

IONIC CONDUCTIVITY, STRUCTURAL ANALYSIS AND BIODEGRADABLE PROPERTIES OF 2-HYDROXYETHYL CELLULOSE DOPED AMMONIUM CHLORIDE WITH PLASTICIZED ETHYLENE CARBONATE SOLID BIOPOLYMER ELECTROLYTE

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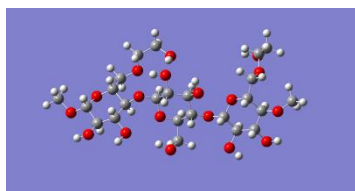
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Graphical abstract



Abstract

Solid biopolymer electrolytes (SBEs) systems based on 2-hydroxyethyl cellulose (2-HEC), doped with ammonium chloride (AC) and various amounts (2-6 wt%) of ethylene carbonate (EC) as a plasticizer, were prepared using the solution casting technique. The ionic conductivity of the SBEs was analyzed using electrical impedance spectroscopy (EIS), with the sample containing 2 wt% of EC showing the highest conductivity at 3.43×10^{-4} S/cm. The physical appearance and structural properties of the SBEs were also tested. Fourier transform infrared spectroscopy (FTIR) demonstrated a vibrational spectrum of composite SBEs, confirmed through comparison with Gaussian analysis, with a peak value at 2928 cm^{-1} indicating the C-H stretching of 2-HEC. Additionally, X-ray diffraction (XRD) revealed a broadened amorphous peak for 2-HEC and a distinct peak indicating AC crystallinity. The biodegradable properties of the SBEs were assessed through soil burial. From this, decomposition of material can be observed in natural soil to prevent long-term pollution. The hydroxyl groups in the HEC molecule facilitates enzymatic breakdown from cellulase.

Keywords: Solid biopolymer electrolytes, Ionic conductivity, X-ray diffraction, Fourier transform infrared spectroscopy, Biodegradable

Abstrak

Sistem elektrolit biopolimer pepejal (SBEs) berasaskan selulosa 2-hidroksietil (2-HEC), didopkan dengan ammonium klorida (AC) dan pelbagai jumlah (2-6 wt%) etilena karbonat (EC) sebagai agen pemplastik telah disediakan menggunakan teknik penuangan larutan. Kekonduksian ionik SBEs dianalisis menggunakan spektroskopi impedans elektrik, dengan sampel mengandungi 2% berat EC menunjukkan kekonduksian tertinggi pada 3.43×10^{-4} S/cm. Penampilan fizikal dan sifat struktur SBEs juga telah diuji. Spektroskopi inframerah transformasi Fourier menunjukkan spektrum getaran, disahkan melalui perbandingan dengan analisis Gaussian, dengan nilai puncak pada 2928 cm^{-1} menunjukkan regangan C-H selulosa 2- hidroksietil. Selain itu, pembelauan sinar-X mendedahkan puncak amorfus yang diperluaskan untuk 2-HEC dan puncak yang berbeza yang menunjukkan kehabluran AC. Sifat biodegradasi SBEs telah dinilai melalui pengebumian tanah. Daripada ini, penguraian bahan tersebut dapat diperhatikan dalam tanah semula jadi untuk mengelakkan pencemaran jangka panjang. Kumpulan hidroksil dalam molekul HEC memudahkan pemecahan enzimatik daripada selulase.

Kata kunci: Elektrolit Biopolimer Pepejal, Kekonduksian Ion, Pembelauan X-Ray, Spektroskopi Inframerah Transformasi Fourier, Boleh Terbiodegradasi

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1.0 INTRODUCTION

Electrochemical devices such as batteries have been used around the world to produce energy through a chemical reaction known as oxidation and reduction. The atoms of anode materials will release electrons during the oxidation process, which will then travel to the cathode through a wire that connects the two electrodes. The reduction process takes place at the cathode, where positive ions in the electrolyte can receive electrons and transform into atoms [1]. Ions move between the anode and cathode contacts in a battery, which is made up of electrolyte [2].

