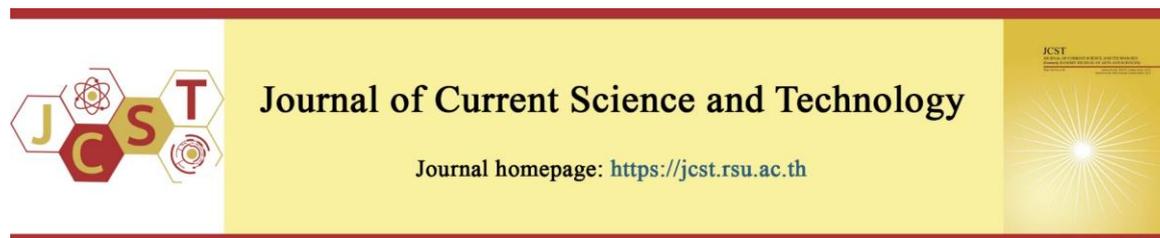


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Ionic transport study of PVDF-PEO/ NaNO₃ based solid electrolyte

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Abstract

The invention of solid electrolytes appears to be cutting-edge technology for the battery system as they conquer the majority of the issues associated with liquid electrolytes. However, the most significant constraints for solid electrolytes are poor ionic conductivity and inadequate power density. In the current study, an initiative is being taken to enhance the ionic conductivity of solid electrolytes. In this view, several samples of solid electrolytes are prepared by differing the proportion of PVDF and PEO; also, NaNO₃ is utilized as a conductivity booster in various ratios. Due to numerous advantages, the cast solution technique is opted to prepare all the examined electrolytes. Further, the homogeneity of all the substances existing in the electrolytes is observed with XRD. Also, all the ready samples' ionic conductivity and dielectric constant are tested to analyze their performance in lithium-ion batteries. A homogeneous bonding between PVDF and PEO is observed in the electrolyte matrix, but the presence of NaNO₃ is not observed thoroughly. The ionic conductivity of all samples looks stable and increases with the concentration of NaNO₃ in the compound matrix. Therefore, it is being concluded that using NaNO₃ in the PVDF-PEO-based polymer electrolytes has favorably impacted conductivity and stability. The current research is a reasonable attempt to improve the ionic conductivity of PVDF-PEO-based electrolytes with one-of-a-kind proportions.

Keywords: ionic conductivity; NaNO₃; PVDF-PEO; solid polymer electrolytes.

1. Introduction

The petroleum-based oil prices have reached an all-time high in India, which is the primary energy source in the present scenario, also the combustion of fossil fuels releases hazardous gases (Kumar & Kumar, 2016, 2018). Therefore, it is the right time to accelerate the research in the field of alternative sources of energy for energy and environmental sustainability (Kumar, 2020; Kumar, & Ramprasad Mangishetti, 2020). One of the best renewable energy sources is solar energy, available in abundance worldwide. But, the major problem with solar power is its duration of availability (Owusu & Asumadu-Sarkodie, 2016) because solar energy is available during the daytime only.

Furthermore, the intensity of energy during the day duration also varies periodically with the geometric location of the place. Therefore, energy storage becomes crucial and essential to utilize solar energy when solar energy is

unavailable (Shaqui, Sopian, & Al-Hinai, 2020). In such a condition, the storage process and storage device play a vital role in using stored energy more effectively and efficiently. In this regard, the batteries are the first preference for the users to store electrical power because of their compatibility, easy operation, compact size, and economical (Sharma, Panwar, & Tripathi, 2020).

Although the batteries have carried many benefits over the other storage devices, they are still far from the most optimized alternative to energy-producing machines such as IC engines. The batteries have a higher weight-to-energy ratio, limited operating condition parameter ranges, and are dangerous and less efficient when operating at higher temperatures (Duan et al., 2019; May, Davidson, & Monahov, 2018). Most of these problems are directly or indirectly associated with electrolytes and electrodes of the batteries (Pender et al., 2020). After the invention of lithium-ion

batteries, the problems related to the electrodes are almost addressed, but the electrolytes still have enough chances of modification. In the recent past, much work has been done on electrolytes. Researchers introduce solid polymer-based electrolytes that are more stable and safe than liquid ones (Castillo et al., 2021). Also, liquid electrolytes form dendrites near the electrodes, which are very hard and brittle and cause leakage that leads to accidents such as explosions in the battery (Zhang, Li, Yang, & Chen., 2021). The solid polymer electrolytes provide safety to the batteries and add extra strength (Chen et al., 2021). Additionally, solid electrolytes offer a wide range of electrochemical windows along with more substantial chemical stability when compared with liquid electrolytes (Gancarz, Zorębski, & Dzida, 2021).

