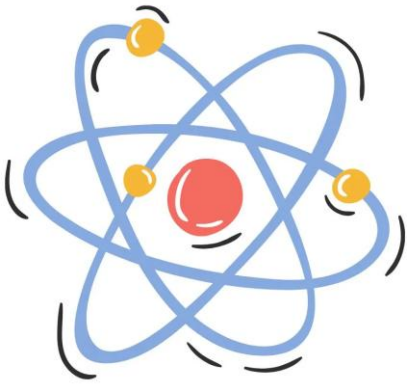

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ABSTRACT: The development of an electrolyte system using a biopolymer composite with pectin and an ionic salt (KI) is revealed in this communication. The solution cast method has been used to create composite electrolyte films of pectin biopolymer that has been produced with KI salt at different concentrations. The conductivity for pectin biopolymer-based electrolyte sheets can reach orders of 7.8×10^{-6} S/cm, a three-order increase above the conductivity of pure pectin (2.7×10^{-9} S/cm). The current communication describes electrical, structural, and optical analyses of the biopolymer electrolyte films. The electrical conductivity of the produced biopolymer electrolyte sheets is demonstrated by the Nyquist plot. The optical characteristics are represented by the POM images, which emphasize the amorphous nature of the electrolyte layers. The greatest cationic transference number reveals the ionic composition of biopolymer electrolyte sheets. The optical properties were investigated using UV-visible optical absorption spectroscopy in the 300–800 nm wavelength range.

KEYWORDS: Polymer Electrolytes, Biopolymer, Electrochemical Devices, UV Spectroscopy, Transference Number

1. INTRODUCTION

The solid-state device industry has grown significantly as a result of the smart energy storage devices' recent rapid advancements. Solid polymer electrolytes are essential to electrochemical devices because of their long lifespan, safety, dimensional stability, processability, and flexibility [1]. Poly(ethylene oxide) (PEO), poly(vinyl alcohol) (PVA), poly(vinyl pyrrolidone) (PVP), and poly(ethylene glycol) (PEG) are examples of synthetic polymers that have been widely used to create solid polymer electrolytes for a variety of applications in different electrochemical devices [2].

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These electrolytes are the building blocks of many electrochemical devices, including solar cells, batteries, fuel cells, and supercapacitors. Ionic conductors, polymer electrolytes are a special family of materials that combine the properties of polymers with electrolytes. Their conductivity usually ranges from 10^{-2} to 10^{-5} S/cm. While the flow of charge in external circuits is controlled by electrons to maintain electrical equilibrium within the cell, the movement of charge within electrochemical cells depends on the presence of cations and anions in the electrolyte [3,4]. Traditional electrolytes usually comprise diluted strong acid/base solutions, 1–2 molar salt solutions, and ionic liquids like 1-ethyl, 2-methyl imidazoliumthiocyanate. Although these solutions furnish the required cations and anions for device functionality, they suffer from issues such as volatility, corrosiveness, and evaporation, resulting in diminished device performance [5,6].

Researchers have created polymer electrolytes with a number of benefits over their liquid counterparts to address these maintenance-related issues [7]. These electrolytes, which are made by combining salts, weak acids and bases, and ionic liquids into various synthetic, natural, semi-synthetic, or biopolymer matrices, can exist in gel, quasi-solid, or solid states. The electrolytes produced by this synthesis procedure are extremely conductive and long-lasting, meeting the electrochemical requirements of the devices. Environmental sustainability is also enhanced by the substitution of biodegradable polymers for non-biodegradable synthetic ones in polymer electrolytes [8–12].

The disadvantages of synthetic polymer electrolytes, especially their high cost and lack of environmental friendliness, have been successfully addressed by biopolymer-based electrolytes. Numerous electrochemical devices, such as fuel cells, solar cells, batteries, sensors, and supercapacitors, have found widespread use for these biopolymer electrolytes [13]. Prominent discoveries highlight biopolymers with improved ionic conductivity. Pectin with an ionic liquid electrolyte, for example, have a remarkable ionic conductivity of 1.43×10^{-6} S/cm at 25°C [14]. Similarly, chitosan exhibits substantial ionic conductivity of 2.44×10^{-3} S/cm at 30°C [15], and proton conducting cellulose acetate-based electrolytes exhibit an ionic conductivity of 2.44×10^{-3} S/cm at 30°C [16]. Advancements continue with proton-conducting chitosan-sodium alginate polyion complexes, displaying an ionic conductivity of 4.2×10^{-3} S/cm at 32°C [17]. A notable composite, the corn starch: $LiClO_4$: $BaTiO_3$ polymer composite electrolyte, boasts an ionic

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conductivity of 1.84×10^{-4} S/cm at 25°C [18]. Additionally, agar: NH_4SCN biopolymer electrolyte demonstrates an ionic conductivity of 1.03×10^{-3} S/cm at 30°C [19].