Liquid, gel, and solid electrolytes are the three main types of electrolytes. Solid electrolytes are useful for making energy storage devices like supercapacitors and batteries that are safe, flexible, stretchable, wearable, and self-healing [3]. There is a growing need for SBEs because of the rising number of expensive and non-environmentally friendly battery products. Bio-based polymers have become more important in recent years because they could be used instead of or in place of regular polymers. Natural polymers are another name for bio-based polymers. It is naturally made by living things and can be found in our surroundings. Instead of using non-biodegradable polymers, they can help solve many problems, such as lowering costs, slowing down global warming, and other economic and environmental issues [4]. Since natural polymer is non-toxic, good in thermal stability, renewable and safe for humans, it has been studied as the best alternative from synthetic polymer.

Using the SBEs, the researchers are finding solutions to reduce the environmental effect since it showed a positive aspect [5]. To make SBEs, inorganic salts must be dissolved in a functional polar polymer framework [6]. There are two types of polymers which were natural polymers and synthetic polymers. Cellulose is an example of a natural polymer [7]. Due to its capacity to decompose, cellulose is one of the most abundant materials on Earth. It is also one of the biopolymers that are used for this purpose. Research into cellulose derivatives, including nanocomposite membranes, is at an all-time high because of its biodegradability, affordability, and low production costs [8].

2-hydroxyethyl cellulose (2-HEC) is one of the celluloses proven to be suitable for SBEs host materials [9]. As a biocompatible, good thickening and stabilising agent, and a tendency for adhesion products and film-formation, it has gotten a lot of attention from both industry and study groups [10]. 2-HEC is a very water-soluble carbohydrate because it has a hydroxyethyl group ($\text{CH}_2\text{-CH}_2\text{-OH}$) [11]. This is due to the chemical alteration that 2-HEC goes through, where some of the hydroxyl groups on the glucose units are replaced with hydroxyethyl groups [12]. The helical microfibril structure of cellulose gives it both a crystalline and an amorphous form, which enhances ion mobility. These

characteristics make cellulose an excellent choice for the host material of SBEs [9]. Pure 2-HEC SBEs has an ionic conductivity of about 10^{-6} Scm^{-1} , which is not enough for industrials use [10].

To increase conductivity, cellulose based biopolymer electrolyte requires an ionic donor. One approach to improve ionic conductivity with ionic dopant is ammonium chloride (AC) [13]. AC was chosen as ionic dopant due to the ionic conductivity of the SBEs is enhanced by the H^+ of the ammonium ion group NH_4 from ammonium salt [14]. Furthermore, plasticizers were introduced in polymer-salt system to enhance the ionic conductivity. A good plasticizer should possess high dielectric constant, low volatility, and high molecular weight. EC is selected as a plasticizer due to its ability to dissolve a significant amount of electrolyte, greater boiling temperature (248°C), and high dielectric constant ($\epsilon = 89.1$) [15]. The present work aiming to develop a new biodegradable SBEs with 2HEC doped AC plasticized with EC. The effect of AC and various amount of EC towards the ionic conductivity of SBEs were further analyzed.

2.0 METHODOLOGY

There are 3 types of systems that were prepared; pure polymer (H0), polymer-salt (HA0), and polymer-salt-plasticizer (HAE). SBEs were prepared by using a solution casting technique using 2-HEC as a polymer host, AC as an ionic dopant and EC as a plasticizer. The formulation of AC and EC was determined using weight percentage calculation. Table 1 shows the designation and formulation of SBEs. 2.0g of 2-HEC was added into distilled water and stirred until it was completely dissolved. The 10wt% of AC were added to the 2-HEC solution and stirred again until the solutions became homogeneous. The mixture was cast into petri dish and were kept drying at room temperature inside desiccator with silica gel. For the plasticized sample, the highest conductivity of 2-HEC-AC (10wt%) SBEs was chosen to be added with plasticizer. The same method was used to produce 2-HEC-AC-EC SBEs by adding different amount of EC as plasticizer into 2-HEC-AC mixture.

