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SYNTHESIS AND CHARACTERIZATION OF PEO/PVDF POLYMER BLEND ELECTROLYTES DOPED WITH LIFEPO$_4$ NANOFILLER AND EPOXIDIZED NATURAL RUBBER PLASTICIZER

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ABSTRACT

This research focuses on the preparation and characterization of polymer blend electrolytes based on polyethylene oxide (PEO) and polyvinylidene fluoride (PVDF), incorporating LiFePO$_4$ as a nanofiller and epoxidized natural rubber as a plasticizer. The incorporation of LiFePO$_4$ aims to enhance the electrochemical performance of the polymer blend by improving ionic conductivity and mechanical stability. The polymer blends were prepared using a solution-casting technique, where varying concentrations of LiFePO$_4$ were introduced into the PEO/PVDF matrix. The XRD reveals the degree of crystallinity and the effect of LiFePO$_4$ on the polymer structure. The undoped sample shows distinct crystalline peaks corresponding to the PEO and PVDF phases. A reduction in intensity is observed as the doping level increases, which indicates a reduction in crystallinity due to the disruption caused by the LiFePO$_4$ particles. For the TGA/DTA graph, the undoped polymer blend (PEO75/PVDF25) shows a characteristic decomposition at (400–600 °C), (5% of LiFePO$_4$) curve, which shows early decomposition than other doped samples. The increased LiFePO$_4$ content acts as a thermal barrier at higher concentrations, delaying the degradation process. The results indicate that the inclusion of LiFePO$_4$ significantly influences the thermal properties of the PEO/PVDF blend electrolytes, making them promising candidates for use in high-performance lithium-ion batteries.

Keywords: Polymer electrolyte, polyethylene(oxide) (PEO), polyvinylidene florid (PVDF), lithium iron phosphate (LiFePO$_4$), XRD, TGA/DTA

INTRODUCTION

As the world energy demand increases, energy storage has gained substantial interest as it plays a key role in many fields, from flexible electronics to electric vehicles and renewable energy. Lithium-ion is the most common battery technology (Gucci et al., 2023). Despite these advantages, numerous incidents of the explosion of Lithium-ion batteries have been reported, and improvements in terms of safety have consistently been in demand (Kim & Lim, 2021), developing a stable and safe electrolyte that works at voltages as high as 5 V is a formidable challenge in present Lithium ion battery research because such high voltages are beyond the electrochemical stability of the conventional carbonate-based solvents available. In the past few years, extensive efforts have been carried out by the research community toward the exploration of high-voltage electrolytes (Tan *et al*., 2014).

To address these issues, researchers have turned to solid-state polymer electrolytes (SPEs), which combine ionic conductivity with mechanical robustness and enhanced safety features. Solid polymer electrolytes (SPEs) offer several advantages over liquid electrolytes, including excellent processability (Zhou et al., 2025). Solid Polymer Electrolytes (SPE) are capable of eliminating leakage and mitigating the risks of thermal runaway, making them attractive candidates for next-generation lithium-ion batteries. These solid-state electrolytes offer advantages such as reduced risk of leakage, enhanced safety, and better compatibility with lithium metal anodes (D. Zhang et al., 2023). Recently, development in polymer science has attracted the attention of the research community due to its advantages offered by polymers, such as unique and imperative properties for advanced applications (Mohammed *et al*., 2019). Recently, development in polymer science has attracted the attention of the research community due to its advantages offered by polymers, such as unique and imperative properties for advanced applications (Mohammed *et al*., 2019). Among the various materials studied, polyethylene oxide (PEO) and polyvinylidene fluoride (PVDF) stand out due to their complementary properties. PEO, with its high ionic conductivity in the amorphous phase, is often blended with PVDF, known for its mechanical strength and chemical stability, to achieve a synergistic effect. Polymer solid electrolytes have low ionic conductivity at room temperature, which limits their application in solid-state batteries (Amudha & Suthanthiraraj, 2015). These materials are being explored rapidly as a substitute for metal or ceramic-based materials in a lot of applications such as automotive, aerospace, marine, sporting goods, and electronic industries. Polymers are materials made up of joining a large number of monomers; they are made up of long chains or networks of smaller molecules called monomers (Ngudsuntear *et al*., 2022).. Much later, polymer science emerged into the academic field (Koul *et al*., 2001). In addition to their economic superiority, Polymers possess a vast variety of favorable physical, chemical, and mechanical properties, such as flexibility, lightweight, optical transparency, along ease of processing, which have made the study of polymers an attractive field of research (Singh et al., 2015). Blending of two polymers not only results in improving the mechanical strength but also helps in increasing the conductivity (Pradeepa et al., 2015). Among various materials investigated for SPEs, polyethylene oxide (PEO) stands out due to its remarkable ability to solvate lithium salts and enable ion transport through its amorphous regions (Ye *et al*., 2021). However, the practical application of PEO is limited by its high crystallinity at room temperature, which reduces ionic conductivity to suboptimal levels

