

Biodegradable poly (lactic acid)/ZnO/Pluronic composite films: Mechanical properties, antibacterial performance, and fruit preservation

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Received 21 Dec. 2025; Revised 06 Feb. 2026; Accepted 10 Feb. 2026; Published 25 Feb. 2026.

DOI: <https://doi.org/10.54939/1859-1043.j.mst.109.2026.78-86>

ABSTRACT

*In this study, composite films based on Poly(lactic acid) (PLA), Zinc Oxide (NPs) and Pluronic P123 (0.5–1.5 wt% ZnO/Plu) were fabricated and characterized with respect to structure, mechanics, antibacterial behavior, and fruit-preservation performance. An optimum was observed at 1.0 wt% ZnO/Plu, where tensile strength (15.7 MPa), elongation at break (~0.7%), and reduced 24-h water solubility coincided with uniformly dispersed ZnO/Plu domains confirmed by SEM/EDX and FTIR. The films exhibited strong, dose-dependent antibacterial activity against *Escherichia coli* and *Bacillus subtilis*, with maximal inhibition at 1.0 wt% and a slight decline at 1.5 wt% due to nanoparticle aggregation. When used as active packaging for bananas, the 1.0 wt% ZnO/Plu film most effectively limited weight loss, maintained titratable acidity and vitamin C, and preserved visual quality, indicating its potential as a biodegradable active packaging material for postharvest applications.*

Keywords: PLA; ZnO NPs; Pluronic P123; Active food packaging; Fruit preservation.

1. INTRODUCTION

Poly(lactic acid) (PLA) is a leading biopolymer for sustainable food packaging because of its renewable origin, biodegradability, and good film-forming ability [1]. However, its brittleness, relatively high gas and water vapor permeability, and lack of intrinsic antimicrobial activity restrict its use for actively protecting fresh, highly respiring fruits [2]. Consequently, current research focuses on PLA-based nanocomposite films containing functional fillers to enhance mechanical strength, barrier properties, and antimicrobial performance, thereby extending shelf life. Among inorganic nanomaterials, zinc oxide nanoparticles (ZnO NPs) are particularly attractive since they are generally recognized as safe at appropriate doses, provide broad-spectrum effectively antimicrobial activity, and confer UV-shielding and mechanical reinforcement to polymer matrices [3].

ZnO-based active packaging can effectively inhibit both Gram-positive and Gram-negative bacteria on food surfaces, reduce weight loss, and delay physicochemical deterioration of fruits during storage [4]. PLA/ZnO bionanocomposite films, for example, have achieved complete inhibition of *Escherichia coli* at relatively low ZnO loadings, underscoring the importance of nanoparticle–bacteria interactions and reactive oxygen species in antimicrobial activity [5]. Comparable antimicrobial and quality-preserving effects have been reported for ZnO-containing biopolymer films on grapes, pomegranate arils, and other fruits, where the nanocomposite layer functions as a semi-permeable barrier regulating moisture and gas transfer while limiting microbial spoilage [6]. Nonetheless, realizing their full potential requires achieving homogeneous ZnO dispersion and strong interfacial compatibility within PLA, as excessive nanoparticle loading promotes aggregation and degrades mechanical properties.

Pluronic block copolymers, particularly Pluronic P123, offer an attractive strategy to address these compatibility issues due to their amphiphilic PEO–PPO–PEO structure, excellent biocompatibility, and their ability to act as non-ionic surfactants and soft segments in polymer blends [7]. In polymeric films and hydrogels, Pluronic-based systems have been shown to

modulate mechanical behavior, tune swelling and erosion profiles, and improve the dispersion of embedded particles or active agents through steric stabilization and interfacial interactions [8]. Furthermore, studies on PLA/Pluronic-type systems have demonstrated that incorporating Pluronic segments into PLA matrices can alter hydration, microstructure, and degradation behavior, thereby enabling the design of degradable polymeric systems with controlled-release characteristics [9]. By analogy, these findings suggest that Pluronic P123 could serve as a compatibilizer or film agent for ZnO nanoparticles within PLA, improving nanoparticle dispersion and interfacial adhesion and, consequently, enabling a more favorable balance between strength, flexibility, and antimicrobial performance of the resulting composite films [10].

