Improved mechanical and electrochemical properties of PVDF/PEO/LiClO$_4$ based solid polymer electrolyte by using TiO$_2$ and MgO nanoparticles

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ABSTRACT

The poly (vinylidene fluoride) PVDF and poly (ethylene oxide) PEO-based solid polymer electrolytes are gaining popularity due to their good electrochemical and mechanical characteristics. In the present work the polymer electrolyte is prepared by taking 80% PVDF, 20% PEO, and 20% LiClO$_4$ through rigorous mixing. Further, the properties of PVDF/PEO/LiClO$_4$ based electrolyte are improved by adding TiO$_2$ and MgO nanoparticles in different proportions. The results show that the addition of TiO$_2$ and MgO nanoparticles has a significant effect on the electrochemical characteristics and mechanical properties of the electrolyte. The maximum current onset potential of 4.97 V is observed with a 4% concentration of TiO$_2$ nanoparticles. Also, the mechanical properties such as ultimate stress and failure stress are increased with the addition of nanoparticles. The ionic conductivity of the electrolyte first increases with an increase in the concentration of nanoparticles but it starts decreasing after 2% concentration.

Key words: TiO$_2$;MgO; Solid Polymer Electrolyte; PVDF/PEO; Nanoparticles.

INTRODUCTION

Risersthe invention of the Lithium-ion battery brings a revolution in energy storage techniques. The lithium-ion batteries have two main components i.e. cathode and anode make up of good conductive materials (lithium) and electrolyte have good conductivity (Subba Reddy et al., 2007). Oneseparator is also used to transfer the ions. The high demand for

lithium-ion batteries is because of their greater energy concentration, low self-discharge rate, higher operational power, and longer life. The electrolytes are the most important part of lithium-ion batteries that decides all the mentioned characteristics of the battery. Liquid electrolytes are mostly used in almost all types of batteries which are very economical and good conductors to the electrons and ions. During the operation, dendrites are formed at the metallic electrodes that can pierce the strainer of the battery and might cause short-circuit (Hangchao Wang et al., 2020). The same cause might leads to an explosion in batteries several times. Because of this, the use of liquid electrolyte batteries was very rare, especially in heavy-duty applications (Liang, Han, Wang, Lan, & Mao, 2018). However, in recent years, the patterns have shifted dramatically, with the creation and effective deployment of solid polymer electrolytes in lithium-ion batteries being the primary drivers. Polymer electrolytes improve the safety and response speed of lithium-ion batteries, paving the way for renewable energy to succeed. solar energy, wind energy, fuel cells, and other renewable energy sources (Yu & Manthiram, 2021). All these renewable energy sources and technologies are very much needed for the self-sustainability of developing countries such as India in terms of energy requirements. Presently, most of the energy demand is completed by fossil fuels which are present in a very limited amount (Parvesh Kumar & Kumar, 2016a, 2018, 2019). Further, the emissions of these fossil fuels release hazardous gases that cause air pollution and results in global warming and various air-borne diseases (Gautam, Kumar, Pali, & Kumar, 2016; P Kumar & Kumar, 2018; Parvesh Kumar & Kumar, 2016b).

All these problems can be sorted out with the use of cleaner and renewable energy sources which are heavily dependent upon energy storage with the use of cleaner and renewable energy sources which are heavily dependent upon energy storage systems. The highly valuable advantages of solid polymer electrolytes for energy storage system works as a torch in the dark path of the storage technology (Yao et al., 2019). One of the most effective

