



A Study on Electrolyte Interactions With Polymer and Nanofiller for Dye Sensitized Solar Cells

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Abstract

In the current scenario, electrolyte plays a crucial role in the fabrication of Dye-Sensitized Solar Cells (DSSCs). To advance the performance of DSSCs, it is essential to explore electrolyte with polymers. Polyvinyl alcohol (PVA) is non toxic, bio compatible, bio degradable, thermally and chemically stable water-soluble polymer with excellent mechanical strength and transparency, which is anticipated to be an excellent candidate in DSSCs. An attempt was made on Polymer based electrolyte with nano filler thin films which are prepared using dip coating technique. The properties of the prepared films are characterized by various experimental techniques such as FTIR, UV-Vis and Electro Chemical Impedance spectroscopy. The Fourier Transform Infrared (FTIR) analysis reveals the complexation of the polymer nanocomposite network and the UV-visible spectral study shows the optical band gap. Besides, the impedance analysis indicates high ionic conductivity which is due to ionic mobility and segment motion of polymer network. The outcome of the studies reveals the interaction of electrolyte with polymer and nano filler for Dye -Sensitized Solar Cells.

Keywords: FTIR, UV-Visible, Impedance, conductivity, band gap.

1. Introduction

There is a sustained interest for clean/alternative energy strategies and for environmental remediation. Growing environmental concerns related to the extensive use of non-sustainable fossil fuels and a

constantly increasing energy demand will force mankind, sooner or later to tap into clean and sustainable source of energy. Solar cell is expected to make a great contribution to both environmental treatment and renewable energy. Solar energy is widely considered to be future clean energy carriers in many applications such as environmentally friendly vehicles, domestic heating and stationary power generation [1-2].

Dye – Sensitized Solar Cells (DSSCs) have been under investigation for the past decade due to their attractive features such as high energy conversion efficiency and low production cost [3-4]. Polymers have played an integral role in the advancement of DSSCs. Regenerable redox couples (eg I/I_3^-) are usually dissolved in an organic solvent, which results in high energy conversion efficiency but it has got some drawbacks such as leakage and evaporation of the solvent [5-6]. Therefore, several attempts have been made to substitute liquid electrolyte with solid or quasi-solid state electrolyte such as solid polymer electrolyte (SPEs) [7], Polymer gel electrolyte (PGEs) [8] and organic hole-transport materials [9].

Among them nanocomposite solid polymer electrolyte have achieved considerable attention because of their excellent properties such as easy fabrication, low cost and good stability. So they have obtained growing interest in solid state batteries, electrochromic devices and fundamental research of ion transport in disordered phases [10].

Polyvinyl alcohol (PVA) based polymer electrolytes also attracted much interest due to their good film forming capability, hydrogen bonding enhanced

mechanical strength, wide temperature window in which they are stable in gel phase and biodegradability.

Recently, a new class of materials in which an inorganic/ceramic nanosized filler or high molecular weight organic filler was dispersed into the polymer electrolyte matrix as the third component, seems to be an attractive approach for the formation of nanocomposite polymer electrolyte.

In the present study, the polymeric material used to prepare the nanocomposite thin film is Polyvinyl Alcohol (PVA), and the electrolyte material is Potassium Iodide (KI) and Iodine (I₂) and also the nanofiller is Zinc Oxide.

In this study, we report on the preparation and characterization of PVA based electrolyte with nanofiller. Polymer nano composite electrolyte system composed of PVA as a host polymer, Potassium iodide and iodine as salt and nano sized ZnO as filler. The FTIR, UV and ionic conduction study for the polymer nanocomposite are investigated.

2. Experimental

2.1. Materials

Poly (Vinyl Alcohol) PVA having molecular weight 1,20,000 and Potassium Iodide (KI), Iodine (I₂) and Zinc Oxide (-30 nm) from sigma Aldrich were used. Dimethyl Sulfoxide (DMSO) of Purity > 99% obtained from Merck is used as solvent.

