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MORPHOLOGICAL INSIGHTS INTO STARCH-SODIUM HYDROGEN SULFATE COMPLEX MEMBRANES VIA MACRO AND MICRO-SCALE ANALYSIS

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ABSTRACT

This research investigates the transformation of starch-based membranes when combined with sodium salts, focusing on their physical and structural changes. Membranes were fabricated using a solution casting method, incorporating starch, sodium hydrogen sulfate, distilled water, and glycerin. The pristine membrane exhibited a clear and even surface, reflecting a homogeneous polymer network. However, introducing sodium hydrogen sulfate at different levels led to noticeable alterations in the membrane's appearance, such as increased cloudiness and structural irregularities. Advanced imaging techniques, including high-resolution photography, scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS), were employed to analyze the material. These methods revealed the development of both large-scale and fine-scale structures, as well as the even dispersion of sodium ions throughout the polymer network. The interaction between the salt ions and the polymer chains disrupted the balance between crystalline and amorphous regions, promoting a shift toward greater amorphous character. This change affected the membrane's transparency and light-diffusing capabilities. The results offer valuable insights into how varying salt concentrations can alter the physical and structural attributes of starch-based membranes, potentially expanding their utility in various material science applications.

Keywords: solid biopolymer electrolyte, starch, sodium hydrogen sulphate, morphology.

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1. INTRODUCTION

The increasing global focus on sustainability and environmental preservation has driven significant interest in the development of materials derived from natural sources [1]. Starch, a naturally occurring biopolymer, has gained considerable attention as a viable alternative to synthetic polymers due to its abundance, cost-effectiveness, and ability to break down naturally in the environment [2]. Composed of two primary components, amylose and amylopectin, starch exhibits a unique molecular structure that lends itself to a wide range of applications. While its potential has been extensively studied in fields like biodegradable packaging and medical devices, its role in more advanced technological applications, such as solid-state electrolytes, remains relatively unexplored [3,4]. This gap presents an opportunity to expand the use of starch-based materials into areas requiring innovative, sustainable, and high-performance solutions, particularly in energy storage and electrochemical systems.

The integration of sodium salts into starch-based systems presents a promising strategy to improve the performance and versatility of starch-derived materials. Sodium salts, such as sodium hydrogen sulfate, function as effective modifiers that interact with the starch polymer network, breaking down its crystalline structure and increasing its amorphous character. These interactions result in notable alterations to the membrane's properties, including enhanced ionic conductivity, improved mechanical durability, and changes in surface texture. In starch-sodium systems, the formation of granular voids and reduced polymer chain entanglement amplifies the impact of salts on thermal and structural behaviors, with higher salt concentrations preventing the recrystallization of amylose-rich matrices. These interactions between salt and starch molecules play a critical role in modifying key properties, such as gelatinization and gel strength. [5]. Importantly, these effects are driven by the accessibility of salts and their direct interactions with the polymer, rather than changes in hydration, providing valuable guidance for the development of starch-based solid polymer electrolytes. Research by Chiotelli on wheat and potato starches combined with sodium chloride further underscores the influence of salt concentration on starch properties. The study revealed that sodium chloride alters gelatinization parameters, including onset, peak, and end temperatures, as well as gelatinization enthalpy and storage modulus. These changes were linked to the dual role of salts in modifying water properties and directly interacting with starch polymers, highlighting the intricate relationship between solute concentration and starch behavior [6]. Similarly, studies have shown that at low concentrations, salts are excluded from the starch matrix, while at higher concentrations, they bind more effectively, significantly impacting the structural integrity of the material [7]. In systems where starch was combined with various salts, such as sodium chloride, sodium sulfate, potassium chloride, calcium chloride, and sodium phosphate, researchers observed notable changes in viscosity and gelatinization temperature. Rheological

analyses demonstrated that these salts influence starch-gum interactions under different shear conditions, emphasizing their role in altering the texture and structural dynamics of starch-based complexes [8]. Further evidence from Vilwock and BeMiller supports the transformative effects of sodium salts on starch. Their work illustrated that salts, depending on their position in the lyotropic series, exert distinct effects on starch gelatinization. For instance, sodium sulfate limited granule swelling due to its lyotropic nature and the creation of a Donnan potential, while sodium chloride failed to inhibit swelling, leading to granule pasting. These findings underscore the complex mechanisms by which salts influence starch behavior, including granule swelling, gelatinization inhibition, and reaction efficiency [9]. By carefully adjusting salt concentrations, it is possible to fine-tune the structural and functional properties of starch-salt complexes, making them highly adaptable for a range of applications, particularly in advanced energy storage technologies. This approach not only enhances the performance of starch-based materials but also opens new avenues for their use in innovative and sustainable solutions.

