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Controlled-release cinnamon oil in flexible poly(lactic acid) films via mesoporous SBA-15 encapsulation

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Abstract

Poly(lactic acid) (PLA) has critical limitations in food packaging applications because of its inherent brittleness and lack of active functionality. To address these limitations, we developed flexible PLA films with sustained-release antimicrobial activity via mesoporous Santa Barbara Amorphous-15 (SBA-15) encapsulation of cinnamon oil (MAO). SBA-15 (Brunauer–Emmett–Teller surface area: 568.9 m²/g; pore diameter: 7.5 nm) demonstrated exceptional MAO loading capacity (801.5 mg/g). MAO@SBA-15 was incorporated into the polyethylene glycol-plasticized PLA via solution casting. The 3 wt% MAO@SBA-15 composite exhibited the highest tensile strength (16.0 MPa) and crystallinity (32.4%) owing to the uniform filler dispersion and nucleation effects. At 7 wt% loading, films achieved superior barrier properties [water vapor permeability: 2.6 × 10⁻¹³ g·cm/(cm²·s·Pa); oxygen transmission rate: 4.5 × 10⁻¹³ cm³·cm/(cm²·s·Pa)], antioxidant activity (1,1-diphenyl-2-trinitrophenylhydrazine scavenging: 34.4%), and preservation efficacy for mulberries (weight loss rate: 3.6%; hardness: 0.82 N) over seven days. The MAO release followed Higuchi model (R² = 0.9738), confirming controlled diffusion. This study established a potentially scalable strategy for fabricating multifunctional PLA packaging with enhanced flexibility, barrier performance, and sustained bioactive delivery.

Keywords: Poly(lactic acid), mesoporous silica, sustained release, active packaging, cinnamon oil



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INTRODUCTION

The global shift toward sustainable packaging has positioned poly(lactic acid) (PLA) as a leading bio-based alternative to petroleum-based plastics owing to its biocompatibility, compostability, and approval by the U.S. Food and Drug Administration (FDA) for food contact^[1]. PLA is often used in food packaging, 3D printing, agricultural films, medical devices, and many other fields^[2]. However, the pure PLA films exhibit low toughness, limiting their use in food packaging^[3]. Therefore, plasticizers such as citrate esters and polyethylene glycol (PEG) are essential for enhancing the flexibility, ductility, and processability of the PLA matrix^[4-6]. PEG is the most commonly used plasticizer in PLA, owing to its superior biocompatibility and interfacial compatibility^[7].

PLA-based packaging materials still cannot effectively extend the shelf life of food due to their single function. Active ingredients such as silver nanoparticles, plant essential oils and antibacterial polypeptides can effectively extend the shelf life of food by inhibiting microbial growth^[8]. Plant essential oils are regarded as the safest and most popular active ingredients. Cinnamon oil (MAO), an essential plant oil rich in cinnamaldehyde and eugenol, exhibits antifungal, anticancer, antioxidant, and anti-inflammatory properties^[9]. MAO exhibits superior broad-spectrum antimicrobial efficacy against critical food-spoilage pathogens compared to other essential oils at equivalent concentrations^[10]. Furthermore, its potent antioxidant capacity directly addresses oxidative spoilage mechanisms in oxygen-sensitive foods, such as fruits^[10]. However, MAO has several limitations, including high volatility and instability^[11]. Essential oil encapsulation technologies, especially chitosan, gelatin, metal-organic frameworks (MOFs), covalent organic frameworks (COFs), and mesoporous molecular sieves, have potential application value in addressing the instability and release rate of essential oils^[12]. Negi *et al.* reported a chitosan nanoparticle-based encapsulation of plant essential oils that exhibited excellent bacteriostatic activity^[13]; however, chitosan exhibited a low loading capacity for plant essential oils (< 200 mg/g) and was incompatible with PLA. New porous materials, such as MOFs and COFs, exhibit high loading capacities, but are often costly and difficult to commercialize^[14,15].

Mesoporous molecular sieves, such as Santa Barbara Amorphous (SBA)-15, SBA-16, and Mobil Composition of Matter No. 41 (MCM-41), with high specific surface areas, regular and orderly pore structures, narrow pore size distributions, and continuously adjustable pore sizes, have broad application prospects in catalysis, separation, biology, and nanomaterials fields. Loading thyme essential oil (TEO) onto mesoporous nanosilica can enhance the mechanical strength and antioxidant activity of starch-based films, while also slowing down the release rate of TEO^[16]. Recent studies have demonstrated the efficacy of TEO within starch films^[16], yet its synergy with PLA, especially in achieving concurrent flexibility, controlled release, and barrier enhancement, remains unexplored.

In this study, we hypothesized that SBA-15 encapsulation mitigates the volatility of MAOs while enabling the sustained release of PLA, synergistically enhancing flexibility, barrier properties, and bioactivity. A mesoporous silica, SBA-15, a type of mesoporous silica, was synthesized and loaded with MAO. The filler (MAO@SBA-15) was then combined with PLA and PEG to fabricate active PLA-based packaging films via solution casting. The mechanical properties, light transmittance, water vapor and oxygen permeability, antioxidant and antibacterial activities, and preservation of mulberries in PLA-based active packaging films were measured. Finally, the release kinetics of MAO in the PLA-based active packaging films were studied. This study pioneered the integration of mesoporous carriers into PLA to resolve the long-standing conflict between the flexibility and active functionality of biopolymer packaging.

EXPERIMENTAL

Materials and chemicals

Poly(ethylene oxide)-*co*-poly(propylene oxide)-*co*-poly(propylene oxide) (P123), hydrochloric acid (HCl), tetraethyl orthosilicate (TEOS), PLA, polyethylene glycol 2000 (PEG), trichloromethane, MAO, anhydrous ethanol, calcium oxide (CaO), ascorbic acid, and 1,1-diphenyl-2-trinitrophenylhydrazine (DPPH) were purchased from Titan Technology Co. Ltd. (Shanghai, China). All chemicals were of analytical grade. The normal human liver cell line LO2 was purchased from the Shanghai Cell Bank of the Chinese Academy of Sciences (Shanghai, China). Cells were cultured in Dulbecco's modified Eagle's medium with 10% fetal bovine serum in a cell culture incubator (Thermo Fisher Scientific, Waltham, MA, USA) at 37 °C and 5% CO₂.