2.2. Preparation of Polymer Electrolyte films

Polymer electrolyte solution containing of 0.5g of PVA was dissolved in Dimethyl Sulfoxide (DMSO). Different amount of KI (0.04, 0.06, 0.08, 0.1g) were then added to the solution. The solution was then heated to 393K and maintained for 30 minute until a viscous like solution was formed. The solution was allowed to cool down to 323K and some iodine (10% of weight) crystals were added with continuous stirring until a homogeneous polymer electrolyte was produced. It was then casted on FTO glass slides as thin films by using dip coating unit (Model: SPEKTRODIP Dip coater. The

prepared thin films are stored safely in desiccators for further study.

UV visible spectra (UV-Vis recording spectrophotometer Lambda 35) is taken. The transmission (T%) is measured in the spectral range 250 to 1100 nm at room temperature. The thickness of the thin films are measured by a digital micrometer screw gauge. Electrochemical impedance spectroscopy (EIS) is done to determine the impedance of the electrolytes.

3. Results and Discussion

3.1. FTIR study of PVA, PVA /KI, PVA/KI/I₂ and PVA/KI/I₂/ZnO

FTIR transmission spectra of Pure PVA (a) PVA/KI/I₂ (b) PVA/KI/I₂/ZnO (c) are shown in figure 1.

Pure PVA

The spectra of 0.5 g of PVA shows the broad band observed between 3500 and 3200 cm⁻¹ refers to the intermolecular hydrogen bonding and O-H stretching vibration. The vibrational band between 3000 cm⁻¹ and 2888 cm⁻¹ is associated with the C-H asymmetric stretching from alkyl groups and absorption peaks between 1700 cm⁻¹ and 1690cm⁻¹ which is due to stretching of C-O from acetate group remaining in PVA [11-12].

Coates has mentioned in his paper that stretching hydroxyl (O-H) group occurs between the regions of 3570-3200 cm⁻¹. Coates [13] and Awadia and Agarwal [14] have also mentioned that O-H stretching occurs at 3615-3050 cm⁻¹.

The Peak at 3402 cm⁻¹ in pure PVA is ascribed to hydroxyl group (O-H) stretching and it is not due to absorbed solvent. The band corresponding to methylene (CH₂) group asymmetric stretching and aliphatic C-H stretching vibration of pure PVA occurs at about 2997, 2913 cm⁻¹ [15-18].

2.3. Characterization

The FTIR (FTIR Spectrophotometer -Jasco 6300) is done for wavenumber range between 400 to

4000 cm^{-1} , to study the functional groups and the interaction between the polymer and electrolyte.

PVA with KI & I₂

From the table 1 it is inferred that the O-H stretching vibration at 3402 cm^{-1} (28%) is shifted to 3533 cm^{-1} (24%). For a change in amount of I₂ from 0.002g, 0.004g, 0.006g and 0.008g, the shift in vibrations are 3407, 3560, 3241 and 3509 cm^{-1} are shown in fig 1 b. These changes indicate that I₂ can act as a secondary dopant on PVA, the maximum influence of I₂ is observed at 0.006g of I₂, the O-H peak intensity and position both being affected tremendously at this concentration. This effect begins at 0.004g of I₂, as evident from large number of bands in the region 2596 cm^{-1} to 1911 cm^{-1} at this concentration.

The C-C stretching vibration in PVA located at 1577 cm^{-1} and 1578 cm^{-1} in PVA-KI mixtures are shifted to 1663, 1655, 1660 cm^{-1} on adding I₂ (0.002, 0.004, 0.006 & 0.008g). These changes are attributed to strong interaction of the secondary dopant I₂ on PVA-KI doped polymer matrix. The changes in the C-C stretching & O-H in plane bending mode at 1430 cm^{-1} is shifted from 1436 cm^{-1} to 1418, 1436, 1433 cm^{-1} as I₂ concentration changes. The maximum effect of I₂ on changing polymer configuration is evident at 0.004g of I₂ & 0.006g of I₂ as evident from the initial blue shift (18 cm^{-1} with decreased intensity 27%) then red shift (18 cm^{-1} with increased intensity 7%) then a blue shift 93 cm^{-1} with increased intensity of 16%) as concentration of I₂ changes from 0.002, 0.004, 0.006, 0.008g.

