

Development of nanocurcumin-incorporated biopolymer films for enhanced stability and pH-responsive monitoring

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ABSTRACT

This study developed biopolymer films using chitosan (CS), carboxymethyl cellulose (CMC), and pectin (Pec) incorporated with nanocurcumin (NCur). FE-SEM and ATR-FTIR analyses confirmed that NCur integration created denser microstructures through strengthened molecular interactions, improving tensile strength and elongation by up to 17% and 20%, respectively. Furthermore, NCur reduced moisture content and doubled antioxidant radical-scavenging activity. The films exhibited distinct pH-dependent colour transitions from yellow (acidic) to reddish-brown (alkaline). In beef storage trials, the films successfully monitored spoilage via recognisable colour shifts over two days. Overall, these NCur-loaded films demonstrate strong potential as sustainable, intelligent packaging for active preservation and real-time freshness monitoring.

Keywords: Biodegradable film; Nanocurcumin; pH indicator; Smart packaging.

1. INTRODUCTION

The accumulation of non-degradable plastic waste constitutes a critical global security and environmental challenge, notably within food packaging and logistics [1]. Petroleum-based plastics persist for centuries, threatening ecosystems and human health via microplastic release [2], making the development of biodegradable alternatives a strategic necessity to alleviate waste management burdens.

Biopolymers such as chitosan (CS), carboxymethyl cellulose (CMC), and pectin (Pec) have gained prominence as sustainable substitutes due to their biocompatibility [3, 4]. For instance, J. Fiallos-Núñez et al. [5] developed chitosan/gelatin films that degraded within 31 days in soil. Similarly, our previous research [6] confirmed that pectin films derived from Vietnamese grapefruit peel degrade rapidly within 30 days, significantly outperforming commercial polyethylene films in environmental compatibility. However, biopolymer films often suffer from inherent limitations, including low mechanical strength and high moisture absorption, which hinder their use in long-term storage applications [7, 8]. Strategies such as crosslinking and polymer blending have been employed to mitigate these issues [9,10]. In which blending with bioactive compounds is particularly effective, enhancing both structural integrity and functionality. S. Ojagh et al. [11] strengthened chitosan films using cinnamon essential oil, while S. Tie et al. [12] improved the mechanical and antioxidant profiles of CMC/chitosan/pectin films using procyanidins, thereby extending the shelf life of fresh produce.

Building on this trend, curcumin (Cur) has emerged as a multifunctional additive, offering

antioxidant activity and pH-responsive colour changes, making it suitable for smart packaging [13–15]. Several studies have demonstrated these advantages; for instance, P. Rachtanapun et al. [16] showed that Cur enhances antioxidant and antibacterial properties in chitosan films, while F. Wang et al. [17] reported photodynamic antibacterial activity in gelatin/chitosan - curcumin systems.

Nevertheless, Cur is limited by photosensitivity, aggregation, and poor dispersibility, which compromise film uniformity and colourimetric reliability [18]. To address these challenges, nanocurcumin (NCur) has emerged as a more efficient alternative. At the nanoscale, NCur addresses these limitations, exhibiting superior dispersibility and photostability at the nanoscale [19,20]. However, its potential role as a structural reinforcing agent within biopolymer matrices remains underexplored. Furthermore, there is a lack of comprehensive studies comparing the interactions of NCur with biopolymer matrices of distinct ionic characteristics and structures, specifically cationic Chitosan (CS), anionic Carboxymethyl Cellulose (CMC), and anionic/neutral Pectin (Pec). Understanding how NCur interacts with these diverse chemical environments is crucial for tailoring packaging materials with optimal performance.

Therefore, this study aims to develop and characterise pH-indicator films based on CS, CMC, and Pec incorporated with NCur. The films were evaluated for their morphological (FE-SEM), chemical (ATR-FTIR), and mechanical properties to assess structural stability. Furthermore, physical parameters, including adhesion, moisture content, and antioxidant capacity, were analysed. Finally, the practical efficacy of the NCur-indicator films was validated through the real-time monitoring of beef spoilage under storage conditions.