Analyzing the physical structure of starch-sodium salt membranes is essential for uncovering how their composition and properties evolve. Advanced imaging tools, such as scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS), offer a comprehensive view of surface characteristics and the distribution of elements within the material. When combined with traditional optical imaging, these techniques enable researchers to assess how varying salt concentrations influence the membrane's visual and textural properties, such as transparency, roughness, and uniformity. Observable changes, like heightened cloudiness or the emergence of unique microscopic patterns, can be attributed to the dynamic interactions between starch polymers and salt ions. While earlier research has briefly addressed these physical transformations, there is a significant lack of detailed studies utilizing advanced imaging to investigate the morphology of starch-sodium salt composites. This gap underscores the need for further exploration into how cutting-edge imaging technologies can uncover the complex structural details of these materials, offering new insights into their behavior and potential applications.

This research seeks to explore the structural changes in starch-sodium salt composite membranes, with a focus on their physical and morphological characteristics. By examining how varying salt concentrations influence the membrane's structure, the study aims to uncover the fundamental interactions that drive these transformations. The results will establish a connection between surface features and the internal structural modifications, shedding light on the mechanisms that determine the material's properties. These insights are anticipated to guide the design and optimization of innovative biopolymer-based materials, particularly for use in energy storage systems and other advanced technological applications.

2. MATERIALS AND METHODS

The raw materials, starch ($C_6H_{10}O_5$) and sodium hydrogen sulfate (SHS), were obtained from Sigma-Aldrich. To produce the solid biopolymer electrolytes (SBEs), sodium hydrogen sulfate was added in varying concentrations, ranging from 5% to 45% by weight (as detailed in Table-1). The preparation method involved dissolving the specified amounts of SHS in a solvent blend of distilled water and glycerin. The mixture was stirred continuously until a clear, uniform solution formed, ensuring complete dissolution of all components with no remaining particles. The weight percentages (wt.%) of the constituents were calculated using Equation 1, which provides a systematic approach for determining the SBE composition.

$$\text{wt.}\,\% = \frac{x}{x+y} \times 100 \quad (1)$$

where, x is the amount of SHS (g), and y is the amount of polymer host (g).

Table-1. Compositions of polymer and ionic dopant.

SBE sample	Sodium hydrogen sulfate (SHS)		Distilled water (ml)	Glycerin (ml)
	wt.%	g		
A	0	0.000	20	0.6
B	5	0.053	20	0.6
C	10	0.111	20	0.6
D	15	0.176	20	0.6
E	20	0.250	20	0.6
F	25	0.333	20	0.6
G	30	0.429	20	0.6
H	35	0.538	20	0.6
I	40	0.667	20	0.6
J	45	0.818	20	0.6

The next step in the preparation process involved adding 1 g of starch to the pre-mixed solution of distilled water, glycerin, and sodium hydrogen sulfate. The mixture was stirred continuously at a temperature between 60-70°C until a smooth, uniform solution was obtained. This solution was then poured into a clean glass petri dish and left to dry naturally at room temperature, allowing the formation of solid biopolymer electrolyte (SBE) membranes. To eliminate any remaining moisture and maintain the solid-state integrity of the membranes, they were placed in a desiccator containing silica gel. This drying phase was essential for stabilizing the membranes and preventing degradation caused by moisture. The entire procedure, known as the solution casting technique, is depicted schematically in Figure-1.

