

Structural and Electrical Studies of Chitosan: Polyvinyl Alcohol with Blend Polymer Electrolyte Doped with Potassium Iodide

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Abstract

The technique of solution casting was employed to produce a flexible and uniform blend polymer electrolyte film (BPE) comprising Chitosan (Cs): polyvinyl alcohol (PVA) doped with potassium iodide (KI) at varying concentrations ($X = 0\%$, 10% , 20% , 30%). X-ray diffraction (XRD) was utilized to examine the structural characteristics of the BPE and assess their crystallinity and amorphous nature. Fourier-transform infrared spectroscopy (FTIR) was used to identify the functional groups and ensure the homogeneous mixing of PVA/CS/KI. The electrical properties of the BPE were assessed to determine the capacitance and potential window using cyclic voltammetry (CV). It was observed that the BPE doped with 20% KI (sample PCK20) exhibited a highly crystalline nature and demonstrated the highest capacitance of BPE 449.1 pF . The potential window for the BPE ranged from 1.45 V to 2 V . These BPE materials show potential for use in environmentally friendly energy storage applications. The acquired results show good crystallinity, potential stability, and capacitance as evaluated by cyclic voltammetry (CV), and they could be beneficial for energy storage applications.

Keywords- BPE, Structural and electrical properties, Green energy.

1. Introduction

Renewable energy sources come from natural sources such as sun, oceans, seeds, algae, wind, storms, geothermal and recyclable polymeric materials and have attracted great attention due to the growing oil disaster and environmentally friendly concerns (Wang et al., 2020; Zhou et al., 2021). Solid polymer electrolyte (SPE) has been used in electrochemical devices used multiple times. They replaced liquid electrolytes due to the ease of fabrication of films and broad electrochemical stability (Wu et al., 2009, Ramesh et al., 2010; Winie and Shahril, 2015). SPEs, which are becoming increasingly popular, are gaining increasing importance in recent years due to their potential use in the energy industry, especially in storage and production (Tarascon and Armand, 2001; Cheng et al., 2010; Ferrari et al., 2010). SPEs have lower conductivity for use in real-world battery applications, but outperform traditional liquid electrolytes in thermal and mechanical durability. Blending polymers (Kumar et al., 2014, Patla et al., 2018), cross-linking polymers (Garcia et al., 2016), and adding ionic liquids (Liew et al., 2015), inorganic fillers (Muchakayala et al., 2017), and plasticizers (Alves et al., 2016) are just some of the proposed approaches to improve SPE ionic conductivity. Blending polymers has attracted many people due to their cheap price, easy assembly and predictability (Tiong et al., 2016; Demir et al., 2017; Anilkumar et al., 2017; Farid et al., 2018). SPE film properties depend upon the blend miscibility (Premalatha et al., 2016; Abdelghany et al., 2018). Many more previous studies mentioned proton conducting solid blend polymer electrolyte (SBPE) systems, including PVDF-PVA-NH₄SCN (Muthuvinayagam and Gopinathan, 2015),

MC-PVA-NH₄NO₃ (Zainuddin et al., 2018), PVA-PAN-NH₄SCN (Sivadevi et al., 2015), starch-chitosan-NH₄Cl (Shukur et al., 2014), dextran-chitosan-NH₄SCN (Kadir and Hamsan, 2018), CdCl₂ – doped - PVA/PVAc-PVP (Baraker and Lobo, 2019), and PVA-CS-GL-NaBr (Sadiq et al., 2022).

The selection of a PE involves an assessment of its electrical, structural, and morphological characteristics based on the specific application. Choosing the right host polymer depends on several factors, two of which include: (i) In the host polymer's backbone has the presence of a functional group with sufficient electron donor capacity to form a coordination bond with the cations of the doped salt, and (ii) minimal resistance to bond rotation. Biodegradable polymers such as starch, chitosan, cellulose, and synthetic biodegradable polymers like poly (vinyl alcohol) (PVA) have been employed in electrochemical processes to produce SPEs (Kumar et al., 2023).