2. METHODOLOGY

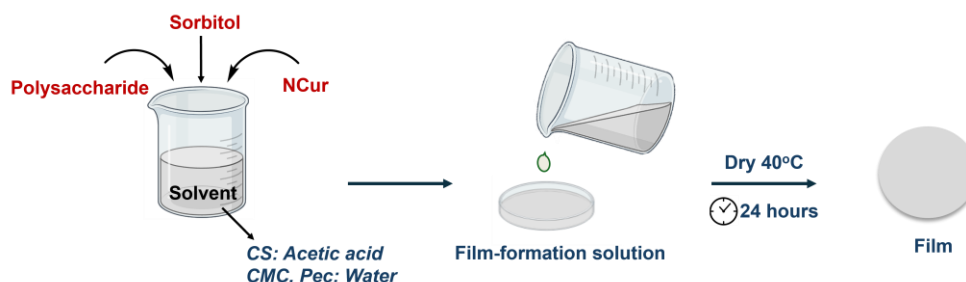
2.1. Materials

All chemicals used in this study were of analytical grade.

Commercial Chitosan (CS, degree of deacetylation $\geq 90\%$, $M_w \approx 150$ kDa) and Carboxymethyl Cellulose (CMC, purity $\geq 99\%$, $M_w \approx 250$ kDa) were used. Pectin (Pec, $M_w = 29.427$ g/mol, purity = 84.02%, and degree of esterification = 86.40%) was extracted from grapefruit peel [21]. Nanocurcumin (NCur, 50–70 nm, Vietnam Academy of Science and Technology) served as the pH indicator. Acetic acid (99.5%) and distilled water were used as solvents, with Sorbitol ($\geq 98\%$) as the plasticiser. Buffer solutions were prepared using boric acid, sodium borate, citric acid, and phosphate salts (all $\geq 99\%$, Xilong, China).

2.2. Preparation of film-forming solutions and film casting

Films were fabricated by solvent casting (*Scheme 1*). Base polymer solutions (2.0% w/v) were prepared by dissolving CS in 1.0% (w/v) acetic acid, while CMC and Pec were dissolved in distilled water. Sorbitol (0.4% w/v) was added to all solutions as a plasticiser. For indicator films, NCur (1.0% w/v) was dispersed into the polymer solutions under continuous stirring. The mixtures (10 mL) were cast into Petri dishes and dried at 40 °C for 24 h. The resulting films were designated as CS/NCur, CMC/NCur, and Pec/NCur [22].



Scheme 1. Film fabrication process.

2.3. Characterisation, morphology and structure

Visual appearance was photographed under visible light.

Surface morphology was examined using Field Emission Scanning Electron Microscopy (FE-SEM) on platinum-coated samples.

The chemical structure was analysed using ATR-FTIR spectroscopy (100 scans at a resolution of 4 cm^{-1}) on vacuum-dried films.

2.4. Mechanical properties

Film thickness was measured using a digital gauge. Tensile strength and elongation at break were evaluated using a universal testing machine (Zwick Z2.5, Germany) at $24 \pm 1\text{ }^\circ\text{C}$ and $50 \pm 1\%$ RH. Results were averaged from five replicates.

2.5. Physical properties

The adhesion strength of the films was determined by the peel-off force of the films applied to pig skin using the Zwick machine.

The moisture content (MC) was determined according to the method of Jelena Jovanovic and colleagues [23]. Films were weighed after drying (m_1), conditioned in 80% humidity for 24 hours, and weighed again (m_2). Moisture content (%) was calculated using:

$$\text{MC (\%)} = [(m_2 - m_1)/m_1] \times 100\% \quad (1)$$

pH sensitivity was assessed by mixing film solutions with buffers (pH 3–12) and measuring UV-Vis absorbance (350–780 nm) [24].

Antioxidant capacity was evaluated via hydrogen peroxide (H_2O_2) scavenging assay. Films were incubated in 40 mM H_2O_2 , and absorbance reduction at 230 nm was measured [25].

H_2O_2 reduction (%) was calculated by:

$$\text{H}_2\text{O}_2 \text{ reduction} = [(A_1 - A_2)/A_1] \times 100\% \quad (2)$$

where A_1 is the control absorbance, and A_2 is the absorbance after film treatment.