The C-O stretching vibration is affected by about 8 cm^{-1} with random changes in the intensity from 26% to 40%, 42% to 21% as I₂ concentration increases uniformly by 0.002g. The bending vibrations in the region 950 cm^{-1} - 600 cm^{-1} appear to become broad with increased intensity. The C-I vibration expected at 550 cm^{-1} due to doping by I₂ is not separately found as a band. Hence I₂ should be present in between the polymer chain converting the polymer to the expanded coil

arrangement. KI & I₂ may interact to produce I₃ ions that may bind to O-H groups of polymer, favouring expanded coil networking of PVA-KI₃ skeleton.

PVA-KI-I₂-ZnO

FTIR spectra of PVA-KI-I₂ -ZnO is shown in figure 1c. The Spectra of Polymer electrolyte/ Zinc Oxide nanocomposite films clearly exhibit characteristics absorption peaks corresponding to only polymeric groups of Pure PVA with PVA-KI-I₂. That is, the FTIR spectra of the nanocomposite films show no apparent change or shift in the characteristic peaks of PVA with the addition of ZnO nanoparticles. The pure PVA, PVA-KI-I₂ and PVA- KI-I₂-ZnO nano composite films yielded the same FTIR spectra which means a poor chemical interaction between the polymer electrolyte and Zinc oxide occurred or the ZnO characteristics peaks is overlapped by the polymer electrolyte characteristic peaks (Table 1).

ZnO nanoparticles are generally polar and thus strong self - interaction is induced between the nanoparticles. Such a scenario results in hard agglomerates [22]. The high polarity of ZnO nanoparticles impedes the chemical interaction between ZnO and the polymer matrix. This in turn causes no change or shift in the characteristic peak found in the FTIR analysis. The changes in the position, shape, intensity, formation of new peaks and disappearance of peak infers the interaction between PVA, KI, I₂ and ZnO. Therefore it can be concluded that electrolyte is associated in the polymer matrix. IR spectra results prove that the complexation of PVA with KI & I₂ are summarized in Table 1.