Preparation of SBA-15 and MAO@SBA-15

SBA-15 was synthesized as follows: Initially, 2.86 g of P123 was dispersed in 100 mL of 1.1 mol/L HCl. The mixture was heated to 45 °C under continuous stirring for 2 h, after which 6 g of TEOS was added. After thorough mixing for 1 h, the solution was transferred to a reactor and heated at 105 °C for 48 h. The resulting white solid was collected by filtration, washed sequentially with deionized water and ethanol three times, and then dried at 60 °C for 12 h. Finally, the material was calcined in a muffle furnace at 550 °C for 5 h in air (with a heating rate of 1 °C/min) to remove the template, yielding the desired SBA-15.

SBA-15 was subjected to vacuum drying at 150 °C for 12 h, and was subsequently mixed with MAO in a bottle and ultrasonicated for 5 min to ensure thorough dispersion. Excess MAO was then filtered out, and the resulting sample was dried at room temperature for three days to obtain the final MAO@SBA-15 product. The MAO loading capacity of SBA-15 (q , mg/g) was calculated using

$$q \text{ (mg/g)} = \frac{W_1}{W_0} \quad (1)$$

where W_1 (mg) and W_0 (g) represent the weight of MAO and SBA-15, respectively.

Structural characterization of SBA-15 and MAO@SBA-15

The functional groups of SBA-15 and MAO@SBA-15 were analyzed using an AVATAR 370 Fourier transform infrared spectrometer (FT-IR, Thermo, USA) using the KBr pressing method. The samples were scanned from 400 to 4,000 cm⁻¹ at 4 cm⁻¹ resolution with 32 scans. The crystal structures were determined using an Ultima IV X-ray diffractometer (XRD, Rigaku, Japan). The diffraction angles ranged from 0.5° to 10° at a scan rate of 1°/min. The surface morphology was observed using a Sigma 500 field-emission scanning electron microscope (SEM, Zeiss, Germany). The acceleration voltage was 10 kV. The permanent porosity of samples was analyzed using an ASAP 2020 HD88 automatic physical adsorption instrument [Brunauer–Emmett–Teller (BET), Micromeritics, USA] by N₂ adsorption-desorption measurements after activation at 200 °C for 12 h under vacuum.

Preparation of PLA-based active packaging films

The preparation method of PLA-based packaging film was as follows. PLA, PEG, and chloroform (7.9/2.1/100, w/w/w) were added into the beaker. The PLA/PEG film at the ratio maintained substantial toughness without phase separation^[17]. Then, 0, 1, 3, 5, or 7 wt% MAO@SBA-15 based on the weight of PLA was incorporated into the PLA-chloroform solution under stirring for 2 h, respectively. The uniformly dispersed solution was poured into a glass dish and dried at room temperature for 12 h. The samples were named PLA, PLA/1%MAO@SBA-15, PLA/3%MAO@SBA-15, PLA/5%MAO@SBA-15, and PLA/7%MAO@SBA-15, respectively. The PLA film with 3 wt% SBA15 (PLA/3%SBA15) was prepared in the same way as a control.

Structural characterization of PLA-based active packaging films

The functional groups of the PLA-based active packaging films were determined using AVATAR 370 FT-IR in the reflection mode following ASTM E1252. The samples were scanned from 400 to 4,000 cm^{-1} at a resolution of 4 cm^{-1} with 32 scans. The crystal structures of the PLA-based active packaging films were determined by Ultima IV XRD with Cu K α radiation ($\lambda = 1.54 \text{ \AA}$) at 40 kV and 40 mA according to ASTM E915. The scanning was carried out in the diffraction angle range of 5°-80° at a scanning rate of 10 °/min. The cross-section morphology of PLA-based active packaging films was observed using Sigma 500 field-emission SEM according to ISO 16700. The cross-sections were sprayed with gold before observation. The acceleration voltage was 15 kV. The elemental analysis of PLA-based active packaging films was performed using X-Max 80 energy-dispersive spectroscopy (EDS, Oxford Instruments, UK). The acceleration voltage was 20 kV. The differential scanning calorimetry (DSC) curves of the PLA-based active packaging films were obtained using a DSC 300 instrument (NETZSCH, Germany). Temperature ranged from -25 to 230 °C at a heating rate of 10 °C/min.

Performances of PLA-based active packaging films

The transmittance properties of the PLA-based active packaging films were evaluated using a UV-2600 ultraviolet (UV)-visible spectrophotometer (Shimadzu, Japan) with a wavelength range of 200-800 nm, and observed through photos.

The mechanical properties of the PLA-based active packaging films were evaluated using a YG061-1500 electronic strength tester according to ASTM D882-10. Thicknesses of all films were obtained by averaging five different positions measured with a thickness gauge (QST EXPRESS, China). The clamp spacing and crosshead speed were 50 mm and 50 mm/min, respectively. Each sample was tested five times and then averaged.

Water contact angle of the PLA-based packaging films was measured using a CA-100D automatic contact angle measuring instrument (Shanghai Jiezhun Instrument, China). Each sample was tested three times and then averaged.

The water vapor permeability [WVP, $\text{g}\cdot\text{cm}/(\text{cm}^2\cdot\text{s}\cdot\text{Pa})$] of the PLA-based packaging films was measured using a C360M water vapor transmission rate tester (Languang, China) according to the Chinese national standard GB/T 1037-2021, and calculated using

$$WVP [\text{g}\cdot\text{cm}/(\text{cm}^2\cdot\text{s}\cdot\text{Pa})] = \frac{(W_5 - W_4) \times X}{A \times t \times \Delta p} \quad (2)$$

where ($W_5 - W_4$) and t represented the absorbed weight (g) and time (s) of water vapor passing through the PLA-based packaging film, respectively. A and X denoted the area (cm^2) and thickness (cm) of the sample, respectively, and Δp indicated water vapor pressure difference between the two sides of the sample (Pa). The thicknesses of all films were obtained by averaging measurements from five different positions using a thickness gauge. Each sample was tested three times and then averaged. CaO (2 g) was then added to the moisture-permeable cup. The mouth of each moisture-permeable cup was sealed with a circular PLA-based packaging film with an area of 5 cm^2 , and then weighed. Finally, the sealed bottle was placed in 90% relative humidity (RH) for one day, and weighed. Each sample was tested three times and then averaged.

