Research Square

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Enhanced Ionic Conductivity in Polyvinyl Alcohol-Based Gel Polymer Electrolytes with Titanium dioxide Nanofillers

A. A. Rahim
Universiti Sains Malaysia
P. Q. B. Mazlan
Universiti Sains Malaysia
C. G. Er
Universiti Sains Malaysia
M. F. Shukur
Universiti Teknologi Petronas
M. H. Buraidah
University of Malaya
M. F. Aziz
fareezuanaziz@usm.my

Universiti Sains Malaysia

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Abstract

The development for advanced polymer electrolytes with high ionic conductivity is crucial for replacing traditional liquid electrolytes in energy conversion systems that often suffer from leakage. Gel polymer electrolytes (GPEs) offer a safer and more stable alternative, yet their performance is restricted by the high crystallinity of polymer. In this study, a polyvinyl alcohol (PVA)-based electrolyte system was developed comprising potassium iodide (KI) in ethylene carbonate (EC) and propylene carbonate (PC), and dimethyl sulfoxide (DMSO) solvent. Titanium dioxide (TiO_2) nanofillers at varying concentrations were introduced to analyze their effect on electrolyte properties. Structural and electrochemical characterizations were carried out using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and electrochemical impedance spectroscopy (EIS). XRD analysis revealed a significant reduction in crystallinity with TiO_2 incorporation, whereas FTIR demonstrated strong interactions between polymer, salt, and filler. Ionic conductivity increased significantly with nanofiller addition, reaching a maximum of 10.4 mS cm^{-1} at 4 wt.% TiO_2. These findings highlight the beneficial effect of nanofillers in high-conductivity GPEs, offering the promising potential for developing a safe and efficient gel electrolyte in electrochemical systems.

1.0 Introduction

Polymer electrolytes have emerged as promising alternatives to traditional liquid electrolytes in a range of electrochemical devices, including lithium-ion batteries, supercapacitors, and solar cells. Unlike liquid electrolytes, their practical deployment is severely hampered by inherent issues of solvent evaporation, leakage, corrosion, and poor long-term thermal stability, which lead to rapid device degradation [1]. However, polymer electrolytes offer improved mechanical stability, enhanced safety, and flexible processing options [1, 2]. Their solid or semi-solid nature reduces risks associated with device failure and enables the development of thin, lightweight, and flexible energy conversion systems.

Among polymer electrolytes, gel polymer electrolytes (GPEs) represent a superior compromise, combining the high ionic conductivity of liquids with the mechanical integrity and enhanced safety [3–5]. These quasi-solid-state electrolytes consist of a liquid electrolyte entrapped within a solid polymer matrix. This hybrid nature not only mitigates the risk of leakage but also provides excellent interfacial contact with electrodes, resulting in improved device performance and stability. There are many polymers available that can be utilized as polymer host in synthesis of GPE which include polyethylene oxide (PEO) [6], polyacrylonitrile (PAN) [7], polyvinyl alcohol (PVA) [8], carboxymethylcellulose (CMC) [9] to name a few. PVA is a semicrystalline polymer which commonly used in GPE synthesis due to its excellent film-forming ability, chemical stability, non-toxicity, and abundance of hydroxyl groups that can facilitate ion transport [10–12]. However, a significant drawback of polymer is its high degree of crystallinity, which severely restricts ionic mobility by creating rigid, impenetrable regions within the polymer matrix. Despite these limitations, GPEs are increasingly used in advanced electrochemical applications due to their balanced properties and potential for safer, more efficient energy conversion solutions.

Thus, to overcome this limitation, several strategies and modifications can be employed to provide better performance of GPEs. One effective approach is the introduction of plasticizers, such as ethylene carbonate (EC) and propylene carbonate (PC), which disrupt crystalline domains and enhance the amorphous content, thereby facilitating ion transport [13, 14]. Moreover, to further increase the GPEs performance is by incorporation of a dopant salt. Previous studies have been used iodide salt particularly potassium iodide (KI), which is notable for improving the performance and ionic conductivity of GPEs by providing additional charge carrier [15]. KI salt also aids in maintaining the amorphous nature of the polymer matrix by disrupting crystalline regions, which improved ion migration and overall charge transport [16].