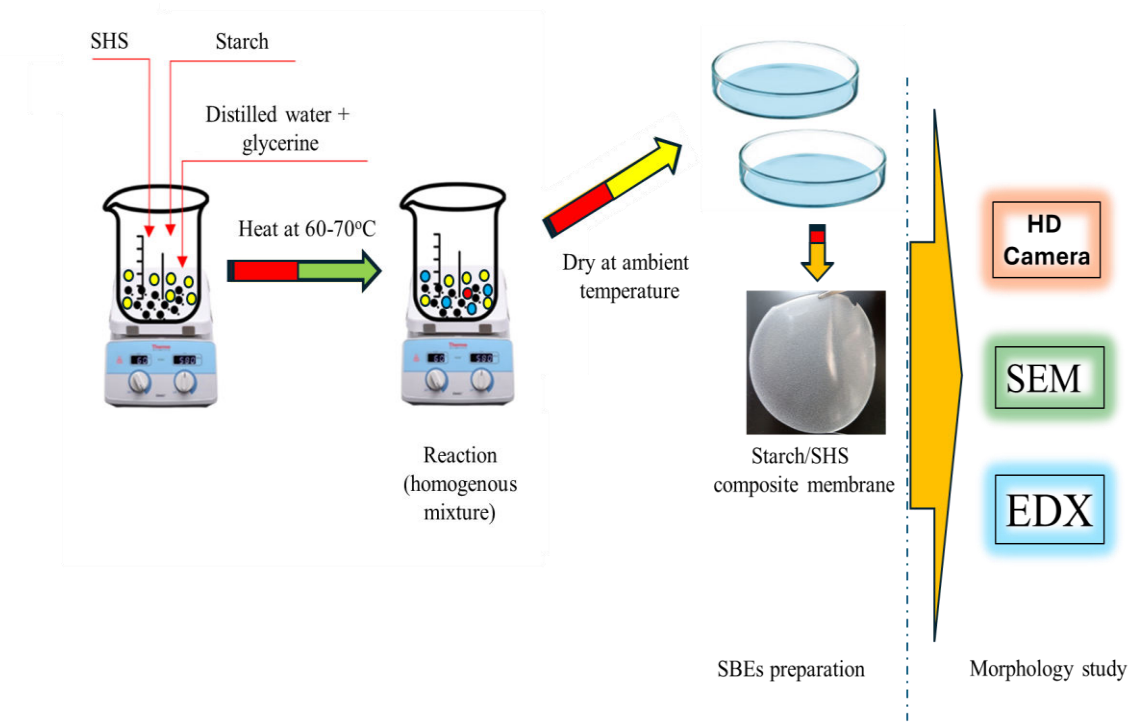

Figure-1. A schematic diagram for preparation and morphology study of solid biopolymer electrolyte membrane.

The fabricated samples were captured using a 20-megapixel high-definition camera to examine the macroscopic impacts of sodium salt integration, including alterations in opacity, surface texture, and color. This approach offers a qualitative assessment of the physical transformations resulting from different salt

concentrations, acting as an initial indicator of structural changes within the membranes.

To conduct a thorough examination of surface morphology, scanning electron microscopy (SEM) was utilized. The analysis was carried out using the JEOL JSM-6350LA SEM model, operating at an acceleration voltage of 20 kV to obtain high-resolution images of the membrane's microstructure. SEM allows for the observation of surface characteristics, including roughness, pore formation, and morphological alterations caused by the incorporation of sodium salt. Changes in surface morphology, such as the shift from a smoother to a more uneven and textured structure, were documented at magnifications reaching up to 1,000×. These microstructural variations provide valuable insights into the interactions between the polymer matrix and the sodium salt.

The elemental composition and distribution across the membranes were further investigated using energy dispersive spectroscopy (EDS), a tool integrated with the SEM system. EDS mapping offered detailed insights into the spatial distribution of essential elements, including sodium (Na), oxygen (O), and carbon (C), within the membranes. This technique was crucial for verifying the uniform dispersion of sodium hydrogen sulfate throughout the polymer matrix and detecting any potential regions of phase separation. The EDS data not only confirmed the successful integration of salt into the matrix but also provided valuable information on how ionic interactions influenced the microstructural changes.

To guarantee precise SEM and EDS measurements, the sample surfaces were coated with a thin layer of gold using a sputter coater. This step was essential to avoid electrostatic charging during analysis, which could otherwise distort the images and compromise the quality of the data. The coated samples were securely mounted onto specimen holders using double layers of cellophane tape to ensure stability throughout the observation process.

Furthermore, the SEM images were examined to identify crystalline and amorphous regions within the samples. These structural characteristics are closely tied to the ionic conductivity of the membranes, as a shift from a crystalline to a more amorphous structure typically improves the movement of charge carriers. This analysis established a clear connection between the surface morphology and the electrochemical performance of the membranes.

3. RESULTS AND DISCUSSIONS

The solid biopolymer electrolytes (SBEs) based on starch were successfully synthesized via the solution casting method, incorporating starch, glycerin, and sodium hydrogen sulfate (SHS) as key components. The morphological characteristics of the resulting membranes, analyzed through visual inspection and macroscopy, reveal significant structural transformations as a function of SHS concentration.

