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Research Paper

Preparation and Application of Bifunctional Cellulose Aerogel Loaded with Anthocyanin Derived from Purple Corncob

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Abstract. This study constructed dual-functional aerogels with pH-responsive colorimetry and antibacterial preservation by extracting cellulose and anthocyanins from purple corncobs and loading them with a cinnamon essential oil/β-cyclodextrin inclusion complex. The three-dimensional porous network structure endowed the aerogels with superior mechanical properties, achieving a compressive stress of 5.9 MPa at 90% strain. Compared to the control cellulose film material, the dual-functional aerogel exhibited faster pH responsiveness, with its steady-state color difference (ΔE) being approximately 1.25 times that of the film material. The aerogel loaded with the cinnamon oil/β-cyclodextrin inclusion complex showed a significantly slower decline in DPPH radical scavenging activity (12.48% over 60 hours) compared to the system with free essential oil (28.25% over 60 hours), representing a 55% improvement in sustained-release efficiency. Application experiments demonstrated that the bifunctional aerogel possessed more significant responsiveness and preservation effects for monitoring shrimp freshness. This research provides a theoretical foundation for developing materials for seafood freshness monitoring and preservation from agricultural waste.

Keywords: *Purple corn cob; Freshness monitoring; Food preservation; Functional packaging materials*

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1 Introduction

The global food industry is undergoing a transformative shift, driven by escalating consumer demand for high-quality, safe, and nutritious products. Seafood, prized for its rich proteins, omega-3 fatty acids, and essential micronutrients, has become a cornerstone of modern diets. However, its inherent perishability poses a monumental challenge to the supply chain [1-3]. Spoilage in seafood, primarily instigated by microbial proliferation, endogenous enzyme activity, and oxidative rancidity, accelerates rapidly post-harvest, leading to significant economic losses, resource wastage, and potential public health risks from foodborne pathogens [4,5]. Traditional preservation methods, such as refrigeration, freezing, and chemical preservatives, while effective to a degree, often come with drawbacks including high energy consumption, potential nutrient degradation, and growing consumer aversion towards synthetic additives. Consequently, the quest for innovative, intelligent, and sustainable packaging solutions that can actively monitor product quality and extend shelf life has emerged as a critical frontier in food science and technology [6-8].

In this context, intelligent packaging systems, capable of providing real-time, visual information about the freshness and safety of packaged content without the need for destructive testing, have garnered tremendous research interest [9]. Among various intelligent packaging strategies, pH-responsive colorimetric indicators are particularly promising for monitoring protein-rich foods like seafood. The spoilage of such products is frequently accompanied by the accumulation of basic volatile amines (e.g., trimethylamine, ammonia) due to microbial

decarboxylation of amino acids and autolytic processes, leading to a discernible increase in the headspace pH [10]. Therefore, a material that undergoes a visible color change in response to these alkaline volatiles can serve as a direct, user-friendly freshness indicator. Natural pigments, especially anthocyanins, have become the focal point for developing such indicators due to their vibrant, pH-dependent colorimetric properties, non-toxicity, and inherent biodegradability [11-13]. Derived from various fruits, vegetables, and flowers, anthocyanins exhibit structural transformations across different pH levels, changing color from red in acidic conditions to purple, blue, or even green/yellow in alkaline environments. This unique property has been exploited in several studies to create freshness indicators; however, many existing systems rely on anthocyanins extracted from common sources like purple sweet potato or red cabbage and are often integrated into dense film matrices, which can suffer from slow response times, poor mechanical properties, and single functionality limited to mere indication.

Simultaneously, the paradigm of food packaging is evolving from passive barrier systems to active packaging that can intervene in the preservation process. The incorporation of antimicrobial agents, such as plant-derived essential oils (EOs) like cinnamon oil, which possess broad-spectrum antimicrobial and antioxidant activities, represents a powerful strategy to actively inhibit spoilage microorganisms and lipid oxidation [14,15]. However, the direct application of EOs is hampered by their volatility, sensitivity to light and oxygen, and strong odor, which can affect food sensory qualities. Encapsulation technologies, particularly using β-cyclodextrin (β-CD) to form inclusion complexes, offer an elegant solution by protecting the active compounds, masking strong odors, and enabling controlled release, thereby prolonging the antimicrobial efficacy. The ideal next-generation packaging material would thus synergistically combine the real-time, visual monitoring capability of a pH-sensitive indicator with the sustained, active preservation action of an antimicrobial agent within a robust, eco-friendly matrix.

This pursuit aligns perfectly with the principles of a circular bio-economy, emphasizing the valorization of agricultural and processing by-products. Purple corn cob, a significant waste stream from the corn processing industry, presents a remarkable and underutilized resource. It is not only a rich source of cellulose—a renewable, biodegradable [16], and mechanically competent polymer ideal for constructing packaging scaffolds—but also contains substantial amounts of valuable anthocyanins within its cob. Traditionally, these anthocyanins are discarded or underutilized. An integrated approach that extracts both cellulose and anthocyanins from the same purple corn cob feedstock embodies a holistic and sustainable valorization strategy, maximizing resource efficiency and reducing waste [17]. Cellulose, when combined with polyvinyl alcohol (PVA), can form physically cross-linked networks with enhanced mechanical integrity through freeze-thaw cycles, suitable for creating three-dimensional aerogel structures [18-20].

Aerogels, with their ultra-lightweight nature, high porosity, and enormous specific surface area, offer distinct advantages over conventional dense films for such applications. Their interconnected porous network facilitates the rapid diffusion of target gas molecules (like ammonia), leading to faster and more pronounced colorimetric responses from embedded indicators [21,22]. Furthermore, the high surface area and tunable pore structure provide an ideal platform for the high loading and controlled release of active compounds like encapsulated EOs. Therefore, engineering a multifunctional aerogel that leverages the inherent properties of all components—cellulose as a sustainable scaffold, in-situextracted anthocyanin as a responsive pigment, and a β-CD-cinnamon oil inclusion complex as a stabilized antimicrobial reservoir—represents a highly innovative and integrated solution.

Against this backdrop, the present study is designed to develop a novel, bifunctional aerogel for intelligent food packaging by comprehensively utilizing purple corn cob. The research is built on a cascade valorization process: first, anthocyanins (PCCA) and cellulose (CF) are sequentially extracted from the same purple corn cob. The cellulose is then combined with PVA to form a base gel, into which the naturally derived PCCA and a pre-formed cinnamon oil/β-cyclodextrin inclusion complex (EOC) are incorporated [23-25]. Through a process involving freeze-thaw cycling, foaming, and subsequent freeze-drying, a three-dimensional, porous aerogel is constructed. This work systematically investigates the formation mechanism, physicochemical properties (morphology, crystallinity, mechanical strength), and the dual functionalities of the resulting aerogel. The colorimetric responsiveness to ammonia and acetic acid vapors is quantified, and the reversible sensing capability is evaluated. The sustained-release profile and enhanced antioxidant stability imparted by the β-CD encapsulation are analyzed. Finally, the practical efficacy of the aerogel is demonstrated in a real-food application by monitoring its color change and evaluating its preservative effect on shrimp during storage. This integrated approach aims to

create a high-performance, multifunctional packaging material that not only addresses the critical needs for seafood freshness monitoring and extension but also offers a sustainable pathway for the high-value utilization of agricultural waste, contributing to the development of advanced, eco-conscious smart packaging systems. The following sections detail the materials, methods, and comprehensive results elucidating the performance of this novel bifunctional aerogel.