A further advanced approach to enhance electrolyte performance is the incorporation of inorganic nanofillers. Among of the nanofillers, titanium dioxide (TiO_2) is a particularly promising candidate due to its high dielectric constant, which promotes greater salt dissociation [17]. Moreover, its ability to disrupt polymer chain packing, help reducing overall crystallinity and creating additional ion conduction pathways [18–20]. This nanocomposite approach can significantly improve the ionic conductivity and electrochemical stability of the GPE. In this study, the TiO_2 was used as nanofillers in PVA-based GPE incorporated with KI salt. Various characterization techniques were carried out to investigate the effect of nanofillers on electrolyte performance. This research aims to contribute toward the design of safe, efficient, and scalable electrolytes for next-generation energy conversion technologies.

2.0 Methodology

2.1 Materials

Polyvinyl alcohol (PVA) (99% hydrolyzed), ethylene carbonate (EC) and propylene carbonate (PC), titanium dioxide (TiO_2), and iodine (I_2) crystals were purchased from Sigma Aldrich. Dimethyl sulfoxide (DMSO) was obtained from R&M chemicals. Potassium iodide (KI) was from Univar Analytical Reagent.

2.2 Gel polymer electrolyte preparation

Gel polymer electrolyte was prepared by mixing the polyvinyl alcohol (PVA) as host polymer with potassium iodide (KI) dopant salt and addition of various concentration of titanium dioxide (TiO_2) nanofiller in ethylene carbonate (EC), propylene carbonate (PC) potassium iodide and dimethyl sulfoxide (DMSO) solvent. 0.42 g of KI salt was first being dissolved in EC-PC solvent and stirred continuously at 30˚C for 15 minutes. At the same time, PVA was also being dissolved in DMSO solvent at 80˚C for 20 minutes. Simultaneously, before mixed both KI-EC-PC and PVA-DMSO mixed solution, TiO_2 was dissolved in DMSO for 30 minutes at 80°C under continuous stirring of hotplate. After varied concentration of TiO_2 was dissolved, the TiO_2 solution was added in KI-EC-PC mixed solution and stirred at 30˚C for 20 minutes. KI-TiO_2 mixed solution was poured gradually into PVA-DMSO solution and stirred until a gel-like form mixture obtained at 100°C in closed container. When a gel form achieved, iodine crystals (I_2) were added and stirred continuously at 45°C till completely dissolved. This procedure

resulted in the form of gel and the final GPE samples was placed in a veil and stored in a desiccator before sent for characterization. The sample composition of PVA-KI-EC-PC-TiO_2 GPE system listed in Table 1.

Table 1
The variation of weight percentage and weight of the TiO_2 in the GPEs with fixed amount of PVA (0.20 g), EC (0.30 g), PC (0.4 g), KI (0.42 g) and DMSO (2.2 ml).

Designation	TiO_2 (wt.%)	Weight (g)
TD1	0	0.000
TD2	2	0.143
TD3	4	0.287
TD4	6	0.430
TD5	8	0.574

2.3 Fourier Transform Infrared Spectroscopy

FTIR spectrophotometer (iS10 Thermo Scientific) was used to study the chemical composition and molecular interactions in gel polymer electrolytes (GPEs) by analysing the infrared absorption spectra. The data revealed the information about functional groups and molecular structures. The interaction and coordination between salt, polymer and the nanofiller of GPE were also studied by FTIR characterization. The measurement of the wavenumber was in the range of 500 to 4000 cm^{-1} with resolution of 2 cm^{-1} which are suitable for analysing solid and liquid state of GPE.