Further, the improvement in the interaction between electrodes and electrolytes is also noticed with solid polymer electrolytes (Meng, Zhu, & Lian, 2022). However, the solid electrolytes are limited to lower ionic conductivity than liquid electrolytes, which decreases the efficiency of the batteries (Talip, Yahya, & Bustam, 2020; Xu, Deng, Guo, Zhou, & Yao, 2021). To improve the ionic conductivity of the solid electrolyte, the researchers utilized several techniques, including blending of different polymers and salts, doping of highly conductive salt, doping of organic or nonorganic nanoparticles, etc. (Caradant et al., 2020; Liang et al., 2021; Zhang & Huo, 2021). The mixing of different polymers helps improve the ionic conductivity and supports achieving the desired range of other properties such as mechanical strength and interface compatibilities (Wang, Shen, Deng, Cui, & Yao, 2021). Further, the ionic conductivity of the battery is also modified by alteration in the separators; in general, solid-state electrolytes and inorganic filler electrolytes separators are used (Barbosa et al., 2021; Yu & Manthiram, 2021). The inorganic fillers are not directly involved in conductivity enhancement like organic electrolytes (Feng et al., 2021; Zhao, Yi, He, & Zhou, 2019). Another crucial aspect of batteries is donor number. A higher donor number and Lithium-ion Lewis acidic cation enhance the solubility of lithium polysulfide. As a result, the redox sulfur species concentration is increased (Suriyakumar, Kathiresan, & Stephan, 2019). Wang et al. (2022) researched to identify the suitable solid polymer thin electrolyte for a higher range of

operational temperature. The authors synthesized the PVDF@PEG solid polymer electrolyte via the thermo-curing method; the ionic conductivity of the prepared sample was 8.03×10^{-5} S/cm at room temperature. The authors also noticed an excellent electrochemical stability window of 5.2V for the same sample. Many researchers try several different polymers, salts, and nanoparticles to get the desired properties and results. Wu et al. (2009) investigated the ionic conductivity of PVDF-HFP-based polymer and lithium salts. They observed that the ionic conductivity reached 1.64×10^{-4} S/cm near the room temperature and 1.75×10^{-3} S/cm at a higher operating temperature of 80 °C. The authors also noticed that the presence of PHEMO in the polymer matrix helps improve the thermal stability of the solid polymer electrolyte. T

Gulino et al. (2021) used $\text{LiBH}_4\text{-MgO}$ -based electrolyte to investigate the ionic conductivity of solid lithium-ion batteries. The highest conductivity of 2.86×10^{-4} S/cm is observed for the 53% MgO concentrated sample. According to the authors, the main reason for this is the filling of MgO pores with LiBH_4 . Lue et al. (2015) explored the effect of TiO_2 nanoparticles on the enhancement of electrical conductance. The authors identified that the presence of TiO_2 nanoparticles improves ionic conductivity and increases the mechanical strength of the solid polymer electrolyte. Very similar findings are quoted by Ponam and Singh (2021). Feng et al. (2021) evaluated two different mechanisms to enhance the ionic conductivity of PEO-based electrolytes through a polymer filler interface. The authors identified that the active fillers are more effective in enhancing ceramic additives' conductivity, as these provide extra express channels to Li^+ . Ponam and Singh (2022) also investigated the effect of different polymers in the matrix on the various properties of the solid polymer electrolyte. The authors identified that the polymers such as PVDF and PEO have their signatures on different properties; therefore, the proportion of polymer can be changed to obtain the desired property. Xia, Wu, Zhang, Cui, & Liu (2019) evaluated the future prospective of solid electrolyte-based Li^+ batteries. According to the article published, the biggest challenge for the researchers is to produce affordable electrolytes with competitive interfacial impedance, better ionic conductivity, higher mechanical strength, and compatibility with most of the electrodes.