(Wang *et al*., 2020). Therefore, great attention has been paid to the modification of PEO via different approaches, such as blending PEO with another material or utilizing PEO derivatives, to improve the ionic conductivity and comprehensive properties (Wang *et al*., 2020). By combining PEO with polyvinylidene fluoride (PVDF), a polymer known for its exceptional chemical and thermal stability, researchers can create a synergistic matrix that balances ionic conductivity, mechanical integrity, and thermal stability (Zhou et al., 2025). PVDF contributes to the reduction of crystallinity in the blend while maintaining the structural stability required for long-term battery operation., PVDF is mainly used as a semi-crystalline polymer with relatively good mechanical properties, excellent swelling characteristics, high dielectric constant, and thermal stabilities (Liu et al., 2019). The resulting PEO/PVDF blend is a promising candidate for polymer electrolytes, offering enhanced performance compared to individual polymers (Patla *et al*., 2018).

Polymer electrolytes are of great interest because of their potential application in electrochemical devices such as batteries, fuel cells, supercapacitors, etc. Polymer electrolytes have attracted attention towards it, because of their various advantageous properties in solar cells and energy storage devices. The required conductivity must be as high as 10^{-3} to 10^{-2} S/cm, which is not easily achievable with a polymer at room temperature. In order to improve the ionic conductivity of polymer electrolytes, many methods, like doping of ionic salts, plasticizers, and ionic liquids into a polymer host matrix, were used by researchers (Bello *et al*., 2020).

Attempts to increase the ionic conductivity of second-generation SPEs involved the application of various strategies, including the study of a wide variety of elastomeric host polymers, alternative lithium salts, and the use of a range of conductivity-enhancing additives (Manuela et al., 2002). lithium salt is compatible with nearly all common cathode materials, including $LiMn_2O_4$, $LiFePO_4$, and $LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$, and assembled cells can realize stable cycling without significant fade at 60 °C (L. Zhang et al., 2018).To further enhance the properties of PEO/PVDF blends, nanofillers such as lithium iron phosphate ($LiFePO_4$) are incorporated into the polymer matrix. $LiFePO_4$ is widely recognized for its stability, compatibility with lithium-iron systems, and ability to enhance ionic conductivity. By forming interactions with the polymer chains, $LiFePO_4$ reduces the crystalline regions in PEO, thereby increasing the proportion of amorphous regions responsible for ion transport. Additionally, $LiFePO_4$ improves the thermal and mechanical properties of the polymer blend, ensuring stability under operational conditions (Patla *et al*., 2018). The addition of a plasticizer is another option because it can improve the crystallinity of PEO, which in turn increases the ionic conductivity (Wang et al., 2020).

Wang *et al*. (2020) investigated on enhancement of the Ionic conductivity of polymer electrolytes, where they studied solid polymer electrolytes (SPE) comprising a blend of poly (ethylene oxide) (PEO) and epoxidized natural rubber as a polymer host and $LiCF_3SO_3$ as a dopant, where it was prepared by the solution-casting technique. The SPE films were characterized by field emission scanning electron micros-copy to determine the surface morphology, X-ray diffraction, and differential scanning calorimeter to determine the crystallinity and thermogravimetric analysis to confirm the mass decrease caused by loss of the solvent, while the presence of complexes was investigated by attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy and electrochemical impudence spectroscopy was conducted to determine the ionic conductivity. All the prepared hydrogels showed a significantly larger swelling ratio and improved mechanical strength (Wang *et al*., 2020). The authors reported that the conductivity was enhanced by the addition of dibutyl phthalate (DBP) plasticizer and ZrO_2 nanofiller with maximum conductivity (1.38×10^{-4} Scm^{-1}). The absorption edge and band gap values showed decreases upon addition of $LiSO_3CF_3$, DBP, and ZrO_2 due to the formation of localized states in the SPE and the degree of disorder in the films was increased.

