Optical and electrical conductivity studies of VO$_{2}^+$ doped polyvinyl pyrrolidone (PVP) polymer electrolytes

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1. Introduction

Polymer electrolytes have been a subject of great interest in the past few decades due to their importance in theoretical studies as well as their potential in practical applications such as sensors, electronic devices and components like light-emitting diodes, solar cells, thin film transistors, in micro electronics, batteries and linear or non-linear optics, photonics devices etc. [1–3]. Solid polymer electrolytes, compared to conventional liquid ones, can be easily fabricated with several advantages like safety, leakage-free nature, low cost and easy fabrication into flexible geometries [4,5]. Solid polymer electrolytes exhibit a reasonable ionic conductivity, the dimensional stability, processability and flexibility under ambient conditions. These features have been achieved by a variety of polymer electrolytes such as polymer–salt complexes, gel polymer electrolytes, composite polymer electrolytes and blend based polymer electrolytes.

PVP is one of the attractive polymers due to the strong affinity of the pyridine group and its ability to undergo the hydrogen bonding with polar species [6,7]. The chemical structure of PVP is shown in Fig. 1. As PVP binds to polar molecules exceptionally well, it is used as a binder in many pharmaceutical industrial process for the manufacuation of drugs. This binding nature leads to the application of PVP in coatings for photo-quality transparencies. PVP can be thermally cross-linked, thus ensuring a good thermal stability and mechanical strength and also because of the stability of PVP in water [8,9]. It should be noted that the formation of the transition metal ions and the polymer was conducted by the Fourier transform infrared spectroscopy (FTIR) and the UV-Vis spectroscopy. Room temperature impedance measurements in the frequency range 42 Hz to 5 MHz revealed that the ionic conductivity increased with the increasing the VO$_{2}^+$ ion concentration. The maximum ionic conductivity of 5.39 × 10$^{-3}$ Scm$^{-1}$ at 303 K was observed for the 5 mol% VO$_{2}^+$ ions doped PVP polymer electrolyte film. From the UV-Visible absorption spectra in the wavelength range of 200–800 nm the direct and indirect optical energy band gaps and optical absorption edges were found decreased with the increase in the VO$_{2}^+$ ion concentration. FTIR studies on pure and VO$_{2}^+$ doped PVP polymer films revealed the vibrational changes to occur due to the effect of the dopant VO$_{2}^+$ ions in the polymer. It is suggested that VO$_{2}^+$, as a dopant, is a good choice to improve the electrical properties of the PVP polymer electrolyte.
metal oxides, $V_2O_5$ is of much interest for the contemporary and emerging technology, i.e., applications in microelectronics, solid state ionics and optoelectronics [16–18]. It is also found that the addition of transition metal ions have considerable effects on the structural, optical, electrical and magnetic properties of the polymer. The main object of the present work is to develop a new kind of polymer electrolyte system doped with VO$_{2^+}$ ions, which can be used in the production of novel, solar cells, optical and electronic devices.

2. Experimental

The Polyvinyl Pyrrolidone (S. D. Fine — Chemical Ltd, India) based films were prepared by solution casting technique using the polymer solution doped with VOSO$_4$ (MOLY CHEM LTD) one, in which double distilled water was used as a solvent. For this purpose, a stock solution of 5% of PVP and solutions of various VOSO$_4$ concentrations (1, 2, 3, 4 and 5 mol%) were prepared separately. These solutions were stirred individually at room temperature using a magnetic stirrer for an hour to obtain transparent solutions. The aqueous solutions of pure PVP and one of the solutions of various VOSO$_4$ concentrations were then mixed and thoroughly stirred to form well homogeneous and clear solutions. The stirred solutions were cast onto polypropylene disks and allowed to slowly evaporate at room temperature until the polymer films were obtained on the bottom of the disks. The optical absorption curves of the as prepared films were recorded in the range 200–800 nm at room temperature using the JASCO UV — VIS — NIR Spectrometer (of the Model — V.700, Japan). The FTIR spectra were recorded using a FTIR spectrometer (Perkin—Elmer model-1605, USA), over the wave number range of 500–4000 cm$^{-1}$. The electrical properties of the as prepared polymer electrolyte films at room temperature were studied by using (The Hioki 3532-50 LCR Hi-Tester, Hioki, Japan) over the frequency range 42 Hz – 5 MHz.