2 Materials and Methods

2.1. Materials

Purple corn cob was sourced from Caoxian County, Shandong Province. Cinnamon essential oil (EO) was purchased from Jiangxi Xuesong Natural Medicinal Oil Co., Ltd. Polyvinyl alcohol 1788 and β-cyclodextrin (β-CD, 97%) were purchased from Shanghai Macklin Biochemical Technology Co., Ltd. Sodium hydroxide, glacial acetic acid, sodium chlorite, and citric acid were purchased from Chengdu Kelong Chemical Reagent Co., Ltd., all of analytical grade.

Vertex70 Fourier transform infrared spectrometer (FT-IR) and D8 Advance X-ray diffractometer (XRD) were from Bruker, Germany. S4800 field emission scanning electron microscope (SEM) was from Rigaku, Japan. Cary5000 UV-Vis-NIR spectrophotometer was from Agilent, USA. AI-7000-NGD servo-controlled high-low temperature tensile testing machine was from Tianjin Gaoteweer Testing Instrument Co., Ltd.

2.2. Methods

2.2.1. Preparation of Purple Corn Cob Anthocyanin (PCCA), Purple Corn Cob Cellulose (CF), and Cinnamon Essential Oil/β-Cyclodextrin Inclusion Complex (EOC)

The purple corn cob was cleaned, dried, and crushed, then sieved through a 60-mesh sieve. The sieved powder was added to acidified ethanol, heated and ultrasonically treated at 40°C for 30 minutes, filtered, and the filtrate was dried in an oven at 40°C to obtain PCCA powder. CF was prepared from the filter residue from the above step through alkali treatment and bleaching. The preparation of the cinnamon essential oil/β-cyclodextrin inclusion complex (EOC) referred to the method of Ayala Zavala et al. for preparing β-cyclodextrin-cinnamon essential oil/garlic essential oil microcapsules.

2.2.2. Preparation of Cellulose-based Intelligent Responsive Bifunctional Aerogels

Cellulose-Polyvinyl Alcohol (PVA) Composite Aerogel (CF(aerogel)): 1.5 g (dry weight) of CF was mixed with 20 g of a 5 wt% PVA solution and 13.5 mL of deionized water, stirred uniformly, frozen for 2 hours, and thawed at room temperature. This freeze-thaw cycle was repeated three times to obtain a CF-PVA solution. The frozen-thawed mixture was stirred at high speed for 3 minutes for foaming. The fully foamed material was poured into a mold and freeze-dried for 36 hours.

Cellulose-Polyvinyl Alcohol-Purple Corn Cob Anthocyanin Composite Aerogel (CF-P(aerogel)): 0.46 g of PCCA powder was added to 20 g of the CF-PVA solution, mixed uniformly, stirred at high speed for 3 minutes for foaming, poured into a mold, and freeze-dried for 36 hours.

Cellulose-Polyvinyl Alcohol-Purple Corn Cob Anthocyanin-Cinnamon Essential Oil Composite Aerogel (CF-P-EO(aerogel)): 0.46 g of PCCA powder and 64 mg of EO were added to 20 g of the CF-PVA solution. The mixture was stirred uniformly, foamed at high speed for 3 minutes, poured into a mold, and freeze-dried for 36 hours.

Cellulose-Polyvinyl Alcohol-Purple Corn Cob Anthocyanin-Cinnamon Essential Oil Inclusion Complex Composite Aerogel (CF-P-EOC(aerogel)): 0.46 g of PCCA powder and 2.3 g of EOC were added to 20 g of the CF-PVA solution. The mixture was stirred uniformly, foamed at high speed for 3 minutes, poured into a mold, and freeze-dried for 36 hours.

Cellulose-Polyvinyl Alcohol-Purple Corn Cob Anthocyanin-Cinnamon Essential Oil Inclusion Complex Composite Film (CF-P-EOC(film)): 0.1 g of PCCA powder and 0.5 g of EOC were added to 20 g of the CF-PVA solution, mixed

uniformly, poured into a mold with the same quantity as the aerogel, and dried in an oven at 40°C for 6 hours.

2.2.3. Characterization of Anthocyanin

Equal volumes of PCCA solution were placed in PBS buffers with pH values ranging from 3.0 to 10.0 to observe and record the color of the PCCA solution at different pH values. The UV-Vis absorption spectra of the PCCA solution were measured using a Cary5000 UV-Vis-NIR spectrophotometer with a scanning range of 400-800 nm. The anthocyanin content in the samples was determined using the UV-Vis spectrophotometric method.

2.2.4. Physicochemical Characterization of Bifunctional Aerogels

The surface morphology of the materials was characterized using scanning electron microscopy (SEM). X-ray diffraction (XRD) analysis was performed using a diffractometer with a scanning rate of 10°/min and a scanning angle range of 10° to 40°. The compressive properties of the aerogel materials were tested using a tensile testing machine to measure the stress of different aerogel materials at 90% compressive strain.

2.2.5. Antioxidant Activity of Bifunctional Aerogels

The antioxidant activity of the materials was evaluated using the DPPH radical scavenging method: DPPH was dissolved in methanol at a concentration of 0.025 g/L. 200 μL of the methanol solution impregnated with the aerogel was mixed with 4 mL of the DPPH-methanol solution, stored at room temperature in the dark for 30 minutes, and the absorbance at 517 nm was measured to calculate the DPPH radical scavenging rate.

2.2.6. Color Response of Aerogels to Acidic and Alkaline Gases

A benchtop spectrophotometer was used to comparatively study the color response changes of the materials to acidic and alkaline gases. 10 mL of 20 wt% ammonia water and 20 wt% acetic acid were placed in separate 100 mL glass bottles, and the aerogel was suspended in the bottle to measure the color response to NH_3 and acetic acid vapors. Measurements were taken at intervals over 3 minutes. To study the reversibility of the aerogel's color, the aerogel was exposed to 50 mL of 20 wt% ammonia vapor for 1 minute, then exposed to 50 mL of 20 wt% acetic acid vapor for 1 minute. The test was conducted for 9 cycles.

2.2.7. Effect of Environmental pH on Essential Oil Release from Aerogels

To study the relationship between the release rate of essential oil from the aerogel and environmental pH, CF-P-EO(aerogel) and CF-P-EOC(aerogel) were exposed to 50 mL of 20 wt% ammonia vapor, 50 mL of 20 wt% acetic acid vapor, and an environment with 98% relative humidity, respectively. At regular intervals, 20 mg samples were taken, dissolved in 5 mL of ethanol, and ultrasonicated for 30 minutes. The supernatant was collected to determine the UV absorbance of EO at 286 nm and calculate the EO release rate from the aerogel.

2.2.8. Color Responsiveness and Preservation Effect of Aerogels for Shrimp Freshness Detection

In a 10 cm diameter Petri dish, fresh shrimp were stored at 25°C with CF(aerogel), CF-P(aerogel), CF-P-EOC(aerogel), and CF-P-EOC(film), respectively. Changes in shrimp freshness and the color changes of the aerogels were monitored.