Table 1 Designation of SBEs

Designation	Formulation				
	2-HEC (g)	AC (wt.%)	AC (g)	EC (wt.%)	EC (g)
H0		0	0	0	0
HA0				0	0
HAE2	2	10	0.222	2	0.045
HAE4				4	0.092
HAE6				6	0.141

Characterization techniques include electrical impedance spectroscopy (EIS), X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, computational methods (Gaussian09W), and evaluation of biodegradation properties, aiming to understand their electrochemical, structural, chemical, and environmental aspects. The impedance spectra of EIS were measured in a range frequency of 50 Hz to 1 MHz. For XRD technique, Cu K α radiation source with diffraction angle of 5 – 60° was used to determine amorphousness of SBEs. FTIR-ATR (Attenuated Total Reflection) using germanium crystal was used to observe the vibrational spectra of SBEs system. Soil-burial test was done to evaluate the biodegradation of SBEs system. It was done by measuring the rate of change of SBEs' mass buried in soil for 4 weeks period.

3.0 RESULTS AND DISCUSSION

3.1 Physical Appearance of SBEs

The SBEs sample was seen to be clear, flexible, and sticky. As the amount of plasticizer increased, it became easier to remove from the petri dishes, and the film turned clear, transparent, and yellowish. The sample and its physical look of 2-HEC-AC-EC SBEs are shown in Figure 1.

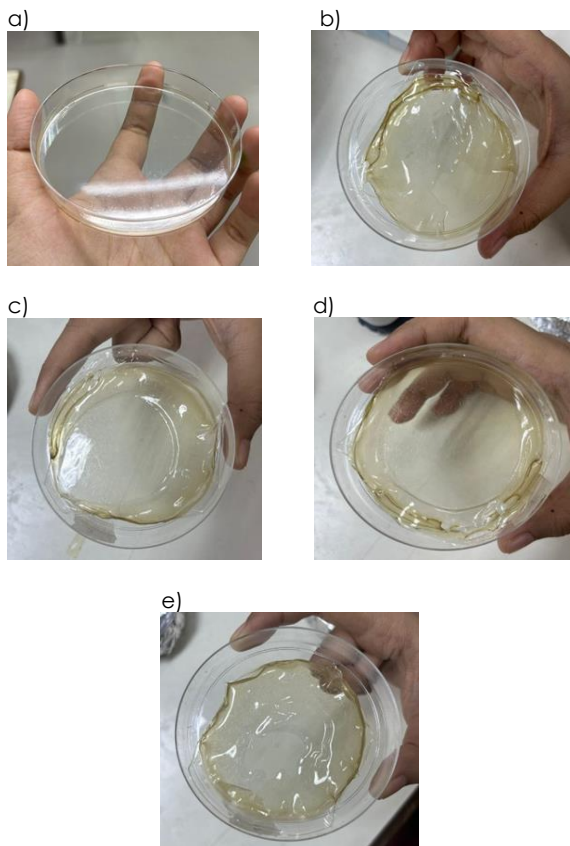


Figure 1 Clear and flexible SBEs film of (a) H0, (b) HA0, (c) HAE2, (d) HAE4, (e) HAE6

3.1.1 Impedance Analysis

Cole-Cole Plot at Room Temperature

The impedance of SBEs was analyzed using Cole-Cole plots at room temperature. Figure 2 shows the plot for 2-HEC-AC-EC, used to determine resistance (R) and reactance (X) as real impedance (Z_r) and negative imaginary impedance ($-Z_i$). Figure 2 illustrates SBEs Cole-Cole plot, displaying a reduced semicircle at high frequencies and inclined spikes at low frequencies. These features indicate the bulk effect and electrode-electrolyte interface, respectively. The study assessed ionic conductivity by deriving bulk resistance (R_b) from the semicircle's intercept on the Z_r axis for each sample.