Reduction in H_2O_2 concentration indicates the antioxidant potential of NCur-indicator films, supporting their use in smart food packaging.

All measurements were conducted in triplicate, and results were expressed as mean \pm standard deviation. Statistical differences between the base and NCur-incorporated films, as well as among different polymer matrices (CS, CMC, Pec), were evaluated using analysis of variance (ANOVA).

2.6. Spoilage monitoring applications

The colour evolution was monitored daily to correlate film response with meat freshness [26]. Fresh beef slices ($3 \times 3\text{ cm}$) were covered with indicator films and stored in sterile Petri dishes at $34\text{ }^\circ\text{C}$ and 80% RH to simulate spoilage.

3. RESULTS AND DISCUSSION

3.1. Characterisation, morphology and structure

Visual appearance (Figure 1) revealed that the base CS, CMC, and Pec films were opaque white with relatively smooth surfaces, indicating stable network formation. The incorporation of NCur imparted a uniform orange colour to all samples, confirming successful integration and excellent miscibility of nanocurcumin within the biopolymer matrices.

Morphology (FE-SEM) micrographs (Figure 2) revealed distinct structural differences. Base films (a-1, a-2, a-3) displayed loosely packed structures with large, irregular pores and minor cracking. Conversely, NCur-incorporated films (b-1, b-2, b-3) exhibited significantly denser, more homogeneous morphologies with finer pores. This densification is attributed to enhanced hydrogen

bonding between NCur and polysaccharide chains, which improves packing density. Notably, CMC/NCur and Pec/NCur surfaces were smoother than CS/NCur, suggesting superior polymer-filler compatibility in these systems.



Figure 1. Visual appearance of the films.

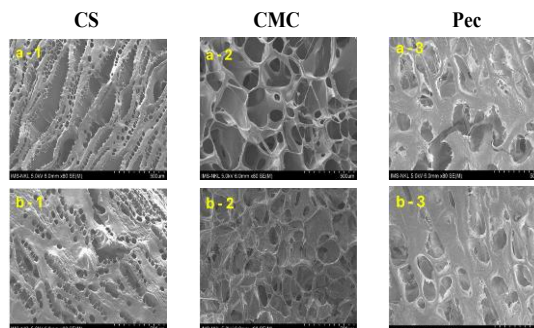
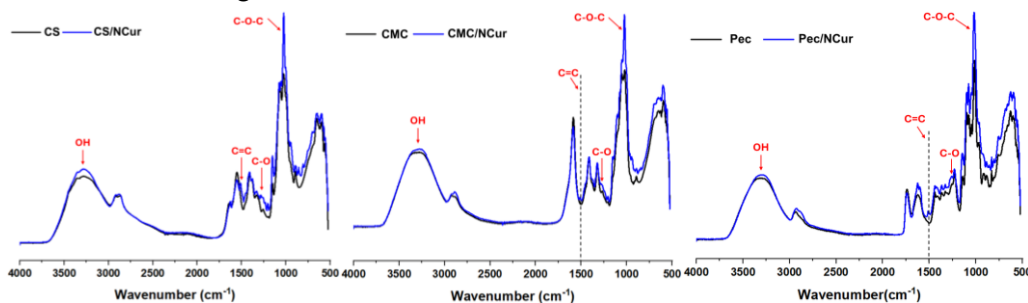


Figure 2. The FE-SEM of the films.

ATR-FTIR spectra (Figures 3–5) confirm the successful incorporation of NCur and reveal strong molecular interactions within the polymer matrices. Specifically, the broadening of the -OH band (3200–3600 cm^{-1}) indicates strengthened hydrogen bonding between NCur and the polymer chains [27]. Furthermore, the intensified peak at 1022 cm^{-1} (C-O-C stretching) suggests additional polar interactions between NCur diketone groups and the polysaccharide backbone [28]. Finally, the emergence of new bands at 1275 cm^{-1} (C-O) and 1500 cm^{-1} (aromatic C=C) in all NCur-loaded films unequivocally validates the presence of NCur’s aromatic structure [16, 29].