3 Results and Discussion

3.1 Analysis of Dendrocalamus sinicus Cellulose Component Content

3.1. Characterization of Purple Corn Cob Anthocyanin (PCCA)

The successful extraction of anthocyanins from the agricultural by-product purple corn cob was the foundational step for developing the intelligent indicator component. Spectrophotometric quantification confirmed a substantial anthocyanin content of 18.99 mg/g in the cob powder, validating its viability as a rich and economical source for this natural pigment. This finding aligns with and supports previous literature highlighting purple corn as a significant reservoir of these valuable compounds, often underutilized in industrial processing. The core

functionality of anthocyanins as pH-responsive chromophores stems from their intricate structural equilibria (Figure 1), which are highly sensitive to the proton concentration in their environment. The visual color transitions observed in the PCCA solutions—from red at pH 3.0 to orange-brown at pH 10.0—are direct macroscopic manifestations of these reversible molecular transformations.

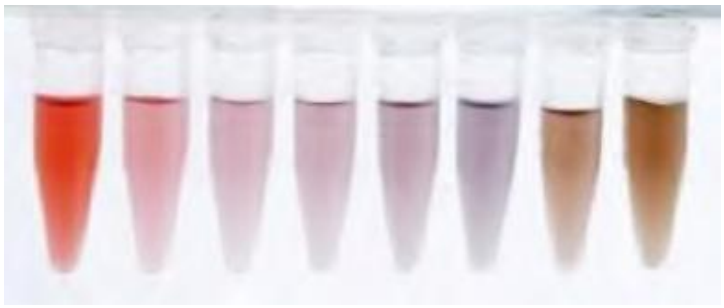

Figure 1 Color changes of PCCA under different pH environments

The underlying mechanism, corroborated by UV-Vis spectroscopic analysis, involves a series of protonation, hydration, and isomerization reactions (Figure 2).

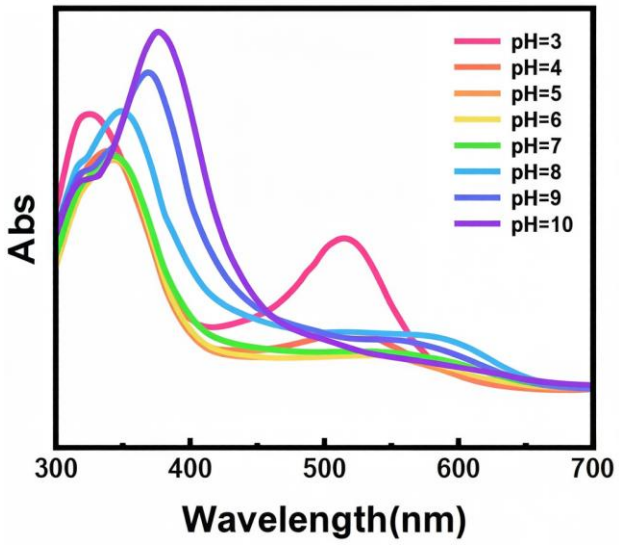

Figure 2 UV visible spectra of PCCA under different pH environments

In highly acidic conditions (pH 3.0), the flavylium cation form predominates, characterized by its red color and a strong absorption maximum at approximately 514 nm, corresponding to a π→π* electronic transition within its conjugated system. As the pH increases to the range of 4.0–5.0, a deprotonation equilibrium is established, leading to the formation of the quinonoidal base. This form exhibits a distinct blue-shift in its absorption maximum to around 340 nm. The initial increase in absorbance within this pH range can be attributed to the formation of a high-molar-absorptivity intermediate during the conversion. Further elevation of the pH to neutral or weakly alkaline conditions (6.0–7.0) promotes the hydration of the quinonoidal base, resulting in the opening of the pyrylium ring to form the pseudobase or carbinol pseudobase, which lacks the extended conjugation, leading to a significant decrease in visible light absorption and a fade in color. Under strong alkaline conditions (pH 9.0–10.0), the pseudobase can undergo a tautomerization to form the chalcone, evidenced by the emergence of a new absorption peak near 370 nm (n→π* transition) [26]. The chalcone's structure has a broken conjugation path compared to the flavylium ion, resulting in the final orange-brown hue. This predictable and reversible structural chemistry makes anthocyanins, particularly those from a sustainable source like purple corn cob, ideal candidates for fabricating visual freshness indicators responsive to basic spoilage volatiles like ammonia and trimethylamine [27].

DOI:

3.2. Physicochemical Characterization of Bifunctional Aerogels

3.2.1. Microstructure Analysis

The integration of multiple components—cellulose (CF), polyvinyl alcohol (PVA), anthocyanin (PCCA), and cinnamon essential oil (EO) or its β-cyclodextrin inclusion complex (EOC)—into a cohesive three-dimensional network was critically examined through scanning electron microscopy (SEM) (Figure 3).