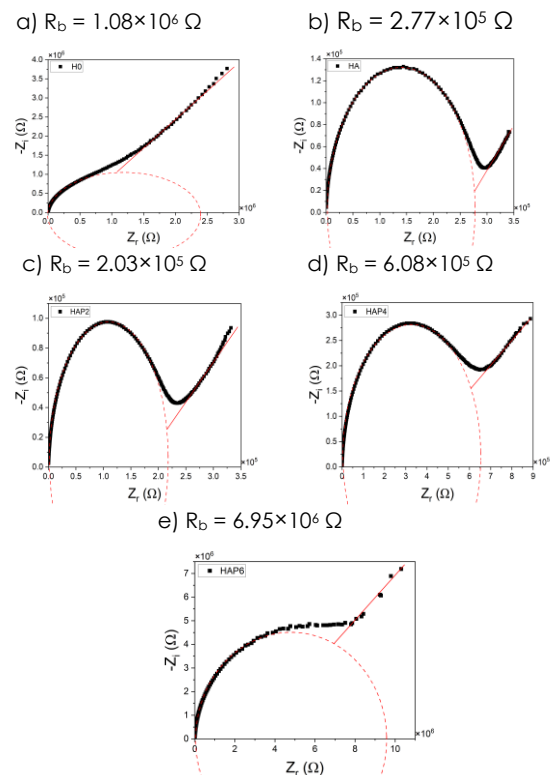


Figure 2 Cole-cole plot of SBEs for (a) H0, (b) HA0, (c) HAE2, (d) HAE4 and (e) HAE6 at room temperature

Ionic conductivity at Room Temperature

Figure 3 shows the room temperature conductivity for 2-HEC-AC-EC SBEs. It found that the sample with 2wt% ethylene carbonate (EC) exhibited the highest conductivity, measuring 3.43×10^{-4} S/cm. This increase in conductivity is attributed to enhanced salt dissociation, which facilitates greater ion mobility. Conversely, the lowest conductivity, 2.93×10^{-5} S/cm, was observed at 6wt% EC concentration within the SBEs.

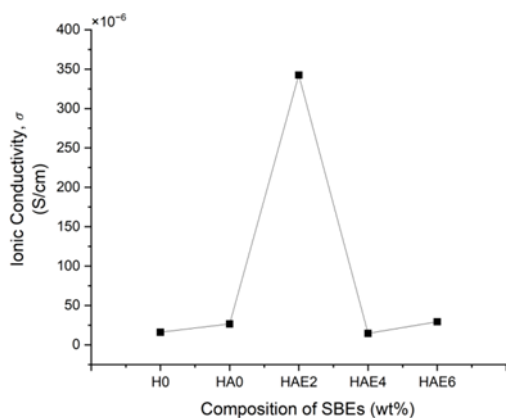


Figure 3 Room temperature conductivity for 2-HEC-AC-EC SBEs

The higher EC content in HAE SBEs increased ionic conductivity by boosting charge carriers [16]. However, above 2wt% EC, conductivity decreased due to fewer charge carriers and reduced mobility. This is due to formation of ion pairs. Excess plasticizers stabilize ion pairs of dissociated AC. Another reason is that high content of plasticizers can disrupt the polymer matrix structure leading to less organized system where ions cannot move freely.

3.2 IR Analysis

IR analysis was conducted on pure 2-HEC, AC, EC, 2-HEC-AC, and 2-HEC-AC-EC to study their functional groups and molecular interactions in SBEs. Both computational simulations using Gaussian 09W software and experimental using FTIR were employed. All of the computational IR need to multiplied by 0.95 to closely align with experimental IR. Figure 5 depicted the graph of IR spectrum of pure 2-HEC which comparable to the experimental IR spectrum in Figure 4. Theoretical data can be used to validate experimental data. Combining theoretical and experimental approaches can provide more comprehensive understanding of SBEs system whereas FTIR provides empirical data that reflects real-world interactions while Gaussian09W allows the exploration of molecular properties under idealized conditions.

Pure 2-HEC's structure was optimized using Gaussian software to simulate its IR spectrum, ranging from 0 to 3500 cm^{-1} . Experimental and simulated results aligned, showing key peaks: C-H stretching at 2928 cm^{-1} and O-H stretching at 3281 cm^{-1} . Figure 4 illustrated the FTIR analysis of pure 2HEC. It revealed that the significant peaks: O-H stretching at 3390 cm^{-1} , axial symmetric C-H stretching at 2921 cm^{-1} , symmetric C-H stretching at 2867 cm^{-1} , CH_2 bending at 1448 cm^{-1} , OH bending at 1353 cm^{-1} and 1311 cm^{-1} , C-O stretching at 1049 cm^{-1} , and C-O-C stretching at 884 cm^{-1} .