2.4 X-ray diffraction

X-ray diffraction (XRD) is a crucial technique for analysing the structural properties and presence of impurity of phases in gel polymer electrolytes (GPEs). The structure of GPE samples were investigated using an entirely computerized X-ray diffractometer (PANalytical X'Pert PRO MRD PW3040). The diffraction patterns were recorded over a 2θ range of 5°–55° to identify both crystalline and amorphous phase within GPE samples. The source of X-ray is Cu-kα radiation with wavelength λ = 0.154 nm. The crystallite size (D) was estimated using the Debye–Scherrer equation [21]:

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad (1)$$

where K is the Scherrer constant (0.98), and β is the full width at half maximum (FWHM).

2.5 Electrochemical impedance spectroscopy

Electrochemical impedance spectroscopy (EIS) used to study ionic conductivity of GPE samples by evaluating the impedance response to a sinusoidal disturbance. The electrical properties of GPE were measured with impedance analyzer is HIOKI 3532-50 LCR HITESTER. The sample was conducted at a range of frequencies from 50 Hz to 100 MHz and was performed at room temperature (25°C). Ionic conductivity (σ) was calculated using the following equation [22]:

$$\sigma = \frac{t}{R_b A} \quad (2)$$

where, *t* is gel polymer electrolyte thickness (0.2 cm), R_b is the bulk resistance which can be obtained from the intersection of Nyquist plot with the real impedance axis and *A* is the electrolyte surface area (2.011 cm^2).

3.0 Results and discussion

3.1 Fourier Transform Infrared Spectroscopy

The FTIR spectroscopy was employed to study the complexation between the materials in the gel polymer electrolyte system. The FTIR spectra of the PVA-EC-PC-KI-TiO_2 GPE system as shown in Fig. 1 revealed critical insights into polymer-salt-filler interactions. The O-H stretching (3326–3241 cm^{-1}) exhibit a clear shift downwards from TD1 to TD5, indicating enhanced strong hydrogen bonding interactions between PVA's hydroxyl groups and KI-TiO_2. This shift confirms successful complexation of K^+ ions within the polymer matrix.

To show that incorporating nanofiller has been increased the amorphous phase in the GPE system, the interaction of the nanofiller with the polymer host and salt was further investigated. When the TiO_2 nanofillers were introduced, OH-anions of TiO_2 and the molecules of polymer matrix are drawn to one another, thus a hydrogen bond is created which implies to Lewis acid-base interaction [23]. These additional interactions within the polymer matrix can weaken hydrogen bonds in OH groups, lowering their vibrational frequency. In another words, the incorporation of TiO_2, disrupts the crystalline region by reducing the intermolecular interaction between polymer chains which facilitates easier movement of ions and subsequently can improve the ionic conductivity [23, 24]. The hydroxyl band peak being observed that it was shifted to lower band from 3324 cm^{-1} (TD1) to 3241 cm^{-1} (TD5).

The C-H stretching peaks (2936–2910 cm^{-1}) showed minor shifts, suggesting subtle rearrangements in the polymer matrix induced by salt dissolution. The C = O stretching (1777–1780 cm^{-1}), attributed to carbonate groups in EC-PC plasticizers, remains stable but with slight wavenumber variations, likely reflecting a weak coordination between K^+ ion hole maintaining their structural integrity. Meanwhile, the C = C stretching (1651–1653 cm^{-1}) is demonstrate the stability of the PVA backbone during these interactions such as hydration and dehydration processes. These C = C groups were present in the chemical structure of PVA due to residual acetate groups and terminal groups of polymer chains [25].

The consistent observation of these bands, even with improved spectral resolution through deconvolution, indicates that the fundamental structure of the polymer remains intact [25, 26]. It is also showed the S = O stretching (1416–1422 cm^{-1}), which may indicate residual DMSO or its interactions. DMSO contains an S = O double bond in its chemical structure, which contributes to its strong electron-donating or hydrogen-accepting capabilities, leading to significant attractive interactions with PVA [27]. The absence of major peak disappearances or new bands confirms no chemical degradation, emphasizing the system's suitability for electrolyte applications. The summary of the FTIR peaks for the GPE system is shown in Table 2.