Despite extensive work on PLA/ZnO nanocomposites and the broad biomedical use of Pluronic-based materials, PLA–ZnO/Pluronic composite films have rarely been investigated as active packaging for fresh fruit, particularly with respect to linking their physicochemical properties to visual quality attributes during storage [11, 12]. This study, therefore, develops PLA–ZnO/Pluronic composite films and evaluates their mechanical and barrier properties together with their ability to preserve fruit appearance and quality. The focus is (i) to clarify how Pluronic-assisted ZnO incorporation affects film structure and properties and (ii) to visually and quantitatively assess the films' performance in maintaining fruit quality, thereby informing the design of multifunctional, biodegradable postharvest packaging materials.

2. EXPERIMENTAL

2.1. Materials

All chemicals used were of analytical grade unless otherwise specified. Poly lactic acid (PLA, 98% purity), dichloromethane (CH_2Cl_2 , 99.5% purity), sodium hydroxide (NaOH, 96% purity), ethanol ($\text{C}_2\text{H}_5\text{OH}$, 99.7% purity), potassium iodide (KI, 99% purity), soluble starch ($\text{C}_6\text{H}_{10}\text{O}_5$)_n, silica gel ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$) and phenolphthalein ($\text{C}_{20}\text{H}_{14}\text{O}_4$) were purchased from Xilong Scientific (Jiangsu, China). Zinc acetate dihydrate ($(\text{CH}_3\text{COO})_2\text{Zn} \cdot 2\text{H}_2\text{O}$, 99% purity) and ammonia solution (25% NH_4OH) were obtained from Merck (Darmstadt, Germany). Polyethylene-polypropylene glycol (Pluronic P123) was purchased from Sigma-Aldrich (St. Louis, MO, USA). Zinc oxide nanoparticles (ZnO NPs, white, particle size range of 20 – 40 nm) were synthesized in-house via a previously established sol-gel method [13].

2.2. Experimental methods

2.2.1. ZnO/Pluronic composite synthesis

A precursor solution (4 mM) was prepared by dissolving 2.4 g of Pluronic P123 in 500 mL of a 1:1 (v/v) ethanol-to-water mixture. ZnO NPs were initially dispersed in a 1:1 (w/w) ethanol-water mixture and stirred uniformly for 30 minutes. The solution (4 mM) was then added to the ZnO suspension, and the resulting mixture was sonicated with an ultrasonic probe to achieve uniform dispersion, then centrifuged, washed multiple times, and dried to obtain the powder.

2.2.2. PLA-ZnO/Pluronic composite film synthesis

The ZnO/Pluronic nanoparticles (0.2 g) were dispersed and sonicated in 20 mL of ethanol. PLA (0.8 g) was dissolved in 38 mL of dichloromethane, then 0.5 mL of the ZnO/Pluronic suspension was added and sonicated to ensure homogeneous dispersion. 10 mL of the solution was cast onto a petri dish (radius 5 cm) and left at ambient conditions for 2 h. The film was then peeled off and dried under vacuum at constant temperature (50 °C) for 12 h. The films were stored in sealed containers, protected from moisture and light. For mechanical testing, films were prepared on plastic substrates (15 cm × 25 cm) following the same procedure.

2.2.3. Characterization of PLA/ZnO/Pluronic P123 composite films

a) *Spectroscopic and morphological analysis.* Molecular structure was analyzed by FT-IR

(Bruker spectrometer). Surface morphology and elemental composition were characterized by SEM-EDX (JEOL JMS-6490 and Hitachi S-4800).

b) Mechanical testing. Film thickness was measured with a Mitutoyo micrometer (PCM 137). Tensile strength was determined according to ASTM D882-95. For solubility, films (3 × 3 cm) were dried at 60 °C under vacuum to constant mass (W_o), immersed in 20 mL distilled water at 25 °C for 24 h with agitation, then blotted, and redried under the same conditions to constant mass (W_s). Water solubility was calculated from W_o and W_s , using the following equation:

$$\%H = \frac{W_o - W_s}{W_o} \cdot 100\% \quad (1)$$

2.2.4. Assessment of antibacterial properties

The antimicrobial potential of PLA/ZnO-Plu composite films was evaluated against two bacterial strains: gram-positive *Bacillus subtilis* and gram-negative *Escherichia coli*.