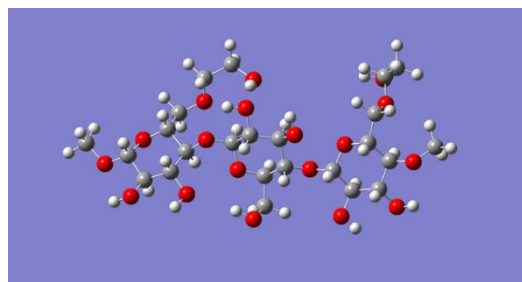


Figure 4 Optimized structure of pure 2-HEC from GaussView (white - hydrogen, red - oxygen, grey - carbon)

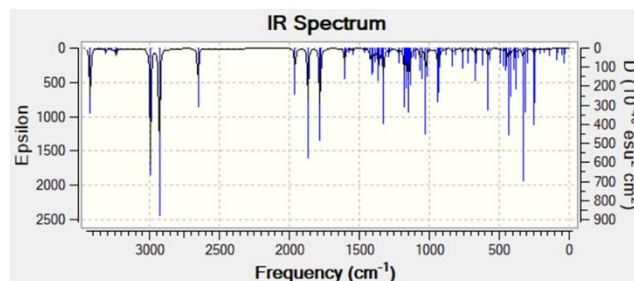


Figure 5 IR spectrum of pure 2-HEC by Gaussian analysis

The structure of pure AC was drew using GaussView (Figure 6) and calculated using Gaussian analysis (Figure 7). The Gaussian IR spectrum of pure AC, highlighting N-H stretching at 1291 cm^{-1} and N-H bending at 1147 cm^{-1} . In the FTIR spectra, wavenumbers at 3183 cm^{-1} indicate the asymmetrical stretching of the N-H bond in the NH_4 group. The N-H stretching is detected at 2973 cm^{-1} with a lower wavenumber. The N-H bending vibration is represented at 1639 cm^{-1} , and the NH_4 stretching vibration is indicated by a strong peak at 1402 cm^{-1} .

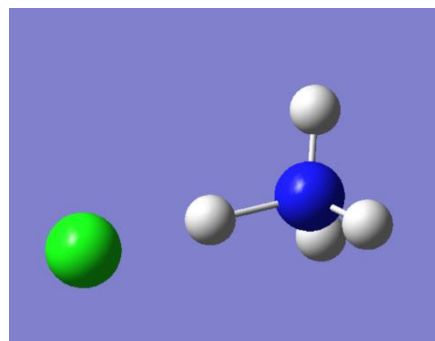


Figure 6 Optimize structure of Pure AC from GaussView (white - hydrogen, green - chlorine, blue - nitrogen)

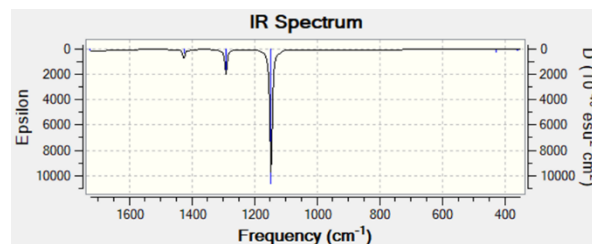


Figure 7 IR spectrum of pure AC by Gaussian analysis

The Gaussian program effectively modeled and tested the structure of pure EC as illustrated in Figure 8. Figure 9 highlighted the peaks at 1892 cm^{-1} for C=O stretching, 1454 cm^{-1} for C-H wagging, 1046 cm^{-1} for C-H twisting, and 718 cm^{-1} for C-C stretching. Functional groups in pure EC were identified using experimental techniques. The C-H bending of EC appeared as peaks at 1391 cm^{-1} . Experimental spectra showed a doublet peak for C=O stretching at 1788 cm^{-1} and 1769 cm^{-1} . Additionally, CH₂ twisting modes were observed as merged peaks forming a shoulder at 1230 cm^{-1} and 1216 cm^{-1} .

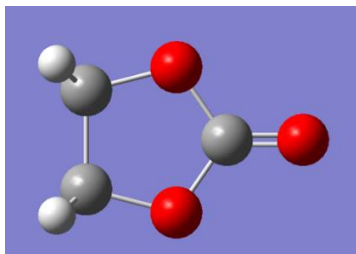


Figure 8 Structure of pure EC from GaussView (red - oxygen, grey - carbon, white - hydrogen)