solutions to the problem of liquid electrolytes is the use of solid electrolytes. Solid electrolytes not only address the safety issue, but they also give the battery more strength by increasing its energy density. Solid electrolytes are not only inflammable, but they also inhibit the production of lithium dendrites. Solid electrolytes also have excellent mechanical, thermal, and electrical properties. Solid polymer electrolytes, on the other hand, have yet to be fully explored since they have some limitations that researchers must overcome (Arya & Sharma, 2018; Rao, Koutavarapu, & Kumar, 2019; Hangchao Wang et al., 2020).The limitations vary with the combination of polymer and salt. The most common limitation that was observed during research is lower thermodynamically stability. The thermodynamic stability of solid polymer electrolytes is dependent upon operating temperature. The poor thermodynamic stability might lead to moderately reversible disrobing–plating behavior (Sementa, Maria Vaglieco, & Catapano, 2012). The ion conductivity of the solid polymer electrolytes is another problem that limited their use in the batteries. However, this problem can be overcome with the manufacturing of solid electrolytes through polyanionic block copolymer (Loh & Wang, 2012). On the other hand, the problem associated with thermodynamic stability cab be resolved with blending of highly thermodynamic stable material during production (Luo et al., 2019). It is found that the blending of metallic nanoparticles in solid electrolytes enhance the thermodynamic properties of electrolyte without affecting the ionic conductivity (Dhatarwal & Sengwa, 2020; R. Kumar, Subramania, Sundaram, Kumar, & Baskaran, 2007; Song et al., 2015). I some case, the ionic conductivity of the solid electrolytes is improved by adding the nanoparticles during the preparation of solid electrolytes.Most of the trial results propose that the homogeneously discrete ceramic-filler such as TiO_2, Al_2O_3, etc. may deliver stronger and more winding boundaries impeding the dissemination of nucleated dendrites.

The solid polymer electrolyte is made with poly (vinylidene fluoride) PVDF and poly (ethylene oxide) PEO in varied quantities in this study. The optimal fraction is chosen for

further investigation. During the preparation of the solid electrolyte, nanoparticles of TiO2 and MgO are scattered in the electrolyte to improve its thermal and ionic properties. The sol-gel approach has been chosen to create nanoparticles. The weight of each nanoparticles varies between 1% and 4% of the total weight of PVDF and PEO.Three samples for each nanoparticle are prepared for the testing by taking1%, 2%, and 4% by weight. The properties of all the prepared samples are tested with their respective standards. The dielectric properties and ionic conductivity of the prepared samples are measures through impedance spectroscopy. While the thermal properties and molecular structures of the prepared solid electrolytes are measured with differential scanning calorimetry-thermogravimetric analysis (DSC-TGA) and X-ray diffraction (XRD) respectively. The detailed results are shown in the result and discussion section.

LITERATURE REVIEW

The research is going on to find out the compatibility of various nanoparticles in enhancing the conductive and mechanical properties of solid polymer electrolytes prepared with a different combination of salts and polymers. The nanoparticles such as Mn_2O_3, nano-grained Sr and Zr, MgO, TiO_2, Al_2O_3, etc. were analyzed by different researchers during their studies. The trend of results appears after analyses do not have a uniform pattern. In this section, the exhaustive literature review is conducted and results obtained by various researchers are analyzed which gives strength to the current research. Alagar et al. (Alagar, Madhuvilakku, Mariappan, & Piraman, 2019) analyze the performance of symmetric supercapacitor with solid electrolytes. The authors used PVA- $LiNO_3$ gel electrolyte with Mn_2O_3-s/Mn_2O_3–c positive electrode and rGO as the negative electrode. During the analyses, the authors analyzed that the three-electrode arrangement made up of permeable Mn_2O_3nanocubes and nanosphere displays an outstanding specific capacity of 345 F g^{-1} and 321 F g^{-1} at 0.2 A g^{-1} respectively. Further, the cyclic stability of 89% was observed for nanocubes and nano-

grained Sr and Zr on the properties of $BaCeO_3$ at intermediate temperature conditions for solid oxide fuel cells. The authors found that the $Ba_{1-x}Sr_xCe_{1-y}Zr_yO_{3-\delta}$ electrolytes for (x=y=0.3 and x=y=0.2) exhibits peak conductivity of 0.008 and 0.007 S cm^{-1} respectively.