a) Semi-Solid agar overlay technique. Nutrient broth (20 g/L) was prepared in distilled water and solidified with either 8 or 20 g/L agar. Media were sterilized at 121 °C, 15 psi for 20 min, cooled to 45–50 °C, inoculated with bacteria, and poured into sterile Petri dishes. After solidification, circular film discs (1 cm diameter) were placed on the agar and incubated at 37 °C for 24 h, after which inhibition zones and bacterial growth were recorded.

b) Bacterial suspension immersion and disk diffusion assay. Bacterial inoculum was added to sterile test tubes containing nutrient broth. Rectangular film samples (dimensions: 1 cm × 2 cm) were simultaneously placed into the inoculated medium in each test tube. After incubation periods of 6 hours and 24 hours, samples were withdrawn.

2.2.5. Assessment of fruit storage preservation efficacy

a) Mass loss during storage period

Bananas were weighed at 24-hour intervals throughout the storage period. Mass loss measurements were recorded in a data table to enable continuous monitoring of water loss and fruit degradation kinetics.

b) Total titratable acidity (TTA) content

Total titratable acidity was determined by neutralization titration following Vietnamese Standard TCVN 5483:2007. Banana samples were homogenized in 40 mL of distilled water, centrifuged at 6000 rpm for 10 min, and the supernatant was filtered. A 5 mL aliquot was mixed with 2–3 drops of 0.1% phenolphthalein and titrated with 0.1 N NaOH to a persistent pink endpoint. Acidity, expressed as % malic acid equivalent, was then calculated similarly to the previous report [14].

c) Vitamin C content determination

Vitamin C content was determined using iodometric titration according to TCVN 11168:2015. Five grams of the orange sample were homogenized with 10 mL of 2% HCl solution and subsequently diluted to 50 mL in a volumetric flask. An aliquot of 20 mL was transferred to a conical flask, starch indicator solution (1%) was added, and the mixture was titrated with 0.01 N iodine solution until a persistent blue color appeared for approximately 20 seconds. The experiment was repeated three times, and the mean value was recorded.

Vitamin C content (mg/100 g) was calculated using the following equation:

$$X = \frac{V \times V_1 \times 0.00088 \times 100}{V_2 \times m} \quad (2)$$

where X = vitamin C content (mg/100 g); V = volume of 0.01 N iodine solution consumed (mL); V_1 = total volume of diluted sample (mL); V_2 = volume of diluted sample used for

titration (mL); m = mass of sample (g); 0.00088 = mass of vitamin C (g) equivalent to 1 mL of 0.01 N iodine solution.

3. RESULTS AND DISCUSSION

3.1. Optimization of PLA-ZnO/Plu film synthesis

From the results summarized in table 1, it can be observed that the film thickness increases progressively with increasing ZnO/Plu content. Within the investigated concentration range, the thickness increases, suggesting that the films' stiffness is likely to increase, which may adversely affect their flexibility and, consequently, their suitability for packaging applications [15, 16]. Therefore, the tensile strength and elongation at break of the films were subsequently evaluated.

Table 1. Film thickness corresponding to different ZnO/Plu concentrations.

Conc/Time	1	2	3	4	Avg.
PLA-0.5% ZnO/Plu	0.038	0.042	0.038	0.055	0.04325
PLA-1.0% ZnO/Plu	0.088	0.076	0.071	0.052	0.07175
PLA-1.5% ZnO/Plu	0.071	0.083	0.089	0.068	0.07775

As shown in figure 1, both tensile strength and elongation at break increase with ZnO/Plu content relative to pure PLA, but in a nonlinear manner. At 0.5% ZnO/Plu, the yield and ultimate tensile strength increase to 5.85 and 6.56 MPa, respectively, with a slight rise in elongation at break and a marked reduction in solubility, indicating the onset of mechanical reinforcement and improved stability. The optimum is obtained at 1.0% ZnO/Plu, where yield strength, tensile strength (~15.7 MPa), and elongation at break (~0.7%) reach their maximum values, while 24-hour solubility is greatly reduced, consistent with an optimally dispersed filler phase and strong interfacial interactions. Further increasing the loading to 1.5% ZnO/Plu lowers both strengths to 5.22 MPa and reduces elongation at break to 0.3%, despite a slight additional decrease in solubility, suggesting nanoparticle aggregation or phase separation that introduces microdefects and weak interfaces, thereby degrading the mechanical performance [17, 18].

