl_2$ doped TSP films.

This implies that the higher the value of α, smaller the length of light, that permeates through the material before it is absorbed. The rate of absorption of light for negligible scattering is given by Beer-Lambert law [20].

According to this law, whenever light radiation with an intensity "*I*" falls normally (at 90°) on the substance, ratio of the intensity of light I_0 that enters the cubical of thickness "*d*" is absorbed and is given by the following relation

$$I = I_0 e^{-\alpha t}. \quad (4)$$

Since polymer electrolytes have minimal absorption, the value of α is derived from absorbance. Thus, the absorption coefficient, also called as power fraction of absorption of a material, is analysed using the following formula [22]:

$$\alpha = \left(\frac{2.3032}{t}\right) \cdot A, \quad (5)$$

where *A* denotes the optical absorbance and *t* denotes its thickness.

The optical absorption edge (or absorption limit) is a sharp increase in the absorption spectrum of a material. This rise occurs at the wavelengths, wherein the energy of absorbed photon is comparable to the energy needed for an electronic transition within the material. It is important to note that these transitions are not the only factors which influence the absorption edge, but are the major contributors. The absorption edge data is calculated from the plot of *h*ν and α.

Figure 4 shows the plot of photon energy vs. absorption coefficient for pure TSP and various concentrations of $MgCl_2$ incorporated biopolymer electrolyte films. From the plots, the values of absorption edge are derived through the extrapolation the straight-line portion of the curve of the steeply rising region of the absorption curve onto the energy axis. The intercept of this line on the energy axis corresponds to the photon energy at absorption edge. The corresponding values of photon energies are presented in Table 1. From the table, it is seen that the film TSP:$MgCl_2$ (70:30 wt.%) has the lowest value for absorbed photon energy.

A dip in the photon energy from value 4.7 eV reported for pure TSP to the value 4.08 eV for TSP:$MgCl_2$ (70:30 wt.%) film was seen, which again increases slightly to the value 4.19 eV for the film TSP: $MgCl_2$ (70:30 wt.%).

The lowest absorbed photon energy reported here determines the ability of the material to interact with light. This enhances the suitability of the material for application in optical sensors through the increase of sensitivity and the efficiency of light detection, as reported in the work of Eldhose et al. [23]. The shift in the absorption edge to a slightly lower energy levels could be stated due to the establishment of new confinement regions in optical bands that push electrons from the valence band into the conduction band through the newly formed levels. Further, as the concentration of the salt rises, a decrease in the band gap is observed. Similar results had been reported in the work of Aziz et al. [24], where CND was the dopant salt for the polymer PMMA.

3.2.4. Analysis of optical refractive index

Optical reflectance (denoted by *R*) is defined as the ability and efficiency of an optical surface to reflect the energy radiant on it. Typically, this occurs due to the variation of refractive index, where the electromagnetic radiation penetrates the surface. From the optical transmittance optical and absorbance data, optical reflectance is calculated, which later gives refractive index and further yields several parameters.

Table 1 Optical absorption coefficients and refractive indices for different compositions of TSP:$MgCl_2$ films.

S. No	Composition (TSP: $MgCl_2$)	*h*ν vs. α (eV)	Refractive index μ
1	100:0	4.7	2.38
2	90:10	4.48	2.74
3	80:20	4.41	3.53
4	70:30	4.08	4.92
5	60:40	4.19	4.51

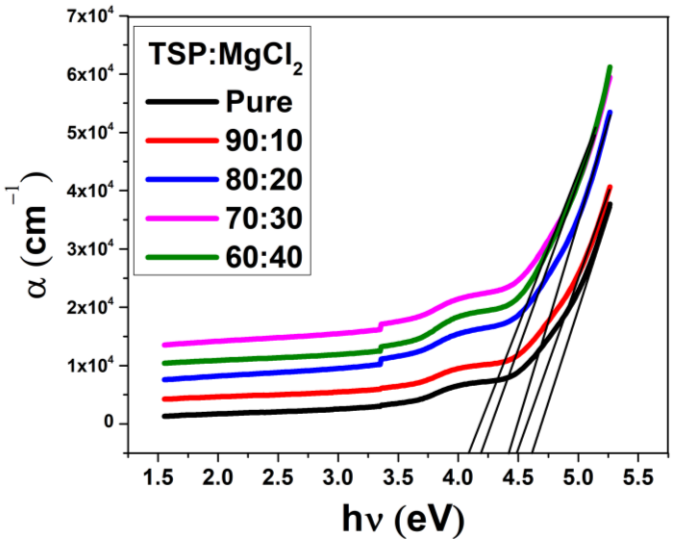

Figure 4 Optical absorption coefficient for $MgCl_2$ doped TSP films.