Table 2
Assignments of each functional group in PVA-EC-PC-KI-TiO_2 GPE system.

Wavenumber (cm^{-1})						
Assignments	PVA-EC-PC-KI-TIO_2					References
	TD1	TD2	TD3	TD4	TD5	
O-H stretching	3326	3294	3282	3261	3241	[26–29]
C-H stretching	2936	2939	2928	2910	2928	[26–29]
C = O stretching	1777	1779	1776	1780	1779	[28–30]
C = C stretching	1653	1651	1652	1652	1652	[28–30]
S = O stretching	1418	1416	1419	1419	1422	[28–30]
C-O stretching	1009	1011	1015	1013	1013	[26–30]
C = C bending	949	950	950	949	947	[26–30]

3.2 X-ray diffraction

X-ray diffraction is a useful tool to determine the structural phase by observing the appearance and disappearance of crystalline or amorphous region. The XRD patterns of PVA-EC-PC-DMSO-KI-TiO_2 GPEs with different amounts of TiO_2 have been shown in Fig. 2. The pure TiO_2 (PT) peaks indicate that the TiO_2 is crystalline in nature with highest intensity at $2\theta \sim 25$. When TiO_2 nanofillers were introduced, the XRD patterns of GPEs from TD1 to TD5 showed a broad peak at $2\theta = 20$ which indicates that the electrolytes are in amorphous state. The amorphous nature implies that the GPE samples are semi-crystalline and therefore more flexible to assist ion transport [31]. It can be observed that TD5 has the highest intensity and broader peak compared to the other samples, which implies that the TD5 is highly amorphous. This also revealed that by increasing the TiO_2 content in GPE, may increase the amorphous phase in polymer matrix.

This is evidenced by the increase in the Full Width at Half Maximum (FWHM) values observed in the XRD patterns as the TiO_2 content increases. It can be observed that the value of FWHM decreases slightly

starting from 0.023 rad (TD1) to 0.022 rad (TD2), then the value of FWHM increases from 0.022 rad (TD3) to 0.089 rad (TD5). A broader FWHM indicates a greater degree of amorphousness in the GPE [32]. The enhanced amorphousness of the gel electrolyte facilitates better ionic mobility, which is beneficial for ionic conductivity. The FWHM values and crystallite size of the XRD patterns for PVA-EC-PC-DMSO KI-TiO_2 GPEs have been listed in Table 3.

Table 3
2θ, FWHM and crystallite size for gel polymer electrolytes contain different amount of TiO_2.

Sample	**2θ (°)**	**FWHM, β (rad)**	**Crystallite size, D (nm)**
Pure TiO_2 (PT)	25.22	0.023	6.252
TD1	20.07	0.022	6.533
TD2	21.69	0.026	5.439
TD3	21.36	0.028	5.101
TD4	21.72	0.051	2.791
TD5	21.38	0.088	1.606

The crystallite size shows a decrease trend with increase TiO_2.These overall peaks show that the crystalline nature of PVA decreases with TiO_2 addition. The semicrystalline nature of PVA has been shifting slightly towards amorphous nature with addition of TiO_2, due to the apparent fall of PVA peak height and increases in peak broadness. This might be happened due to decrease in intermolecular interaction between PVA chains and TiO_2 particles via hydroxyl bond thereby reduces the crystalline nature of PVA [33]. Overall, XRD results clearly indicates that the addition of TiO_2 nanofillers revealed to be plays a crucial role in tuning the structural morphology of the GPEs, which beneficial for application in next-generation energy conversion applications.