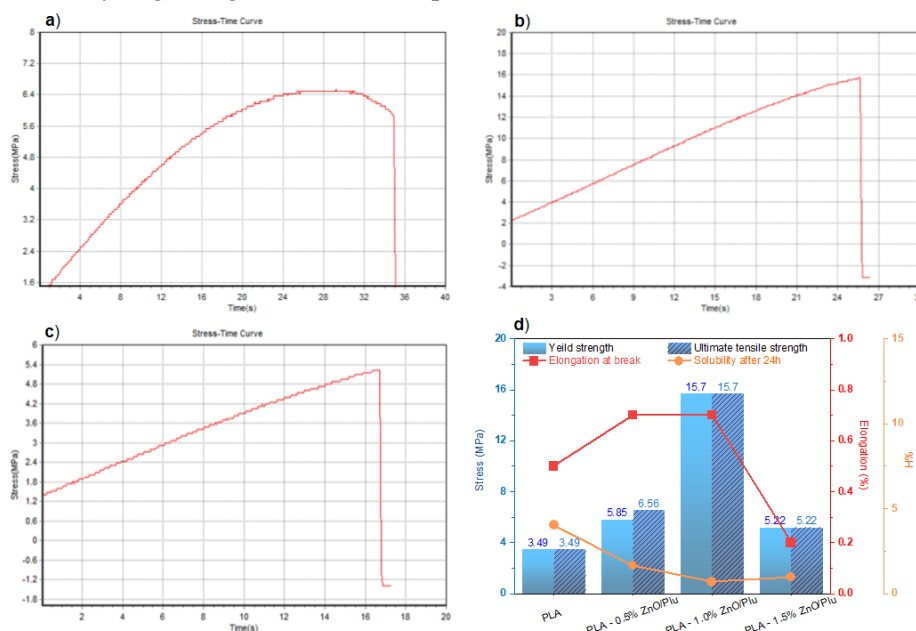


Figure 1. Tensile strength (R_b) measurement results of the film at ZnO/Plu concentrations from 0.5 to 1.5% (a-c) and corresponding mechanical properties (d).

It is observed in figure 2 that the yield strength and ultimate tensile strength increase only

slightly relative to pure PLA, while the elongation at break also exhibits no significant improvement. This indicates that ZnO at low loading provides only limited reinforcement, mainly because the extent of interaction and interfacial adhesion between the ZnO particles and the PLA matrix is not yet optimized. In contrast, the sample containing only Pluronic (PLA-1% Plu) shows a clear deterioration in mechanical performance: both the yield strength and ultimate tensile strength are lower than those of pure PLA, and the elongation at break decreases to around 0.3%. These findings suggest that free Pluronic tends to plasticize or weaken the PLA network, possibly due to phase separation, leading to a softer but not stronger material and even imparting increased brittleness, in agreement with previous reports in the literature [19, 20].

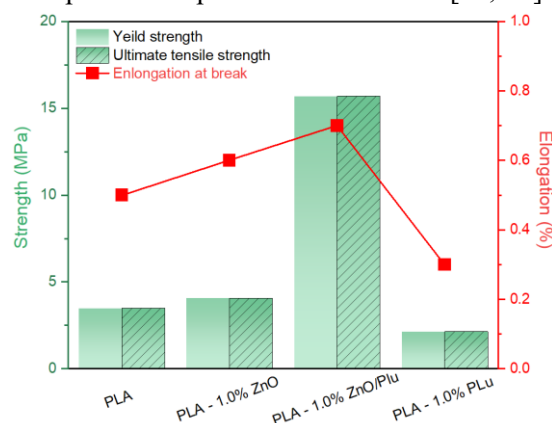


Figure 2. Comparison of mechanical properties of pure PLA, PLA-1.0% ZnO, PLA- 1.0% ZnO/Plu, and PLA-1.0%Plu films.