Figure-2(a) illustrates the pure starch membrane, exhibiting a smooth and uniform texture with high transparency, indicative of a well-integrated polymer matrix. A similar surface quality is observed in the 5 wt.% SHS-incorporated membrane (Figure-2b), suggesting minimal disruption to the polymer network at this concentration. However, as the SHS content increases from 10 wt.% to 45 wt.% (Figures 2c-2j), distinct morphological changes emerge, demonstrating the influence of salt incorporation on the polymer structure. At lower concentrations (10-20 wt.% SHS), the membranes undergo a slight shift in appearance, transitioning from clear to a faint yellowish tint. This color change may be attributed to enhanced ionic interactions between SHS and the starch backbone, subtly altering the polymer arrangement. With further SHS addition (25-35 wt.%), minor surface irregularities begin to appear, possibly due to increased disruption in the polymeric chains. At the highest salt loadings (40-45 wt.%), the membranes exhibit noticeable phase separation, roughened textures, and opacity, suggesting the saturation limit of salt solubility within the polymer matrix. These findings highlight the delicate balance between salt concentration and polymer stability, influencing the overall electrochemical properties of the SBE system [10, 11].

As the sodium hydrogen sulfate concentration increased beyond 25 wt.%, more pronounced morphological transformations were observed. The membranes exhibited a distinctly heterogeneous surface, marked by visible irregularities and textural changes. These alterations can be attributed to the progressive incorporation of sodium ions into the polymer matrix, which disrupted the crystalline regions of starch. The sodium ions interacted with the hydroxyl groups of starch, reducing intermolecular hydrogen bonding and promoting a more amorphous structure. Consequently, the membranes developed a yellowish hue and increased surface roughness, indicating significant structural modifications [12].

At concentrations between 30 wt.% and 45 wt.%, the membranes displayed pronounced heterogeneity, with small, lump-like structural features emerging. This phenomenon suggests phase separation within the polymer matrix, likely caused by the uneven distribution of sodium hydrogen sulfate. At higher concentrations, regions enriched with salt coexisted with polymer-rich domains, a common occurrence in polymer-salt systems with limited salt solubility. The disruption of the uniform polymer network altered both mechanical and optical properties, leading to increased light scattering and further intensifying the yellowish appearance [13, 14].

Notably, beyond 45 wt.% salt content, the membranes became increasingly fragile and brittle. This brittleness likely stemmed from excessive phase separation and precipitation of sodium hydrogen sulfate, which compromised the structural integrity of the polymer network. These findings underscore the intricate interplay between polymer-salt interactions, phase behavior, and the

resulting physical characteristics of the membranes. The observed morphological evolution highlights the critical threshold at which excessive salt loading transitions from beneficial structural modification to detrimental phase instability [15].