In the cell walls of different plant tissues, pectin, a naturally occurring polysaccharide, acts as a binding agent [14]. Figure-1[20] shows the chemical makeup of pectin polymers. Fruit pulp, which contains high levels of pectin, is one of the major byproducts produced by the fruit and vegetable industries. Terrestrial plant cell walls and the primary and middle lamella are the main locations for pectin, a special acidic hetero-polysaccharide with a structural function. It is made up of chains of 300–1000 galacturonic acid units, which are poly(galacturonic acid) methyl esters [21,22].

Pectin consists of intricate chains linked by covalent bonds along their backbones. The specific composition of pectin varies based on factors like the source plant's species and type, the originating tissue, ripeness stage, and extraction method [23].

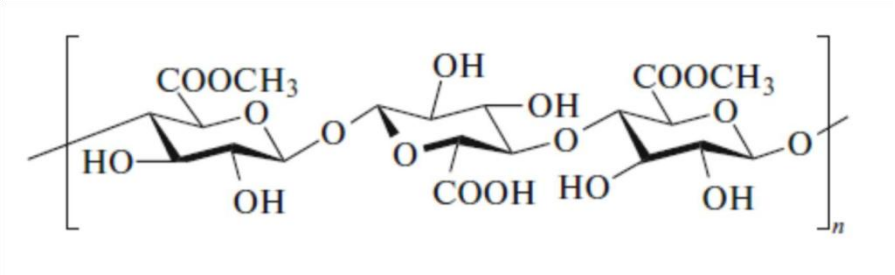

Figure:1 Chemical Structure of Pectin bio polymer [20].

In order to extract pectin, a number of intricate physicochemical processes must occur, including the breakdown and release of pectin from plant tissues and its subsequent solubility in an appropriate solvent. Temperature, pH, and time are the main determinants of this complex process. Pectin has been extracted from fruit peels using a variety of acids, including organic acids like citric acid and tartaric acid as well as mineral choices like sulfuric acid, nitric acid, and hydrochloric acid. From an economic and environmentally sustainable perspective, citric acid is the best option among these options [24].

The synthesis of the pectin biopolymer has been carried out in this article. With the addition of various potassium iodide (KI) compositions, the produced pectin is used in polymer electrolytes. The electrical and optical characteristics of the resulting biopolymer electrolytes are described.

The conductivity has been carried out usingCHI604Dinstrument. Ionic transference number has been measured with the help of CHI604D instrument and UV and POM have been discussed in detail for optical studies.

2. EXPERIMENTAL

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2.1 Synthesis of Pectin

Sample Collection: Peels from Mosambi (Citrus limetta) were sourced from local juice centers in Moradabad market. To eliminate dirt, dust, and pesticide residue, the peels were thoroughly washed. Subsequently, they were cut into small segments (depicted in Figure-2a) and blanched using boiling water to deactivate enzymes. After blanching, the mixture was manually filtered through two layers of cheesecloth or muslin cloth to separate insoluble components (pieces). The remaining insoluble materials were treated with warm absolute ethanol for 30 minutes to extract oil from the peel, followed by a washing step. To eliminate excess water, the materials were then pressed using hand pressure [14].

2.2 Method of extraction

This study used a version of the acidic extraction process (shown in Figure 2a). Initially, 25g of Citrus limetta peels were sliced into small pieces, with the white areas separated for individual weight measurement. The peels were subsequently cooked in 100 ml of deionized (DI) water, as shown in Figure 2b. Following the heating process, dilute HCL was added to the mixture to maintain a specified pH level.