3.3 Electrochemical impedance spectroscopy

The R_b of the electrolyte was determined by extrapolating the intercept of the Nyquist plot on the horizontal axis as shown in Fig. 3. These plots exhibit an inclined spike at the low frequency region without a semicircle in high frequency region. The non-existent presence of semicircles at high frequencies suggests ions are the main charge carriers, indicating that conductivity primarily results from ion conduction [34]. The addition of 2 wt.% TiO_2 (TD2) enhanced the R_b value, implying that a small amount of filler initially limits ion mobility, likely due to insufficient interaction between the filler surface and polymer matrix. Then R_b dropping to 9.59 Ω at 4 wt.% (TD3), indicates the improvement in ionic transport. TiO_2 content of TD3 in polymer matrix disrupts polymer crystallinity and enhances the amorphous phase, allowing better ions mobility within the electrolyte [35]. Further increase to 6 wt.% TiO_2 (TD4), the R_b value remains relatively constant, demonstrating that the nanofiller continues to aid in

maintaining ion conduction pathways. However, when the TiO_2 concentration reached at 8 wt.% (TD5), the R_b increases again due to agglomeration of TiO_2 nanofillers at higher loading, which limits the effective conduction pathways and restricts ion mobility [36].

Figure 4 shows the effect of TiO_2 concentration on the conductivity. Initially, the GPE system without the TiO_2 had been observed to exhibit the highest ionic conductivity, then when an amount of 2 wt.% of TiO_2 was added, the ionic conductivity being reduced. This reduction can be attributed to the interaction between the TiO_2 nanofiller in polymer matrix which lower content of nanofiller was still insufficient to significantly modify the polymer structure, leading to restricted ion mobility which consequently lower the ionic conductivity [37]. As the TiO_2 concentration content had been further increased, it was observed that the ionic conductivity also increased. The highest ionic conductivity was reached at 4 wt.% of TiO_2 at (10.4 ± 0.5) mS cm^{-1}. This enhancement can be attributed to improved polymer-filler interactions, which disturb the ordered regions of the polymer matrix and increase the amorphous phase of the electrolyte. The Lewis acid characteristic and large surface area of TiO_2 significantly enhanced ion mobility by facilitating the transportation of more conducting pathways [38]. Further TiO_2 content addition, which are TD4 and TD5 resulted in conductivity reductions ascribed to excessive filler inducing agglomeration. Therefore, the rise in ionic conductivity at 4 wt.% TiO_2 demonstrated the optimal beneficial effect of nanofiller dispersion on GPE system where it enhances amorphous content and polymer flexibility.

A similar observation compared to previous study by Moorti et al. [39] who also incorporated TiO_2 nanofiller as additive in PVA-ammonium thiocyanate (NH_4SCN)-based GPE obtained highest conductivity of 4.52 mS cm^{-1} at 3 wt.% of TiO_2 content. The authors demonstrated the addition of nanofiller in polymer electrolyte was beneficial in improvement of ionic conductivity due to their ability to reduce the degree of crystallinity of polymer for ion mobility. In another work by Yap et al. [40], they also study the TiO_2 nanofiller effect on PVA-lithium trifluoromethanesulfonate (LiTf) GPE system and found to have maximum conductivity of 0.108 mS cm^{-1} with addition of 10 wt.% of TiO_2. Study by Rahim et al. [41] reported to also achieve optimum ionic conductivity of 0.33 mS cm^{-1} when 8 wt.% of TiO_2 added to PVA-cellulose acetate (CA) blend GPE system. Based on previous studies, the present PVA-based GPE system was achieved higher ionic conductivity value which proved the TiO_2 nanofiller may further improve ionic conductivity for next-generation energy conversion applications.

4.0 Conclusion

PVA-based gel polymer electrolytes incorporating TiO_2 nanofiller have been successfully prepared and characterized. This research has comprehensively analyzed the structural and electrical properties of the PVA-EC-PC-KI system with addition of TiO_2 nanofillers. The FTIR analysis strongly confirmed the interaction between the components, where shifts in the O-H signified enhanced complexation and an increase in the amorphous content of the polymer matrix. The XRD studies supported these findings,

revealing a decrease in crystallite size and enhanced amorphous nature with the addition of TiO_2. The impedance studies discovered that the R_b decreased with increasing TiO_2 concentration, leading to an optimum ionic R_b conductivity of (104.0 ± 1.0) × 10^{-4} S cm^{-1} at 4 wt.% TiO_2. Overall results obtained revealed that incorporating TiO_2 nanofiller into the PVA-based GPE system was shown to support and enhance the gel electrolyte's conductivity by disrupting polymer crystallinity and providing additional ion transport pathways. Thus, the proposed GPE system with a TiO_2 nanofiller in the present study has shown an excellent potential to be employed as an efficient and stable electrolyte material for next-generation energy conversion development.