Many nanofillers were identified and experimented by the researchers. They showed a significant improvement in the ionic conductivity of the electrolyte by a certain degree. Some of them are cited in the current section. However, a few very rare fillers are also available, which have a better impact on several properties of electrolytes, including ionic conductivity, and one of these fillers is sodium nitrate (NaNO_3). NaNO_3 is a chemical compound that comes from the alkali metal salt family, and this is a very rarely available source of nitrate anion (NO_3^-) Ward et al. (2018). The nitrate anion bonds with hydrogen, which help the free electrons pass through the structure. The NaNO_3 is soluble in most liquid compounds, but the concentration varies with every liquid (Reynolds, 2018). The NaNO_3 splits into Na^+ and NO_3^- ions in the solution and is trapped in the same polymer-based electrolytes. The presence of ions helps the flow of charge through the electrolytes, and as a result, the ionic conductivity increases. The ionic conductivity depends upon the concentration of NaNO_3 , so the current conductivity of the electrolyte increases with increasing the percentage of NaNO_3 . Rao, Reddy, & Chary (2005) investigated that the ionic conductivity of an electrolyte can be increased by two degrees for direct current by doping the NaNO_3 into the solution. A similar trend in the improvement of ionic conductivity for LiNaSO_4 -based electrolytes was observed by Slavkova et al. (2018) in their results. Shetty et al. (2021) conducted an ion transport study for NaCMC-based solid electrolyte film doped with NaNO_3 salt. The authors investigated that the increasing concentration of NaNO_3 improves the ionic conductivity of the solid electrolyte and enhances the glass temperature. The authors studied the crystalline nature of the NaNO_3 by analyzing the XRD patterns of the solid electrolyte samples.

In the present research work, an effort is put into finding the solution for a few mentioned problems. Subsequently, a few samples of solid electrolytes are prepared by blending various polymers, salts, and different materials. In this research, the impact of changing the proportion of PEO and PVDF polymers on the ionic conductivity of the solid polymer-based electrolytes is measured. Also, the effect of the presence of NaNO_3 in the electrolyte matrix is analyzed. The results obtained are satisfactory compared with those obtained from other polymers, salt, and nanoparticles

combinations. The presence of NaNO_3 in the matrix enhances the electric conductivity and thermal stability, whereas the changing composition of polymers shows a noticeable impact on the mechanical properties of the electrolyte. The polymer used in this work has various advantages and disadvantages. Therefore, the percentage of each polymer is selected so that the benefits of that polymer dominate in the electrolyte. The detailed results and finds are presented in other sections.

2. Objectives

The prime objective of the present study is to prepare thermally stable solid electrolytes for lithium-ion batteries and test them for their ionic conductivity.

3. Materials and Methods

3.1 Materials

The chemicals and salts used in the present work to prepare the electrolyte are purchased from Sigma-Aldrich, USA. The molecular weight of the PEO used in the current research is 900,000 g/mol, and the PVDF, which is the second component for electrolyte production, has a molecular weight of 534,000 g/mol. The NaNO_3 , dimethylformamide (DMF), and other required chemicals are purchased from the local chemical supplier of the institute.

3.2 Synthesis of Polymer Electrolytes

In the present work, four electrolytes are prepared with different proportions of polymers and NaNO_3 . The PEO proportions selected are 10% and 20% with PVDF, and the concentration of NaNO_3 kept is 5% and 10% in the PEO-PVDF polymers. First, the mixture of PEO and PVDF is prepared by taking in 10% PEO-90% PVDF and 20% PEO-80% PVDF. The solid polymer electrolytes are prepared through the cast solution method. At first, the defined mixture of PEO-PVDF is dissolved in a specified volume of DMF through a magnetic stirrer by keeping the speed of agitation and reaction temperature constant at 350 rpm and 50°C. The solution is put for stirring untouched for at least 2 hours. Once the homogeneous solution appears; the desired quantity of NaNO_3 is added into the solution and put for stirring again at the same speed and temperature for another couple of hours. After that, heating is stopped but the agitation is continued for another 30 hours or until the uniform solution is appeared. After obtaining the homogeneous mixture, the solution is put for vacuum drying in a