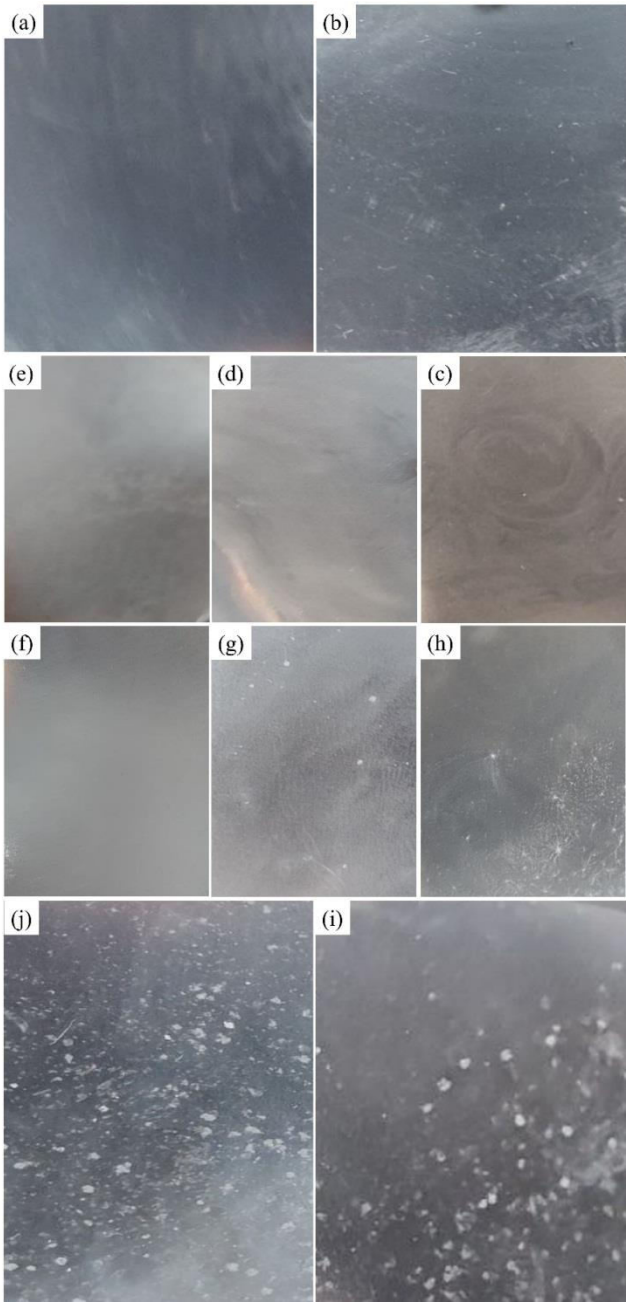

Figure-2. SBE membranes are at different weight % of SHS. Corresponding image for (a) pure starch, (b) 5 wt.%, (c) 10 wt. %, (d) 15 wt. %, (e) 20 wt. %, (f) 25 wt. %, (g) 30 wt. %, (h) 35 wt.%, (i) 40 wt.%, (j) 45 wt.% of SHS.

The impact of sodium hydrogen sulfate (SHS) concentration on the microstructure of starch-based solid biopolymer electrolytes (SBEs) was analyzed using scanning electron microscopy (SEM) at 1000× magnification. This analysis provided a detailed visualization of the structural evolution within the polymer matrix as a function of salt incorporation.

The SEM images (Figures 3(a)-3(j)) illustrate how increasing SHS content from 0 wt.% to 45 wt.% alters the surface characteristics of the membranes. The pristine starch membrane (Figure 3a) exhibited a highly uniform and smooth surface, indicative of a well-organized polymer network with minimal structural irregularities. The absence of visible disruptions suggests that, in its pure form, the starch matrix remains intact, with strong intermolecular hydrogen bonding maintaining its homogeneous morphology. With the addition of 5 wt.% SHS (Figure-3b), subtle textural variations began to emerge. The previously smooth surface displayed fine grain-like formations, suggesting the initial stages of polymer-salt interactions. This change is likely due to sodium ions engaging with the hydroxyl functional groups of starch, modifying the native hydrogen bonding network and creating localized structural distortions. These early morphological shifts indicate the onset of ion-induced rearrangements within the polymer framework. As the SHS concentration increased further, the membranes exhibited progressively more pronounced modifications, with the emergence of distinct surface irregularities and increased roughness. These transformations reflect the growing influence of salt ions on the polymer architecture, disrupting its crystalline regions and promoting a more amorphous character. The observed morphological evolution underscores the critical role of salt content in shaping the structural integrity and physical properties of starch-based SBEs. [10, 11].

The influence of sodium hydrogen sulfate (SHS) concentration on the microstructure of starch-based solid biopolymer electrolytes (SBEs) was systematically examined using SEM imaging (Figures 3(c)-3(j)). A progressive transformation in surface morphology was observed as the SHS content increased, revealing significant structural dynamics within the polymer matrix. At 10 wt.% SHS (Figure 3c), the membrane surface began to display intricate flower-like patterns interspersed with emerging grain boundaries. This distinctive morphology suggests localized phase separation, where clusters of sodium hydrogen sulfate began to form discrete domains within the predominantly polymer-rich regions. Such distribution indicates an evolving interaction between the salt and polymer chains, subtly disrupting the matrix's crystalline order and introducing heterogeneity.

Further increasing the SHS concentration to 15 wt.% (Figure 3d), the membrane revealed a more complex topology characterized by fine, grain-boundary-like features scattered across the surface. These patterns likely result from the salt exceeding its solubility threshold, precipitating out as discrete crystalline domains. This precipitation not only disrupted the uniformity of the polymer network but also introduced mechanical weaknesses, altering the material’s optical clarity and flexibility.