The oxygen permeability [OTR, $\text{cm}^3\cdot\text{cm}/(\text{cm}^2\cdot\text{s}\cdot\text{Pa})$] of the PLA-based packaging films was measured using a GTT gas permeation meter (Brugger, Germany) according to the Chinese national standard GB/T 1038.1-2022. The test films, with an area of 5 cm^2 , were evaluated under conditions of 23 °C and 0% RH.

Measurements were conducted in triplicate for each sample using automated endpoint detection by the instrument.

The antioxidant activity of the PLA-based packaging films was expressed as the DPPH free radical scavenging rate (R_{DPPH} , %), as given in

$$R_{\text{DPPH}} (\%) = \frac{A_s - A_c}{A_c} \times 100 \quad (3)$$

where A_s and A_c represented the absorbance of the DPPH solution with and without the PLA-based packaging film, respectively. PLA-based packaging films with dimensions of 4 cm × 4 cm were immersed in 100 mL of ethanol for 2 h, and then filtered. Then, 1 mL filtrate was mixed with 4 mL DPPH-ethanol solution (75 μmol/L). The mixture was incubated in the dark for 60 min. Finally, the absorbance of the mixture was measured at 517 nm using a UV spectrophotometer.

The antibacterial activities of the PLA-based packaging films were as follows. PLA/PEG, 1MAO@SBA-15 or 3MAO@SBA-15 packaging film with a size of 2 cm × 2 cm was added into a container containing *Escherichia coli* (*E. coli*) or *Staphylococcus aureus* (*S. aureus*) suspension and liquid medium at 37 °C for 2 h. Then, 20 μL aliquots were pipetted onto solid agar medium. The inoculum was evenly spread across the plate surface, and incubated at 37 °C for 12 h. Finally, the morphology was observed. The inhibition rates of the PLA-based film against *E. coli* and *S. aureus* are determined according to the method reported by Shah^[18].

Different types of PLA materials (0.2 g) were sterilized by UV irradiation, cut into small fragments, and placed into LO2 cell culture dishes for 24-hour treatment, and phosphate-buffered saline (PBS) was used in the control group. LO2 cells were digested and LO2 cells (5×10^3 cells/well) were plated into 96-well plates. After 24 h, cell viability was assessed using a CCK8 assay. The cells were cultured for 1 h with CCK8, and the optical density (OD) was measured at 450 nm using an enzyme-labeling instrument.

After being treated with different types of PLA materials, LO2 cells were digested, and 50 μL of cell suspension was mixed with 50 μL trypan blue staining solution. The mixture was placed in a hemocytometer, and cells were counted under a microscope for 10 min.

The preservation of mulberries using PLA-based packaging films was performed as described below. The fresh mulberries were weighed, and packaged using PLA-based packaging films. The mulberries were stored in PLA-based packaging films at room temperature for seven days, and weighed. The water loss rate of the mulberries (%) was calculated using

$$\text{Water loss rate}(\%) = \frac{W_7 - W_6}{W_7} \times 100 \quad (4)$$

where W_7 (g) and W_6 (g) are the mulberry weights before and after storage, respectively. Hardness was determined with a GY-4 fruit hardness tester (Puyan, China).

Release kinetics

A PLA/3%MAO@SBA-15 film with a size of 4 cm × 4 cm was encased in dialysis tubing (12–14 kDa), and then placed in a flask containing 100 mL of ethanol to simulate controlled release conditions. At predetermined time intervals (0, 2, 4, 6, 8, 12, 24, 48, and 72 h), 3 mL of the solution was collected, and the absorbance of the solution at 287 nm was measured using a UV-2600 spectrophotometer. The cumulative

release profile of MAO from the PLA/3%MAO@SBA-15 film was analyzed using the Avrami equation [Equation (5)] and Higuchi equation [Equation (6)].

$$R(\%) = \frac{M_t}{M_\infty} = 1 - \exp(-(kt)^n) \quad (5)$$

where R denoted the release ratio of MAO (%), which was the ratio of the release weight of MAO at time t (M_t , g) to the release weight of MAO at infinite time (M_∞ , g). k , t , and n denoted the release rate constant (s^{-1}), the release time (s), and the release mechanism parameters, respectively.

$$Q/A = 2C_0(Dt/\pi)^{1/2} \quad (6)$$

where Q and C_0 denoted the release weight (mg) and initial concentration (mg/mL) of MAO, respectively. A , D , and t denoted the diffusion area (cm^2), apparent diffusion coefficient (cm^2/min), and the diffusion time (min), respectively.

Statistic analysis

All the performances of PLA-based packaging films were presented as a mean value of at least three replicates $\pm$ standard deviation^[19]. Data were analyzed by Tukey's honest significant difference test using the IBM SPSS Statistics program. Different letters represent statistically significant differences ($P < 0.05$).