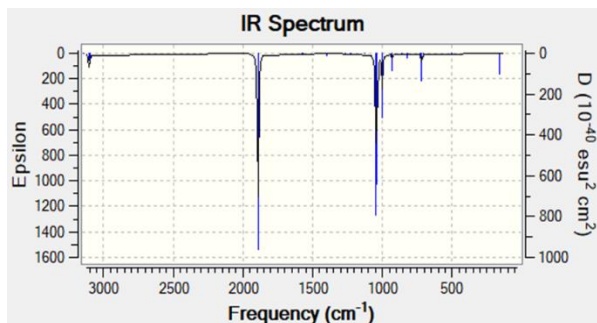


Figure 9 IR spectrum of pure EC by Gaussian analysis

Figure 10 shows the IR spectrum of 2-HEC-AC shows key peaks: O-H stretching at 3354 cm^{-1} , C-O stretching at 1636 cm^{-1} (shifted from 1590 cm^{-1} in 2-HEC), asymmetric C-H stretching at 2931 cm^{-1} , and symmetrical stretching of O-H at 1456 cm^{-1} and C-H at 1355 cm^{-1} . The changes in C-O stretching peak are attributed to the interaction between salt and polymer. The addition of NH₄Cl strengthens the C-O stretching peak at 1051 cm^{-1} . A weak peak at 888 cm^{-1} indicates C-H bending due to interactions between 2-HEC and AC.

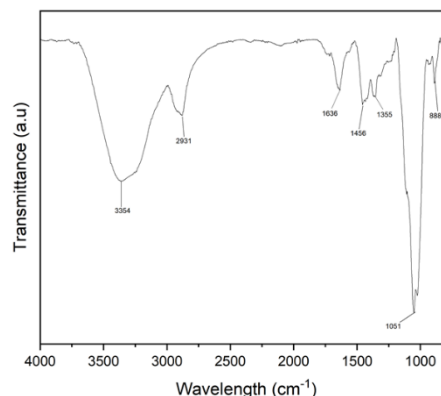


Figure 10 FTIR spectra of 2-HEC-AC (HA0) SBEs

Figure 11 displays experimental FTIR results for 2-HEC-AC-EC SBEs with varying EC concentrations (2-6wt%). The spectra range from 4000 to 650 cm^{-1} , showing five prominent peaks at 3413 cm^{-1} , 1649 cm^{-1} , 1453 cm^{-1} , 1357 cm^{-1} , and 1050 cm^{-1} . The peak at 3413 cm^{-1} indicates O-H stretching bonds. EC concentration increased, this peak shifted to lower wavenumbers and broadened.

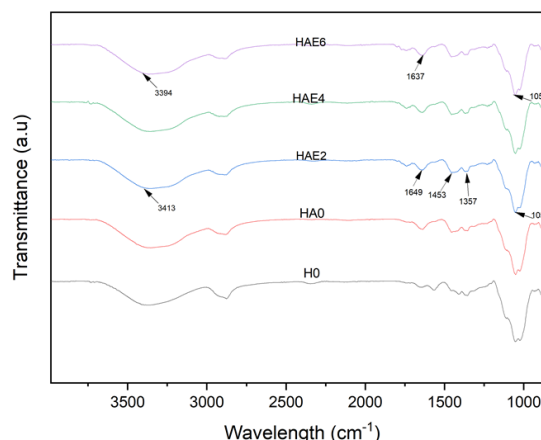


Figure 11 FTIR results for 2-HEC-AC-EC SBEs

In previous research, the band at 1700 cm^{-1} corresponds to C=O stretching in EC, shifting to 1637 cm^{-1} in HAE6 due to interactions with 2-HEC. When EC is introduced into the 2-HEC-AC SBEs system, the C=O peak disappears due to polymer complexation. Figure 11 shows peaks at 1453 cm^{-1} (O-H stretching) and 1357 cm^{-1} (symmetrical C-H stretching) in 2-HEC. These peaks decrease in intensity with increasing EC concentration. In HAE2, the C-O stretching band shifts to 1055 cm^{-1} , attributed to the COOH group in EC, showing a shift from 1050 cm^{-1} [17].

3.3 XRD Analysis

Figure 12 is illustrated the XRD spectra of 2-HEC, 2-HEC-AC and 2-HEC-AC-EC SBEs. The XRD patterns of pure 2-HEC SBEs, showing a broad peak indicative of an amorphous structure between 15° and 28° . This broad diffraction peak at 21.0° confirms the amorphous nature of pure 2-HEC, characteristic of cellulose-based materials [18]. Converting pure 2-HEC into SBEs and incorporating salt and plasticizers reorganizes the 2-HEC chain arrangement, further enhancing its amorphous nature [19].