petri dish. The sample is kept in the vacuum for 60 hours at a constant temperature of 40°C. The same process is repeated to prepare all the other samples. All the electrolytes are cut into desired shape and size and kept in a silicon environment and airtight

container. The pieces are tested after two weeks of production. The whole procedure of preparing solid polymer electrolytes is illustrated in figure 1, and the terminology of all the tested samples is given in table 1.

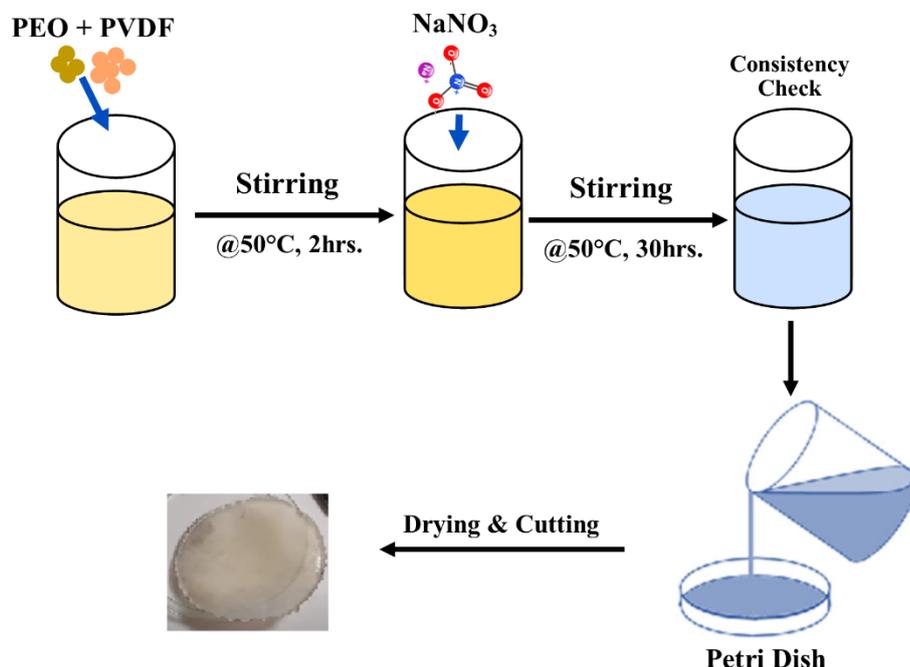


Figure 1 Electrolyte preparation procedure

Table 1 Terminology of prepared electrolytes

Representation	Nomenclature
BP1	90% PVDF + 10% PEO
BP2	80% PVDF + 20% PEO
0.95BP1-0.05SN	95% BP1 + 5% NaNO ₃
0.9BP1-0.1SN	90% BP1 + 10% NaNO ₃
0.95BP2-0.05SN	95% BP2 + 5% NaNO ₃
0.9BP2-0.1SN	90% BP2 + 10% NaNO ₃

3.3 Characterization of Prepared Electrolytes

The methods opted to measure the various properties are similar to our previous research. Different properties, including surface patterns and ionic conductivity at various operating temperatures and frequencies, are measured for all the prepared electrolytes. An X-ray diffractometer (XRD) determines the surface structures of electrolytes, and the measuring range selected in the current test is 10° to 70°. All the major compounds showed their presence in the matrix, which XRD observes with the intensity. The XRD patterns are