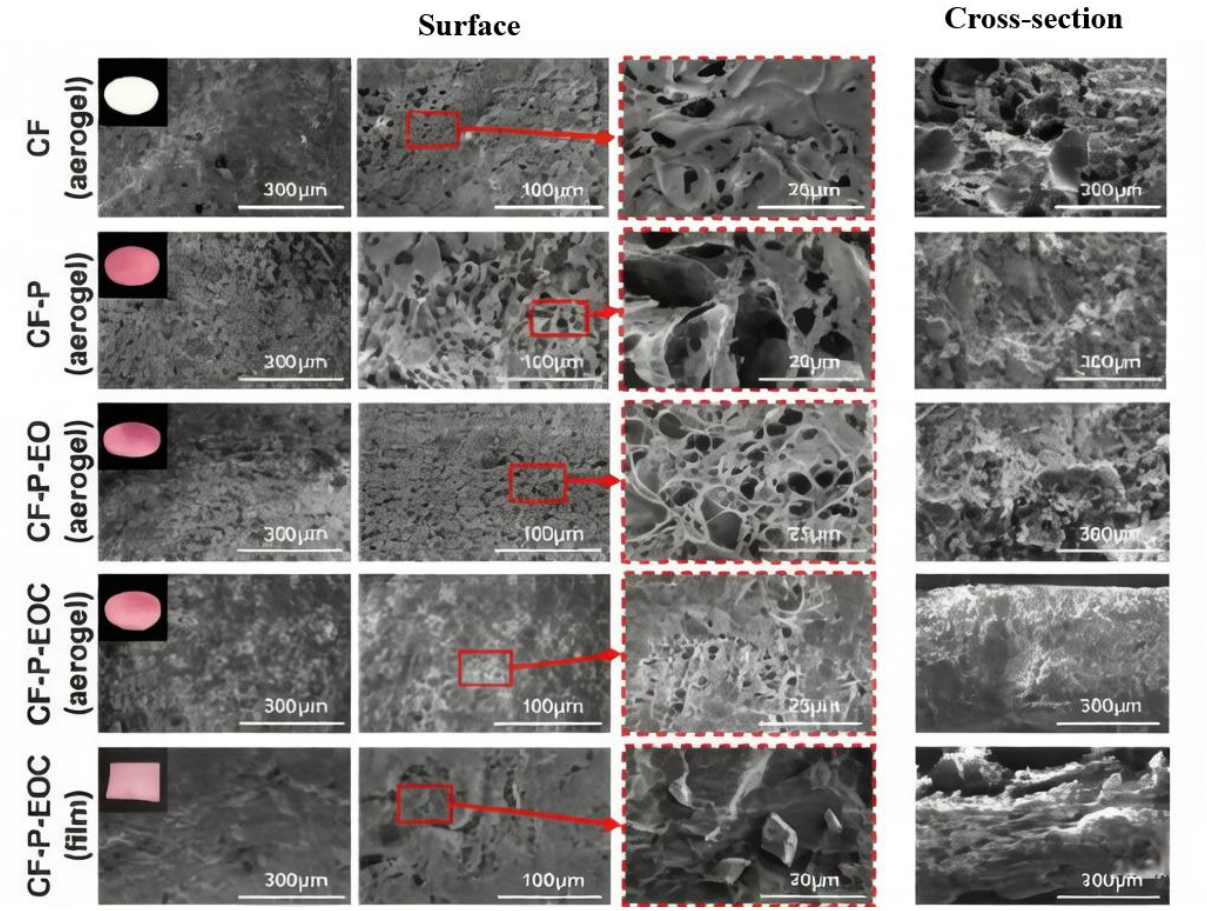

Figure 3 SEM images of the macroscopic morphology, surface, and cross-section of CF (aerogel), CF-P (aerogel), CF-P-EO (aerogel), CF-P-EOC (aerogel), and CF-P-EOC (film)

The pure CF-PVA aerogel (CF(aerogel)) exhibited a porous but non-uniform network structure with irregular pore sizes. This morphology is typical for freeze-dried polymer blends where phase separation and ice crystal growth during freezing can create heterogeneous pores. The introduction of PCCA into the matrix to form CF-P(aerogel) resulted in a markedly more uniform and refined porous architecture. This significant improvement can be mechanistically attributed to the role of PCCA as a dynamic cross-linker and rheology modifier. The abundant phenolic hydroxyl groups on the anthocyanin molecules can form extensive intermolecular hydrogen bonds with the hydroxyl groups on both the cellulose and PVA chains [28]. This interaction increases the solution viscosity and creates a more robust, physically cross-linked network prior to freezing. This enhanced network strength likely restricts the uncontrolled growth and coalescence of air bubbles during the high-speed foaming step and regulates the size of ice crystals formed during subsequent freezing, leading to a more homogeneous pore structure after sublimation.

The addition of free cinnamon essential oil (CF-P-EO(aerogel)) led to a further refinement and homogenization of the pore size distribution, resulting in predominantly micron-sized pores. Cinnamaldehyde, the primary component of cinnamon oil, is hydrophobic. When introduced into the hydrophilic CF-PVA-PCCA aqueous

mixture, it likely forms localized hydrophobic microdomains through emulsion droplets or molecular aggregation. These microdomains act as nucleation sites and physical barriers during the foaming and freezing processes, templating and limiting the expansion of gas bubbles and the growth of ice crystals, thereby yielding a finer and more controlled pore morphology [29].

The most striking structural transformation was observed in the aerogel incorporating the β-cyclodextrin-cinnamon oil inclusion complex (CF-P-EOC(aerogel)). This sample displayed a highly uniform, sub-micron scale porous structure. This can be explained by the dual role of the EOC complex. First, the β-CD shell encapsulates the hydrophobic EO, enhancing its dispersibility and stability within the aqueous precursor solution. Second, the external hydrophilic surface of the β-CD molecules can participate in the hydrogen-bonding network with CF, PVA, and PCCA [30]. This improved compatibility and the nano/micro-scale size of the EOC particles act as highly effective and uniformly distributed nucleation agents during the freezing step, promoting the formation of an exceptionally regular and fine ice crystal template, which after lyophilization translates into a remarkably uniform aerogel scaffold [31]. In stark contrast, the solvent-cast film (CF-P-EOC(film)) prepared from the same components showed a dense, non-porous, and flat morphology in cross-section, highlighting the fundamental role of the freeze-foaming and lyophilization process in creating the crucial three-dimensional porous network.

3.2.2. Crystal Phase Structure Analysis

Analysis of Fig. 4 shows that the XRD patterns of all aerogels exhibit a broad diffuse peak at 20 = 22° and a diffraction peak is observed at 14°, indicating that they all possess cellulose I characteristics and are primarily amorphous. Comparing the XRD patterns of CF-P(aerogel) and CF-P-EO(aerogel) with CF(aerogel), it can be found that the introduction of anthocyanin and cinnamon essential oil did not significantly alter the amorphous nature of the materials. Notably, the introduction of the cinnamon essential oil/β-cyclodextrin inclusion complex in CF-P-EOC(aerogel) led to the appearance of typical crystalline form II diffraction peaks at 11.4° and 17.5°, confirming the successful introduction of the cyclodextrin-cinnamon essential oil complex in the aerogel [32].

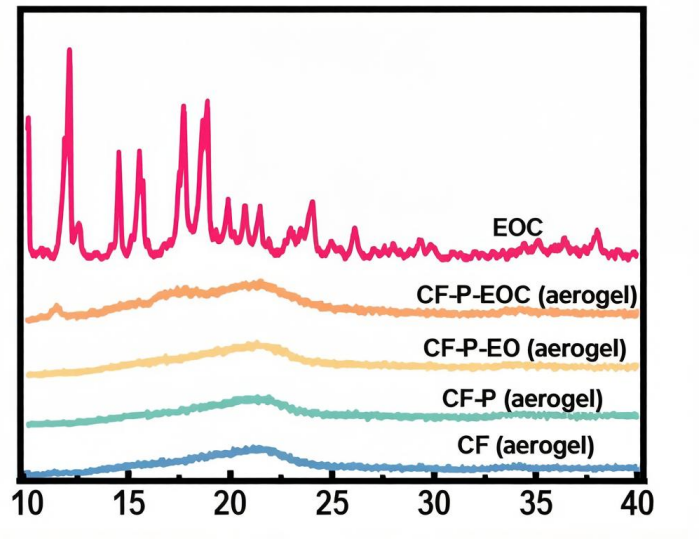

Figure 4 XRD images of EOC, CF (aerogel), CF-P (aerogel), CF-P-EO (aerogel), and CF-P-EOC (aerogel)

3.2.3. Mechanical Properties of Aerogels

The mechanical integrity of the aerogels under compression is paramount for practical handling and application. The baseline CF(aerogel), relying solely on physical entanglement and hydrogen bonding between CF and PVA chains, showed a compressive stress of 2.85 MPa at 90% strain. The incorporation of PCCA increased this value to 3.86 MPa, a 35.5% enhancement. This significant improvement is ascribed to the multi-dentate hydrogen bonding capability of anthocyanin molecules. Acting as a natural polyphenolic cross-linker, PCCA forms additional hydrogen bonds between the CF fibrils and PVA chains, effectively reinforcing the polymeric network and enhancing stress transfer throughout the structure, thereby increasing stiffness and strength [31-34].