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The authors declare that they have no competing interests.

Consent to Publish declaration

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Ethics declaration

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Author contribution declaration

A. A., P. Q. B., and C. G. wrote the main manuscript text. A. A., P. Q. B., and C. G. analyzed the EIS and XRD data. M. F. and M. H. provided the FTIR data and performed the corresponding analysis. M. F. also edited and paraphrased the manuscript. All authors reviewed the manuscript.

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Figures

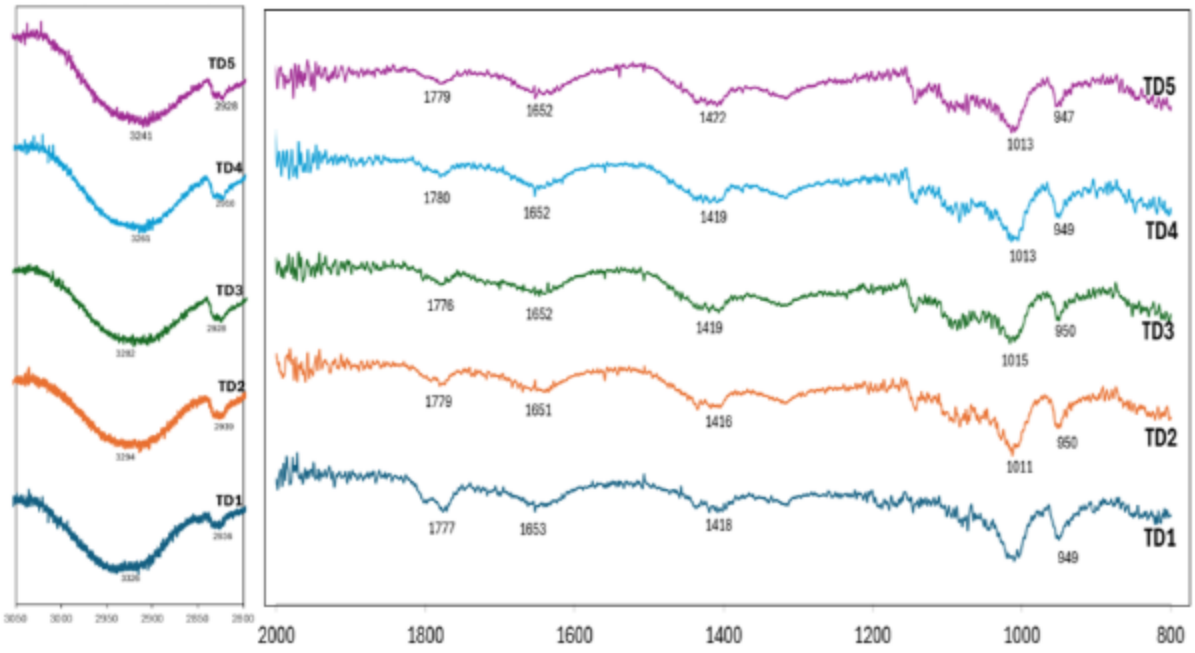


Figure 1

FTIR spectra of GPE samples.