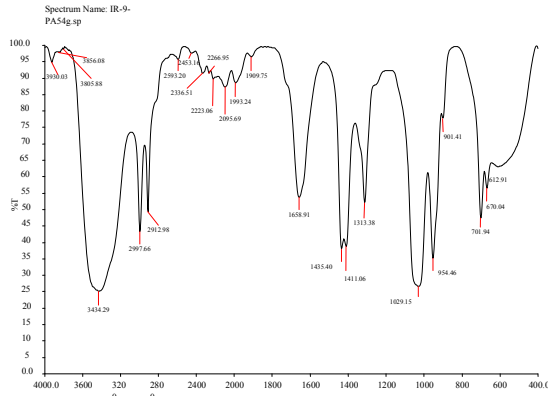


Figure 1a. FTIR spectra of PVA

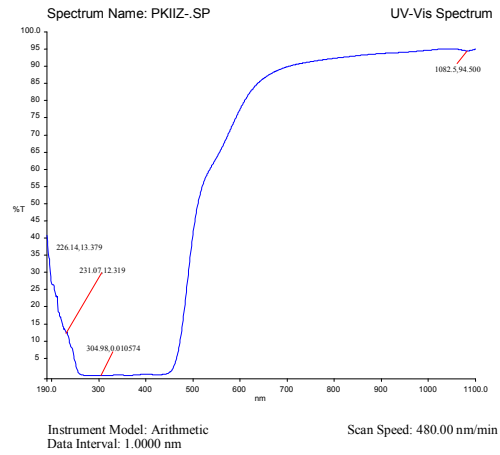


Figure 2. UV-Visible spectra of PVA composite film

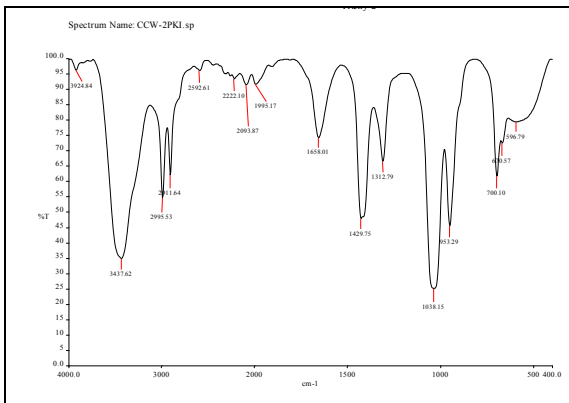


Figure-1b. FTIR spectra of PVA/KI/I2

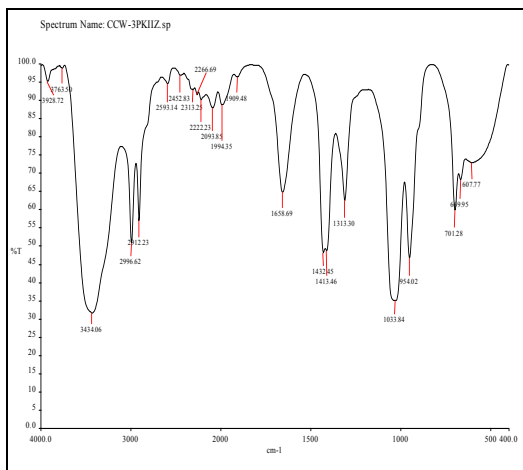


Figure1.c. FTIR spectra of PVA/KI/I2/ZnO

3.2. Optical Properties

The Optical Properties of Polymers can be suitably modified by the addition of dopants depending on their reactivity with the host matrix [23]. The optical absorption spectrum is an important tool to obtain optical band gap energy of crystalline and amorphous materials. The fundamental absorption which corresponds to the electron excitation from the valence band to the conduction band can be used to determine the nature and value of the optical band gap. The optical transmittance spectra of composite films of PVA-KI-I2-ZnO is shown in figure 2.

It is inferred that the film possess transmittance of above 80% in the wavelength region $\lambda > 400$ nm and absorption edges lies in the ultraviolet region of the spectrum. The relation between the absorption coefficient (α) and incident photon energy ($h\nu$) can be written as [24, 25].

$$\alpha h\nu = c (h\nu - E_0)^m \text{-----(1)}$$

where c is a constant, E_0 is the optical band gap of the material and the exponent m depends on the type of transition, m is an index which can be assumed to have values of $1/2$, $3/2$, 2 and 3 depending on the nature of the electronic transition responsible for absorption, m is equal to $1/2$ for allowed direct transitions, $3/2$ for direct forbidden transitions, 2 for allowed indirect

transitions and 3 for forbidden indirect transitions. The indirect band gaps of films E_0 can be obtained from equation (1) by extrapolating linear portion of $(\alpha hv)^{1/2}$ to zero absorption in the $(\alpha hv)^{1/2}$ Vs hv plot as shown in the figure 3.

The addition of (KI & I₂) & ZnO causes a decrease in E_0 which may be explained on the basis of incorporation of amount of dopant forms charge

transfer complexes (CTCS) in the host lattice, which enhance the lower energy transitions leading to the observed change in optical band gap. These CTCs increase the electrical conductivity by providing additional charges in the lattice and hence a decrease of band gap [26, 27]. The values of direct band gaps of the composite films of PVA, KI, I₂ and ZnO are around 3.33eV.