These results suggest that film formation involves a self-assembly process driven by hydrogen bonding and polar interactions, in which NCur acts as a crosslinker to reinforce film cohesion without toxic chemical agents.



(a) CS and CS/NCur; (b) CMC and CMC/NCur; (c) Pec and Pec/NCur.
Figure 3. The FTIR of the films.

3.2. Mechanical properties

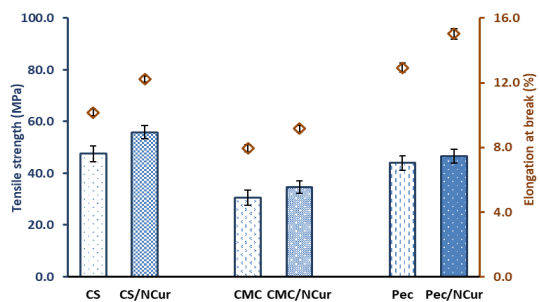


Figure 4. Mechanical strength of films.

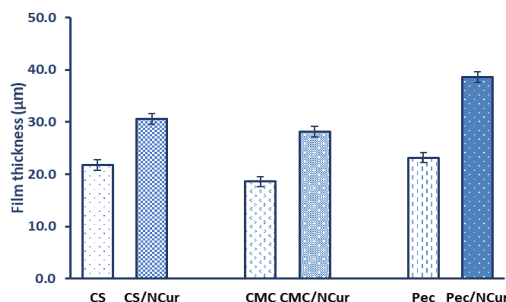


Figure 5. Film thickness of films.

Mechanical testing (Figure 4) showed that NCur incorporation significantly improved tensile strength and elongation at break for all film types. Tensile strength increased from 47.62 to 55.91 MPa for CS, from 30.58 to 34.62 MPa for CMC, and from 43.95 to 46.61 MPa for Pec. Corresponding improvements in elongation at break were also observed, increasing from 10.16% to 12.23% (CS), from 7.95% to 9.16% (CMC), and from 12.94% to 15.02% (Pec).

Film thickness (Figure 5) increased slightly upon NCur incorporation, indicating successful dispersion of NCur within the polymer matrices and resulting in a more compact internal network. This improvement is attributed to the uniform dispersion of NCur nanoparticles, which facilitates stress transfer through the polymer matrix. By functioning as "physical crosslinkers," NCur molecules reinforce the intermolecular forces within the CS, CMC, and Pec networks, resulting in films that are mechanically superior to their neat counterparts.

3.3. Physical properties

3.3.1. Film adhesion testing

Adhesion strength is a crucial parameter for packaging applications, as stable contact between the indicator film and the food surface is a prerequisite for accurate real-time freshness monitoring. As shown in Figures 6 and 7, NCur improved adhesion across all matrices: CS (0.50-0.53 N), CMC (0.57-0.59 N), and Pec (0.56-0.58 N). This enhancement is driven by stronger intermolecular bonding, which ensures better surface contact and greater flexibility for food packaging applications.

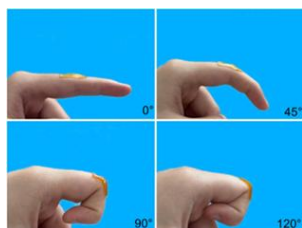


Figure 6. Illustration of the film's adhesion.

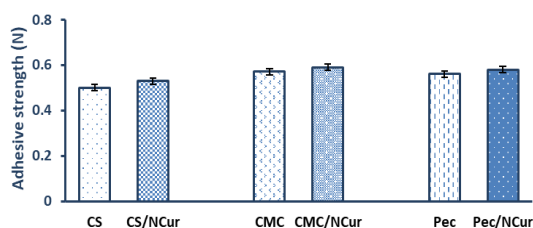


Figure 7. Adhesive strength of the films.

3.3.2. Moisture content

Moisture content is a critical parameter governing water retention and mechanical stability. Lower moisture levels enhance swelling resistance, ensuring the film's suitability for practical food packaging. As shown in Figure 8, NCur incorporation significantly improved moisture resistance. The ANOVA results confirmed that NCur acted as an effective reinforcing agent, resulting in a significant reduction in moisture content ($p < 0.05$) across all modified films, in detail CS (47.01% → 35.08%), CMC (92.02% → 67.20%), and Pec (58.77% → 27.34%). This reduction is due to strong interactions between NCur and the polymer hydroxyl groups, which limit the number of water-binding sites. Lower moisture content enhances structural stability and resistance to swelling [31].