Chitin, the second most common biopolymer globally, originates from crustacean exoskeletons, fungi, and insect cell walls (Mourya and Inamdar, 2008). CS, a biopolymer extract from chitin, is extensively castoff in medical and electrochemical devices due to its harmless, biodegradable, and nontoxic nature (Aziz et al., 2017). CS stands out from other biopolymers because of its unique composition, which includes hydroxyl and amino functional groups (Chagala et al., 2017). Polyvinyl alcohol (PVA) is a water-soluble polymer with high dielectric strength, excellent charge storing capability, and intriguing optical properties (Femandes et al., 2013). The PVA molecule consists of a hydrophobic chain and hydrophilic end groups that extend into the outer aqueous phase, while the hydrophobic chains occupy space at the solid-liquid interface, preventing crystallinity aggregation due to steric hindrance and increased energy barrier (Li et al., 2020). When polar polymers with high electron affinity interact with the cation or surface groups of fillers, a homogeneous nanocomposite is formed (Rao et al., 2012). The hydroxyl groups in PVA's carbon chain backbone facilitate hydrogen bonding and contribute to polymer composite formation. Its exceptional transparency makes PVA an ideal choice for use in multilayer coatings for organic solar cells, providing excellent oxygen barrier properties (Nofal et al., 2021; Sadiq et al., 2022).

The PVA/Chitosan (PVA/CS) blend is highly transparent and serves a wide range of purposes due to its excellent chemical and mechanical properties (Alotaibi et al., 2023). PVA/CS, chosen for its ecological nature and the presence of hydroxyl groups, is effective for durable composite preparation techniques. It is known for being soluble, cost-effective, and environmentally friendly (Shubha and Rao, 2016; El-Aassar et al., 2023). The Blend Polymer electrolyte is utilized to improve the structural and electrical properties of the electrolyte. Structural properties are assessed using XRD, while functional groups are analyzed using FTIR. The electrical properties, including potential stability/window and capacitance, are evaluated using CV. The BPE outperforms other PEs in terms of certain properties.

The aim of this work was to fabricate chemically CS:PVA-based blend films using a traditional solvent casting method. In this study, we used salt KI variations of 0–30 wt% in the constant amount of CS: PVA (1:1). We attempted to assess the relationship between salt KI and a specific material mixing ratio. This was significantly higher than the concentration used in previously published experiments with various other compositions and salt concentrations (Nofal et al., 2020). Furthermore, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and cyclic voltammetry (CV) analysis were employed to study the electrical and structural properties of the prepared BPE films. The BPE films produced are affordable, more environmentally friendly and can be used in green energy applications.

2. Experimental

2.1 Materials

The straightforward polymeric materials are polyvinyl alcohol with the molecular weight 9000-10000 g/mol) and chitosan (CS) with the high molecular weight, both polymer materials are purchased from Sigma-Aldrich. The Salt Potassium Iodide (KI) with molecular weight 166 g/mol purchased from CDH & for Solvent used acetic acid (CH_3COOH) molecular weight 60.05 g/mol purchased from RANKEM.

2.2 Pure/KI BPE Film Preparation

The polymers selected for this purpose were chitosan (CS) and polyvinyl alcohol (PVA) mixed with Potassium Iodide (KI) as a salt using the solution casting method. The different KI concentrations were incorporated into the polymer mixture (0–30%). DI water served as a solvent for PVA and KI, while CS was soaked in acetic acid for 1 h and then dissolved in 5 mL of DI water with stirring for 4 hrs at ambient temperature. Then both solutions are mixed and stirred at ambient temperature for 19 hrs, as shown in **Figure 1**. Further, it is converted into gel form, then poured into a petri dish and dried in a hot air oven at 50°C for 10 hours. as shown in **Figure 1**. The samples were designated PCK0, PCK10, PCK20 and PCK30 (as given in the **Table 1**).

Table 1. Sample preparation of BPE.