Between 20 wt.% and 35 wt.% SHS (Figures 3e-3h), the membranes exhibited pronounced roughness and an increasingly uneven texture. This roughening is attributed to the combined plasticizing effects of sodium hydrogen sulfate and glycerin, which softened the polymer structure, enhancing its flexibility. The increased salt content interfered with the polymer's crystalline regions, resulting in a less ordered, more amorphous matrix. This transformation underscores the role of SHS as both a structural disruptor and a plasticizer. Upon reaching 40 wt.% and 45 wt.% SHS (Figures 3i and 3j), the membranes experienced severe morphological changes, including the formation of cracks and extensive surface irregularities. These defects suggest that the polymer matrix became oversaturated with salt, leading to excessive phase separation and instability [15]. The observed cracking is indicative of the material's inability to accommodate further ionic species, resulting in structural degradation and compromised mechanical integrity. Overall, SEM analysis highlighted a clear morphological trajectory as SHS concentration increased-from smooth, homogeneous surfaces to progressively rougher and more irregular textures, culminating in mechanical failure at higher salt loadings. This progression provides critical insights into the limits of salt incorporation in starch-based SBEs, emphasizing the delicate balance required for optimizing their structural and functional properties for energy storage applications.

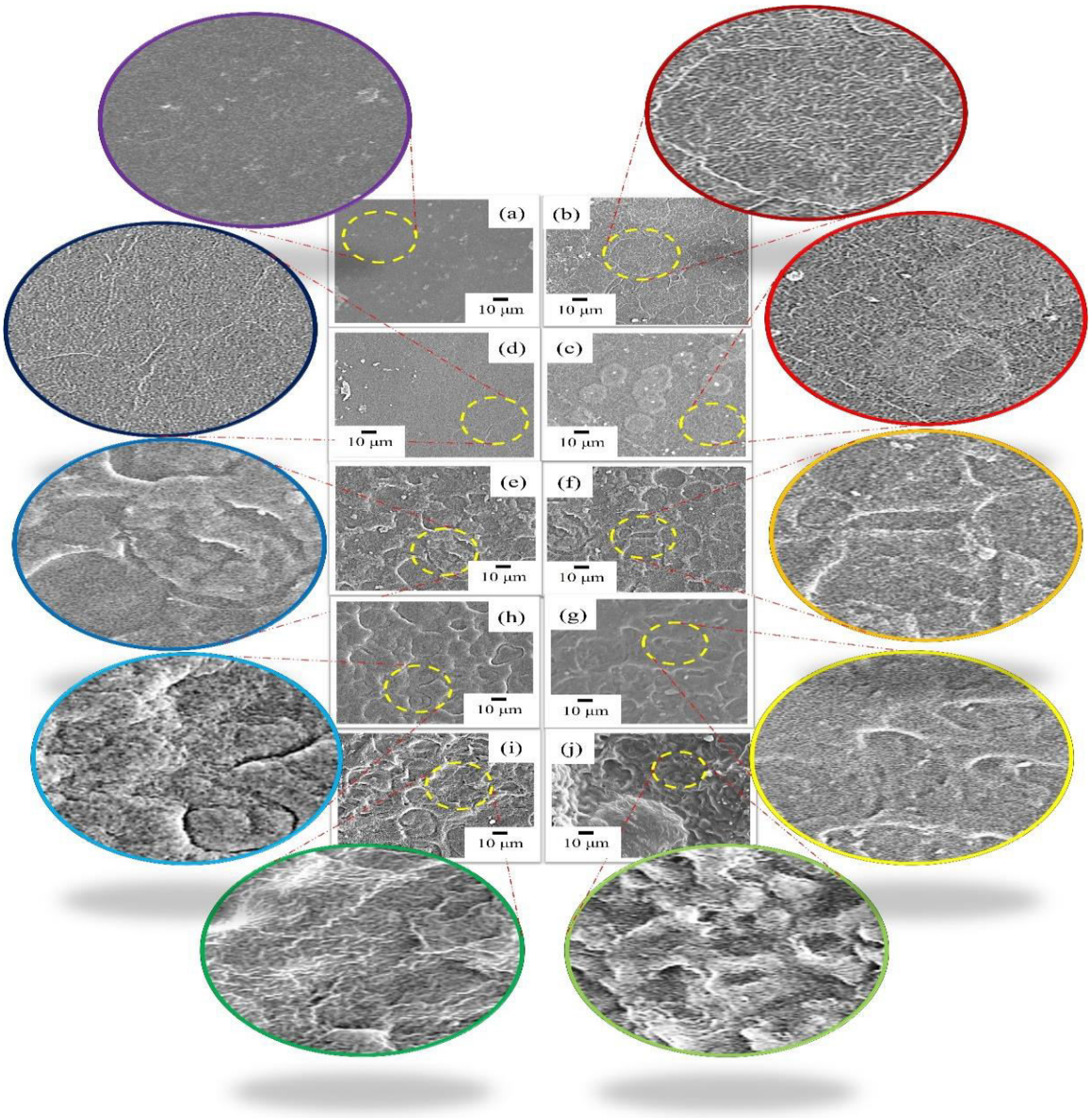

Figure-3. The surface morphology of SBE membranes. Corresponding image for (a) pure starch, (b) 5 wt. %, (c) 10 wt. %, (d) 15 wt. %, (e) 20 wt. %, (f) 25 wt. %, (g) 30 wt. %, (h) 35 wt.%, (i) 40 wt.%, (j) 45 wt.% of SHS.

To evaluate the elemental composition and spatial distribution within the starch-based solid biopolymer electrolyte (SBE) membranes, energy dispersive spectroscopy (EDS) was employed. This technique enables precise identification of key elements, providing crucial insights into the uniformity of sodium hydrogen sulfate (SHS) dispersion within the polymer matrix. Ensuring consistent elemental distribution is fundamental for optimizing the electrochemical properties of the membranes, particularly their ionic conductivity and mechanical stability. [16, 17].