A more substantial mechanical reinforcement was achieved with the addition of bioactive agents. The CF-P-EO(aerogel) and CF-P-EOC(aerogel) exhibited compressive stresses of 5.80 MPa and 5.90 MPa, respectively. The main active component of cinnamon oil, cinnamaldehyde, contains both an aldehyde group and an aromatic ring with electron-rich sites. These functional groups can engage in hydrogen bonding and dipole-dipole interactions with the hydroxyl-rich matrix. More importantly, the hydrophobic cinnamaldehyde molecules or the EOC complexes can act as nano-fillers that physically impede polymer chain mobility and may induce local matrix densification, leading to enhanced load-bearing capacity. The slightly higher strength of the EOC-loaded sample suggests that the well-dispersed, solid inclusion complex particles might act as more effective reinforcing fillers compared to the molecularly dispersed or emulsified free oil [35].

The combination of PVA and cellulose in the prepared aerogels formed a preliminary physical cross-linking network, which provided basic mechanical properties to the aerogel, but the strength was relatively low, manifested as a maximum stress of only 2.85 MPa for CF(aerogel) in Figure 5. The addition of anthocyanin increased the maximum stress of the aerogel from 2.85 MPa to 3.86 MPa, an increase of 35.5%. This significant performance improvement mainly stems from the hydrogen bonds formed between the hydroxyl and carbonyl groups in the anthocyanin molecular structure and the PVA and cellulose molecules, enhancing the internal cross-linking network of the material and improving its mechanical strength. The addition of cinnamon essential oil and the cinnamon essential oil/β-cyclodextrin inclusion complex increased the maximum stress of the aerogel to 5.80 MPa and 5.90 MPa, respectively. This is primarily because cinnamaldehyde, the main component of cinnamon essential oil, contains aldehyde and phenolic hydroxyl groups, which can form hydrogen bonds with PVA and cellulose [36,37].

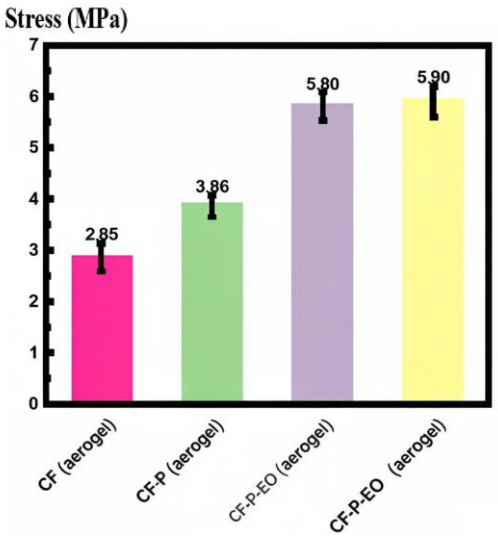

Figure 5 The maximum stress values of CF (aerogel), CF-P (aerogel), CF-P-EO (aerogel), and CF-P-EOC (aerogel) at 90% compression

3.3. Functional Characterization of Bifunctional Aerogels

3.3.1. Color Response of Aerogels to Acidic and Alkaline Gases

The dynamic colorimetric response tests revealed the superior performance of the porous aerogel architecture over dense films for gas sensing (Figure 6). All anthocyanin-containing materials (CF-P(aerogel), CF-P-EOC(aerogel), CF-P-EOC(film)) showed negligible color change (ΔE < 7) upon exposure to acetic acid (CH3COOH) vapor. This is consistent with the anthocyanin chemistry, as the flavylium cation (red) is stable under acidic conditions, and the acetic acid vapor merely maintains or slightly enhances this protonated state, resulting in no significant visible shift from the initial red/ptiink hue [38].

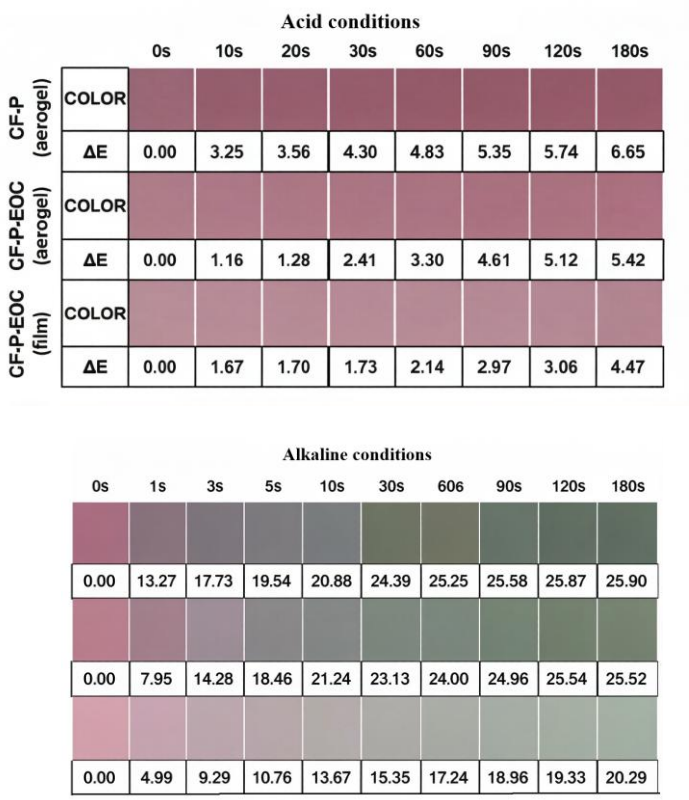

Figure 6 Responsiveness of CF-P (aerogel), CF-P-EOC (aerogel), and CF-P-EOC (film) to acidic and alkaline gases and changes in Δ E

In contrast, exposure to ammonia (NH3) vapor triggered a rapid and pronounced colorimetric response in the aerogels. Both CF-P(aerogel) and CF-P-EOC(aerogel) achieved a ΔE > 7 within 1 second, with a visually discernible color shift from light red to medium gray within 5 seconds, reaching a steady-state ΔE of 25.58 after 90 seconds. This rapid response is a direct consequence of the three-dimensional, highly porous network of the aerogel. The open and interconnected pores provide a vast surface area and short, unobstructed diffusion pathways for the NH3 gas molecules. This allows for instant penetration and interaction with the anthocyanin molecules dispersed throughout the bulk of the material, facilitating the rapid deprotonation reaction that converts the flavylium cation to the quinonoidal base and other alkaline forms (bluish/gray colors) [39-40].

The solvent-cast film (CF-P-EOC(film)), despite containing the same active components, exhibited a significantly slower and weaker response. It showed only a ΔE of 10.76 after 5 seconds, with a final steady-state color difference only 80% of that achieved by the aerogels. The dense, non-porous structure of the film poses a substantial barrier to gas diffusion. The NH3 molecules must slowly permeate through the solid polymer matrix via a solution-diffusion mechanism, making the response kinetics diffusion-controlled and therefore much slower. This comparison unequivocally demonstrates the critical advantage of the aerogel morphology for applications requiring fast visual detection, such as real-time food spoilage monitoring [41].

The aerogels were alternately exposed to 50 mL of 20 wt% ammonia water and 50 mL of 20 wt% acetic acid vapor for 3 minutes each to test their color reversibility. As shown in Figure 7, 8, and 9, the ΔE values of the three materials remained relatively stable over 9 cycles. However, compared to the aerogel materials, the color change of CF-P-EOC(film) after multiple cycles was already somewhat difficult to distinguish with the naked eye, indicating its poorer stability for repeated detection of biogenic amines compared to aerogel materials [42].