Further, the activation energy values were found 0.11 and 0.10 eV respectively with a power density of 250 mW cm^{-2} and 175 mW cm^{-2} also the maximum circuit voltage obtained is0.875 V&0.645 V respectively. Lue et al. (Lue et al., 2015) used TiO_2 as an electric conductance enhancer for a quasi-solid-state electrolyte. The authors found that the use of TiO_2 nanoparticles boosts the long-term performance of the semi-solid electrolytes with improves conductive and mechanical properties. The authors investigated that, the utilization of TiO_2 nanoparticles suppresses the evaporation rate for the solvents and maintains better contact between electrolyte and electrodes. The photo-to-electricity efficiency for the TiO_2 doped solid polymer electrodes is increased to 6.48% after 2400 hours which was observed at 5.08% in the initial state. Pal and Ghosh (Pal & Ghosh, 2018) also investigated the influence of TiO_2 nano-particles on the cell performance and charge carrying capacity of PMMA - $LiClO_4$ based solid electrolyte. The author observed that the maximum conductivity of ions was 3×10^{-4} S cm^{-1} at an operating temperature of 303K with a 1% weight concentration of TiO_2 in the electrolytes. Further addition of TiO_2 in the electrolyte overpowers the ion pair establishment because of solid Lewis acid-base interaction. Ahamad and Gupta (Ahamad & Gupta, 2020) use carbon nanotubes (CNT) and carbon black (CB) to enhance the conductive characteristics of the solid electrolytes interfaces. Authors found that the capacity of electrolytes fade is around 47.7% after 500 rounds while CNT is used as conductive additives for the solid electrolytes. While for CB when used as conductive additives to the solid electrolyte the fade incapacity of electrolyte further decreased to 54.4% after the equal numberMaheshwaran et al. (Maheshwaran, Kanchan, Mishra, Kumar, & Gohel, 2020) investigated the effect of adding a small amount of MgO nanoparticles in magnesium-ion

conducting solid electrolytes. The authors noticed that the ionic conductivity of 1.49×10^{-4} S/cm can be obtained at 297K with 1% by weight dispersion of MgO nanoparticles in the solid electrolytes during preparation. The authors also notice a significant improvement in the mechanical properties of the blended electrolyte. During the study, the mechanical strength of 2.7 kgf, young's modulus of 150 N/mm^2, and stress of 2.6 N/mm^2 were observed at the maximum load of 10 kgf. The mentioned literature results indicate that the use of nanoparticles in the solid electrolytes enhances the ionic conductivity also these nanoparticles help to improve the mechanical properties of the electrolytes which provides extra safety to the device with improving life.

EXPERIMENTAL

MATERIALS

Sigma-Aldrich in the United States provided the PEO (molecular weight 900,000 g/mol), PVDF (molecular weight 534,000 g/mol), and Lithium perchlorate (LiClO4). Dimethyl-formamide (DMF) is bought from the Pon Pure Chemicals Group in Chennai, India. Before use, all of the polymers are vacuum dried. The institute's local vendors provide the additional chemicals, such as titanium (IV) isopropoxide (TTIP), glacial acetic acid, sodium hydroxide (NaOH), magnesium nitrate Mg(NO3)2, urea, acetic acid, and distilled water.

SYNTHESIS OF TiO$_2$&MgO

The effect of dispersion of TiO$_2$ and MgO nanoparticles on the ionic conductivity and mechanical characteristics of the solid polymer electrolytes are analyzed in the present study. The TiO$_2$ and MgO nanoparticles used for the study were also prepared in the institutionallaboratory. The TiO$_2$ nanoparticles are prepared through the most popular sol-gel method while the solution combustion method is opted tosynthesis the MgO nanoparticles. For the synthesis of TiO$_2$, the titanium (IV) isopropoxide (TTIP) is dissolved into distilled water in