RESULTS AND DISCUSSION

Structural analysis of SBA-15 and MAO@SBA-15

The FT-IR spectra of SBA-15 and MAO@SBA-15 are shown in [Figure 1A](#). For pure SBA-15, the peak at 460 cm^{-1} corresponds to the bending vibration of Si–O, while the peaks at 806 and $1,083\text{ cm}^{-1}$ are attributed to the symmetric and asymmetric stretching vibrations of the Si–O–Si bridge, respectively. The broad peak at $3,438\text{ cm}^{-1}$ arises from the O–H stretching vibration of silanol groups on the SBA-15 surface, consistent with previously reported results^[20]. In the spectrum of MAO@SBA-15, new absorption bands emerge in the ranges of $1,676$ – $1,420$ and 748 – 687 cm^{-1} . These bands were assigned to the C=O stretching vibrations of MAO components and the C–H stretching vibrations of aliphatic and aromatic compounds, respectively^[21]. Additionally, the peak at $1,125\text{ cm}^{-1}$ is characteristic of the C–O stretching vibration in phenolic groups. Notably, the O–H stretching vibration peak of SBA-15 shifts from $3,438$ to $3,327\text{ cm}^{-1}$ upon loading with MAO, suggesting strong interactions between MAO components and the surface hydroxyl groups of SBA-15^[22]. These results collectively confirmed the successful incorporation of MAO into the SBA-15 framework.

The XRD patterns of SBA-15 and MAO@SBA-15 are shown in [Figure 1B](#). SBA-15 exhibits distinct diffraction peaks at 1.0° , 1.7° , and 1.9° , which correspond to the (100), (110), and (200) crystal planes, respectively^[23]. Notably, the close resemblance between the XRD profile of MAO@SBA-15 and that of pure SBA-15 clearly indicates that the incorporation of MAO preserved the crystalline structure of the SBA-15 host.

The SEM images revealed the morphology of SBA-15 [[Figure 1C](#)], which exhibited a uniform short rod-like structure^[24]. The nitrogen adsorption-desorption isotherms of SBA-15 are shown in [Figure 1D](#). This material possesses a specific surface area of $568.9\text{ m}^2/\text{g}$, a pore volume of $1.0\text{ cm}^3/\text{g}$, and an average pore diameter of 7.5 nm . These favorable structural parameters, particularly the high porosity and large surface area, are crucial for achieving efficient loading of MAO^[25,26]. Importantly, the measured loading capacity of SBA-15 for MAO is $801.5 \pm 3.7\text{ mg/g}$. Compared to other encapsulation technologies^[13–15], SBA-15 has a higher loading capacity for plant essential oils, which enables active packaging to extend the shelf life of food.

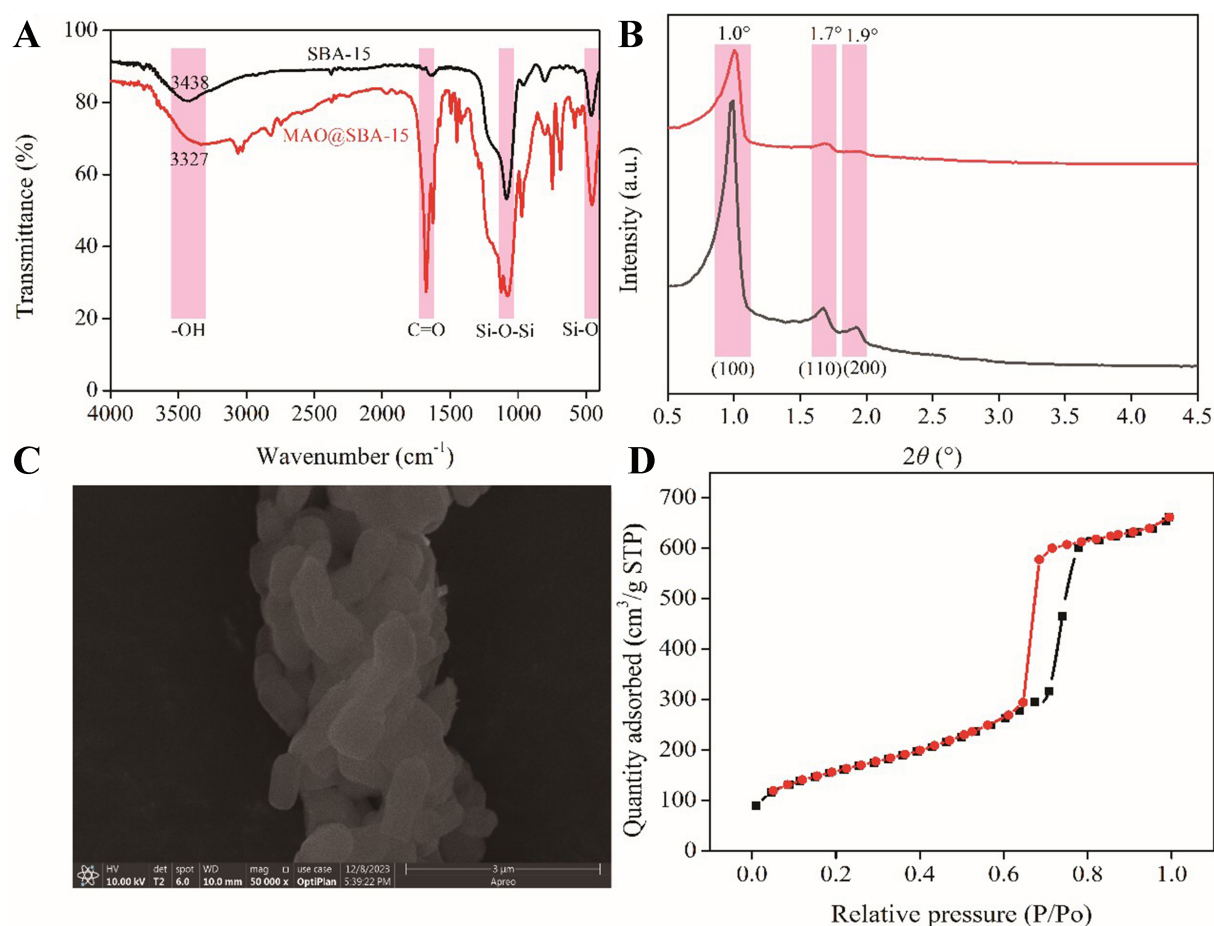


Figure 1. Structural characterization of SBA-15 and MAO@SBA-15. (A) FT-IR; (B) XRD; (C) SEM; and (D) BET. SBA-15: Santa Barbara Amorphous-15; MAO: cinnamon oil; FT-IR: Fourier transform infrared spectrometer; XRD: X-ray diffractometer; SEM: scanning electron microscope; BET: Brunauer–Emmett–Teller.