3.2. Characterization of properties and morphology

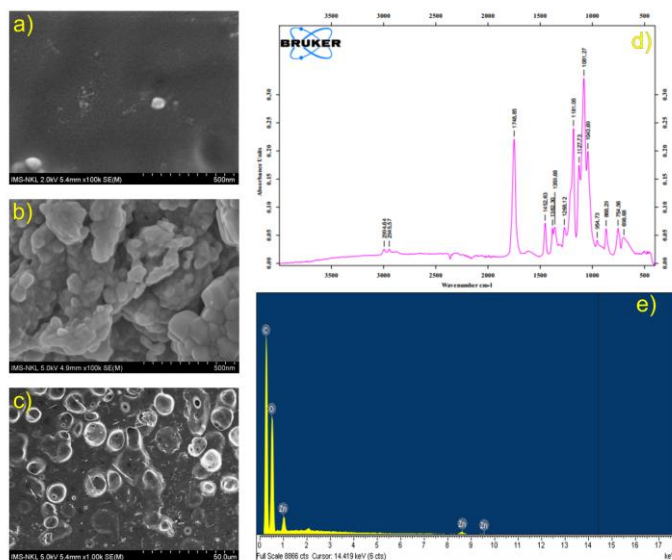


Figure 3. Morphologies of pure PLA film, 1% ZnO/Plu composites, and PLA-1.0% ZnO/Plu film (a-c); FTIR (d) and EDX (e) of PLA-1.0% ZnO/Plu film.

The SEM images in figure 3 (a-c) show that the morphology of the films before and after modification with ZnO-Plu changes markedly. The pure PLA film exhibits a smooth and homogeneous surface, whereas the modified film displays a relatively uniform distribution of spherical ZnO-Plu domains. In the SEM micrograph of the isolated ZnO/Plu, ZnO nanoparticles

with sizes in the range of approximately 50–80 nm are observed on the Plu surface, although they still appear in the form of aggregates or clusters. However, after incorporation into the PLA matrix, the ZnO/Plu phase becomes more uniformly dispersed. This improvement is attributed to the ultrasonication treatment applied to the suspension prior to film casting, which promotes the breakup of aggregates and enhances the dispersion of ZnO nanoparticles within the polymer matrix [21]. The Zn and other element content in the films was further evaluated, and the results are presented in figure 3e.

The FTIR spectra in figure 3d further confirm the presence of the different components in the PLA/ZnO–Plu system. The bands at 1748 cm^{-1} and 1081.27 cm^{-1} are characteristic of the stretching vibrations of the ester -C=O- and -C-O- groups in the PLA backbone [22]. The absorption bands at 2994 and 2945 cm^{-1} are assigned to the stretching vibrations of -C-H- in CH_3 groups present in both PLA and Pluronic. In addition, the peaks at 1359, 1128, and 1181 cm^{-1} correspond to the stretching vibrations of -C-H- in the PLA and Plu chains in the films. In the low-wavenumber region between 700 and 490 cm^{-1} , weak bands attributed to the Zn–O stretching vibration of ZnO nanoparticles are observed. Overall, the infrared spectroscopic analysis confirms the presence of all constituents forming the PLA/ZnO–Plu film system [22, 23].

3.3. Evaluation of the antibacterial activity of PLA/ZnO films

Figure 4 shows that the qualitative observations are consistent with the quantitative bacterial counts presented in the line graph. The viable cell concentration in both strains remains high in the control and PLA samples, confirming the absence of intrinsic antibacterial properties in the pure PLA film. Upon addition of 0.5% ZnO/Plu, the bacterial concentration drops significantly, indicating a strong bacteriostatic effect.