the presence of glacial acetic acid. The reaction is conducted into the flat bottom conical flask with tight closure. The reaction is conducted with a slightly higher amount of distilled water. For the complete mixing, the quantity of distilled water required is 10 times the volume of titanium. In the current experiment, 9 ml of TTIP is dissolved into 100ml of distilled water. The neck of the flask is tightly closed during the reaction to overcome the evaporation losses. The mixture isstirred for 45 minutes with a magnetic bit and magnetic stirrer arrangement. This arrangement also has a heating pad that maintains the temperature of the reaction. After 45 minutes, acetic acid is added to the solution and let react for another 5 hours with constant speed and temperature of 80°C. The heating is stopped after 5 hours and the solution is kept for natural cooling. After the cooling, the suspended particles settle down by changing the pH value of the solution to less than 7 by adding the required amount of NaOH solution in it. Once the pH level reaches below 7, the nanoparticles are allowed to settle down at the bottom of the flask and an excess amount of water is removed from the solution. The collected TiO_2 nanoparticles are then dried in a vacuum at above 150°C for at least 15 hours. Finally, the dried nanoparticles of TiO_2 are stored in an airtight container. The process followed for the preparation of TiO_2 nanoparticles is very much similar to opted of Leyva et al. (Leyva-porras, Toxqui-teran, Vega-becerra, & Miki-yoshida, 2015).As discussed earlier the MgO nanoparticles are synthesized through the solution combustion process. Initially, a mixture of Magnesium nitrate $Mg(NO_3)_2$ and urea is prepared which acts as a fuel in the reaction. The prepared mixture is then dissolved into the distilled water and form a homogeneous solution. The prepared solution is then heating to slightly above 100°C and makes the solution dry. The remaining dried mass of the solution is calcined at around 500°C for 6 hours to get the pure MgO nanopowder. Similar to the TiO_2 nanoparticles the MgO nanopowder is also stored in an airtight container for further use.

SYNTHESIS OF SOLID POLYMER ELECTROLYTES

A mixture of PVDF and PEO in a proportion of 80:20 by weight is prepared before starting the reaction. The mixture is dissolved into DMF at 70°C with the help of a magnetic bit and magnetic stirrer. The reaction is performed in a flat bottom conical flask with airtight closer. The closer will help to overcome any evaporation losses during the reaction. The stirrer speedduring the reaction is kept constant at 300 rpm. Once the homogeneous solution of DMF and polymer mixture is prepared, 20% by weight of $LiClO_4$ to the weight of polymer mixture is added to the solution. The solution is put for agitation again at the same temperature and same stirring speed until $LiClO_4$ is completely mixed in the solution. After complete mixing of polymer and salt different quantities of TiO_2 and MgO nanoparticles are added to the solution differently.The quantity of TiO_2 and MgO nanoparticles is taken 1%, 2%, and 4% by weight to the weight of the polymer mixture. The quantity of polymer mixture used in the reaction is taken as a base for all the measurements taken during the synthesis of the polymer electrolytes. After adding nanoparticles the solution is again stirred for some more time for complete and homogeneous mixing of nanoparticles in the solution. The prepared solutions are cast into glass Petri dishes and put for heating at a constant temperature of 80°C until allthe DMF is evaporated from the prepared electrolytes. The samples are then naturally cooled to ambient temperature. To remove the traces of the DMF in the samples, the samples are put into the vacuum oven for 24 hours at a temperature of 40°C. Finally, the samples are ready for characterization and further studies. The procedure followed for the preparation of solid polymer electrolytes with traces of nanoparticles is similar to the procedure followed by Dhatarwal and Sengwa(Dhatarwal & Sengwa, 2020).

CHARACTERIZATION OF ELECTROLYTES

The created solid polymer electrolytes are put to the test to determine their phase structure, electrochemical properties, and thermal properties, among other things. An X-ray diffractometer (XRD) with a scanning range of 10° to 60° is used to characterise the phase structures. The thermal stability and ionic conductivity of solid polymer electrolytes are measured using a thermogravimetric analyzer and an Autolab. The electrolyte's ionic conductivity is also tested in a nitrogen-controlled atmosphere. To determine the electrolyte's ionic conductivity, the electrolyte is placed between two stainless steel electrodes. The current fluctuates in frequency from 10 Hz to 1 MHz during the experiment. The electrolyte's ionic conductivity () is calculated using the following formula:

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

Where t is the electrolyte thickness, Rbis is the bulk resistance, and A is the electrolyte's cross-sectional area. In a similar study to Wang et al., the ionic conductivity variation is investigated in the temperature range of 298K to 353K. (Hao Wang et al., 2020).