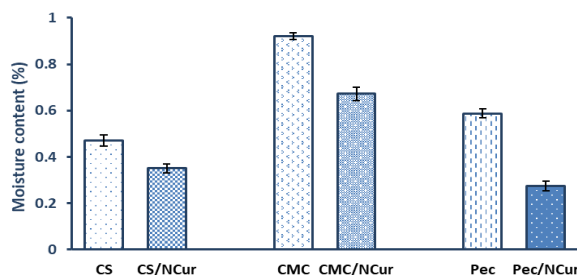


Figure 8. Moisture content of the films.

3.3.3. pH sensitivity of NCur-indicator films

UV-Vis spectroscopy effectively assesses pH-responsiveness, as structural changes (e.g., protonation, tautomerism) directly alter the electronic absorption profile [30]. Since NCur exhibits well-defined pH-dependent spectral shifts, UV-Vis analysis provides an effective means to characterise the pH sensitivity of NCur-indicator films.

The UV-Vis spectra and colour responses of the CS/NCur, CMC/NCur, and Pec/NCur films under different pH conditions (Figures 9–11) demonstrate a clear pH-dependent optical behaviour characteristic of NCur-based indicators. i) Acidic (pH 3–5): Films appeared bright yellow with an absorption peak at 420–430 nm (keto form of NCur) [31]. ii) Neutral (pH 6–8): Films shifted to pale yellow with slightly decreased absorbance [32]. iii) Alkaline (pH > 8): A visible shift to reddish-brown occurred, accompanied by a bathochromic shift in absorbance. This indicates the deprotonation of NCur into its ionised enol form [33].

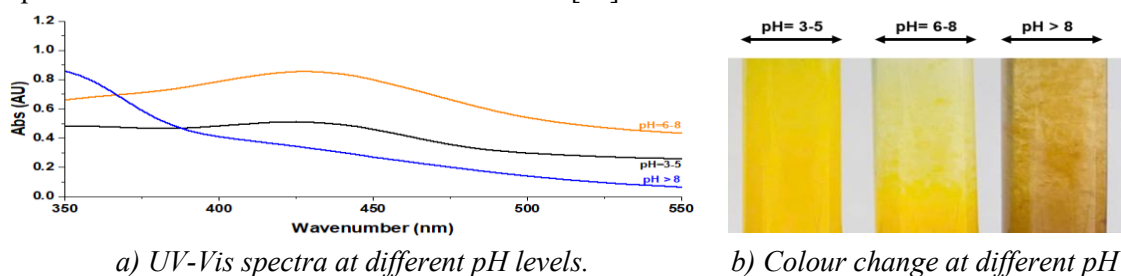


Figure 9. Colour and UV-Vis spectra under different pH conditions of the CS/NCur films.

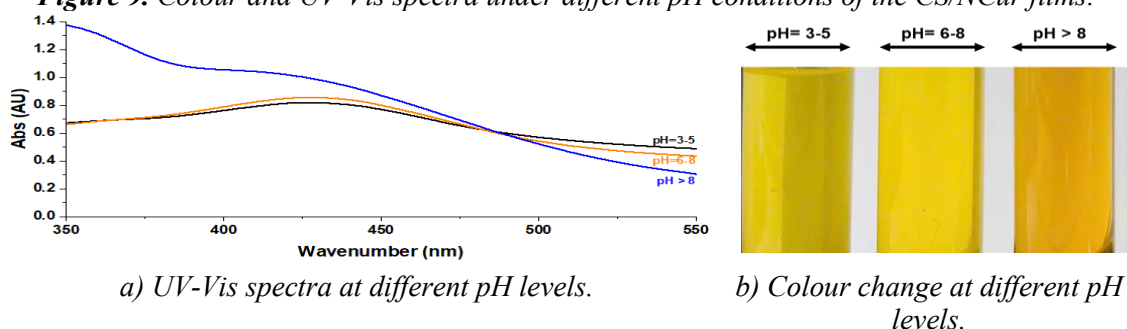


Figure 10. Colour and UV-Vis spectra under different pH conditions of the CMC/NCur films.