observed by using a Philips X-ray diffractometer with Cu α 1 radiation ($\lambda = 1.5405 \text{ \AA}$), the detector position is recorded in a wide range of angle 2θ (the range of angle is; $10 \leq 2\theta \leq 70$) and it has a scanning rate of $3^\circ/\text{min}$. Whereas the ionic conductivity of the prepared samples is measured with an AC impedance spectroscopy analyzer made by Autolab. All these experiments are conducted in a nitrogen environment, and the system is closed to the surrounding. A sandwich of prepared sample and two electrodes are made to check the ionic conductivity; the electrodes used in the present experiments are made up of stainless steel and have constant ionic conductivity concerning the temperature of operation. The current supplied to the experiment has varying frequencies, and the system is capable of altering the current frequency in the range of 10 Hz to 1 MHz. The ionic conductivity (σ) of all the prepared samples is calculated by using the mentioned formula:

$$\sigma = \frac{b}{R_b A}$$

The above formula; b represents the sample's thickness, A represents the tested sample's cross-sectional area, and the bulk resistance is represented by the term R_b .

The trends of dissimilarity in the conductivity against the current between the temperature range of 298 K to 333 K are very much similar to Wang et al. (Wang et al., 2020).

4. Results and Discussions

The X-ray diffraction pattern of all the tested samples (i.e. NaNO_3 , PVDF, PEO, 0.95BP1-0.05SN, 0.9BP1-0.1SN, 0.95BP2-0.05SN, and 0.9BP2-0.1SN) are illustrated in figure 2. The appearances peak of pure NaNO_3 , PEO, and PVDF is noticed at 28.4° , 18.3° , and 19.7° , respectively. However, PEO and PVDF have two peaks; the other peak for PEO appears at 22.7° , whereas the second peak of PVDF appears at 39.1° . On the other side,

the peak of PEO in the blended polymer electrolytes does not appear significantly because the OH group present in the PEO is neutralized by the PVDF. However, a small peak in the presence of PEO in the blended polymer can be observed in figure 2. Moreover, the crystallinity of the prepared samples increases with an increase in the proportion of PEO in the matrix. Similar findings were also noticed by (Wang et al., 2020).

Also, the intense peak of NaNO_3 is not witnessed even in a single prepared solid blended sample, which represents a homogeneous mixture with no extra salt present in the blended polymer electrolytes. All the prepared electrolytes look very stable in the X-ray diffraction patterns. This is witnessed by the compatibility of additives with the parents' organic polymers. Patel & Kumar (2019) noticed similar types of shapes (Nidhi et al., 2019) in their published research.

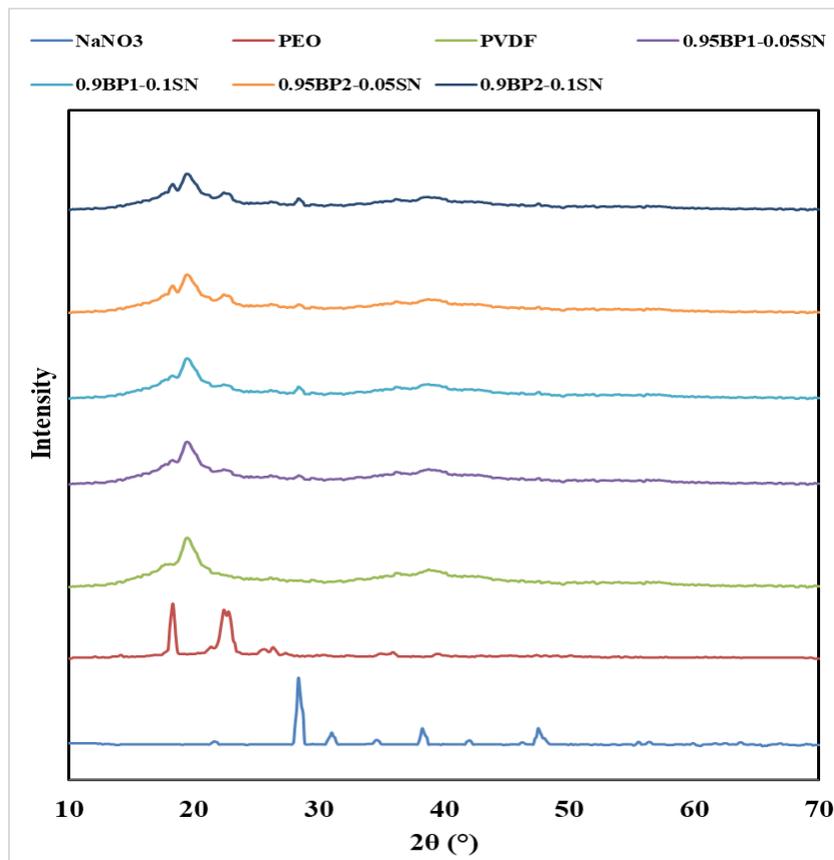


Figure 2 XRD patterns of all tested samples

The ionic conductivity of all blended electrolytes (0.95BP1-0.05SN, 0.9BP1-0.1SN,