Structural analysis of PLA-based packaging films

The FT-IR spectra of the PLA-based packaging films are shown in [Figure 2A](#). The peak at $1,750\text{ cm}^{-1}$ corresponds to the C=O stretching vibration in PLA. The peaks at $2,882$ and $1,083\text{ cm}^{-1}$ were assigned to the –CH stretching vibration and C–O stretching vibration of the PLA backbone, respectively. The characteristic peak at $1,459\text{ cm}^{-1}$ is attributed to the –CH₂ bending vibration in PEG^[27,28].

The XRD patterns of the films are shown in [Figure 2B](#). The PLA film exhibited partial crystallization with distinct diffraction peaks at 16.7° , 19.2° , and 21.6° ^[29]. Compared with the PLA film, all PLA-based films containing SBA-15 or MAO@SBA-15 retained identical diffraction peak positions. This indicates that the MAO@SBA-15 filler did not disrupt the crystalline structure of PLA. The crystallinity values calculated using Jade 6.0 software were 27.5% for PLA, 34.1% for PLA/3%SBA-15, 29.5% for PLA/1%MAO@SBA-15, 32.4% for PLA/3%MAO@SBA-15, 30.9% for PLA/5%MAO@SBA-15, and 30.2% for PLA/7%MAO@SBA-15. Importantly, these results demonstrate that MAO@SBA-15 significantly enhanced the crystallinity of the PLA-based composite films. This enhancement was likely due to MAO@SBA-15 acting as a heterogeneous nucleating agent and promoting nucleation and crystal growth^[30]. Moreover, the film with 3 wt% MAO@SBA-15 exhibited the highest crystallinity among PLA-based active packaging films. This excellent

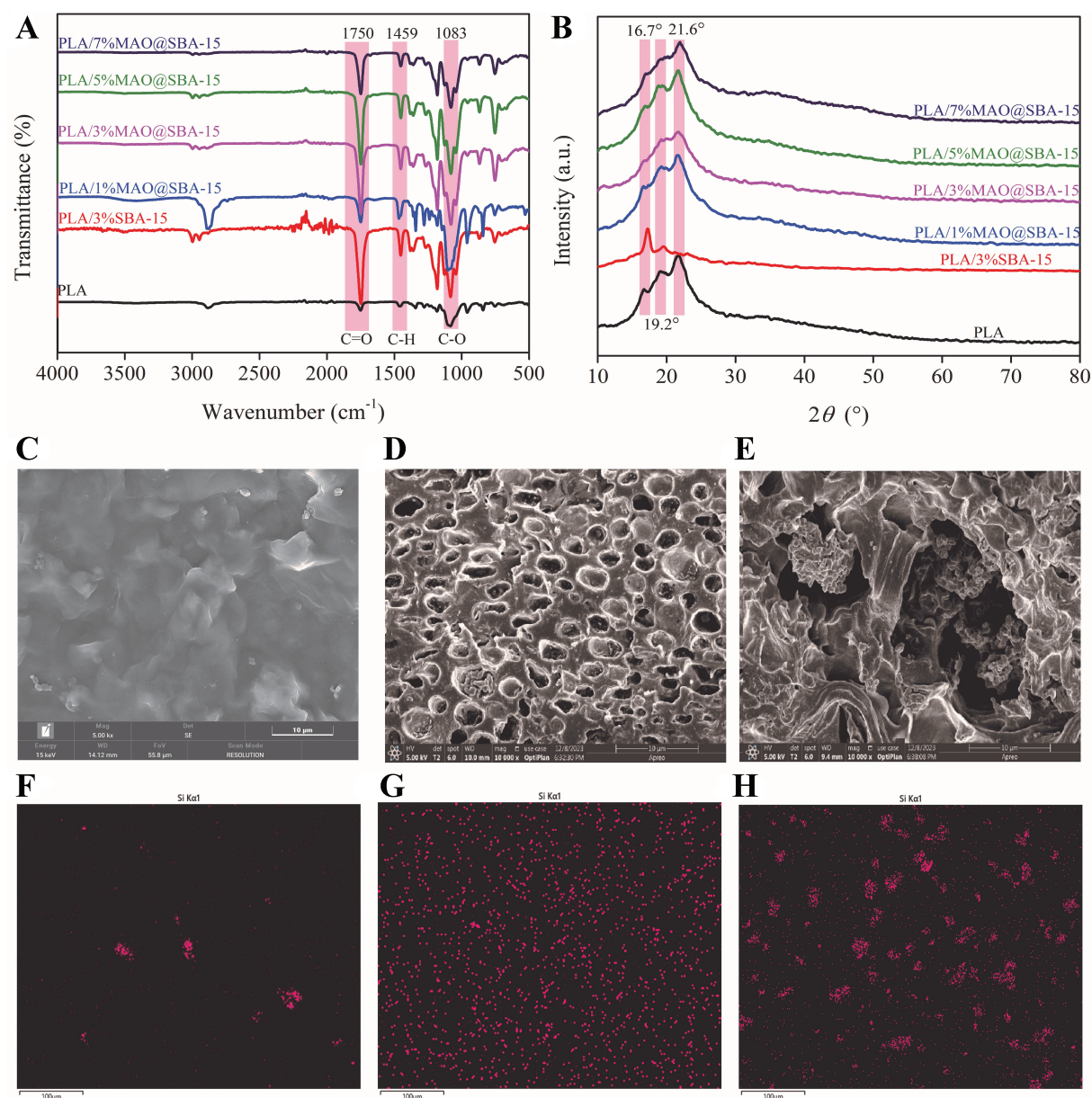


Figure 2. The structural characterization of PLA-based packaging films, (A) FT-IR; (B) XRD; (C) SEM of PLA/1%MAO@SBA-15 film; (D) SEM of PLA/3%MAO@SBA-15 film; (E) SEM of PLA/7%MAO@SBA-15 film; (F) Elemental mapping of PLA/1%MAO@SBA-15 film; (G) Elemental mapping of PLA/3%MAO@SBA-15 film; and (H) Elemental mapping of PLA/7%MAO@SBA-15 film. PLA: Poly(lactic acid); FT-IR: Fourier transform infrared spectrometer; XRD: X-ray diffractometer; SEM: scanning electron microscope; MAO: cinnamon oil; SBA-15: Santa Barbara Amorphous-15.

performance is likely attributed to the effective dispersion of MAO@SBA-15 within the PLA matrix.