Figures 4(a)-4(i) present EDS elemental mappings for sodium (Na), oxygen (O), and carbon (C) in membranes containing 5-45 wt.% SHS. The bright intensity regions in the mappings indicate the presence and distribution of these elements across the membrane surface. At lower salt concentrations, Na appears well-integrated into the polymer matrix, with a relatively uniform dispersion. This suggests effective interaction between sodium ions and the hydroxyl groups of starch, which facilitates homogeneous mixing during the casting process. Comparative studies on polymer-salt systems have shown that achieving uniform salt dispersion is critical for optimizing conductivity pathways [18-20]. The even integration of Na throughout the polymer network helps maintain a continuous ion transport channel, which is essential for electrolyte performance in energy storage applications. The results of this study corroborate previous research on starch-based composites, where the strong affinity between sodium ions and starch molecules was found to enhance the formation of interconnected ion-conducting domains [21, 22]. Furthermore, the influence of SHS concentration on elemental distribution is evident in the shifting balance of O and C signals in EDS spectra. The gradual increase in Na content is accompanied by subtle variations in O and C intensities, indicating modifications in the polymer matrix's structural organization. These changes suggest a progressive transition from a predominantly crystalline arrangement to a more amorphous structure, reinforcing the notion that salt incorporation disrupts the native polymer network. The consistency of the EDS data across multiple membrane samples underscores the reliability of the results. The uniform elemental distribution at moderate SHS levels suggests that solution casting remains an effective fabrication method for ensuring homogeneity in biopolymer-salt composites. However, at higher concentrations, the onset of phase separation highlights the need to optimize salt loading to prevent excessive aggregation. In summary, EDS analysis provides a detailed understanding of elemental dispersion in starch-SHS membranes, confirming the influence of salt concentration on membrane uniformity and structural integrity. These findings serve as a valuable reference for fine-tuning polymer-salt interactions to enhance the performance of solid biopolymer electrolytes in energy storage applications.