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The optical reflectance (denoted by R) of the biopolymer films is calculated through the equation [25]:

$$R = 1 - (T + A), \quad (6)$$

where R is denoted as optical reflectance, T is denoted as transmittance and A is denoted as optical absorbance.

For the desired material, the optical properties of the material are estimated knowing the parameters such as coefficient of extinction and refractive index [26]. Refractive index is considered one of the most fundamental properties of a material that gives information pertaining to optical dispersion for all wavelengths of light. The refractive index of the optical material gives information about molecular polarisation as well as the electronic polarisation of incident light beam. The optical refractive index (denoted by μ) is deduced from the value of R and the optical extinction coefficient (k) as follows [27]:

$$\mu = \left[\frac{1+R}{1-R}\right] + \sqrt{\left(\left[\frac{4R}{(1-R)^2}\right] - k^2\right)}, \quad (7)$$

where μ is the real part of the refractive index, R is the optical reflectance, and k is the imaginary part of the refractive index, known as the optical extinction coefficient.

The plot of the photon energy vs optical refractive index for pure TSP along with those with various fractions of $MgCl_2$ added to the biopolymer matrix is shown in Figure 5, and the values of optical refractive index for these compositions are tabulated in Table 1. From the table, it is seen that by raising the concentration of $MgCl_2$ in the TSP biopolymer, a rise in the refractive index from the value 2.49 (for pure TSP) to 4.92 (for the composition TSP:$MgCl_2$ (70:30 wt.%) film) compared to the other compositions was observed. Upon further increasing the salt content, there is a reduction in the optical refractive index.

From these plots, it was observed that an initial change in refractive index was negligible and constant for all the films up to 5 eV. For greater energies, the plot was found to rise as photon energy increases. This pattern of rise in optical refractive index with the salt concentration is similar to that of the value of absorbance for the films. The film TSP:$MgCl_2$ (70:30 wt.%) possessed the maximum value of the refractive index. The value of the highest refractive index (4.92) obtained for this film shows great potential applications in designing optoelectronic devices like LEDs, optical coatings, etc. [28]. The results reported are similar with the results from the work of Mohamad et al. [29], where PbS nanoparticles were doped into methylcellulose polymer.

3.2.5. Analysis of optical extinction coefficient

The parameter optical extinction coefficient (also referred to as extinction coefficient and denoted by k) relates to the dissipation of smaller proportions of the light radiation, which is affected by the optical absorbance. The optical extinction coefficient is generally denoted as the imaginary part of the optical refractive index. This is obtained from the formula [2]:

$$k = \frac{\alpha\lambda}{4\pi}, \quad (8)$$

where α denotes the optical absorption coefficient, and λ denotes the wavelength. The possible interaction between photon energy and the optical medium is depicted through changes occurring in coefficient of extinction, as it varies with the wavelength.

Figure 6 shows the plot of extinction coefficient vs. wavelength for different concentrations of $MgCl_2$-incorporated TSP biopolymer [30]. From the figure, it is observed that with an increase in the wavelength, the magnitude of the extinction coefficient also rises with the salt concentration. The value of k was reported to be highest for the composition TSP:$MgCl_2$ (70:30 wt.%) as compared to the other ratios. This value was in close agreement with the optical absorbance values. Since k is directly proportional to α, both these values were observed to be maximal for the film TSP:$MgCl_2$ (70:30 wt.%).

This high value of k proves the application of this optical prepared film as a material that can be used for blocking light [31]. Further, with an increase in concentration of the $MgCl_2$ salt, k was found to decrease, as reported in a similar work by Muhammad et al. [32], where sodium nitrate was the dopant salt and PVA was the chosen polymer.