RESULTS AND DISCUSSIONS

The number of samples of PVDF/PEO/LiClO$_4$ solid polymer electrolytes is prepared with different concentrations of TiO$_2$ and MgO nanoparticles. All the samples are tested for their ionic conductivity, heat transfer rate, and mechanical properties. The crystal patterns of the polymers and salt are observed with an XRD analyzer. The concentration of nanoparticles is very small so the presence of nanoparticles in the electrolyte is not visible in the results. Therefore the crystal pattern of nanoparticles and parent electrolyte is presented in different figs. Fig. 1 represents the crystalline structure of polymer electrolyte and fig.2 represents the XRD analysis of TiO$_2$ and MgO nanoparticles. The XRD pattern shown for the parent electrolyte in fig. 1 shows someextreme and several weak deflection peaks which represents

the availability of PVDF, PEO, and LiClO4 in the electrolyte. The first peak at 18.2° shows the presence of PEO in the electrolyte. The peak is not so intensive as compared to the peak present in pure PEO. The reason for that is the presence of LiClO4 in the electrolyte which reacts with the PEO and decreases the intensity of the PEO presence. The highest peak was observed near 19.5° which represents the presence of β- phase crystals of PDVF in the electrolyte. Two narrow and weak peaks are observed at 23.5° and 26.5° which represents the γ-phase crystals of the PVDF and LiClO4. Apart from that several low-intensity peaks are also noticed in the XRD pattern of the parent electrolyte which represents the presence of PVDF, PEO, and LiClO4 in the electrolyte. Thecrystalline patterns of prepared nanoparticles are also analyzed separately through an XRD analyzer which is shown in fig. 2.
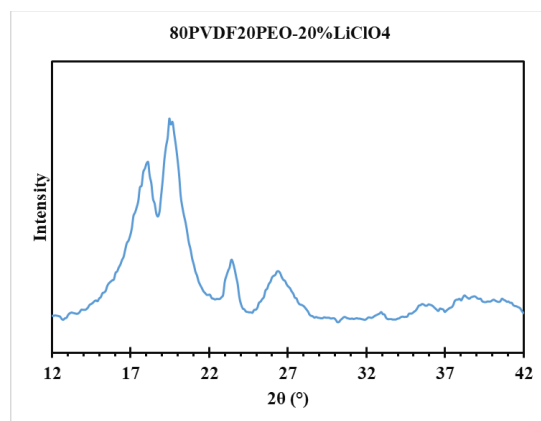

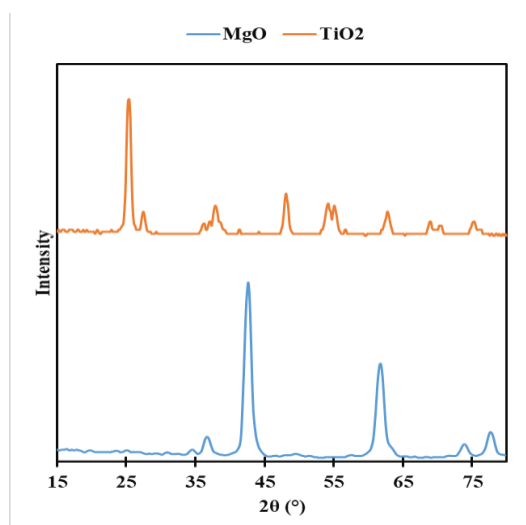**Fig-1**XRD pattern analysis of parent polymer electrolyte

Fig-2XRD patterns for prepared nanoparticles.

Fig. 3 represents the FTIR spectra of parent electrolytes and various electrolytes prepared with blending of TiO_2 and MgO nanoparticles. The FTIR analysis is generally performed to check the crystal phases of various polymers and nanoparticles used and more importantly to check and confirm the changes that occur in ion-dipole interaction (Sengwa & Dhatarwal, 2020). The off-peaks noticed in all the prepared samples of polymer electrolytes at 833 cm^{-1} and 1170 cm^{-1} are represent the presence of β-phase crystals of PDVF. Whereas, the off-peaks at 877, 950, and 1070 represent the α-phase crystals of PDVF. The relation of ionic conductivity and operating temperature of all the prepared solid electrolytes are given in fig. 4. It is analyzed that the addition of 2% TiO_2 nanoparticles in the parent electrolyte solution shows the highest ionicconductivity among all prepared samples. However, further increase inthe nanoparticle concentration in the electrolyte further reduces the ionic conductivity of the electrolyte.