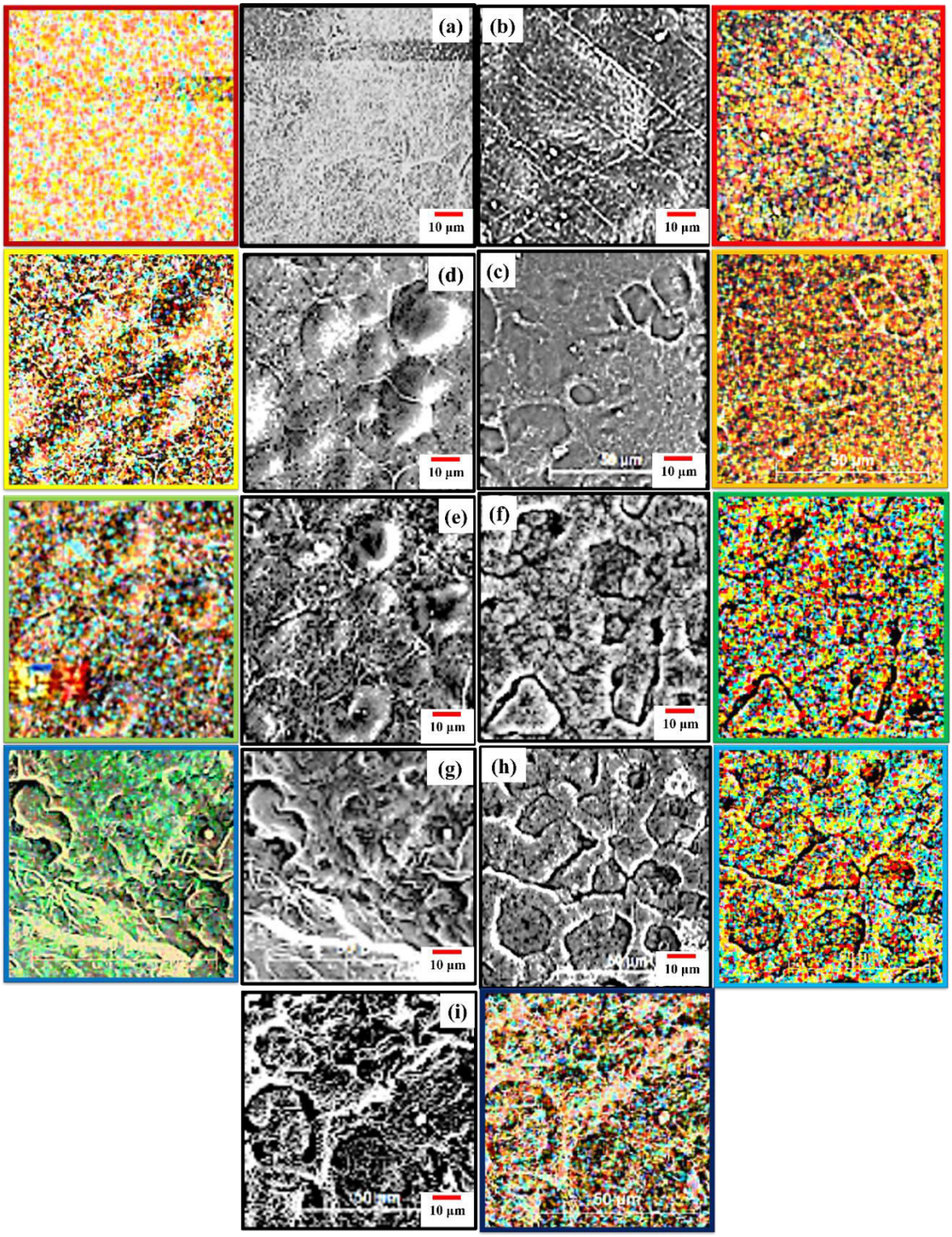

Figure-4. Scanning electron microscope image of starch-SHS complex membranes. Corresponding EDS mappings for sample (a) 5 wt.%, (b) 10wt.%, (c) 15wt.%, (d) 20wt.%, (e) 25wt.%, (f) 30wt.%, (g) 35wt.%, (h) 40wt.% and (i) 45wt.%.

4. CONCLUSIONS

This research successfully developed starch-based solid biopolymer electrolyte (SBE) membranes incorporating sodium hydrogen sulfate (SHS) via the solution casting technique. A comprehensive analysis of the membranes' structural and morphological characteristics was conducted using high-resolution imaging, scanning electron microscopy (SEM), and energy dispersive spectroscopy (EDS). The addition of SHS induced significant modifications in the polymer matrix,

leading to distinct surface features and disrupting the native morphology of starch. These alterations were primarily driven by interactions between SHS and the polymer chains, which influenced the crystalline-to-amorphous transition within the membrane.

The study underscores the critical role of SHS in modifying the structural integrity of starch-based membranes, demonstrating its potential for tailored material applications. The findings offer new perspectives on how salt incorporation affects biopolymer electrolytes, providing essential knowledge for advancing energy storage technologies and other functional material applications. Future investigations should focus on fine-tuning salt concentrations, exploring alternative additives, and evaluating long-term stability under different environmental conditions. This research lays the groundwork for the further development of sustainable biopolymer electrolytes with enhanced performance characteristics.

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