DSC curves of the PLA-based packaging films are shown in [Supplementary Figure 1](#). The melting temperatures of the PLA, PLA/1%MAO@SBA-15, PLA/3%MAO@SBA-15, PLA/5%MAO@SBA-15, and PLA/7%MAO@SBA-15 films are 151.4, 151.8, 152.1, 150.7, and 151.1 °C, respectively. This trend can be attributed to the dual effects of MAO@SBA-15 as a nucleating agent and its potential aggregation at higher loadings. At lower concentrations (1% and 3%), the well-dispersed MAO@SBA-15 particles act as effective nucleating sites, promoting crystallization and resulting in a more perfect crystalline structure with a slightly

elevated melting temperature. However, at higher loading levels (5% and 7%), nanoparticle aggregation may occur, leading to inhomogeneous dispersion within the PLA matrix. These aggregates can disrupt the polymer chain ordering and hinder crystal perfection^[30], thereby reducing the melting temperature. Additionally, increased filler content may introduce interfacial defects and reduce overall crystallinity, contributing to the observed decrease in the melting temperature.

The SEM images and elemental mapping of the PLA-based composite films containing 1 wt%, 3 wt%, and 7 wt% MAO@SBA-15 are presented in [Figure 2C-H](#). Significantly, at a loading of 1 wt% or 3 wt% (based on the PLA weight), MAO@SBA-15 was uniformly dispersed within the PLA matrix [[Figure 2C, D, F, and G](#)]. This uniform dispersion demonstrates the good compatibility between MAO@SBA-15 and the PLA matrix. In contrast, when the loading was increased to 7 wt%, MAO@SBA-15 exhibited agglomeration within the PLA matrix [[Figure 2E and H](#)]. This critical difference in the dispersion behavior is attributed to the high specific surface area of SBA-15. At higher loadings, the SBA-15 particles tended to agglomerate to achieve a lower energy and more stable state.

Transparency of PLA-based packaging films

The light transmittances of the PLA-based packaging films are shown in [Figure 3](#). At 400 nm, the transmittance values of the PLA, PLA/3%SBA-15, PLA/1%MAO@SBA-15, PLA/3%MAO@SBA-15, PLA/5%MAO@SBA-15, and PLA/7%MAO@SBA-15 films were 55.0%, 41.0%, 50.5%, 45.8%, 26.8%, and 18.8%, respectively. Critically, the films exhibited a pronounced and continuous decrease in transmittance with an increase in MAO@SBA-15 content as shown in [Figure 3](#)^[31]. This reduction is attributed to light scattering at the interface between the MAO@SBA-15 particles and the PLA matrix, which compromises the optical homogeneity of the films. Additionally, MAO@SBA-15 enhanced the nucleation density of the PLA/PEG matrix, potentially further increasing light scattering owing to microstructural heterogeneity^[32]. These findings demonstrate that the incorporation of MAO@SBA-15 significantly enhanced the light-blocking capability, particularly in the UV region. This key property renders PLA-based films highly suitable for packaging applications requiring effective UV protection. However, it should be noted that the extent to which this reduction quantitatively contributes to the inhibition of oxidative or microbial degradation in food products remains unclear and would require further specific investigation.

Mechanical properties of PLA-based packaging films

The tensile properties of the PLA-based packaging films are depicted in [Figure 4](#). As shown in [Figure 4A](#), all the films exhibit ductile fracture behavior, which was attributed to the addition of PEG as a plasticizer. The elastic modulus, tensile strength, and elongation at break of the PLA film are (102.0 ± 3.0) MPa, (13.8 ± 0.7) MPa, and $(344.4 \pm 15.4)\%$, respectively. The elastic modulus, tensile strength, and elongation at break of the PLA/3%SBA-15 film are (171.3 ± 4.5) MPa, (16.6 ± 1.0) MPa, and $(312.7 \pm 8.3)\%$, respectively. Compared to the PLA film, the PLA films incorporating SBA-15 or MAO@SBA-15 showed a higher elastic modulus and tensile strength, but lower elongation at break. This difference is likely related to the increased rigidity and crystallinity imparted by SBA-15 or MAO@SBA-15^[33]. [Figure 4B](#) shows that MAO@SBA-15 could significantly improve the elastic modulus of the PLA film. [Figure 4C](#) renders that the tensile strength of the PLA films initially increases and then decreases as the MAO@SBA-15 loading increases. Conversely, [Figure 4D](#) shows that the elongation at break followed a trend opposite to that of tensile strength. Critically, the tensile strength reaches its maximum value of (16.0 ± 1.1) MPa at the MAO@SBA-15 loading of 3 wt%. However, the tensile strength decreased at higher loadings. This decline was attributed to the stress concentrations resulting from the particle agglomeration of MAO@SBA-15^[34]. The nature and amount of filler loading play critical roles in determining the mechanical behavior of the composites. At low concentrations, well-dispersed particles act effectively as reinforcing agents, improving stiffness and strength. However, beyond an optimal threshold, aggregation dominates, resulting in defects and