In another study by Gaabour, (2015) prepared Solid polymer blend films based on Polyethylene oxide (PEO) and polyvinylidene fluoride (PVDF), single and multiwalled carbon nanotubes (SWCNTs-MWCNTs) & CNTs-COOH using the solution casting method. Thermal studies at different heating rates and kinetic parameters were performed using thermogravimetric analysis (TGA) and its first derivative (DTG). The kinetic thermodynamic parameters, such as activation energy, enthalpy, entropy, and Gibbs free energy, were evaluated from TGA data using the Coats-Redfern relation. The lower values of percentage weight loss are from 3.2 to 20% due to splitting or volatilization of small molecules and evaporation of moisture. The main decomposition region in TGA curves has a percentage of weight loss from 33 to 70%. The degradation temperature for the neat blend was lower than that of the nanocomposites after adding both CNTs and an increase of heating rate. The decomposition temperature is moved to higher temperatures, indicating that the thermal stability of the polymer has been improved.

In this research, we prepared a polymer blend using 75 % of PEO and 25 % of PVDF, varying concentrations of $LiFePO_4$ as nanofillers, and a constant percentage of ENR plasticizer. The solution casting method was used to achieve the preparation of this research, and it was sent for characterization. The structural and thermal properties of these samples were obtained, and the incorporation of $LiFePO_4$ nanofillers into PEO/PVDF polymer blends significantly enhances their structural and thermal properties, making them promising candidates for safer and more efficient solid-state lithium-ion batteries.

MATERIALS AND METHODS

Poly (ethylene oxide) (POE), Poly (vinylidene fluoride (PVDF), DMF**(**Dimethylformamide), Lithium iron phosphate ($LiFePO_4$), Epoxidized natural rubber (ENR) were obtained from Sim best scientific & chemicals (Minna, Niger State, Nigeria opposite FUT Minna) and were used without any further purification.

Preparation of Polymer Electrolyte

A stable polymer electrolyte was prepared using the solution casting method. An adequate ratio of an optimized polymer blend PEO and PVDF (75:25), and lithium salt ($LiFePO_4$) at different percentages (5 %, 15 %, 15 %, 20 %), different weights (0.2, 0.3, 0.5, 0.6) g were dissolved in 15 ml of DMF (Dimethylformamide) solvent and 15 % (0.5 g) of plasticizer (epoxidized natural rubber (ENR) which increased the plasticity of the sample was added at constant percentage (grams), the solution was stirred at 50 ^{o}C at 800 rpm for 5 hours and a homogeneous viscous solution was obtained. The resulting homogeneous viscous solution was gently poured on a glass petri dish. The films were dried in a vacuum oven for 20 hours at 50 ^{o}C. Finally, the prepared films are stored in a plastic sample holder for further characterization.

RESULTS AND DISCUSSION

XRD Analysis of Polymer Electrolyte

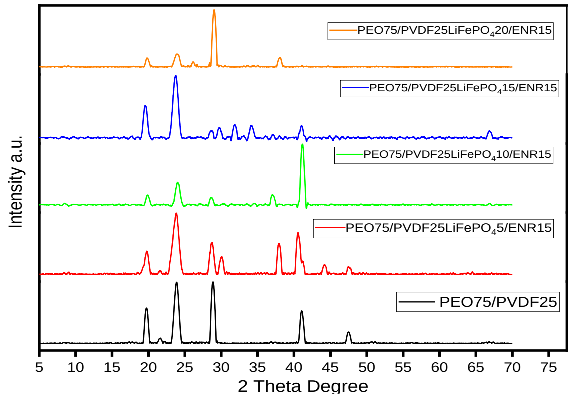

Figure 1 ; XRD curve of polymer blend,nanofiller and platisizer (PEO75/PVDF25, PEO75/PVDF25/$LiFePO_4$10/ENR15,PEO75/PVDF25/$LiFePO_4$15/ENR15,PEO75/PVDF25/$LiFePO_4$20/ENR15),