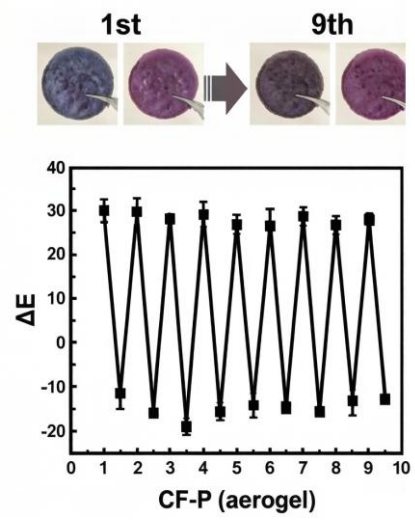

Figure 7 Responsiveness of CF-P (aerogel) after multiple cycles in acidic and alkaline gases and changes in Δ E

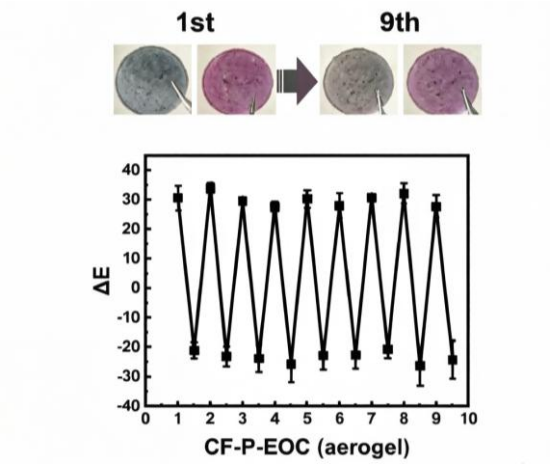

Figure 8 Response of CF-P-EOC (aerogel) after multiple cycles in acidic and alkaline gases and changes in Δ E

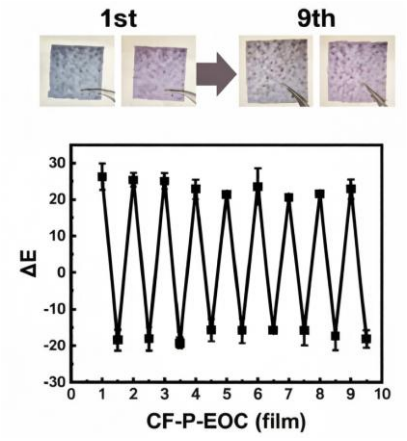

Figure 9 Response of CF-P-EOC (film) after multiple cycles in acidic and alkaline gases and changes in Δ E

3.3.2. Antioxidant Performance

The antioxidant activity, assessed via DPPH radical scavenging assays, provided crucial insights into the stability and release behavior of the active components. The CF-P(aerogel) showed a relatively stable scavenging activity over time, attributed to the inherent but stable antioxidant capacity of the embedded anthocyanins. The CF-P-EO(aerogel), containing free cinnamon oil, exhibited a sharp initial decline in activity, losing 28.25% of its scavenging capacity over 60 hours. This rapid decline is characteristic of volatile and oxidatively unstable compounds like essential oils, which readily evaporate or degrade when exposed to air from the open surface of the aerogel [43-45].

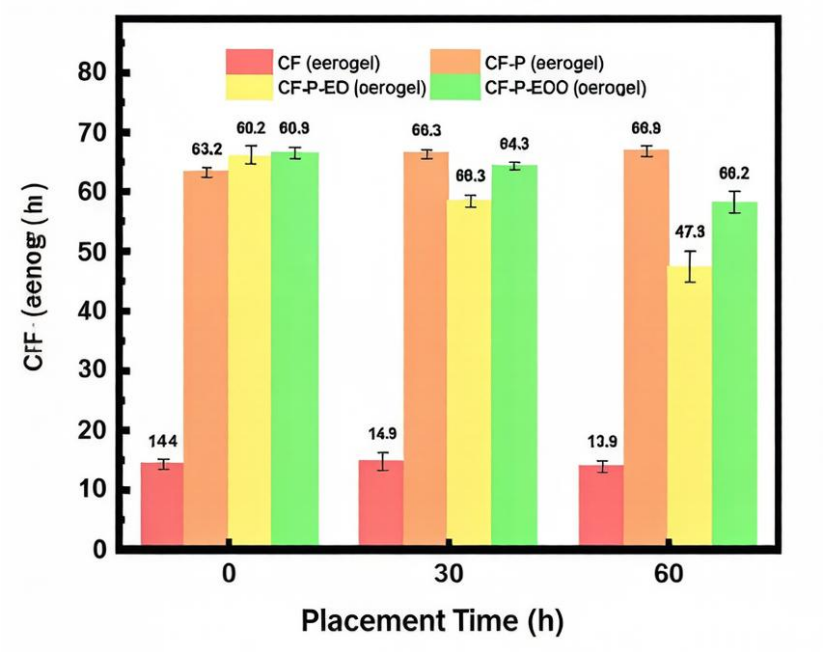

Figure 10 DPPH radical scavenging rates of CF (aerogel), CF-P (aerogel), CF-P-EO (aerogel), and CF-P-EOC (aerogel) at room temperature for 0 h, 30 h, and 60 h

In striking contrast, the CF-P-EOC(aerogel) demonstrated excellent retention of antioxidant activity, with only a 12.48% decline over the same period. This 55% improvement in retention highlights the efficacy of the β-cyclodextrin encapsulation strategy. The hydrophobic cavity of β-CD encapsulates the cinnamaldehyde molecules, forming an inclusion complex. This encapsulation serves multiple protective functions: (1) it significantly reduces the volatility of the essential oil, preventing rapid loss to the atmosphere; (2) it shields the active molecules from direct exposure to light and oxygen, thereby retarding oxidative degradation; and (3) it creates a reservoir from which the oil is released in a slow, controlled manner via dynamic association-dissociation equilibrium at the complex interface. This sustained-release mechanism is vital for extending the active shelf-life of the packaging material [46].

As shown in Figure 10, the aerogel with added anthocyanin (CF-P(aerogel)) exhibited a relatively stable DPPH radical scavenging rate throughout the test period. In contrast, the antioxidant activity of the aerogel with added cinnamon essential oil (CF-P-EO(aerogel)) showed a significant declining trend. The aerogel loaded with the cinnamon essential oil/β-cyclodextrin inclusion complex (CF-P-EOC(aerogel)) significantly improved its antioxidant stability, and the decline in its DPPH radical scavenging rate over time was markedly slowed. Within the 60-hour test period, the DPPH radical scavenging rate decline for the aerogel loaded with free cinnamon essential oil was as high as 28.25%, while for the aerogel loaded with the inclusion complex it was only 12.48%, demonstrating the good antioxidant performance of the CF-P-EOC(aerogel) material.

3.3.3. Essential Oil Release Characteristics

The release behavior of essential oil from the aerogel showed some environmental dependence. From Figure 11, it can be seen that the essential oil exhibited a rapid release phenomenon initially, and the release rate began to decrease significantly after about three days. This phenomenon indicates that essential oil directly loaded onto

the aerogel exhibits instability during storage and quickly reaches distribution equilibrium, making it difficult for the remaining essential oil to continue releasing.