3. Results and discussion

3.1. FTIR analysis

FTIR spectroscopy is an important tool to study the polymer structure changes as well as the formation of the complexes between the VO$_{2^+}$ ions and the host polymer in the polymer electrolyte system. The FTIR spectra were recorded in the range 500–4000 cm$^{-1}$ in the transmittance mode and the results for the pure and the VO$_{2^+}$ (1–5 mol%) doped PVP polymer films are shown in Fig. 2. The FTIR spectrum of the pure PVP exhibits pronounced bands at 3643-2849, 2324, 2123, 1707-1576, 933 cm$^{-1}$ which are assigned to the aliphatic C—H stretching, the CH$_2$ bending, the CH$_2$ wagging, and the O—H stretching groups. The FTIR peak assignment of pure and different concentration (1-5 mol%) VO$_{2^+}$ ions doped PVP polymer electrolyte films are listed in Table 1. The spectrum shows a broad band at 3643-2849 cm$^{-1}$ which is assigned to O—H stretching vibrations of alcohols and phenols [19,20]. The predominant OH vibrational band at 3643-2849 cm$^{-1}$ is shifted towards the lower wave numbers and appears as the broad hydroxyl band in all the doped films as compared to the pure PVP [21]. It gives a strong indication of specific interactions in the polymer matrices. The peak corresponding to the C=O bonding at 1707-1576 cm$^{-1}$ in the pure film is shifted to 1707-1567 cm$^{-1}$ for films of 1 and 2 mol % of VO$_{2^+}$ ions doping [22]. It is also found that this peak disappears in the spectrum for the films of 3–5 mol % VO$_{2^+}$ doping. In other words when VO$_{2^+}$ is added to PVP, there is a broadening of the C=O combination peak, its intensity reduces and the peaks finally disappears, which results from the induced structural rearrangement by adding the dopant to the polymer matrix [23].

The characteristic vibrational peaks at 2324, 2123 and 933 cm$^{-1}$ are assigned to the CH$_2$ deformation, the C=O combination and the C—O stretching of PVP respectively. They are shifted to 2324-2300 cm$^{-1}$, 2130-2147 cm$^{-1}$ and 933-931 cm$^{-1}$ respectively in the cases of the PVP polymer films doped with VO$_{2^+}$ ions. The interaction of the dopant ions with the polymer matrix leads to the shift of these bands. The above results confirm the formation of the complexes of the polymers with the VO$_{2^+}$ ions [24]. This influences the local structure of the polymer backbones and significantly affects their mobility. The compatibility between the polymer matrix and the inorganic dopants influences the optical and ionic conductivity of the PVP polymer electrolytes.

3.2. Optical absorption studies

The measurement of the absorption spectrum is the most direct and the simplest method for investigating the band structure of materials. In the absorption process, an electron gets excited from a lower to a higher energy state by absorbing a photon of known energy in the transmitted radiation. The changes in the transmitted radiation gives information on the types of the possible electron transitions. The fundamental absorption refers to band-to-band or the exciton transition. Furthermore, the fundamental absorption manifests itself by a rapid rise in the absorption known as absorption edge. The absorption edge can be used to determine the optical band gap, $(\varepsilon_0 = \frac{hc}{\lambda})$ as insulator/semiconductors are generally classified into two types (a) direct band gap and (b) indirect band gap materials. In direct band gap semiconductors, the...
top of the valence band and the bottom of the conduction band both lay at same zero crystal momentum (wave vector). If the bottom of the conduction band does not correspond to zero crystal momentum then it is called indirect band gap semiconductor.

Optical absorption spectra of the pure and the VO\textsuperscript{2+} (1–5 mol\%) doped PVP polymer films are shown in Fig. 3. The spectrum of the pure PVP film shows a peak at 220 nm, which is assigned to the carbonyl group of PVP. For the doped film, the intensity of this peak is increased as the absorption coefficient increases and the doping level increases. This may be due to the strong interaction between the PVP and the VO\textsuperscript{2+} ions [25]. The absorption coefficient \( \alpha \) can be determined from the spectra using the formula

\[
\alpha = 2.303 \times \frac{A}{d}
\]

where \( A \) is the absorbance and \( d \) is the thickness of the film. When a direct band gap exists, the absorption coefficient has the following dependence on the energy of the incident photon [26].