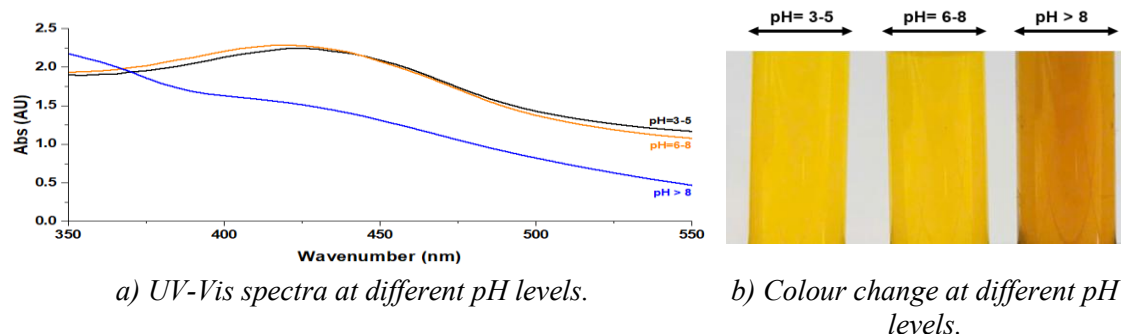


Figure 11. Colour and UV-Vis spectra under different pH conditions of the Pec/NCur films.

These distinct optical transitions confirm that NCur retains its pH-sensitivity within the polymer matrix, making these films suitable for colourimetric monitoring.

3.3.4. Antioxidant capacity using hydrogen peroxide (H₂O₂)

Antioxidant activity is critical for preventing ROS-driven food spoilage. Hydrogen peroxide (H₂O₂), a common ROS precursor that readily generates free radicals, was used as the model

oxidant to evaluate the films' protective efficacy [34].

As shown in Figure 12, statistical analysis indicated that all NCur-incorporated films exhibited significantly higher antioxidant activity ($p < 0.05$) than their base counterparts. H_2O_2 scavenging rates increased from 43.34% to 83.25% for CS, 41.37% to 72.41% for CMC, and 39.41% to 73.40% for Pec. The phenolic and methoxy groups of NCur effectively neutralise free radicals. The superior performance of CS/NCur is likely due to the specific interaction between NCur and the chitosan amine groups, forming a compact structure that inhibits oxidant penetration.

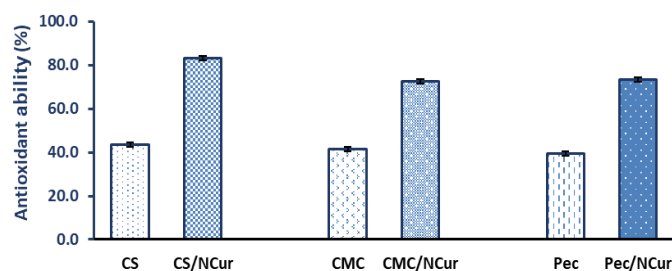


Figure 12. Antioxidant ability of films.

3.4. Spoilage monitoring applications

The colour evolution of the NCur-indicator films during beef storage at 34 °C and 80% relative humidity is presented in Figure 13. When placed on the meat surface, all NCur-indicator films exhibited clear and progressive colour transitions that correspond to the biochemical changes associated with spoilage.