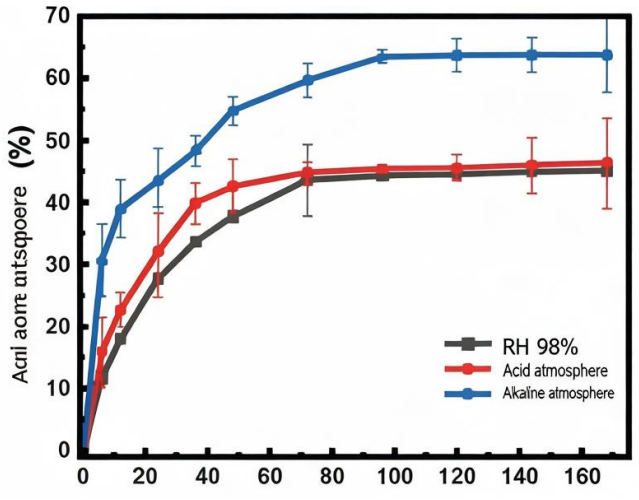

Figure 11 Release curves of cinnamon essential oil from CF-P-EO (aerogel) under relative humidity of 98%, acidic atmosphere, and alkaline atmosphere, respectively

Figure 12 shows that the essential oil treated with β-cyclodextrin encapsulation released more slowly initially compared to the unencapsulated one, and the release rate still showed a slow upward trend after 160 hours. This indicates that β-cyclodextrin encapsulation treatment can effectively slow down the release rate of essential oil, achieving a good sustained-release effect. This is because β-cyclodextrin, with its external hydrophilic and internal hydrophobic cavity structure, dynamically "locks" the essential oil molecules within the cavity through intermolecular hydrogen bonds, thereby enabling the action of β-cyclodextrin inclusion of cinnamon essential oil to further achieve the sustained release of cinnamon essential oil [47].

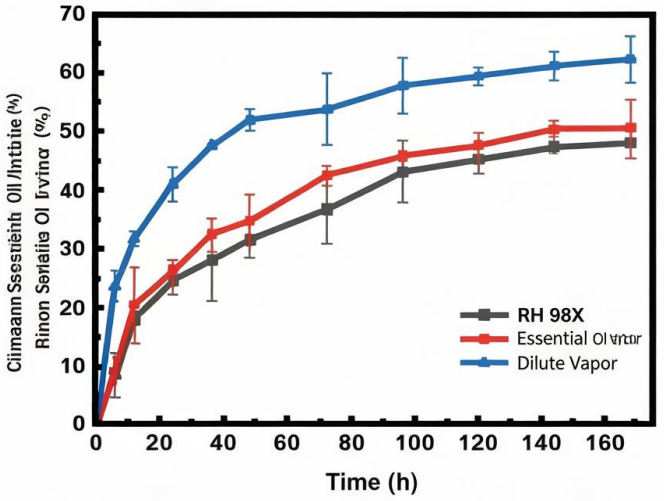

Figure 12 Release curves of cinnamon essential oil from CF-P-EOC (aerogel) under relative humidity of 98%, acidic atmosphere, and alkaline atmosphere, respectively

Comprehensive analysis of Figure 11 and 12 reveals differences in the release behavior of essential oil in different environments. In an ammonia atmosphere, the release rate of essential oil is faster, and the release percentage is higher. This difference is due to the dissociation of anthocyanin molecules under alkaline conditions leading to a reduction in covalent cross-linking sites, and the swelling of cellulose under alkaline and humid conditions. The hydrogen bonds between anthocyanin anions and cellulose hydroxyl groups break, resulting in a decrease in cross-linking degree, a looser aerogel structure, and consequently faster release of the essential oil.

3.3.4. Application of Bifunctional Aerogel for Shrimp Freshness Monitoring and Preservation

This study conducted a comparative investigation on the role of the bifunctional aerogel in shrimp freshness monitoring and preservation (Figure 13). In the control group with CF(aerogel), visible spoilage of the shrimp meat occurred at 12 hours, and both the anthocyanin-doped aerogel and film materials showed obvious color changes, indicating the sensitive responsiveness of anthocyanin to shrimp spoilage. At 48 hours, the shrimp meat was severely spoiled, and all corresponding anthocyanin-doped aerogels and film materials turned gray.

For the aerogel sample containing only anthocyanin, CF-P(aerogel), slight color change occurred at 6 hours, indicating its faster response speed to spoilage. The aerogel containing the essential oil inclusion complex, CF-P-EOC(aerogel), showed no obvious color change at 6 hours, because the addition of essential oil delayed the spoilage process. Comparing the aerogel (CF-P-EOC(aerogel)) with the film material (CF-P-EOC(film)), it can be observed that due to the denser structure of the film material prepared by casting, the response sensitivity of anthocyanin is weaker compared to the aerogel, the degree of color change is lower, and the dense structure is not conducive to the release of essential oil [48]. Therefore, its preservation and monitoring effects are weaker compared to the aerogel.

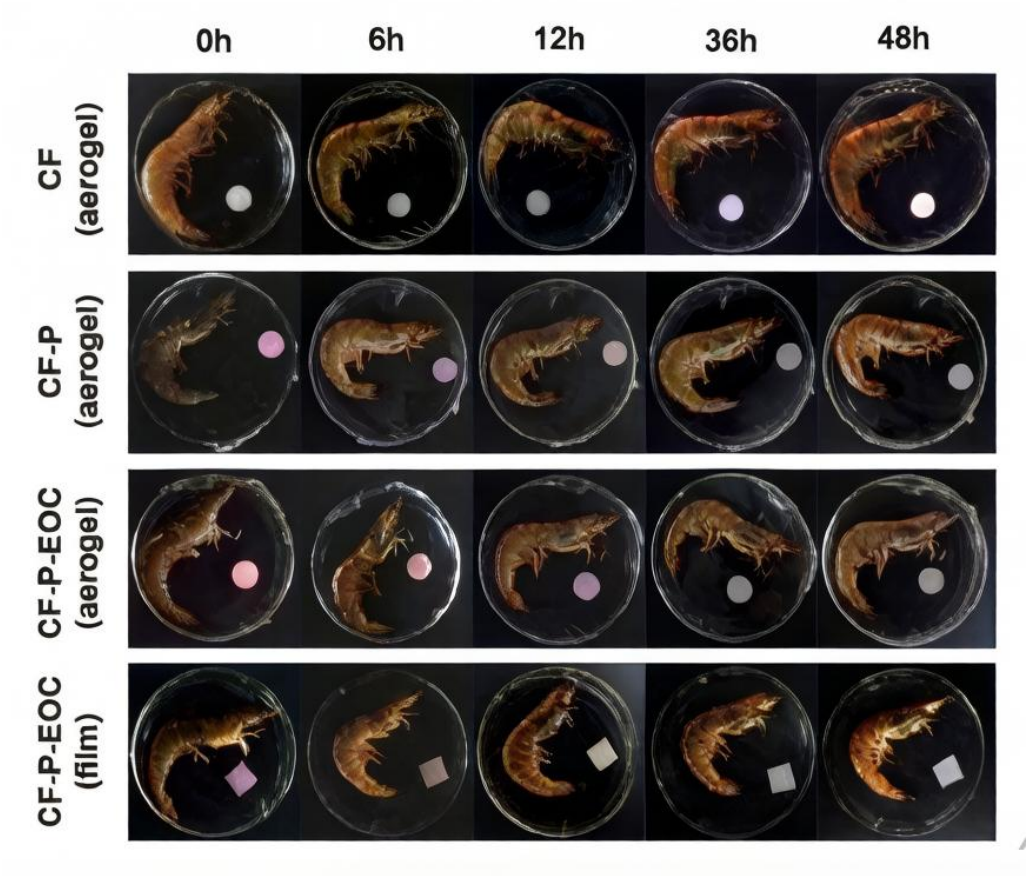

Figure 13 Application of CF (aerogel), CF-P (aerogel), CF-P-EOC (aerogel), and CF-P-EOC (film) for freshness monitoring and preservation of shrimp at 25 ℃