\[
\alpha \cdot h \cdot v = c \cdot (h \cdot v - E_g) \frac{1}{2}
\]  

where \( E_g \) is the band gap energy, \( c \) is constant which is dependent on the specimen structure, \( v \) is the frequency of the incident light and \( h \) is the Planck’s constant. Fig. 4 shows the variation of \( (\alpha \cdot h \cdot v)^2 \) vs \( h \cdot v \), (photon energy). The intercept on the energy axis up on extrapolating the linear portion of the curve to the zero absorption value may be taken as the value of the band gap. The optical band gap energy is obtained by extrapolating the linear region of the curve to the \( h \cdot v \) axis (the X-axis) using the origin software. In the present work, the direct band gap was determined as 3.88 eV for the pure PVP polymer electrolyte, while for the VO\textsuperscript{2+} doped films, values were found to decrease from 3.71 to 3.00 eV. For the indirect transitions which require a photon assistance, the absorption coefficient has the following dependence on the photon energy [26].

\[
\alpha \cdot h \cdot v = A (h \cdot v - E_g + E_P)^2 + B (h \cdot v - E_g - E_P)^2
\]

where \( E_P \) is the energy of the phonon associated with the transition and \( A \) and \( B \) are constants depending on the band structure. The indirect band gaps were obtained from the plots of \( (\alpha \cdot h \cdot v)^2 \) vs \( h \cdot v \) as shown in Fig. 5. For the pure PVP polymer electrolyte, the indirect band gap was 3.48 eV while for the VO\textsuperscript{2+} doped films, the values were found to decrease from 3.30 to 2.43 eV, which are presented in Table 2. The position of the absorption edge was determined by extrapolating the linear portions of the \( \alpha \) vs \( h \cdot v \) plots in Fig. 6 to zero absorption value. For the pure film the absorption edge was at 3.70 eV and for the doped films the values were found to decrease from 3.43 to 2.86 eV. Similar behavior was observed by Morsi et al. [23] and Zidan et al. [27] for the PEO/PVP blended with gold nano particles and the PVA/PVP blended with methylene blue films. The defects in the polymeric matrix might be the reason for the decrease in the band gap values with dopant concentrations, which produce the localized states in the optical band gap, and

which overlaps with the band system. These localized states, are responsible for the decrease in the band gap energy. This indicates that with the increase in the concentration of the VO\textsuperscript{2+} ions, PVP polymer films exhibits more semiconducting nature [28,29]. The process of doping introduces additional defect states in the polymeric matrix. The density of localized states was found to be proportional to the concentration of these defects [30] and consequently, to the VO\textsuperscript{2+} content. The values of the optical constants like the absorption edge, the direct band gap and the indirect band gap decrease due to the formation of the charge transfer complexes between the host polymer matrix and the dopant ions.

3.3 Impedance analysis

Fig. 7 shows the plots of the impedance \( Z' \) as a function of \( Z_\theta \), of the pure and the VO\textsuperscript{2+} ions doped PVP polymer electrolytes at room temperature. These plots known as the Cole–Cole plots, and their equivalent circuits are shown in Fig. 8. The analysis of the Cole—Cole plots has been carried out for the polymer electrolytes using the impedance spectroscopy. A small amount of \( ac \) current
was applied across the sample and the variation of imaginary ($Z''$) and real part ($Z'$) were measured using the 3532-50 LCR Hi-Tester in the frequency range from 42 Hz to 5 MHz. The Nyquist plot (also called Cole–Cole) generally consists of a semicircular arc at higher frequencies and a spike is formed at lower frequencies for the bulk resistance. In the obtained plots, the spike corresponds to the ionic conduction [31] and the semicircle is due to the space charge polarization.

The obtained conductivity values are shown in Table 3. The ionic conductivities of the pure and VO$_2^{+}$ doped PVP polymer electrolytes were calculated from the following relation

$$\sigma = \frac{t}{R_b A}$$  \hspace{1cm} (4)

where $\sigma$ is the ionic conductivity, $R_b$ is the bulk resistance, $t$ is the thickness of the polymer electrolyte and $A$ is the area of an electrode.

From the Cole–Cole plots, it can be seen that as the dopant concentration increases the diameter of the semicircle decreases implying the decrease of the bulk resistance ($R_b$). The depressed semicircle indicates that the semi-crystalline nature in the polymer is converted to the amorphous nature where the transfer of the VO$_2^{+}$ ions takes place in the polymer matrix. This amorphous phase produces a free space volume [32]. The calculated values of the ionic conductivity for the pure and VO$_2^{+}$ doped PVP polymer electrolytes are listed in Table 3. When compared to other dopant concentrations, the 5 mol% VO$_2^{+}$ doped PVP polymer electrolyte shows the highest ionic conductivity. The increment in the ionic conductivity with VO$_2^{+}$ ion concentration is attributed as due to the rise in the number of charge carriers as shown in Fig. 9.