0.95BP2-0.05SN, and 0.9BP2-0.1SN) is calculated and represent in the figure 3. During the analysis of

ionic conductivity data, it is observed that the ionic conductivity of the polymer electrolyte increases with surge with the proportion of the PEO polymer in the blend. The stress-strain curves of PVDF, PEO, BP1, and BP2 are shown in figure 4. A very similar trend was observed in my previous research (Ponam & Singh, 2021). The higher conductivity and better PEO homogeneity are the key factors behind this. However, the mechanical strength of the electrolyte decreases with a higher proportion of PEO as it has poor mechanical properties (Ponam & Singh, 2021). Moreover, the addition of NaNO₃ salt improves the prepared electrolytes' ionic conductivity by improving the polymer matrix's chemical stability and suppressing the semi crystalline peaks (Vahini & Muthuvinayagam,

2019). Therefore, the uppermost conductivity for the current of 1.45×10^{-4} S/cm is observed for 0.9BP2-0.1SN at room temperature (298K), which was higher than the ionic conductivity of 9.5×10^{-5} S/cm get, by Wang et al. (2020) for the same combination of base polymers. The ionic conductivity of all the polymer electrolytes increases with an increase in the operating temperature, but the pattern followed by all the electrolytes is very similar (Chai, Aung, Noor, Lim, & Abdullah, 2022). Therefore, the curves representing the variation in ionic conductivity w.r.t. $1000/T$ remain almost parallel throughout the testing range. The key factor behind it is a reduction of the formation rate of ion-pair that is strengthened by ion aggregation (Ratner & Shriver, 1988).

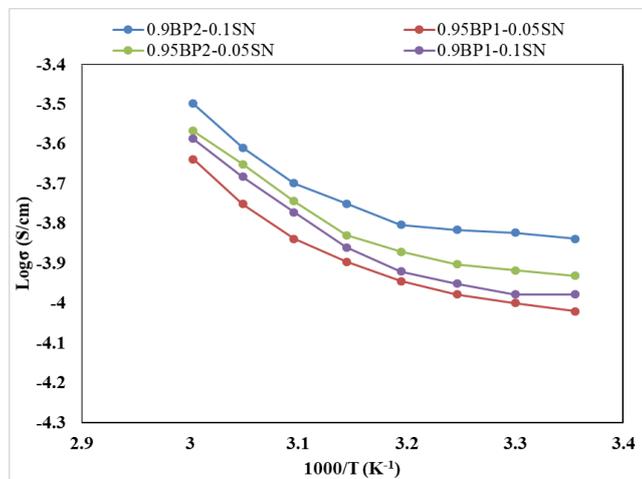


Figure 3 Ionic conductivity of all prepared electrolytes

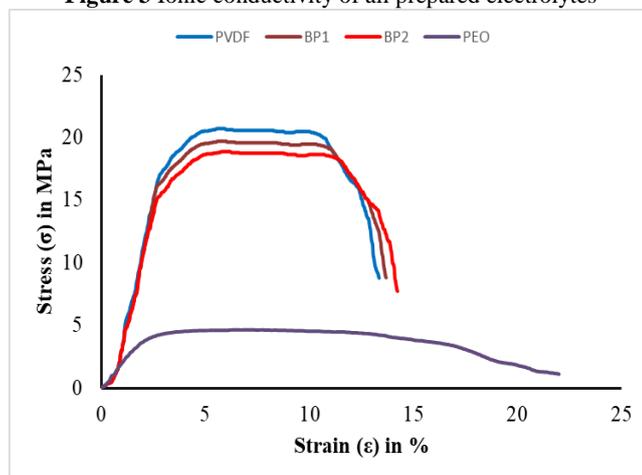


Figure 4 Stress-Strain Curve

The effect of frequency on the ionic AC conductivity for all the prepared electrolytes at room temperature is illustrated in figure 5. The frequency of the current selected in the recent