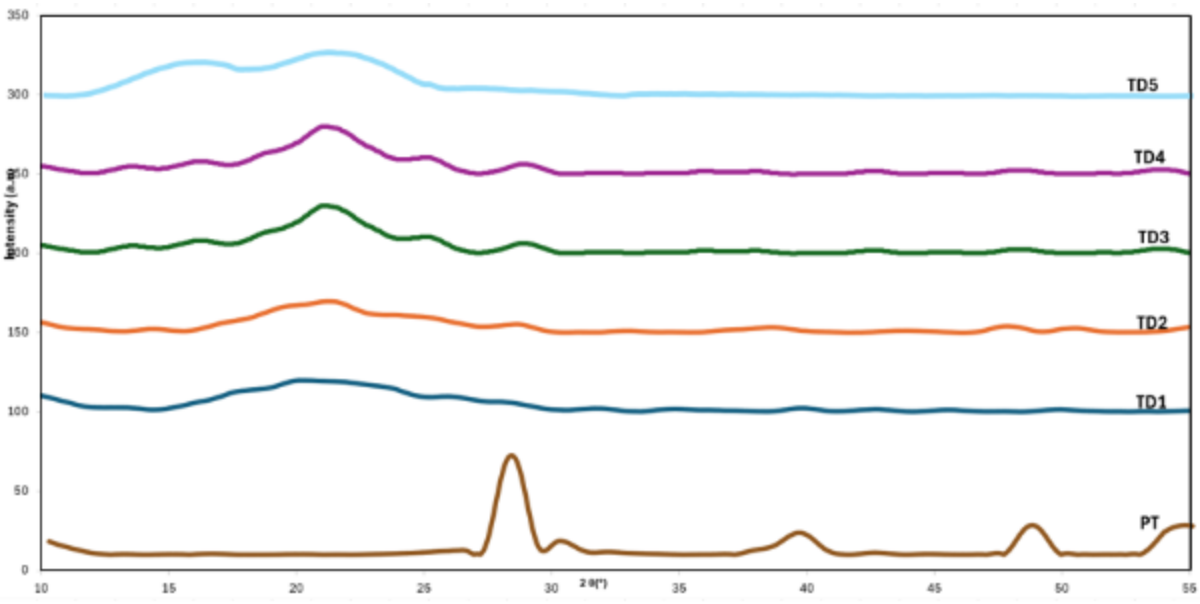


Figure 2

The XRD pattern for PVA-EC-PC-DMSO-KI- TiO_2.