Following the acid extraction, the resulting solution was filtered through cotton. Later, ethanol (96%) was added to the filtrate, as indicated in Figure 2c, and kept undisturbed for an hour to encourage ethanol-induced precipitation. This phase was designed to aid in complete precipitation, potentially concentrating polysaccharides including pectin. The resultant gelatinous precipitate was separated by standard filtering and rinsed three times with 96% ethanol. The ethanol was then recovered through distillation and reused in the pectin extraction process.

The damp pectin was then subjected to stirring to achieve a uniform solution over a span of 3-4 hours. Afterward, the homogeneous pectin gel solution was transferred to petri dishes and allowed to dry in an oven at approximately at 45°C for about 24 hours.

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Figure-2 : Synthesis of gel pectin (a) Pieces of Citrus limetta, (b) Boiling process for extraction of Pectin from cell walls, (c) Pectin floating over Ethanol.

2.3 **Preparation of Electrolyte film**

Pectin, the main polymer, was extracted using a chemical root technique from the peel of Citrus Limetta (Mosambi). Potassium iodide (KI) salt was used to make electrolyte films. Pectin and the salt were used as raw materials, and ionic liquid (IL) 1-ethyl, 2-methyl imidazoliumthiocyanate was used to incorporate biopolymer films.For polymer electrolytes, attaining high ionic conductivity is a crucial prerequisite. While the predominant technique for polyether-based electrolytes involves the inclusion of ionic salts, a recent and innovative strategy outlined in various literatures involve the incorporation of ionic liquids (ILs). Within the context of this research, a biopolymer infused with a specific IL, namely 1-ethyl, 2-methyl imidazoliumthiocyanate, was employed as a substitute for conventional aqueous phase electrolytes. Through a solution cast technique, self-supporting solid biopolymer electrolyte films were created (illustrated in Figure 3(a, b, c, d)). The ratio of the salt (wt %) was systematically altered (5%, 7%, 10% and 10% + 10wt% IL), while maintaining a consistent weight of pectin. Pectin and KI were dissolved in deionized (DI) water, and the mixture was continuously stirred until a homogenous, viscous solution was obtained. These well combined solutions were then put onto polypropylene petri dishes and left in a hot air oven set at 50°C to conduct solvent evaporation. In order to verify the improvement in conductivity, 10wt% of ionic liquid was then applied to a single film containing 10wt% KI.

Following a 24-hour timeframe, free-standing films with a thickness ranging from 130 to 150 µm were successfully obtained.

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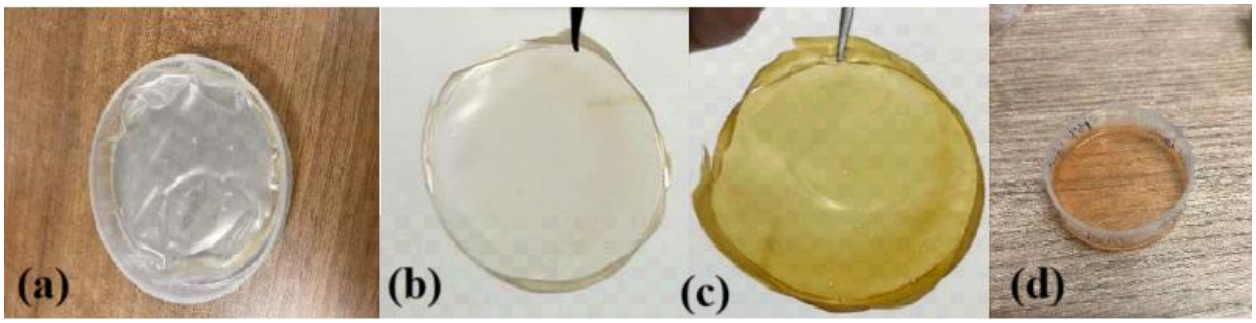

Figure-3: Free standing Biopolymer Electrolyte Films: (a) Pure Pectin, (b) With 7wt % KI, (c) With 10 wt% KI (d) With 10wt% KI + 10wt% IL(1-ethyl, 2-methyl imidazoliumthiocyanate)

3. RESULTS AND DISCUSSION

3.1 Impedance Spectroscopy

The Electrical Impedance Spectroscopy (EIS) with a CHI604D device is used to measure the conductivity of the polymer electrolyte samples. Using spring pressure, the 1x1 cm² samples were divided into smaller pieces and placed between two stainless steel electrodes.