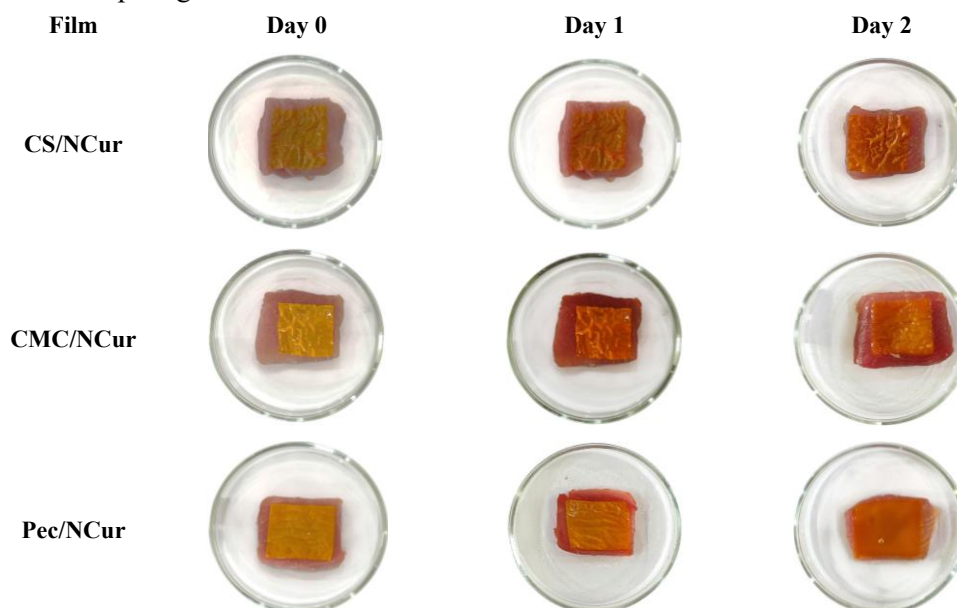


Figure 13. Colour changes of NCur-incorporated films during 2 days of beef storage.

At the beginning of storage (Day 0), the films remained bright/pale yellow, reflecting the slightly acidic/neutral surface pH of fresh meat [35].

By the second day of storage, A distinct colour transition to orange/reddish-brown was observed. This shift corresponds to the release of volatile basic nitrogen compounds (e.g., ammonia, amines) during protein degradation, which creates an alkaline environment and triggers the deprotonation of NCur [36].

The strong correlation between film colour and spoilage progression validates the potential of NCur-

incorporated films as reliable, non-destructive visual indicators for real-time freshness monitoring.

4. CONCLUSIONS

This study successfully fabricated films using chitosan (CS), carboxymethyl cellulose (CMC), and pectin (Pec) incorporated with nanocurcumin (NCur). Acting as both a structural reinforcer and pH indicator, NCur significantly improved the mechanical and physical properties of all matrices. FE-SEM and ATR-FTIR analyses confirmed strong polymer-NCur interactions, resulting in films with superior tensile strength, flexibility, thermal stability, and lower moisture content. Additionally, the films exhibited distinct pH-responsive colour transitions and enhanced antioxidant activity. Practical efficacy was validated through meat storage trials, where colour shifts accurately correlated with spoilage. Overall, NCur integration enhances the physicochemical integrity of biopolymer films while enabling reliable colourimetric sensing.

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TÓM TẮT

Phát triển màng polyme sinh học tích hợp nanocurcumin nhằm tăng cường độ bền và khả năng theo dõi sự biến đổi pH

Nghiên cứu này phát triển các màng biopolymer thông minh từ chitosan (CS), carboxymethyl cellulose (CMC) và pectin (Pec) kết hợp với nanocurcumin (NCur). Các phân tích FE-SEM và ATR-FTIR xác nhận việc tích hợp NCur đã tạo ra cấu trúc đặc biệt hơn, giúp cải thiện độ bền kéo và độ giãn dài lần lượt lên tới 17% và 20%. Bên cạnh việc giảm độ ẩm và tăng gấp đôi hoạt tính chống oxy hóa, màng còn thể hiện khả năng chuyển màu rõ rệt theo pH từ vàng (acid) sang nâu đỏ (kiềm). Thử nghiệm bảo quản thịt bò cho thấy màng theo dõi chính xác quá trình hư hỏng thông qua sự thay đổi màu sắc trong hai ngày. Kết quả khẳng định tiềm năng lớn của màng tích hợp NCur như một giải pháp bao bì thông minh giúp bảo quản chủ động và chỉ thị độ tươi thực phẩm.

Từ khoá: Màng phân hủy sinh học; Nanocurcumin; Chỉ thị pH; Bao bì thông minh.