Sample designation	PVA:CS (in %)	KI (in %)
PCK0	1:1	0
PCK10	1:1	10
PCK20	1:1	20
PCK30	1:1	30

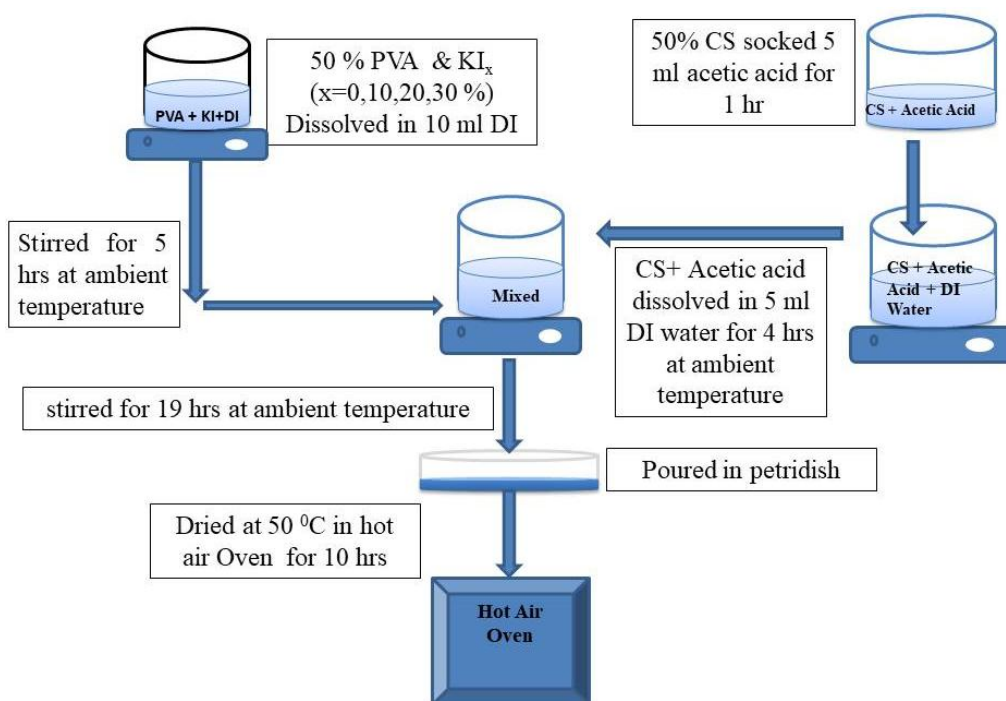


Figure 1. Preparation of BPE film pure CS:PVA (PCK0) and with different KI concentrations (PCK10, PCK20, and PCK30).

2.3 Characterization Technique

The Bruker D8 Advance diffractometer with XRD technique using $\text{CuK}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$) in the 2θ range of 10° – 90° is used to obtain structural information and determine whether BPE is more crystalline or amorphous. Using an FTIR device (PerkinElmer Spectrum IR version 10.7.2), FTIR is used to define the functional group information of pure BPE and with KI-BPE. Using cyclic voltammetry (CV), Squidstat Solo devices determine the potential window and calculate capacitance for the electrical properties of BPE.

3. Result And Discussion

3.1 X-Ray Diffraction (XRD) Analysis

XRD is used to identify the amorphous and crystalline nature of BPE (Nofal et al., 2020). To additional confirm the composite nature with KI concentration, we observed the XRD pattern in the range of 2θ range 10° to 90° . Previously pure PVA and CS (Chen et al., 2007), and pure KI XRD patterns are reported (Jyothi et al., 2016). The recorded XRD pattern of pure blend CS:PVA and with KI concentration as 10, 20, 30 (in percentage) were taken for their analyses. The pure CS:PVA and CS:PVA:KI (PCK0, PCK10, PCK20, PCK30) XRD results were recorded at for room-temperature. Also, the blend CS:PVA film (**Figure 2**) showed two hallow and smaller crystalline peaks in the sample of PCK0. Further, it was observed that 2θ peaks at 12.7° (PCK0 and PCK20 samples) and 13.74° (PCK0) and has a broad peak from 13° to 25.38° in PCK20 sample.