The EIS measurements were carried out at ambient room temperature, spanning the range of 300K to 315K. Subsequently, the conductivity of the electrolyte was determined using a suitable equation.

$$\sigma = \frac{l}{A R_b} \qquad \text{..... (1)}$$

The ionic conductivity of the films was determined based on the considerations of bulk resistance (R_b), film thickness (l), and electrode area (A). The film thickness spanned from 130 to 150µm.

Table-1 illustrates the variation of ionic conductivity with salt concentration. The KI salt was taken initially upto 10% and after that 10wt% ionic liquid was also used along with KI in single film. The findings indicate that the conductivity of polymer electrolytes initially experiences an increase, reaches highest value and subsequently decreases with an increasing concentration of the respective components, and then rises again with addition of ionic liquid in salt concentration. Notably, the highest conductivity value of 7.8×10^{-6} S/cm was attained at 10% weight concentration of potassium iodide along with 10 wt% of Ionic Liquid (Table-1).

The presence of single or double maxima in dispersed polymer electrolyte systems has been documented in the literature [15]. These conductivity enhancements are commonly attributed to the presence of additional charge carriers. Lower maxima typically point to the formation of charge pair ions. The dip in conductivity observed at 5wt% and 10wt% concentrations can be attributed

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to carrier dissociation, while the subsequent increase in conductivity is linked to charge association phenomenon. [25].

Table-1 : Conductivity values of Pectin: KI Polymer Electrolytes from Impedance plot at different concentrations

Salt Concentration (%)	Conductivity
Pure Pectin	1.9×10^{-9}
5	8.6×10^{-7}
7	1.3×10^{-6}
10	2.3×10^{-7}
10+ionic liquid	7.8×10^{-6}

The AC impedance technique stands as an effective method for exploring the electrical traits of materials. In the specific context of pectin/KI biopolymer electrolytes, the determination of ionic conductivity involves employing the Cole-Cole plot, visually depicted in Figure 4.

The ionic conductivity values are calculated by extrapolating the plateau region of the conductance spectra to a zero-frequency point. Notably, the most substantial ionic conductivity emerges from the composition featuring 10wt% of KI + 10wt% (IL). As the concentration of KI within the system, is increased up to 1%, the conductivity of pectin experiences augmentation. Nevertheless, surpassing this point, a further escalation in salt concentration could potentially lead to a subsequent rise in conductivity [16, 17].

Figure 4 illustrate the Cole-Cole plot, exhibiting a semicircular pattern in the high-frequency realm and a spike in the low-frequency realm. These patterns arise from the electric double layer effect. Variations within these semicircles can be attributed to factors like the polymer's amorphous structure, the presence of charge carriers, and the mobility of ions within the electrolytes.

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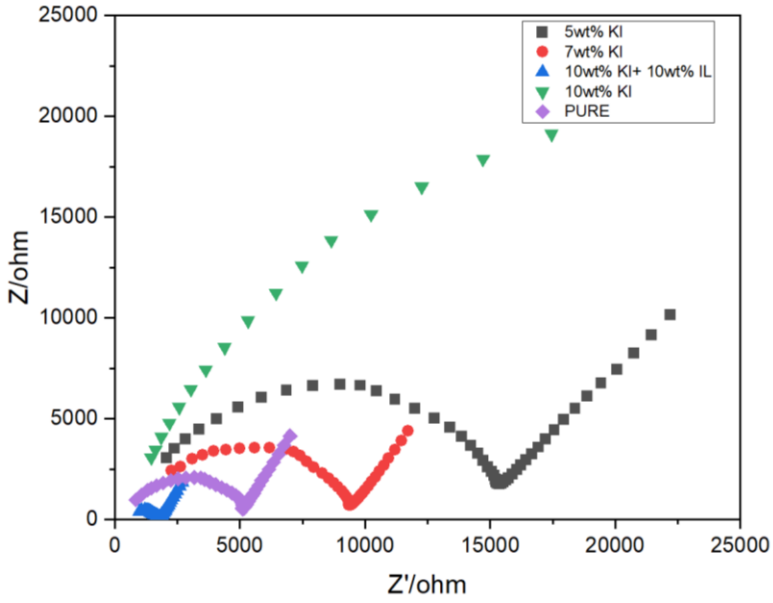

Figure-4: (Color online) Cole-Cole plot of pectin/KI biopolymer electrolytes.