research lies in the range from 10^3 Hz to 10^7 Hz. It can be observed clearly from the figure that the ionic conductivity of all the solid polymer electrolytes increases with an increase in frequency. However, at a higher frequency, the rate of increasing ionic conductivity varies slightly, and as a result, some unpredicted patterns can be observed. The doping procedure and structural disorder are the two major factors on which the conductivity of the solid electrolytes is mainly dependent (Wani & Rafiuddin, 2018). Furthermore, similar to the ionic conductivity w.r.t. temperature, the 0.9BP2-0.1SN polymer electrolyte showed the highest ionic conductivity for most of the frequency range. The

variation in the conductivity of the electrolyte sample concerning frequency is determined by Jonscher's AC power-law, especially for the higher frequency range (Manani, Jethva, & Joshi, 2020). Jonscher's AC power law is given as:

$$\sigma'(\omega) = \sigma_0 + Z\omega^x$$

In this equation, $\sigma'(\omega)$ indicates conductivity that depends upon the frequency, σ_0 is the conductivity independent of the frequency, x is the frequency exponent, and Z is the constant.

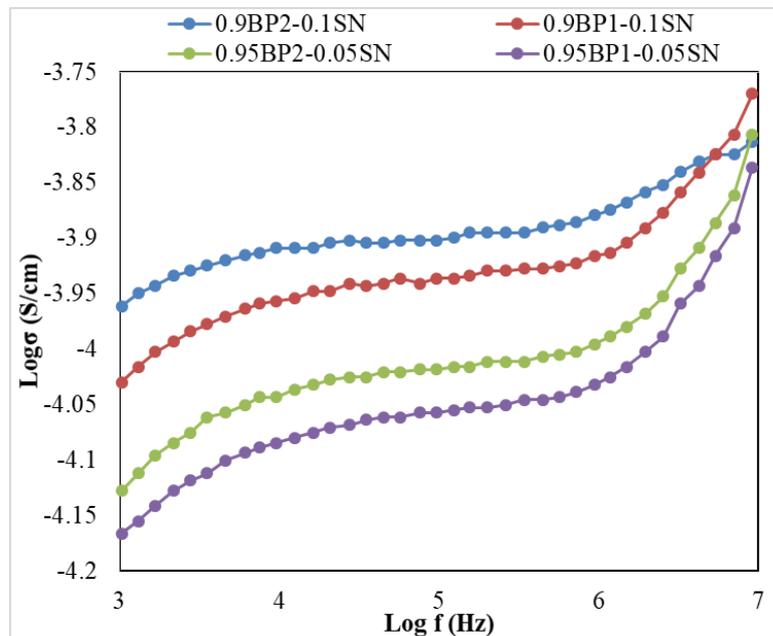


Figure 5 Ionic conductivity w.r.t. frequency

To understand the impact of salt on the ionic conductivity of the solid electrolyte, it is mandatory to study the dielectric constant concerning frequency (Sengwa & Dhatwarwal, 2020). The dielectric constant is a very complex property of the solid polymer electrolyte. The dielectric permittivity of all tested samples of the solid electrolytes is measured for the current frequency and is represented in figure 6. The experimental results illustrated in figure 5 are conducted at room temperature (298K). The ϵ' for the 0.9BP2-0.1SN is the highest among other tested samples at 10^2 Hz frequency, very similar patterns were also observed by Antonio Santoyo-Ramón, Casilari, and Cano-García (2022). In simple words,

the ϵ' value at the initial frequency is enormous compared to other electrolytes. The ϵ' value decreases sharply for all the solid polymer electrolytes between the 10^2 to 10^3 Hz frequency range and becomes a straight line at a higher frequency range. The dielectric permittivity, which depends upon frequency, is calculated with the given formula:

$$\begin{aligned} \epsilon^* &= \epsilon'(\omega) + k\epsilon''(\omega) \\ \epsilon' &= \frac{-Z''}{\omega C_0(Z'^2 + Z''^2)} \\ \epsilon'' &= \frac{Z'}{\omega C_0(Z'^2 + Z''^2)} \end{aligned}$$