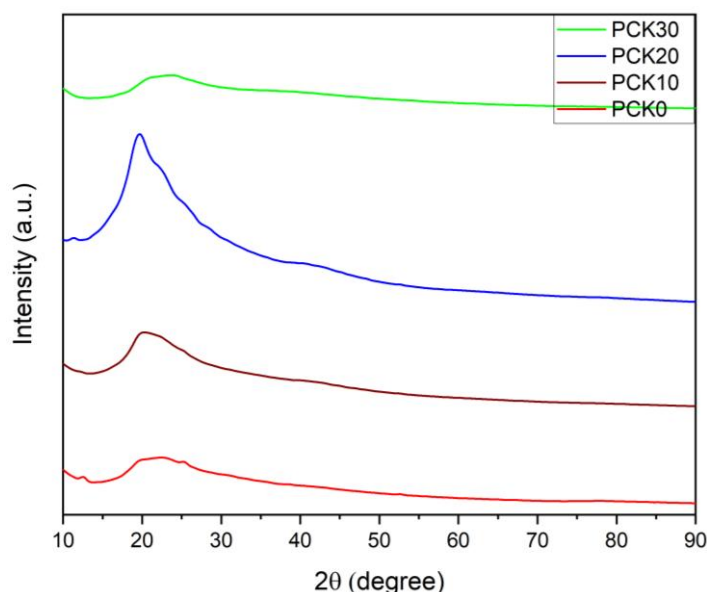


Figure 2. XRD patterns of pure CS:PVA (PCK0) and with different KI concentrations (PCK10, PCK20, and PCK30).

It is worth noting that, as shown by the giant sanctuaries, CS:PVA is less crystalline than pure CS:PVA sample and its structure is practically amorphous (Aziz et al., 2010; Yusuf et al., 2016). According to previous studies, the amorphous structure of polymer electrolytes are associated with large diffraction peaks (Malathi et al., 2010). The pure CS:PVA (1:1) blend polymer pattern shows a decrease in the intensity of main peak (19.30°), indicating intermolecular interactions between the CS:PVA molecules. The intensity of diffraction was found broader than that of CS at $2\theta = 19.30^\circ$, indicating that the presence of CS, reduced the crystallinity of the PVA (Saeedi et al., 2018). The recoded XRD patterns of PCK10,

PCK20 and PCK30 indicate that the changes in KI concentration have an impact on the crystallinity. PCK10 exhibits a lower intensity than PCK0, PCK20 shows a broad peak at 19.80° , suggesting that an increase in intensity leads to an increase in the crystallinity, as shown in **Figure 2**. It is also clear that the further increasing in the concentration of KI enhances the amorphous nature. Therefore, it is revealed that PCK20 is achieved the maximum crystallinity nature which is supporting the capacitance results of CV.

3.2 FTIR Analysis

FTIR transmittance spectra shows the interactions between atoms of pure CS:PVA and with KI electrolyte system. These interactions can cause changes in the vibrational modes of the pure sample and with KI electrolyte system, is shown in **Figure 3**.