To extract ionic conductivity, the bulk resistance is calculated by fitting the Cole-Cole plot with Z-view software (as specified in Table 1). The conduction mechanisms in polymer electrolytes can be explained by two phenomena: (i) polymer segment mobility and (ii) ion migration across coordinated sites within the polymer matrix. The observed increase in conductivity with the addition of KI can be attributed to an increase in the number of mobile charge carriers in the polymer electrolyte system [29].

3.2 Polarized Optical Microscopy

Polarized light transmission microscopy is a widely used technique for investigating the microstructure of transparent, optically anisotropic samples like crystals, liquid crystals, and polymers. In this approach, a microscope image is generated by observing the distribution of light intensity from a light beam that has passed through the specimen onto a viewing plane. The variations in intensity across the image are known as contrast. In most cases, the contrast arises due to the differential absorption of light in distinct regions of the specimen as it traverses them. However, variations in the images produced by polarized light microscopes may also arise from alterations in the phase of light as it traverses the specimen. Since detectors, including the human eye, primarily respond to intensity rather than phase, a meticulous analysis is essential for

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interpreting these images. It is crucial to establish a connection between the contrast observed in these images and the microscopic characteristics inherent to the specimen [30].

Polarized microscope images portray the intensity pattern that results from projecting a specific component of the local electric susceptibility tensor within the object onto the image plane. This tensor component corresponds to the alignment of the polarizer and the analyzer.

Figure 5 showcases Polarized Optical Microscopy (POM) images of three distinct films: pure pectin, pectin doped with 10wt% KI, and pectin doped with 10wt % of KI + 10wt% (IL). In the pure pectin image, noticeable voids are apparent along with shadowy patches. These voids signify the presence of crystalline regions, while the shadowy patches represent amorphous regions. This observation aligns with findings from existing literature.

A comprehensive comparative analysis of pure pectin and pectin doped with various salt concentrations (Figure 5a–c) reveals that the addition of salt amplifies the presence of amorphous shadowy regions as opposed to the crystalline bright regions.

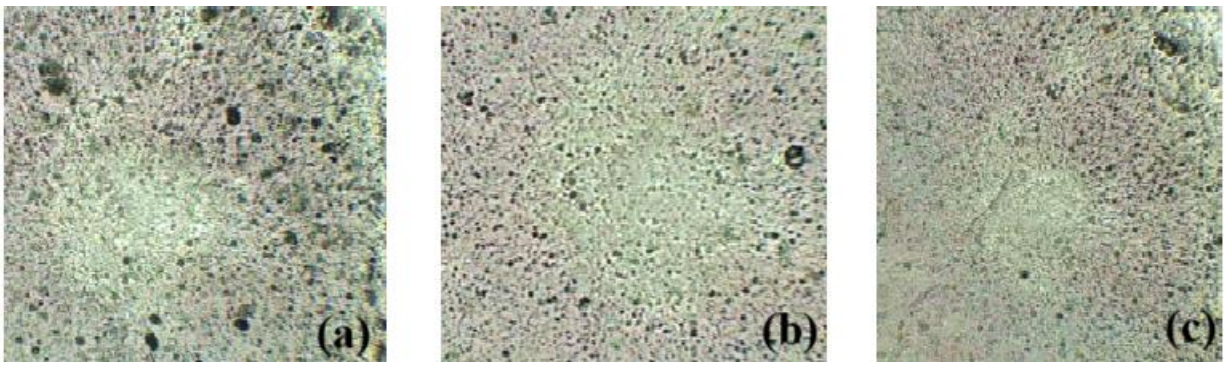

Figure-5 :POM images of (a) pure Pectin, (b) Pectin with 10wt% KI polymer electrolyte film, and (c) Pectin with 10wt % of KI + 10wt% (IL) polymer electrolyte film.

3.3 Transference ion measurement

The determination of the ionic transference number for the most conductive biopolymer electrolyte films was carried out using Wagner's DC polarization method. This method involves applying a low DC voltage amplitude across the film for a duration of 4500 seconds, as illustrated in the accompanying figure.