Moreover, the highest ionic conductivity is observed at room temperature which is taken as 298K in the present research. The reduction in ionic conductivity of all the prepared samples can be observed with an increase in operating temperature and at 353K the difference has become very narrow among all the samples which are significant at room temperature.
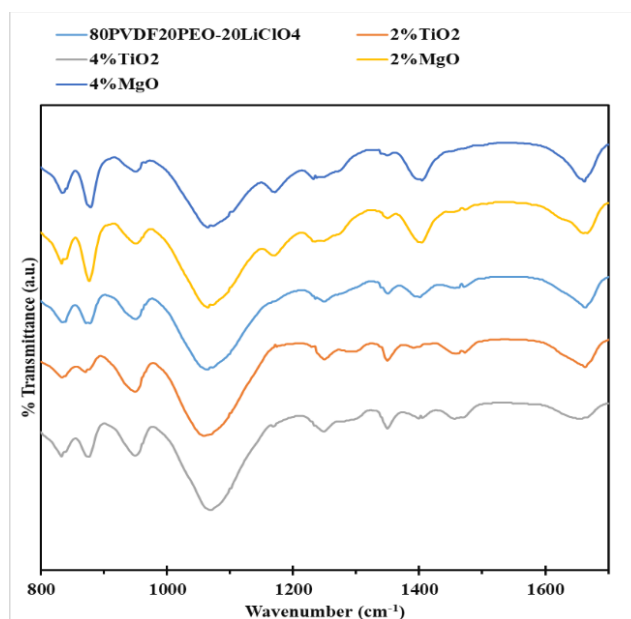

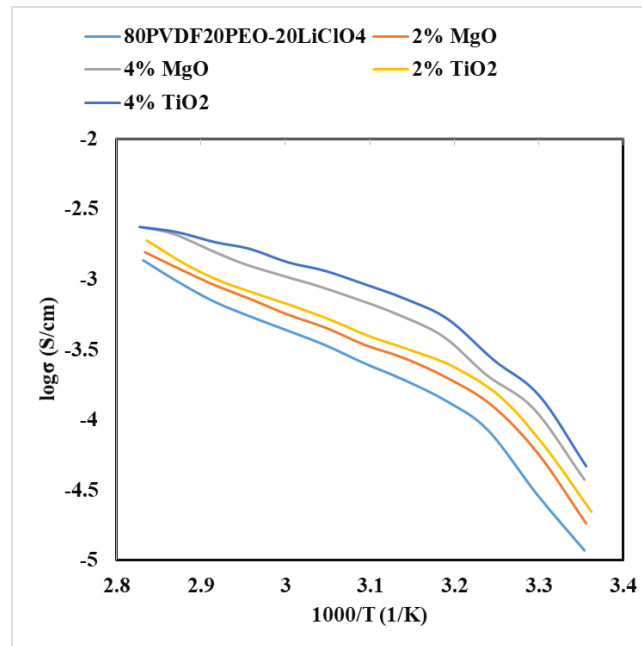
Fig-3FTIR spectra of all prepared samples

Fig-4 Ionic conductivity w.r.t. operating temperature

The mechanical properties such as ultimate stress and failure stress of all the prepared samples are analyzed in the present work and the effect of variation in concentration of nanoparticles on these properties are represented in fig.5 and fig. 6. The nanoparticles have higher mechanical strength than polymers therefore the addition of nanoparticles in the electrolytes improves the mechanical properties of the solid polymer electrolytes. It is observed that the addition of TiO_2 nanoparticles improves the mechanical properties of electrolytes more as compared to MgO nanoparticles and the reason is the higher strength of titanium. The effect of nanoparticle concentration on the current onset voltage is represented in fig.7. It can clearly visible from the fig.7 that the increase in the concentration of nanoparticles in the solid polymer electrolyte will improve the current onset potential of the solid polymer electrolyte. This is suggested that the addition of nanoparticles in the solid polymer electrolytes is an effective way to improve the energy density and electrochemical stability of lithium-ion batteries (Song et al., 2015). Further, the addition of TiO_2 nanoparticles has more impact on the current onset potential of electrolyte as compared to MgO nanoparticles. The onset potential reaches neat to 5 V with 4%