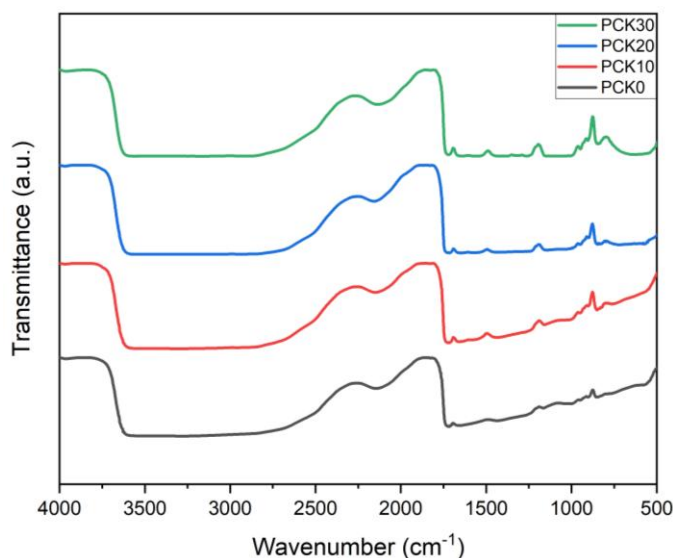


Figure 3. FTIR transmittance spectra of pure CS:PVA (PCK0) and with different KI concentrations (PCK10, PCK20, and PCK30).

Peaks at 873 cm^{-1} associated with C-H stretching were observed (Pavia et al., 2001). This peak was found to be shifted to higher wave numbers with the KI samples. In C-N stretching, the aliphatic amine group was found to have a peak at 1193 cm^{-1} , 873 cm^{-1} , and in C-C stretching, the aromatics group was found to be at 1492 cm^{-1} (Singh et al., 2013a). A vibrational band of the carbonyl group (C=O group) was observed at 1682 cm^{-1} , showing the presence of some H-bonding carbonyl groups (Singh et al., 2013b; Ambika and Hirankumar, 2016; Dong et al., 2018). The very broad peaks at $2800 - 3600\text{ cm}^{-1}$ showed that it is the O-H stretching (Pavia et al., 2001). The peaks of all CS:PVA and CS:PVA with KI, are shown in **Figure 3**.

3.3 Cyclic Voltammetry (CV) Analysis

CV (model name Squidstat Solo) is used for the analysis capacity of BPE. It is used with the stainless steel electrode $1 \times 1\text{ cm}^2$ with the two-electrode configuration of the circuit. Furthermore, the electrical properties such as potential window and capacitance of the prepared BPE were measured using CV (Tolganbek et al., 2021). Squidstat Solo is used to analyze the capacity of BPE. Squidstat Solo is used with the stainless steel electrode $1 \times 1\text{ cm}^2$ and uses the two-electrode configuration of the circuit. The potential window ranges from 1.48 to 2.0 V and is directly proportional to the capacity. The capacity is calculated using this formula (Xiang et al., 2013),

$$C = \frac{A}{2Km(V_2 - V_1)} \quad (1)$$

where, C is the capacitance and K is the scan rate, m , V_1 and V_2 represent mass of the sample, initial and final potential, A area of the CV Curve.