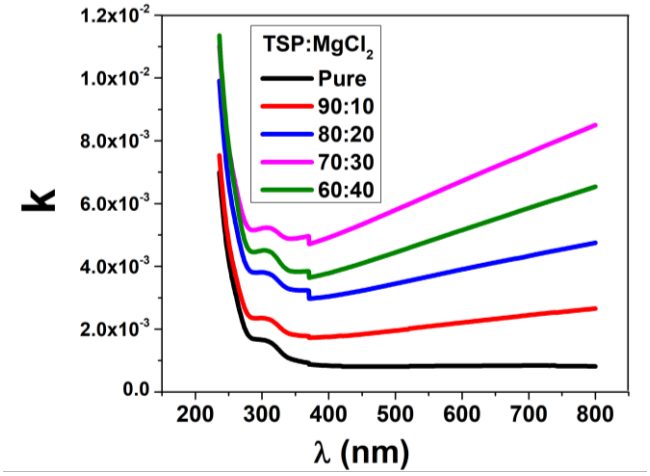

Figure 5 Refractive index for $MgCl_2$ doped TSP films.

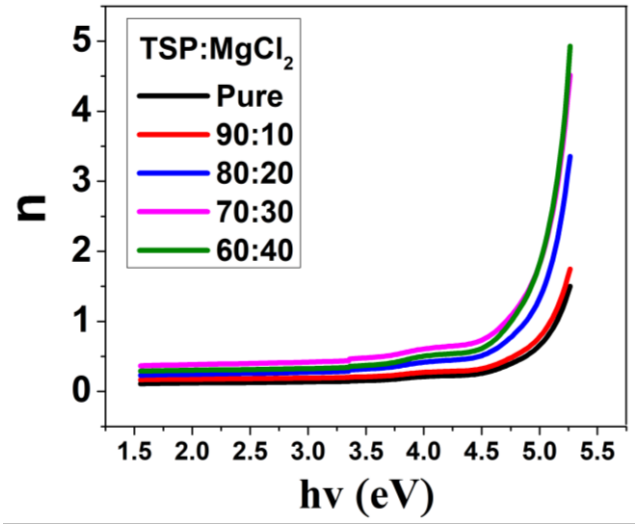

Figure 6 Optical extinction coefficient for $MgCl_2$ doped TSP films.

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4. Limitations

There are no specific limitations in the current study.

5. Conclusions

Using TSP as the host biopolymer and $MgCl_2$ as the dopant salt, solid biopolymer electrolyte films of compositions TSP:$MgCl_2$ (100:0, 90:10, 80:20, 70:30 and 60:40 wt.%) were prepared through the solution casting technique. Through UV visible spectroscopy, optical parameters were calculated. It was reported that for the film TSP:$MgCl_2$ (70:30 wt.%), optimum values of the parameters such as optical absorbance, optical transmittance, optical refractive index, optical absorption coefficient and optical extinction coefficient were calculated. The high values of optical absorbance and optical transmittance exhibited by the film TSP:$MgCl_2$ (70:30) indicate the efficient and optimal light absorption, which is essential for photovoltaic application. The lower values of extinction coefficient of the TSP biopolymer films suggest that the light scattering is minimum, which allows application in maintaining high efficiency in the solar cells. It was observed that the film TSP:$MgCl_2$ (70:30 wt.%) had the lowest value for absorbed photon energy, which indicates the threshold energy needed for the electronic transitions within the material.

Thus, the determination of the optical parameters such as optical absorbance, optical transmittance, optical refractive index, optical absorption coefficient and optical extinction coefficient of TSP biopolymer electrolytes can be correlated with the suitability of the material for the development of organic photovoltaic devices, where efficient light management is essential. Further, analysis of these parameters can help in the fabrication of biopolymer-based membranes for biomedical applications.

Supplementary materials

No supplementary materials are available.

Data Availability Statement

All related data is incorporated in the manuscript. However, raw data which supports the finding of this study cannot be shared at this point in time, as the data forms part of ongoing study.

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Authorship Contributions

Conceptualization: P.V.N.M.
Data curation: M.G.K.
Formal Analysis: P.V.N.M., N.K.J.
Investigation: M.G.K.
Methodology: P.V.N.M., I.S.R.K.
Resources: M.G.K.
Supervision: N.K.J.

Conflict of Interest

The authors hereby declare that they have no known competing financial interests or personal relationships which could have appeared to influence the work reported in this paper.

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