Here Σ'' and Σ' represent the imaginary and fundamental factors of the dielectric permittivity and can be obtained by:

$$\varepsilon' = \varepsilon_{\infty} + \frac{\Delta\varepsilon (1 + \chi^{\alpha} \cos \cos \frac{\alpha\pi}{2})}{1 + 2\chi^{\alpha} \cos \cos \frac{\alpha\pi}{2} + \chi^{2\alpha}}$$

$$\varepsilon'' = \Delta\varepsilon \frac{\chi^{\alpha} \sin \sin \frac{\alpha\pi}{2}}{1 + 2\chi^{\alpha} \cos \cos \frac{\alpha\pi}{2} + \chi^{2\alpha}}$$

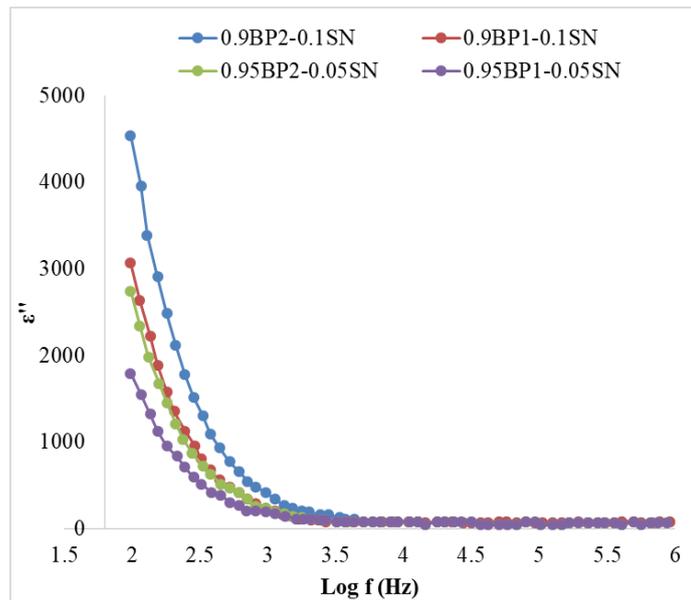


Figure 6 Dielectric constant w.r.t. frequency

Enhancing the concentration of PEO and salt in the polymer matrix increases the dielectric constant of the solid electrolyte. The main factor for higher dielectric constant value at the lower value of frequency is electrode polarization events, which happen due to the complete dissolution of salt with polymers (Gondaliya, Kanchan, Sharma, & Jayswal, 2012; Isaac, Mangani, Devaux, & Bouchet, 2022). At this frequency, the ionic pairs got adequate time to remain in a stable state that binds longer-range motion, and as a result, the dielectric constant increases (Arya & Sharma, 2018).

6. Conclusion

The current work includes the preparation of solid electrolytes with the blended polymer of PVDF and PEO, keeping their proportion 90:10 and 80:20. To enhance the conductivity and current stability, NaNO_3 is doped into the electrolyte in 5% and 10% to the total weight of polymer and salt. The number of properties is tested to check the compatibility of NaNO_3 salt with the polymer and

the ionic conductivity of the prepared solid polymer electrolytes. The key finding noticed during the analysis of properties of tested electrolytes are as follows:

- The X-ray diffraction patterns indicate both the polymers' homogeneity and good compatibility of NaNO_3 salt with the parent polymers. No sharp peaks of any lower percentage polymer and salt appear in the pattern.
- The increase in the proportion of PEO in the electrolyte increases the ionic conductivity of the solid electrolyte. Moreover, the addition of NaNO_3 salt in the matrix also enhances the ionic conductivity of the electrolytes.
- The ionic conductivity and the dielectric permittivity of 0.9BP2-0.1SN are the highest among all the electrolyte samples for all the AC current frequencies.

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