Throughout the course of the experiment, the current exhibited an initial rapid rise followed by a subsequent rapid decline until it eventually reached a stable state. This stabilization of the current is attributed to the occurrence of electrode polarization, a phenomenon arising from the application

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of the DC potential. This polarization induced the deposition of ions onto the surfaces of the electrodes.

The calculation of the ionic transference number (t_{ion}) was executed using Equation (2), utilizing the data extracted from Figure 6.

$$t_{ion} = \frac{initial\ current - final\ current}{initial\ current} \quad \ldots\ldots\ldots\ldots\ldots\ (2)$$

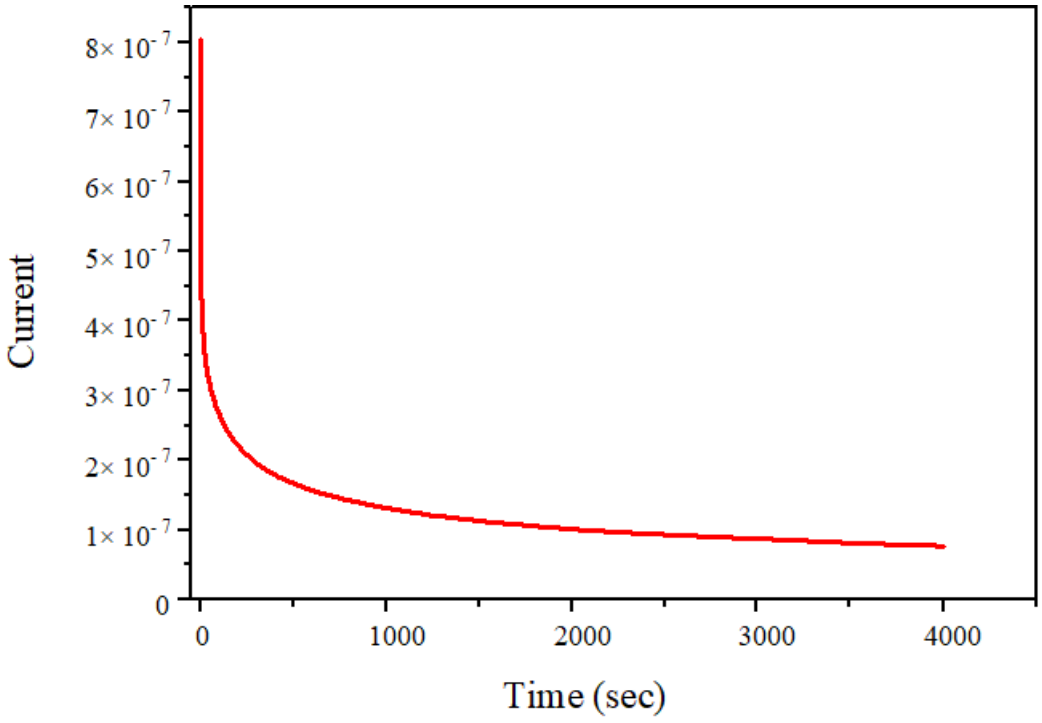

Figure-6: t_{ion} measurement of pectin doped with 10wt % of KI + 10wt% (IL) polymer electrolyte film.

3.4 UV Spectroscopy

A UV spectrum was acquired utilizing a spectrometer at room temperature. The UV Spectrometer was employed to derive the absorption spectra of the provided sample, covering a wavelength range spanning from 300 to 800 nm. This optical absorption analysis was conducted to explore the optically triggered transitions and structural characteristics of the sample. Notably, the optical absorption spectrum exhibited distinct patterns within the 320 to 410 nm range. The absorption spectra of both the pristine and doped KI in Polymer electrolytes were illustrated in Figure 8. Within biopolymers, multiple electronic transitions might occur, as documented in previous studies [31]. These optical absorption spectra could showcase a marked surge in absorption near

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the absorption edge, marking the onset of optical absorption. Figure 7 shows that the assessment of both structural and optical band gaps is accomplished through these optical absorption investigations. Ordinarily, pristine biopolymers display minimal absorption, but through incremental doping, absorption tends to steadily increase.