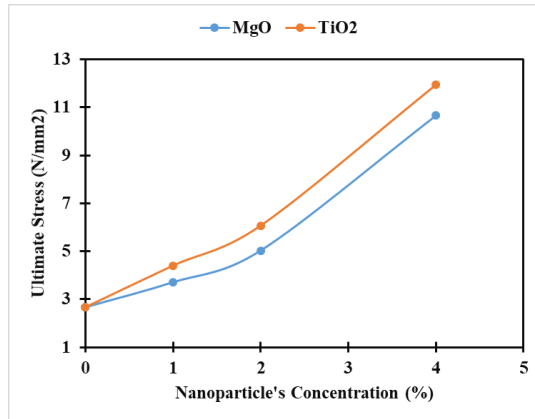TiO_2 concentration in the electrolyte while with the same concentration of MgO the onset potential is around 4.7 V.

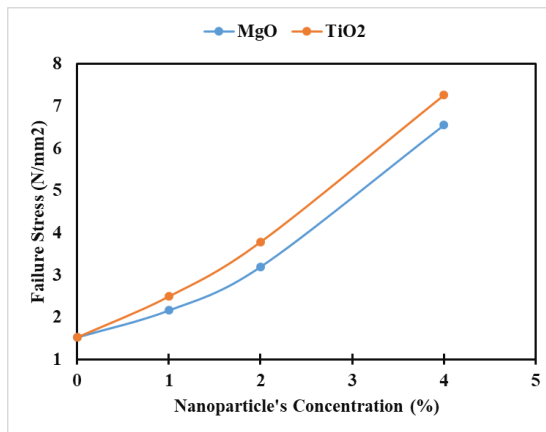**Fig-5** Effect of nanoparticle concentration of ultimate stress of electrolyte

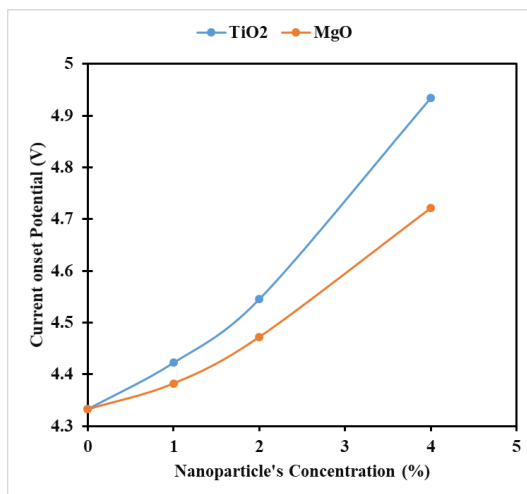**Fig-6** Effect of nanoparticle concentration of failure stress of electrolyte

Fig-7 Current onset potential v/s Nanoparticle concentration

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CONCLUSIONS

In the present study, the effect of adding TiO_2 and MgO nanoparticles on the electrochemical, mechanical, and structural characteristics are analyzed on solid with PVDF/PEO/LiClO$_4$. Several conclusions are drawn from the current studies and some important conclusions are stated here:

- It is very difficult to see the presence of the crystalline pattern of the nanoparticles in the solid electrolytes with very low concentration.

- The addition of nanoparticles improves the ionic conductivity of electrolytes. However, after a certain concentration, the ionic concentration of electrolyte starts decreasing with a further increase in nanoparticle concentration.

- The presence of nanoparticles significantly improves the mechanical properties of solid electrolytes which provide extra strength to the battery.

- The addition of nanoparticles also increases the current onset potential of the electrolyte and as a result the energy density and electrochemical stability of the battery increases.

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