Vibrational Peaks and assignments of (PVA)_{0.5g}, (PVA)_{0.5g}:(KI)_{0.06g}, (PVA)_{0.5g}:(KI)_{0.08g}, (PVA)_{0.5g}:(KI)_{0.08g}:(I₂)_{0.002g}, (PVA)_{0.5g}:(KI)_{0.08g}:(I₂)_{0.004g}, (PVA)_{0.5g}:(KI)_{0.08g}:(I₂)_{0.006g}, (PVA)_{0.5g}:(KI)_{0.08g}:(I₂)_{0.008g}, (PVA)_{0.5g}:(KI)_{0.08g}:(I₂)_{0.006g}:(ZnO)_{0.004g}

Pure (PVA) _{0.5g}	Vibrational Peaks of PVA + KI + I ₂ (cm ⁻¹)							Assignments
	(PVA) _{0.5g} + (KI) _{0.06g}	(PVA) _{0.5g} :(KI) _{0.08g}	(PVA) _{0.5g} :(KI) _{0.08g} :(I ₂) _{0.002g}	(PVA) _{0.5g} :(KI) _{0.08g} :(I ₂) _{0.004g}	(PVA) _{0.5g} :(KI) _{0.08g} :(I ₂) _{0.006g}	(PVA) _{0.5g} :(KI) _{0.08g} :(I ₂) _{0.008g}	(PVA) _{0.5g} :(KI) _{0.08g} :(I ₂) _{0.006g} :(ZnO) _{0.004g}	
3927 3402	3928, 3396	3927 3402	3754 3930 3533	3846 3928 3407	3927 3560 3241	3928 3755 3509	3434 3763 3928	O-H stretching
2997 2913	2999, 2915	2997 2913	2997 2914	2998 2916	2997 2913	2997 2913	2912 2996	C-H asymmetric stretching of CH ₂
2593	2594	2594	2594	2596	-----	2594		C-H stretching
2452	2332	2452 2321	----	2452 2315	-----	-----	2593	
2222	2222	2222	----	2218	-----	-----	2313 2452	
2097	2096	2096	2091	2096	-----	2093	2093 2222 2266	
1994, 1910	1992	1993, 1909	1989, 1908	1991, 1911	1985	1990 1909	1909 1994	
---	---	1727	1727	1725	---	-----	1658	C-O stretching
1577	1577	1578	1663	1665	1665	1660		C=O stretching and C=C stretching
1405	1403	1403	1436	1418	1436	1433	1432 1413	O-H bending and C-H bending of CH ₂
1315	1331	1323	1313	1315	1312	1313		CH-OH bending and CH ₃ in plane deformation
1258	1258	1256, 1117	1251	-----	-----	-----	3434 3763 3928	C-H wagging and C-O stretching
---	1119	1034	1033	1029	1037	1031		C-C, C-O stretch and O-H bending
1037	---	955	953	952	953	952		O-H out of plane bending and CH ₂ rocking vibrations
954	955	831	-----	-----	-----	-----		C-C stretching and CH ₂ rocking
830	830	758	786,702	-----	791	-----		O-H twisting
757	760	699	-----	701	704	701		
700	699	655	-----	----	---	----		

Vibrational Peaks of PVA + KI + I ₂ (cm ⁻¹)								Assignments
Pure (PVA) _{0.5g}	(PVA) _{0.5g} + (KI) _{0.06g}	(PVA) _{0.5g} : (KI) _{0.08g}	(PVA) _{0.5g} : (KI) _{0.08g} : (I ₂) _{0.002g}	(PVA) _{0.5g} : (KI) _{0.08g} : (I ₂) _{0.004g}	(PVA) _{0.5g} : (KI) _{0.08g} : (I ₂) _{0.006g}	(PVA) _{0.5g} : (KI) _{0.08g} : (I ₂) _{0.008g}	(PVA) _{0.5g} : (KI) _{0.08g} : (I ₂) _{0.006g} : (ZnO) _{0.004g}	
-----	426	596	-----	-----	-----	-----	607	C-I stretching