Based on the comprehensive experimental data and characterization results presented in the document, the superior performance of the bifunctional cellulose aerogel is fundamentally governed by a synergistic, multi-scale mechanism that integrates molecular-level chemical responsiveness, nano/micro-scale structural engineering, and macroscopic functional intelligence. The core sensing mechanism originates from the extracted purple corn cob anthocyanin (PCCA), whose molecular structure undergoes reversible, pH-dependent transformations; in the highly acidic state (pH ~3), the flavylium cation predominates, imparting a red color with a strong absorbance at 514 nm, but upon exposure to alkaline volatiles like ammonia (as produced during

seafood spoilage), a deprotonation equilibrium shifts the structure towards the quinonoidal base and eventually to the chalcone form under strong alkaline conditions, resulting in a visible color progression from red to bluish/gray and finally to orange-brown, which provides the direct visual indicator for freshness monitoring. The efficiency of this indicator is dramatically amplified by the engineered three-dimensional porous architecture of the aerogel scaffold, formed through a freeze-thaw and lyophilization process of a cellulose (CF) and polyvinyl alcohol (PVA) mixture; this highly porous and interconnected network, as revealed by SEM analysis, offers a vast surface area and unobstructed diffusion pathways, allowing rapid penetration and distribution of target gas molecules (e.g., NH_3) throughout the entire bulk of the material, which explains why the aerogel (CF-P-EOC(aerogel)) achieved a color difference (ΔE) > 7 within 1 second and a steady-state ΔE of 25.58, significantly outperforming the dense, solvent-cast film where gas permeation is slow and diffusion-controlled. Concurrently, the mechanical reinforcement mechanism is attributed to a multi-component hydrogen-bonding network where PVA and CF chains form a physically cross-linked base, and the introduction of PCCA, with its abundant phenolic hydroxyl groups, acts as a natural polyphenolic cross-linker that forms additional hydrogen bonds with the matrix, increasing the compressive stress from 2.85 MPa to 3.86 MPa; further enhancement to 5.90 MPa with the inclusion complex is due to the cinnamon oil/β-cyclodextrin particles acting as nano-fillers that impede polymer chain mobility and provide additional interfacial interactions. The preservation and sustained-release mechanism is centered on the β-cyclodextrin (β-CD) encapsulation of cinnamon essential oil (EOC); the hydrophobic cavity of β-CD encapsulates the volatile cinnamaldehyde, which serves three critical functions: it drastically reduces the oil's volatility and shields it from oxidative degradation (evidenced by the DPPH radical scavenging rate decline of only 12.48% over 60 hours for CF-P-EOC(aerogel) versus 28.25% for the free-oil system), it creates a reservoir for controlled release via a dynamic association-dissociation equilibrium, and most intelligently, it enables an environmentally triggered release profile where the release rate is accelerated under an alkaline atmosphere. This pH-triggered release is mechanistically linked to the swelling of the cellulose matrix and the breakdown of hydrogen bonds between deprotonated anthocyanin anions and cellulose under alkaline/humid conditions, which loosens the aerogel network and facilitates faster diffusion of the encapsulated oil, meaning the antimicrobial release is autonomously amplified precisely when spoilage alkalinity increases. Therefore, the overall system operates through an integrated "sense-and-act" feedback loop: the porous aerogel structure enables rapid gas sensing and color change via anthocyanin, while the same environmental trigger (alkaline pH) simultaneously promotes the enhanced release of the preserved antimicrobial from the β-CD complex, creating a self-regulating, intelligent packaging material where monitoring and preservation functionalities are intrinsically coupled through the responsive chemistry of its components and the mass-transfer advantages of its engineered nano-porous morphology.

Building upon the compelling visual evidence from the shrimp spoilage monitoring test, which clearly demonstrates the superior and rapid colorimetric response of the porous aerogel structures—particularly the CF-P-EOC (aerogel)—compared to the dense film counterpart, the future application prospects for this bifunctional material are highly promising within the next-generation intelligent food packaging sector. The key differentiator, as visually proven, is the aerogel's three-dimensional, open network which facilitates instantaneous interaction with spoilage volatiles, enabling real-time, visual freshness indication directly on the packaging for consumers and logistics managers. This addresses a critical market need for reducing food waste and enhancing safety in perishable supply chains, especially for seafood, poultry, and high-value meats. Beyond monitoring, the integrated, pH-triggered release of antimicrobials from the β-cyclodextrin inclusion complex—suggested by the delayed spoilage in corresponding samples—allows the packaging to transition from a passive container to an active preservation system that self-regulates antimicrobial deployment in response to spoilage onset. This dual "sense-and-act" functionality positions the material for applications in modified atmosphere packaging (MAP) for fresh cuts, ready-to-eat meals, and even for non-food items like sensitive pharmaceuticals or electronics where moisture and corrosion indication is needed. Future development will focus on scaling the freeze-drying production process, optimizing the aerogel's mechanical flexibility for integration into roll-to-roll packaging lines, and tailoring the anthocyanin source and antimicrobial agent for specific products (e.g., using different plant extracts for varying pH spoilage ranges). Ultimately, leveraging agricultural waste like purple corn cob aligns with circular economy principles, aiming to create affordable, biodegradable, and highly functional smart labels that could replace static expiry dates, significantly cut down global food waste, and provide a tangible, sustainable advancement in active and intelligent packaging technology.

4 Conclusion

Using purple corn cob as the main raw material, this study successfully prepared a bifunctional aerogel material with both seafood freshness monitoring and preservation functions. This bifunctional aerogel has a porous structure and achieves a stress of 5.90 MPa at 90% compressive strain. Compared to the cast film material with the same composition, this aerogel shows significant improvement in both pH response rate and effect (the response rate within 1 second is 2.66 times that of the film, and the steady-state color difference value is about 1.25 times that of the film). The aerogel loaded with the cinnamon essential oil/β-cyclodextrin inclusion complex also exhibited good sustained-release performance (the essential oil release rate of CF-P-EOC(aerogel) at 72 hours was only 90% of that of CF-P-EO(aerogel)). This aerogel material utilizes anthocyanin to achieve visual monitoring of freshness and inhibits microbial growth through the sustained release of essential oil, providing a new approach for intelligent preservation of seafood and enhancing the utilization value of the agricultural by-product purple corn cob.

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