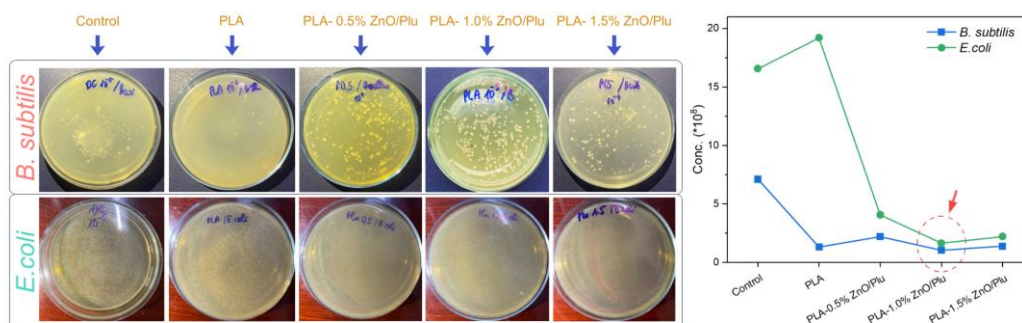


Figure 4. The antibacterial activity of the modified films against *B.subtilis* and *E.coli*, and the corresponding bacterial concentrations after 24 hours.

The minimum concentration is reached for PLA–1.0% ZnO/Plu, where the viable cell concentration for *E. coli* and *B. subtilis* is reduced to a very low level (highlighted by the red arrow), demonstrating an optimum loading at which the antimicrobial activity of the ZnO/Plu phase is most effective. At 1.5 wt% ZnO/Plu, a slight increase in cell concentration is detected, suggesting that excessive Plu P123 may lead to partial aggregation and reduced availability of active ZnO surfaces, thereby slightly diminishing the antibacterial efficiency [24–26]. Overall, the results confirm that incorporation of an appropriate amount of ZnO/Plu endows PLA films with strong, dose-dependent antibacterial activity against both Gram-positive and Gram-negative bacteria, with 1.0 wt% identified as the optimal concentration in this study.

3.4. Assessment of the preservation performance for fruit

Based on figure 5a, after 7 days of storage, bananas wrapped with active films exhibit a markedly better visual quality than those stored under normal (uncoated) conditions. Among all treatments, the PLA/1.0% ZnO–Plu film provides the most effective preservation, as evidenced by

delayed browning and reduced surface defect incidence. This observation is consistent with previous reports showing that ZnO-containing films and nanocomposite packaging can retard senescence and maintain the external appearance of climacteric fruits, including bananas, by forming a semipermeable barrier that moderates gas and moisture transfer and suppresses microbial growth [27].

Consistent with the visual observations, figures 5b and 5c show that the PLA/1.0% ZnO–Plu film markedly reduces weight loss and better preserves total titratable acidity compared with uncoated and neat PLA films. Lower weight loss implies reduced transpiration and respiration, while the slight decrease in acidity indicates delayed ripening and retention of organic acids [28]. The ZnO/Plu-modified films, especially the 1.0% formulation, also mitigate vitamin C loss over 7 days. Similar behavior has been reported for other fruits packaged in ZnO-based nanocomposite films, where improved barrier and antimicrobial properties limit oxidative degradation of ascorbic acid and related nutrients. Overall, the PLA/1.0% ZnO–Plu film, which also exhibits the best mechanical properties, provides an optimal combination of structural integrity and active functionality, ensuring superior preservation of both the physical and nutritional quality of bananas during short-term storage [29].