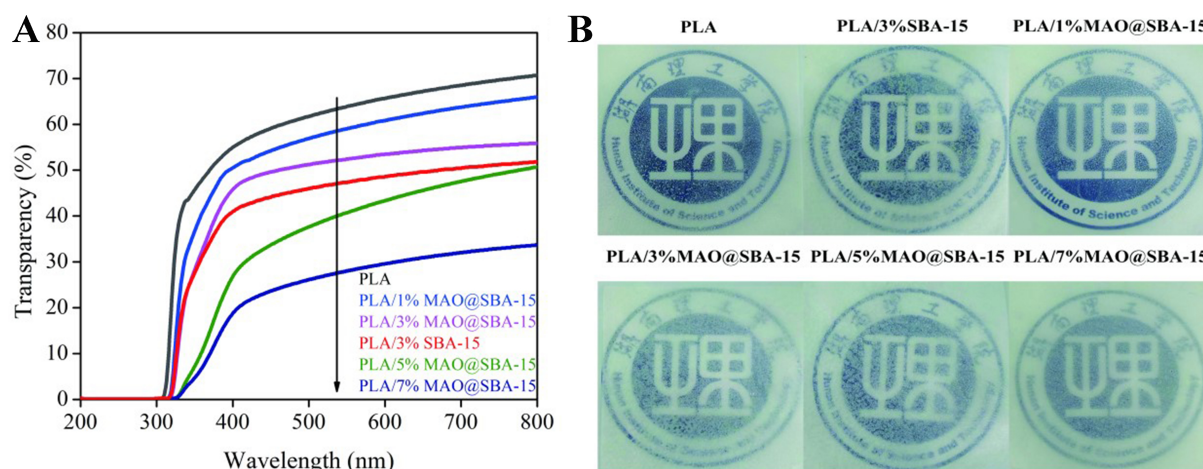


Figure 3. (A) Transmittance and (B) photos of the PLA-based packaging films. PLA: Poly(lactic acid).

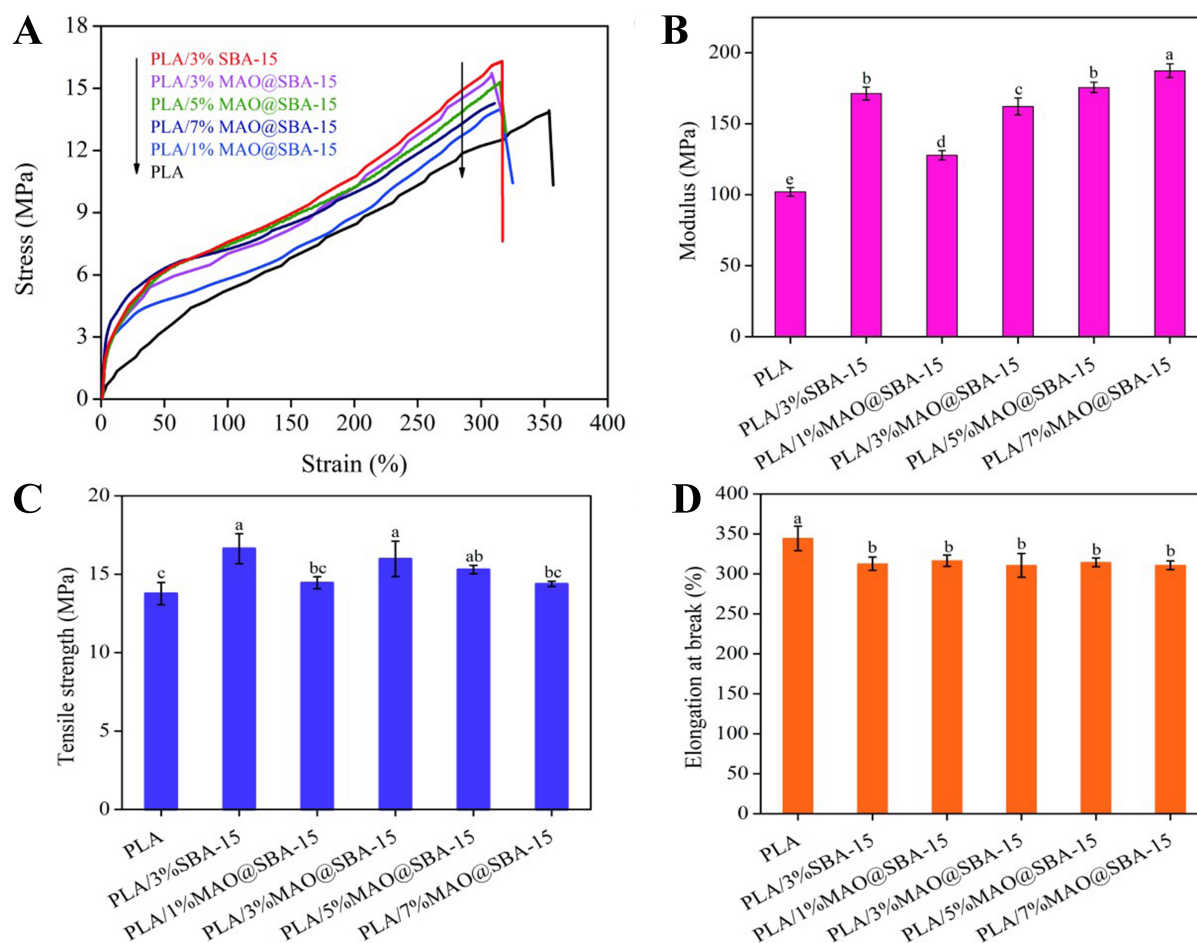


Figure 4. Mechanical properties of PLA-based packaging films. (A) Stress-strain curves; (B) Elastic modulus; (C) Tensile strength; and (D) Elongation at break. Different letters represent statistically significant differences ($P < 0.05$). PLA: Poly(lactic acid).

compromised properties. This dual effect underscores the importance of optimizing filler content to achieve a balanced performance profile between strength and ductility.

Gas barrier of PLA-based packaging film

The water contact angle of the PLA-based packaging films is shown in [Supplementary Figure 2](#). The contact angles of the PLA, PLA/3%SBA-15, PLA/1%MAO@SBA-15, PLA/3%MAO@SBA-15, PLA/5%MAO@SBA-15, and PLA/7%MAO@SBA-15 films are $(68.0 \pm 1.6)^\circ$, $(76.0 \pm 2.8)^\circ$, $(70.1 \pm 2.3)^\circ$, $(73.8 \pm 2.0)^\circ$, $(76.6 \pm 2.5)^\circ$, and $(79.1 \pm 3.1)^\circ$, respectively. These results indicate that the incorporation of SBA-15 or MAO@SBA-15 significantly influences the hydrophobicity of the PLA composite films. The increase in water contact angle relative to neat PLA suggests enhanced hydrophobic character upon the addition of these fillers. Nanofillers such as SBA-15 and MAO@SBA-15 may increase the surface roughness of the composite films, which typically enhances hydrophobicity by reducing the effective contact area between water droplets and the film surface. In summary, the PLA film with higher loadings of MAO@SBA-15 exhibit improved hydrophobic surfaces, which could be beneficial for packaging applications where moisture resistance is desired.