All samples exhibit a broad peak in their XRD spectra, indicating that the SBEs are amorphous. Figure 6 shows minimal to no peak shifting with the addition of EC. This suggests that EC enhances the flexibility (amorphous nature) of the polymer [21] and creates more spaces for ion coordination. These factors facilitate easier movement of ions through the 2-HEC chain, enhancing the ionic conductivity of the SBEs [10].

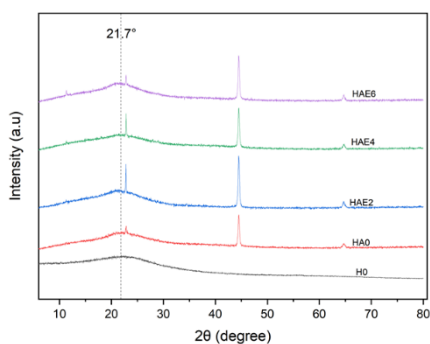


Figure 12 XRD spectrum of 2-HEC, 2-HEC-AC and 2-HEC-AC-EC SBEs

Amorphous properties in SBEs are crucial for facilitating ion movement. The broadness of XRD peaks (FWHM) and smaller crystallite sizes indicate higher amorphous content in SBEs. AC can induce tiny polycrystalline peaks in composite SBEs films around 2θ angles of approximately 44.5° . Increasing plasticizer concentration like EC enhances amorphousness by disrupting polymer chain hydrogen bonds and increasing molecular mobility. This broadens XRD peaks compared to un-plasticized SBEs, suggesting greater amorphous content and smaller crystallite sizes [21]. Improved amorphousness supports ion transport through the polymer backbone, influencing SBEs' ionic conductivity, where ion migration primarily occurs in amorphous regions.

3.4 Biodegradable Properties

SBEs are evaluated for their biodegradability using a soil burial test, which assesses their environmental impact over time through microbial activity. Figure 13 shows the increasing weight loss percentage of 2-HEC-

AC-EC SBEs over 4 weeks, indicating their breakdown in soil.

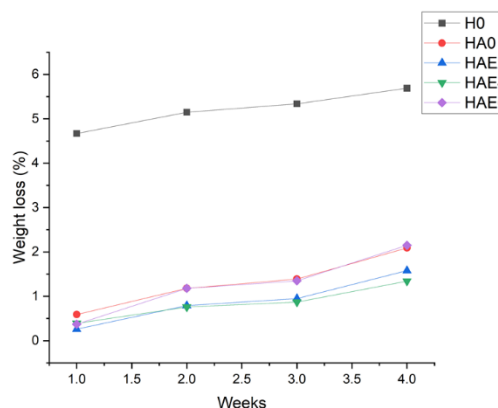


Figure 13 Weight loss of 2-HEC-AC-EC SBEs in weeks

HEC-based SBEs show higher biodegradation compared to those with AC and EC additives. Soil microorganisms break down HEC's cellulose backbone easily, utilizing it as nutrients, leading to significant weight loss [20]. HEC's biodegradability is due to its structure with hydroxyl (-OH) groups that enzymes can readily break down, unlike cellulose with higher substitutions that resist degradation.

4.0 CONCLUSION

In this study, 2-HEC-AC-EC SBEs with varying EC plasticizer percentages (0, 2, 4, and 6 wt. %) were prepared using solution casting. The resulting SBEs were transparent and uniform. EIS measurements showed the highest ionic conductivity at room temperature (303K) was 3.43×10^{-4} S/m for HAE2 SBEs, indicating that adding EC increases conductivity by enhancing mobile charge carriers. FTIR analysis identified O-H, C=O, N-H, and C-H stretching peaks, confirming molecular interactions in the 2-HEC-AC-EC system. XRD confirmed the amorphous nature of H0 and some polycrystalline peaks. Biodegradation tests showed H0 had the highest weight loss, indicating that SBEs with 2-hydroxyethyl cellulose (HEC) degrade more easily due to their structure compared to cellulose with higher substitution levels.

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Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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