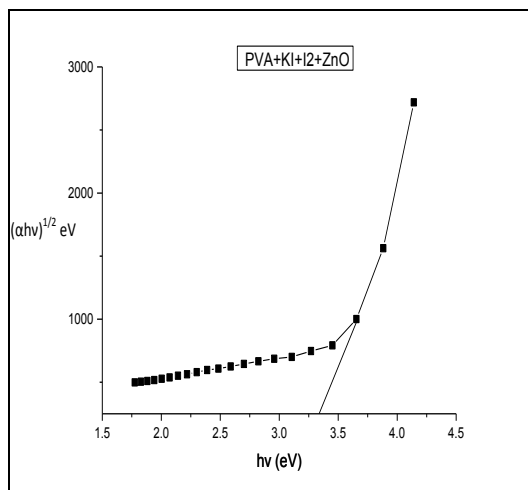


Figure 3 . (αhv) vs $(\alpha hv)^{1/2}$ plots of PVA composite film

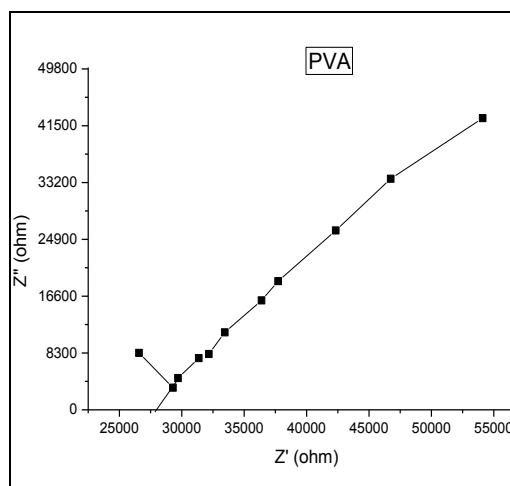


Figure 4 a. Real and imaginary part impedance of pure PVA

3.3. Conductivity studies

The conductivities of the polymer complexes are calculated from the bulk resistance obtained by the intercepts of the typical impedance curves of (Nyquist Plot) for various films of PVA, PVA+KI+I₂ and PVA+KI+I₂+ZnO.

The real and imaginary parts are taken along the x axes and y axes which is shown in figure 4 a, b, c respectively. Intercept of the curve on the real axis gives the bulk resistance (R_b) of the sample. The bulk conductivities σ are calculated using the relation.

$$\sigma = l / R_b A \text{-----(1)}$$

where l is the thickness, R_b is bulk resistance and A is contact area of the electrolyte film during the experiment.