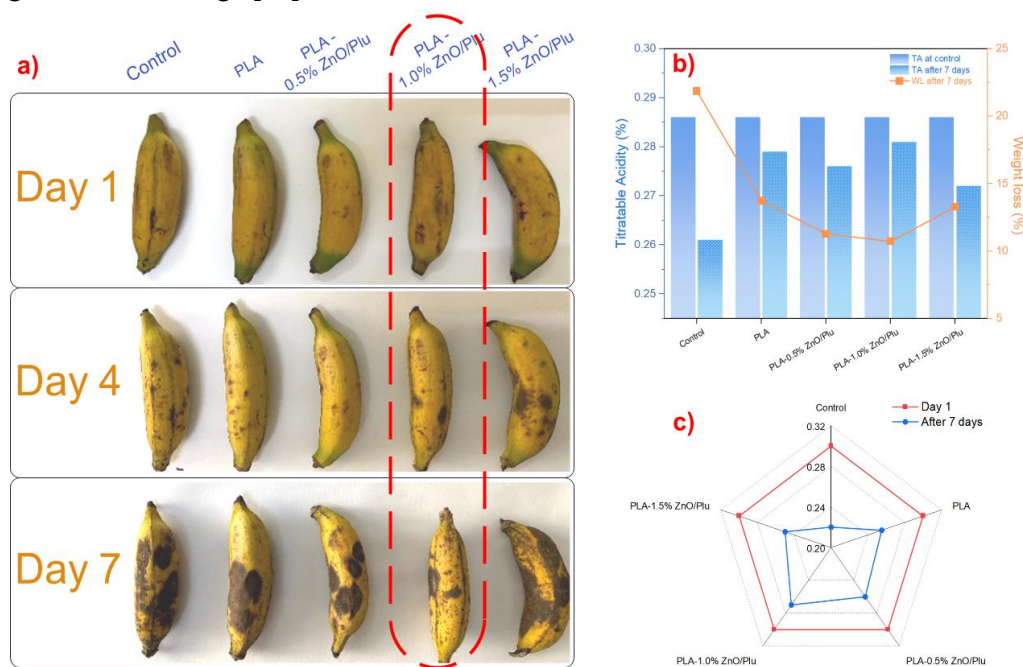


Figure 5. Visual assessment of the banana preservation performance of the modified films (a), correlation between weight change and total titratable acidity after 7 days (b), and changes in vitamin C content after 7 days (c).

4. CONCLUSIONS

This study shows that co-incorporating ZnO nanoparticles and Pluronic into PLA produces composite films whose mechanical, antibacterial, and preservation properties strongly depend on additive concentrations. An intermediate loading of 1.0 wt% ZnO/Plu is optimal, yielding well-dispersed nanoparticles, effective interfacial adhesion, high tensile strength, improved ductility, and reduced water solubility, whereas lower or higher contents give only limited reinforcement or trigger aggregation and performance loss. Functionally, the PLA/1.0 wt% ZnO–Plu films provide robust antibacterial activity against *E. coli* and *B. subtilis* and significantly improve the short-term storage quality of bananas by lowering microbial load, limiting weight loss, and better preserving

acidity and vitamin C. Overall, PLA/ZnO–Pluronic composites emerge as promising biodegradable active packaging materials, capable of bridging the gap between mechanical robustness and functional food preservation, with future work required on scale-up, long-term storage, and regulatory evaluation.

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TÓM TẮT

Màng phân hủy sinh học Poly(axít lactic)/ZnO/Pluronic composite: Tính chất cơ học, hiệu quả kháng khuẩn và ứng dụng trong bảo quản trái cây

Trong nghiên cứu này, các màng composite trên cơ sở poly(lactic acid) (PLA), kẽm oxit dạng hạt nano (ZnO NPs) và Pluronic P123 (0,5–1,5% khối lượng ZnO/Plu) đã được chế tạo và đặc trưng về cấu trúc, tính chất cơ học, khả năng kháng khuẩn và thử nghiệm khả năng bảo quản trái cây. Điều kiện tối ưu được xác định tại hàm lượng 1,0% khối lượng ZnO/Plu, tương ứng với cường độ kéo đứt (15,7 MPa), độ giãn dài khi đứt (~0,7%) và độ hòa tan trong nước sau 24 giờ giảm đều ứng với sự phân tán đồng đều của tác nhân ZnO/Plu, được khẳng định thông qua kết quả phân tích SEM/EDX và FTIR. Các màng chế tạo được cho thấy hoạt tính kháng khuẩn mạnh, phụ thuộc nồng độ đối với *Escherichia coli* và *Bacillus subtilis*, với hiệu quả ức chế cao nhất tại 1,0%. Khi được sử dụng làm bao gói chủ động cho chuối, màng chứa 1,0% ZnO/Plu hạn chế tốt nhất sự hao hụt khối lượng, duy trì độ axit chuẩn độ và hàm lượng vitamin C, đồng thời bảo toàn chất lượng cảm quan, qua đó cho thấy tiềm năng ứng dụng như một vật liệu bao gói chủ động, có khả năng phân hủy sinh học cho giai đoạn sau thu hoạch.

Từ khoá: PLA; ZnO NPs; Pluronic P123; Bao gói thực phẩm; Bảo quản trái cây.