Figure 1. shows the graph of a doped polymer blend of PEO75/PVDF25 with different percentages of $LiFePO_4$ (5,10, 15, 20) % and a constant percentage of Epoxidized Natural Rubber (ENR15) 15 %. It indicates that the addition of plasticizer and lithium salts in the polymer blend suppresses the crystallinity of the polymer. The undoped sample shows distinct crystalline peaks corresponding to the PEO and PVDF phases. This serves as the reference for comparison with the doped samples. With increasing $LiFePO_4$ content (5,10, 15, 20) %, the intensity and position of peaks change. A reduction in intensity is observed as the doping level increases, which may indicate a reduction in crystallinity due to the disruption caused by the $LiFePO_4$ particles. Additional peaks in doped samples suggest the formation of new crystalline phases associated with $LiFePO_4$. The peaks from PEO and PVDF are still visible, indicating that the base structure of the polymer blend is retained. The incorporation of $LiFePO_4$ introduces a heterogeneous structure; the characteristic peaks of PEO shift towards the lower angle side with significant broadening on the addition of the plasticizer, leading to a mixture of crystalline phases. At higher $LiFePO_4$ doping levels (20 %), peak broadening is observed, which signifies a decrease in crystal size or increased amorphous content. and Low doping levels, while at (5 % and 10 %) of $LiFePO_4$ maintain more of the original crystalline structure compared to high doping levels (15 % and 20 %) of $LiFePO_4$ where significant disruption occurs. This shift indicates an increase in interplanar spacing of the (100) plane. PEO (Polyethylene oxide) typically shows a strong peak around 19-20 ° (2θ), which corresponds to a d-spacing of about 4.5-4.7 Å. Polyvinylidene fluoride (PVDF) has characteristic peaks, with one of the strongest usually appearing around 20.3 ° (2θ), corresponding to a d-spacing of about 4.4 Å. According to Wang *et al*, (2020) prepared PVDF, PEO, $LiClO_4$ and SN, the prepared ASPE remains the good ionic conductivity and shows the greatly improved mechanical performance, whose maximum stress is 10 times higher than conventional ASPE without PVDF supporting layer with the decrease in the crystallinity of ASPE leads to the increased ionic conductivity, the reason behind is that the decreased crystallinity improves the binding and distribution of each component, and enables the increased active segments of PEO (Wang *et al*., 2020).

Thermogravimetric Analysis (TGA) of Polymer Electrolyte

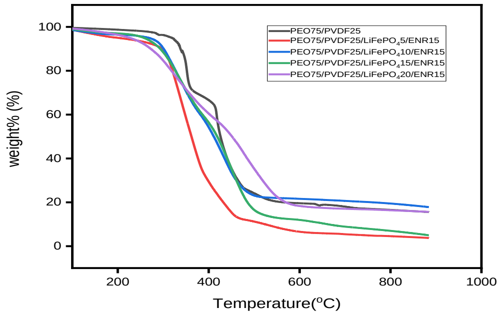

Figure 2; TGA graphycal reperesentation of (PEO75/PVDF25, PEO75/PVDF25/$LiFePO_4$10/ENR15,PEO75/PVDF25/$LiFePO_4$15/ENR15,PEO75/PVDF25/$LiFePO_4$20/ENR15).

Thermal properties, including thermal stability and decomposition behavior, were assessed using thermogravimetric analysis (TGA) and differential thermal analysis (DTA). Figure 2 shows a TGA graph of a polymer blend and doped polymer blends with $LiFePO_4$. The undoped polymer blend (PEO75/PVDF25) showed a characteristic decomposition behavior typical of this polymer blend at (400–600 °C), (5% of $LiFePO_4$) curve shows earlier decomposition than other doped samples, suggesting lower thermal stability for this concentration. For (10 % $LiFePO_4$) and (15 % $LiFePO_4$) curves demonstrate intermediate thermal stability. (20 % $LiFePO_4$) The curve exhibits the highest thermal stability (around 800–1000 °C), and the most significant residual weight, indicating the positive impact of higher $LiFePO_4$ content. All blends stabilize with minimal weight loss after 600 °C it is expected because $LiFePO_4$ is an inorganic salt, a thermally stable material that does not fully decompose under these conditions. The presence of ENR at a constant 15 % contributes to enhanced flexibility and stability in the blends, but the dominant effect observed here is from the varying $LiFePO_4$ content. Basappa *et al*. (2022) prepared polymer blend film with PEO, PVDF and DMF using solution casting techniques, TGA thermogram curve PEO, PEO/PVDF10, PEO/PVDF30 blend films shows two stages decomposition; the first stage of the PEO curve indicating the temperature at 279 °C with weight loss of about 8.71%, PEO/PVDF10 is stable up to 284.38 °C with weight loss is 14.7% and PEO/PVDF 30 is showing a decomposes at 318 °C with weight loss is 13.35, in second stage blend film decomposes fast at 388 °C, 393 °C, and 432 °C with straight weight loss about is 91.2 %, 76.18 %, and 77.44 %. The second stage of weight loss is attributed to the carbon, oxygen, with ether functional group of the polymer blend films (Basappa *et al*., 2022)