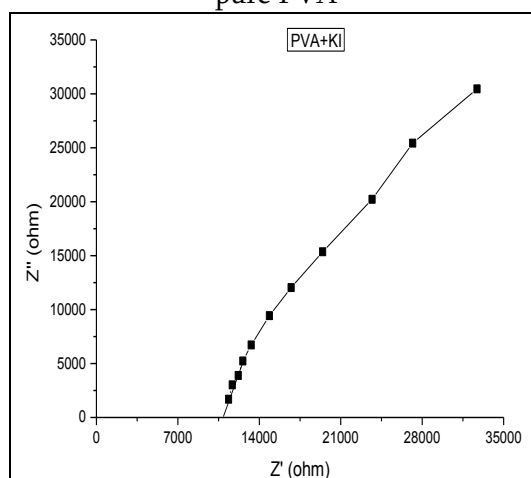


Figure 4 b. Real and imaginary part impedance of PVA+KI

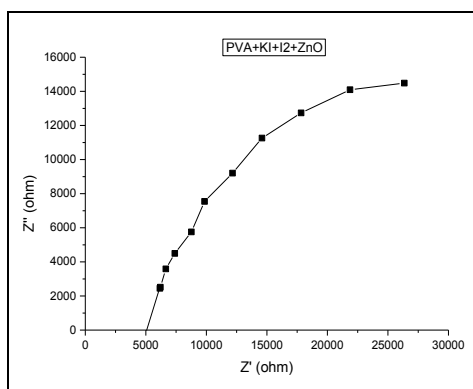


Figure 4 c. Real and imaginary part impedance of PVA+KI+I₂ thin film

The increase in ionic conductivity of polymer electrolyte is due to the reduction in the energy barrier, thereby facilitating the ionic transport. The ionic conductivity of about $0.4115 \times 10^{-3} \text{ S cm}^{-1}$ for pure polymer and $2.307 \times 10^{-3} \text{ S cm}^{-1}$ is observed for the composition of PVA + KI + I₂ + ZnO at 25°C which is shown in the table 2. Table 2. Bulk resistance (R_b), Conductivity (σ), dielectric constant (ϵ) in the Polymer and Polymer nano composite thin films at 100 Hz

Sample	R _b	$\sigma \times 10^{-3}$ (S cm ⁻¹)	ϵ
PVA	27845.23	0.4115	3.7204
PVA+KI	10682.94	1.0727	49.366
PVA+KI+I ₂	5345.24	2.1439	116.4285
PVA+KI+I ₂ +ZnO	4966.659	2.3074	153.8322

3.4. Dielectric Properties

The study of dielectric relaxation in solid polymer electrolytes is a powerful approach for obtaining information about the characteristics of ionic and molecular interactions. The dielectric parameters associated with relaxation processes are of particular significance in ion conducting polymers where the dielectric constant plays a fundamental role which shows the ability of polymer material to dissolve salts. The dielectric constant is used as an indicator to show the

increase in conductivity which is mainly due to an increase in the number density of mobile ions. The frequency-dependent conductivity and dielectric relaxation are both sensitive to the motion of charged species and dipoles of the polymer electrolytes.

The complex dielectric constant of a system ϵ^* is defined by

$$\epsilon^* = \epsilon' - i \epsilon'' \quad \text{-----(2)}$$

Real part of dielectric constant ϵ' of the material is expressed as $\epsilon' = cd / \epsilon_0 A$

where c is the parallel capacitance, d is the thickness of the film, ϵ_0 is the permittivity of the free space and A is the area of the contact area of the film. The variation of the real part of the dielectric constant ϵ' as a function of frequency for the Polymer nanocomposite thin film is shown in figure 5. The observed variation in ϵ' with frequency could be attributed to the formation of a space charge region at the electrode and electrolyte interface, which is known as the non-Debye type of behaviour where the space charge region with respect to the frequency is explained the interaction of ion diffusion. The material electrode interface polarization of the composites masks the other relaxation processes at low frequencies. On the other hand, with increasing frequency there is no time for charge build-up at the interface because of the increasing rate of reversal of the electric field. Therefore, the polarization due to charge accumulation decreases which leads to the decreases in the value of ϵ' .

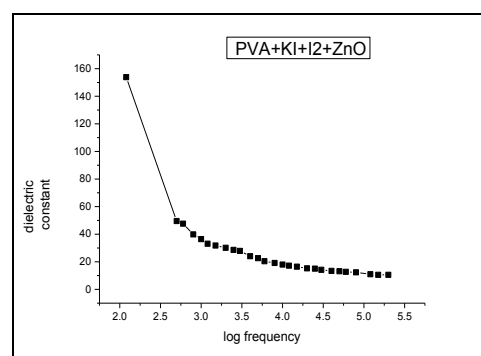


Figure 5. Log frequency Vs dielectric constant of PVA composite film

Conclusion

The polymer electrolyte based on PVA, KI, I₂ and ZnO thin film is obtained using dip coating technique. FTIR spectra prove that the complexation of PVA with KI, I₂ and ZnO. The addition of I₂ and ZnO to the PVA-KI polymer electrolytes has proved to be a convenient method to increase the ionic conductivities of the membranes at 2.307×10^{-3} S/cm at ambient temperature. The increase of degree of amorphousness in the polymeric material increases ϵ' values. The decrease in the optical band gap can be correlated to the formation of the charge transfer complexes within the polymer network on dispersing I₂ in it. From a practical point of view, the polymer based electrolyte is a potential candidate for Dye sensitized solar cells.

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