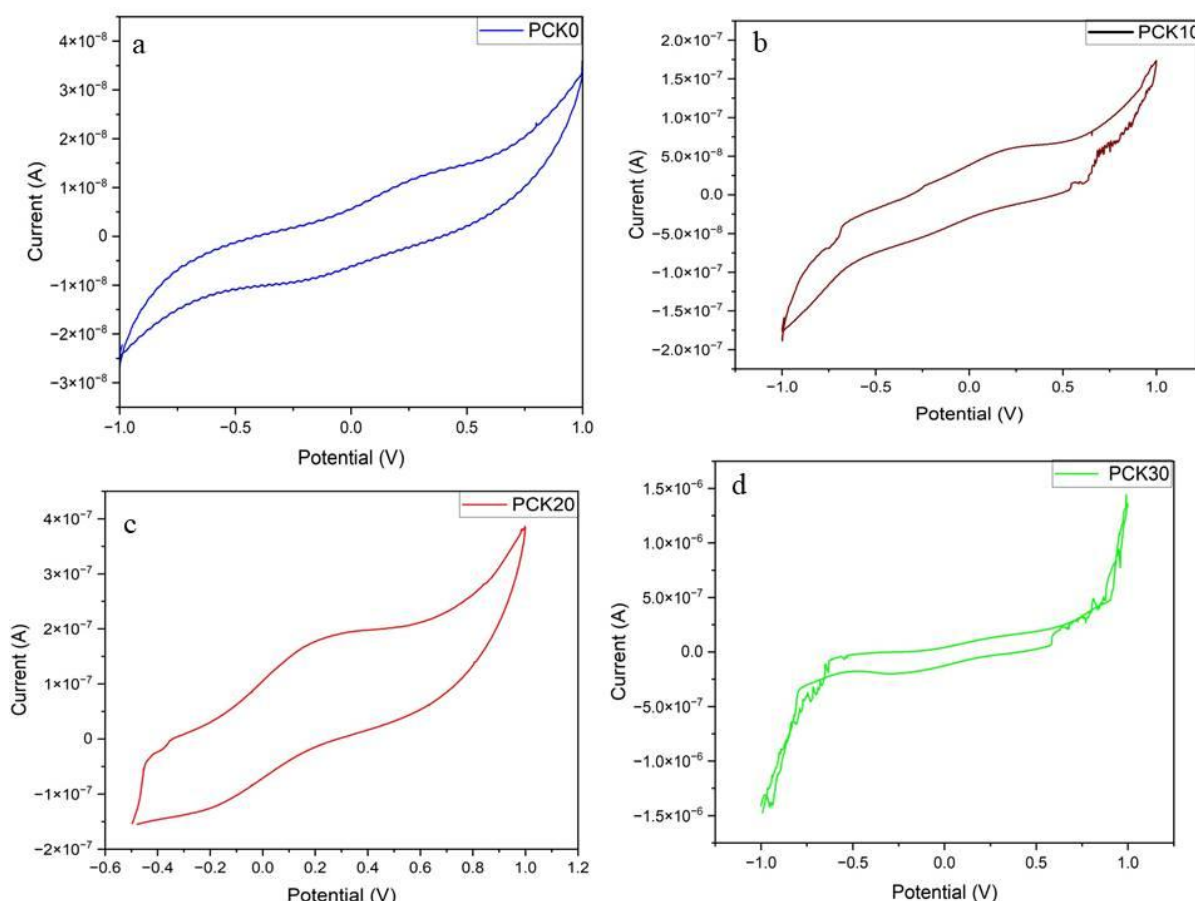


Figure 4. CV curve (a-d) with 10 mV/s scan rate of pure CS:PVA (PCK0) and with different KI concentrations (PCK10, PCK20, and PCK30), respectively.

BPE thin film sandwich between two electrode configurations, in CV mainly with three electrodes as working electrode, reference electrode and counter electrode, but here used in two electrode configurations, the first electrode is a working electrode and the second electrode is made of reference and counter electrode with 10 mV/sec coupled scan rate.

In CV analysis, the observed area under the hysteresis loop reflects the electrochemical processes occurring at the electrode surface. In **Figure 4** we see that the PCK20 is showing bigger area than the pure (PCK0) and doped (PCK10 and PCK30), which is proportional to the capacitance of doped blend materials and indicates the higher charge storage capacity since it increases the charge carrier's ionic mobility (Sharma et al., 2008; Uma et al., 2014; Tholkappiyan et al., 2018, Singh et al., 2024). The highest capacitance of 449.1 pF at 20 wt% KI with a CS:PVA blend was achieved.

4. Conclusion

In summary, XRD reveals the structural nature of the amorphous/crystalline BPE when KI is added as a salt at different concentrations. It also shows that the crystalline nature changed with salt concentration. Furthermore, it was confirmed by FTIR that the CS:PVA interpolymer material was homogeneously mixed with the KI salt, and a change was observed in the peak region of $1500\text{--}1000\text{ cm}^{-1}$, indicating other BPE groups. To understand the electrical behavior, CV calculates the potential window and capacitance of BPE with the salt concentration fluctuations as KI. The potential window is observed at 2V, 1.99V, 1.48V and 1.98V with KI 10%, 20%, 30%. This shows that 20 wt% KI with CS:PVA blended film has a highly crystalline character and supports the result of a capacity of 449.1 pF. These prepared BPE thin film can be used as a membrane for energy storage devices.

Conflict of Interest

There is no conflict interest.

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