Absorption phenomena are typically classified into three categories: $A > 0$ represents cases where measurable absorption occurs, $A = 0$ indicates no absorption, and $A < 0$ is a proposed concept referred to as negative absorption. In the figure it is observed that the pure pectin and 5wt% KI shows negative absorption. This negative absorption is characterized by a phenomenon occurring beneath the baseline (zero line). In such instances, the emergent light intensity surpasses the incident light intensity ($I_0 < I_T$), leading to a calculated transmittance value exceeding 100% ($T = I_T/I_0 > 100\%$). Negative absorption (NA) or negative absorption emission (NAE) is a phenomenon that might arise from emission characteristics in the presence of excitation light [32].

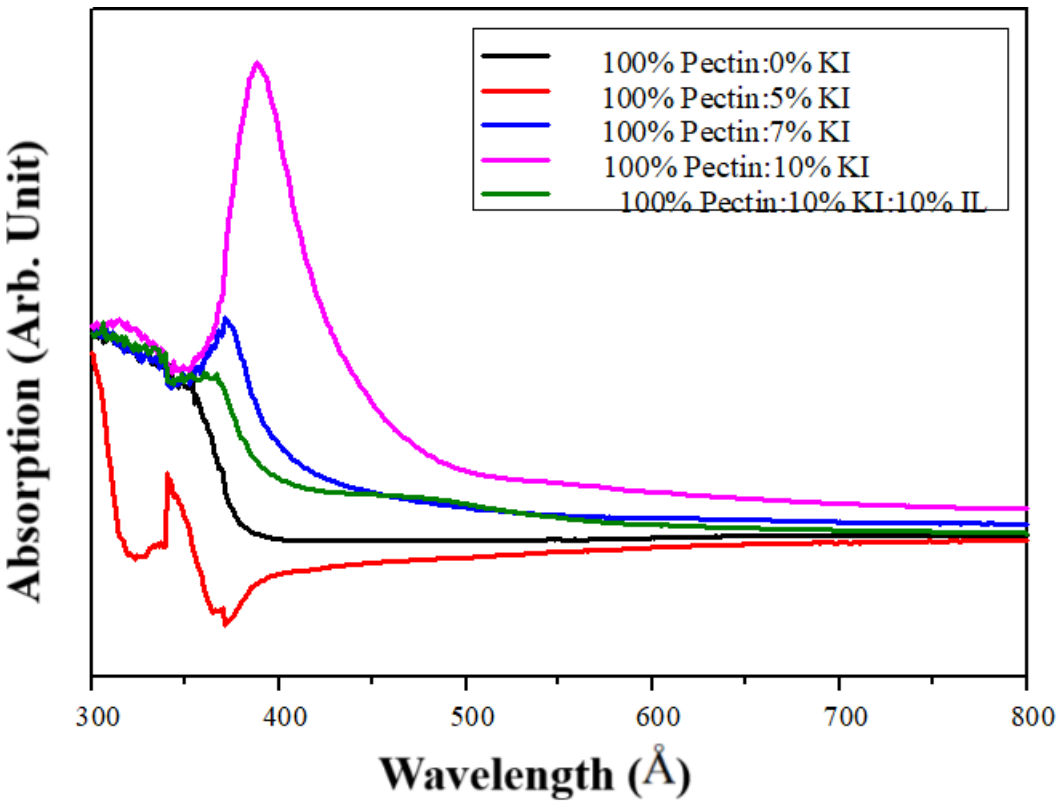

Figure7: U-V Spectroscopy of the prepared polymer electrolyte films.

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4. Conclusion

Using potassium iodide as a salt and IL for conduction, pectin-based biopolymer electrolytes were created using the solution casting technique. Among other electrical properties, ionic conductivity, mobility, and transference numbers were determined. The 10wt% plus 10wt% doping system Impedance study confirmed that IL has the best dielectric properties and the highest ionic conductivity ($7.8x10^{-6}$ S/cm). The greater conductivity zone's lower crystallinity and more amorphous character are confirmed by POM optical measurements. The ion transference number (ion) testing indicates that the biopolymer that was produced has an ionic nature of 0.90. Furthermore, the U-V Spectroscopy illustrates how the addition of salts alters the intensity of absorption spectra, and in a small number of salt concentration cases, negative absorption has also been noted. In order to construct optoelectronic devices, photonic devices, and biointegrated sensors, the emission of light is revealed by negative absorption.

Statements and Declaration

The corresponding author has declared that there is no conflict of any financial or non-financial interest that is directly or indirectly related to the work submitted for publication.

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