Differential Thermal Analysis (DTA) of Polymer Electrolyte

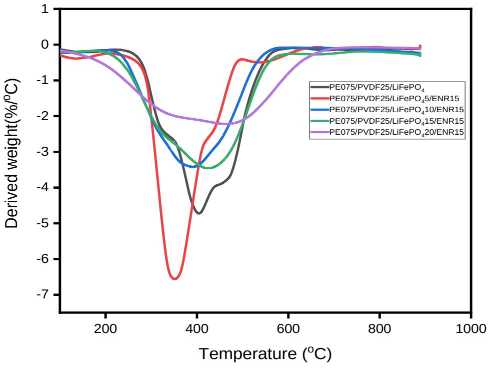

Figure 3; DTA graphycal reperesentation of (PEO75/PVDF25, PEO75/PVDF25/$LiFePO_4$5/ENR15, PEO75/PVDF25/$LiFePO_4$10/ENR15, PEO75/PVDF25/$LiFePO_4$15/ENR15 and PEO75/PVDF25/$LiFePO_4$20/ENR15)

Figure 3 represents the rate of weight loss (% °C) as a function of temperature, identifying the temperatures at which the material undergoes thermal degradation. The base polymer blend (PEO 75/PVDF 25) without $LiFePO_4$ doping shows a single major degradation peak around 450–500 °C. This peak corresponds to the thermal degradation of the PEO and PVDF blend. The absence of additional peaks suggests no external dopants influencing thermal stability. Introducing 5% $LiFePO_4$ leads to an earlier degradation onset compared to the (PEO 75/PVDF 25) curve, with the major peak slightly shifted to lower temperatures (430–450 °C). The incorporation of $LiFePO_4$ slightly reduces thermal stability, likely due to interaction between the polymer matrix and $LiFePO_4$ particles. With (10 % $LiFePO_4$) the degradation peak becomes more pronounced and shifts further to a lower temperature (420–440 °C). This indicates increasing thermal instability as more $LiFePO_4$ is added, possibly due to its catalytic influence on the degradation of the polymer blend. At 15 % $LiFePO_4$, the degradation occurs at similar temperatures (420 °C) but with a broader peak compared to the curve of (10 % $LiFePO_4$), the broader peak suggests complex degradation behavior, due to interactions between the increased $LiFePO_4$ content and ENR.with 20% $LiFePO_4$, the degradation peak shifts back to higher temperatures (450–470 °C), this indicates improved thermal stability compared to lower dopant concentrations. The increased $LiFePO_4$ content acts as a thermal barrier at higher concentrations, delaying the degradation process. The addition of $LiFePO_4$ initially reduces thermal stability (5–15 %), but higher concentrations (20 %) improve its ENR Contribution.

Conclusion

In conclusion, this study investigated the structural and thermal properties of PEO/PVDF polymer blend electrolytes reinforced with varying concentrations of $LiFePO_4$ nanofillers. The findings from XRD Analysis and Thermogravimetric Analysis/Differential Thermal Analysis (TGA/DTA) provided critical insights into the impact of nanofiller incorporation on the blend performance. the incorporation of $LiFePO_4$ nanofillers into PEO/PVDF polymer blends significantly enhances their structural and thermal properties, making them promising candidates for safer and more efficient solid-state lithium-ion batteries. The findings provide a foundation for the continued development of polymer electrolytes tailored for high-performance energy storage applications. By addressing the identified limitations and pursuing the suggested future research directions, this material system has the potential to contribute substantially to the advancement of battery technologies, enabling safer, more sustainable energy solutions.

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