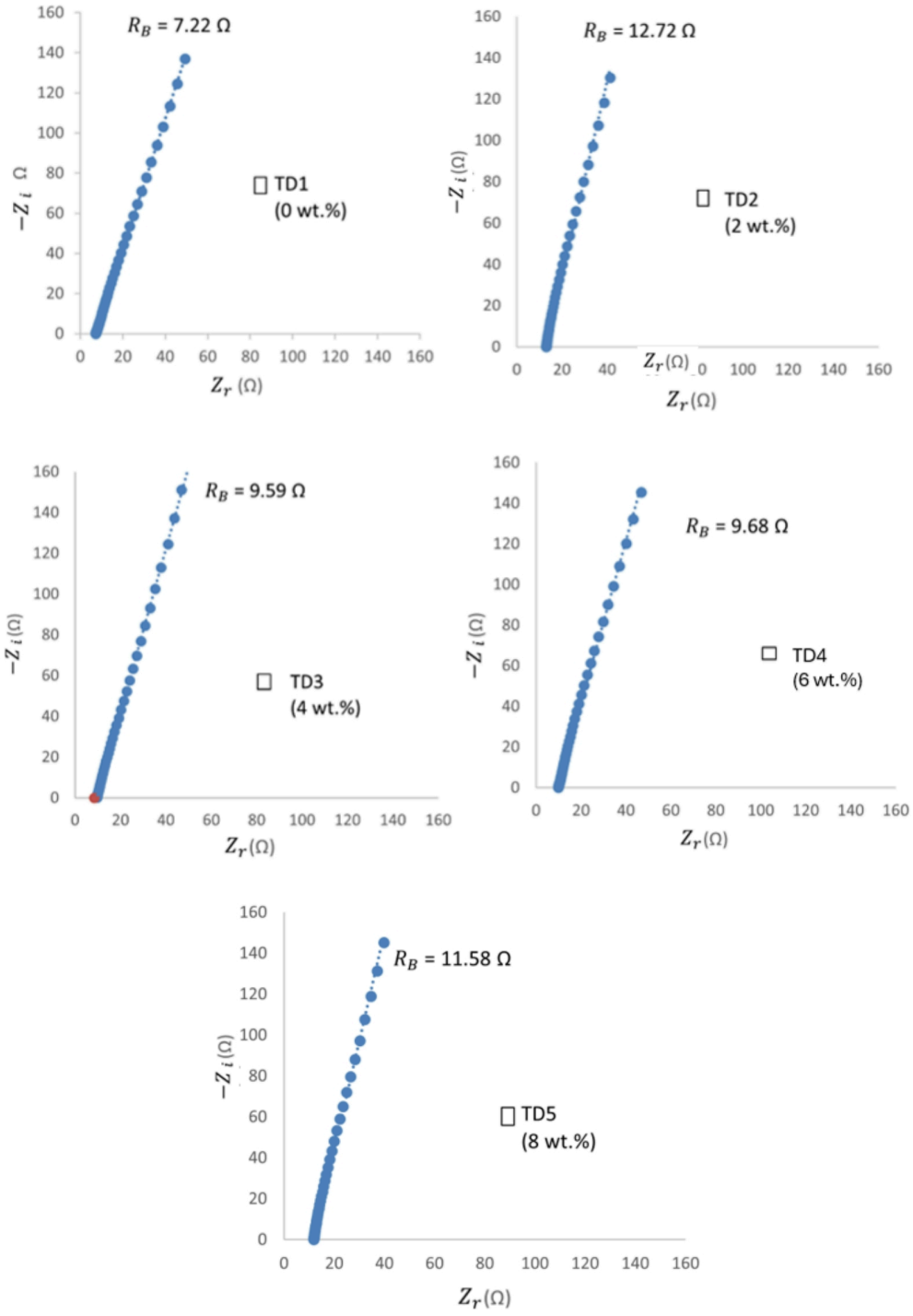


Figure 3

Nyquist plots for PVA-EC-PC-KI-TiO_2 GPE system.

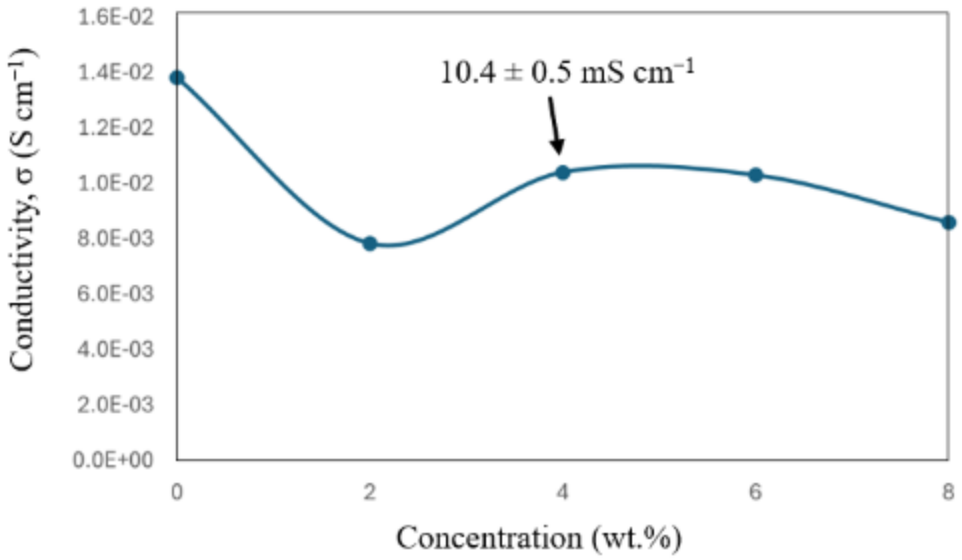


Figure 4

Effect of TiO_2 concentration on conductivity.