### 3.4. Dielectric properties

The dielectric spectroscopy and impedance analysis were carried out using the HIOKI 3532-50 LCR Hi-Tester in the frequency range from 42 Hz to 5 MHz at room temperature. The dielectric constant $\varepsilon'$ and $\tan\delta$ of the present polymer samples were evaluated

| Concentration (mol%) VO$_2^{+}$/PVP | Direct band gap (eV) | Indirect band gap (eV) | Absorption edge (eV) |
|------------------------------------|----------------------|-----------------------|---------------------|
| Pure PVP                           | 3.88                 | 3.48                  | 3.70                |
| 1                                  | 3.71                 | 3.30                  | 3.43                |
| 2                                  | 3.56                 | 3.17                  | 3.26                |
| 3                                  | 3.49                 | 2.76                  | 3.15                |
| 4                                  | 3.36                 | 2.54                  | 3.02                |
| 5                                  | 3.00                 | 2.41                  | 2.86                |
from the capacitance measurements. Fig. 10 shows the variation of $\varepsilon'$ as a function of frequency for the pure and the VO$_2^+$ doped PVP polymer samples at room temperature. From the Fig. 10, it is observed that by increasing the frequency the dielectric constant ($\varepsilon'$) decreases sharply. The decrease in the dielectric constant with the increase in the frequency takes place due to the polarization at the electrode—electrolyte interface and is found to be high for the sample of 5 mol% VO$_2^+$ dopant. This may be due to the drifting of the ions, resulting in a high conductivity. This reveals that the VO$_2^+$ ions are completely dissolved in the polymer chains giving raise to the mobile ions. Due to the formation of the space charge region at the electrode—electrolyte interface a variation is observed in between dielectric constant ($\varepsilon'$) and the frequency which is dependent on the electrolytes, indicating the non-Debye behavior [33–37].

Fig. 11 shows the variation of the dielectric loss factor tan $\delta$ with the frequency ($f$) at room temperature for all the samples. The decrease in the loss factor tan $\delta$ along with the increasing frequency may be due to the reduced proportion of the amorphous nature leading to the reduction in the dispersion magnitude. The appearance of peaks suggests the presence of the relaxing dipoles in the samples and associated with the electrical relaxation process or the inability of dipoles [38].

Fig. 12 shows the variation of the ionic conductivity with respect to the frequency. It explains the conductivity dependence on the relaxation process that is non-exponential in time. The plateau region describes the space charge polarization at the blocking electrode and is associated with ac conductivity ($\sigma_{ac}$) of the complexed polymer electrolyte. As shown in Fig. 11, in the high frequency dispersion region, the ionic conductivity remains nearly constant. However, at high frequencies the conductivity variation at

Table 3
The conductivity values of the pure and the VO$^2^+$ (1, 2, 3, 4 and 5 mol%) doped PVP polymer electrolyte films at room temperature.

| Concentration (mol %) VO$^2^+$: PVP | Conductivity at 303 K (S cm$^{-1}$) |
|-------------------------------------|-----------------------------------|
| Pure PVP                           | $0.96 \times 10^{-8}$             |
| 1                                  | $1.55 \times 10^{-8}$             |
| 2                                  | $1.96 \times 10^{-8}$             |
| 3                                  | $3.02 \times 10^{-8}$             |
| 4                                  | $3.60 \times 10^{-8}$             |
| 5                                  | $5.39 \times 10^{-8}$             |
room temperature is much less than that at lower frequencies [39,40].

4. Conclusion

Solid polymer electrolyte films of polyvinyl pyrrolidone (PVP) complexed with different (1–5 mol%) concentrations of VO$^{2+}$ ions were prepared by the solution cast method. UV-Visible analysis revealed that the value of the optical band gap decreases as the samples were prepared by the solution cast method. FTIR analysis of the sample. From all the characterization results, the 5 mol% VO$_2$ has considerable interaction between the polymer and the VO$_2$ complexed with different (1–5 mol%) concentrations of VO$^{2+}$ ions. Band positions with a change in their intensities. This indicates the formation of the charge transfer complexes between the polymer and the dopant. FTIR spectra show shifts in some of the phous nature and a high ionic conductivity. Hence the VO$_2$ powdery electrolyte films. [8]

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