WVP and oxygen transmission rate (OTR) are critical indices for product packaging applications because they determine the extent to which these gases can permeate the polymer films. Excessive permeation compromises the product quality and shortens the shelf life. The WVP values of the PLA-based packaging films are shown in [Figure 5A](#). The PLA and PLA/3%SBA-15 films exhibit WVP values of 5.3×10^{-13} g·cm/(cm²·s·Pa) and 3.4×10^{-13} g·cm/(cm²·s·Pa), respectively. Significantly, the WVP of the films progressively decreased with increasing MAO@SBA-15 content. Crucially, the film containing 7 wt% MAO@SBA-15 achieves the lowest WVP of 2.6×10^{-13} g·cm/(cm²·s·Pa). This significant reduction is attributed to two key factors: (1) SBA-15 or MAO@SBA-15 extends the diffusion pathway for water molecules within the film^[34]; (2) the incorporation of MAO@SBA-15 promotes a denser and more hydrophobic structure, effectively hindering the diffusion of water molecules^[35].

The OTR results for the PLA-based films are shown in [Figure 5B](#). The PLA and PLA/3%SBA-15 films have OTR values of 9.7×10^{-13} cm³·cm/(cm²·s·Pa) and 6.1×10^{-13} cm³·cm/(cm²·s·Pa), respectively. Notably, the films incorporating SBA-15 or MAO@SBA-15 consistently showed lower OTR values than the PLA film, and the OTR decreased steadily with increasing MAO@SBA-15 content. This effect primarily arises because MAO@SBA-15 or SBA-15 prolongs the diffusion pathways of oxygen molecules^[34]. Of particular note is the PLA film with 7 wt% MAO@SBA-15, which achieves the lowest OTR of 4.5×10^{-13} cm³·cm/(cm²·s·Pa). The substantial reductions in both WVP and OTR, especially at 7 wt% MAO@SBA-15 loading, demonstrate the significant potential of these modified PLA films for high-barrier packaging applications aimed at enhancing product preservation.

The nature and content of the filler play a decisive role in determining the barrier performance. At higher loadings, the number of MAO@SBA-15 particles significantly increases the tortuosity for gas permeation. Moreover, the improved compatibility between the filler and polymer matrix reduces interfacial voids, resulting in a denser structure with fewer diffusion channels. These results underscore the importance of filler and its loading in designing high-barrier PLA packaging films for extended preservation of sensitive products.

Activity and toxicity of PLA-based packaging films

The activity and toxicity of packaging materials are of critical importance in the contemporary food industry. The DPPH scavenging rate serves as a standard indicator for evaluating the antioxidant properties of packaging films, with a higher rate indicating a greater antioxidant capacity. [Figure 6A](#) shows the antioxidant activity of the PLA-based packaging films. The PLA and PLA/3%SBA-15 films exhibit DPPH scavenging rates of $(22.1 \pm 0.1)\%$ and $(22.7 \pm 0.5)\%$, respectively. Importantly, the DPPH scavenging rate of the PLA-based films incorporating MAO@SBA-15 surpassed that of the PLA and PLA/3%SBA-15 films, and significantly increased with rising MAO@SBA-15 content. Notably, the film containing 7 wt% MAO@SBA-

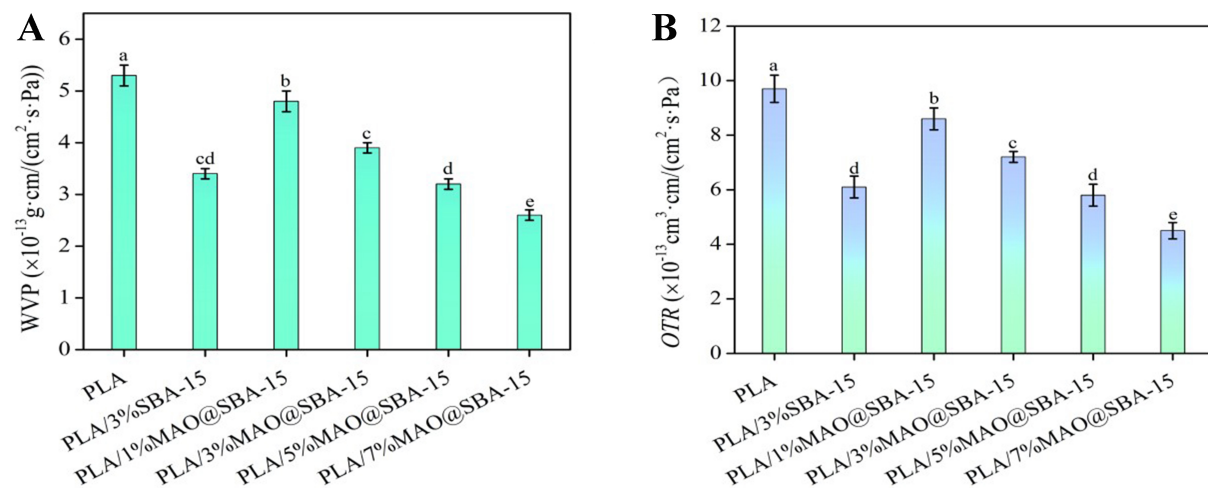


Figure 5. (A) WVP and (B) OTR of PLA-based packaging films. Different letters represent statistically significant differences ($P < 0.05$). WVP: Water vapor permeability; OTR: oxygen transmission rate; PLA: poly(lactic acid).

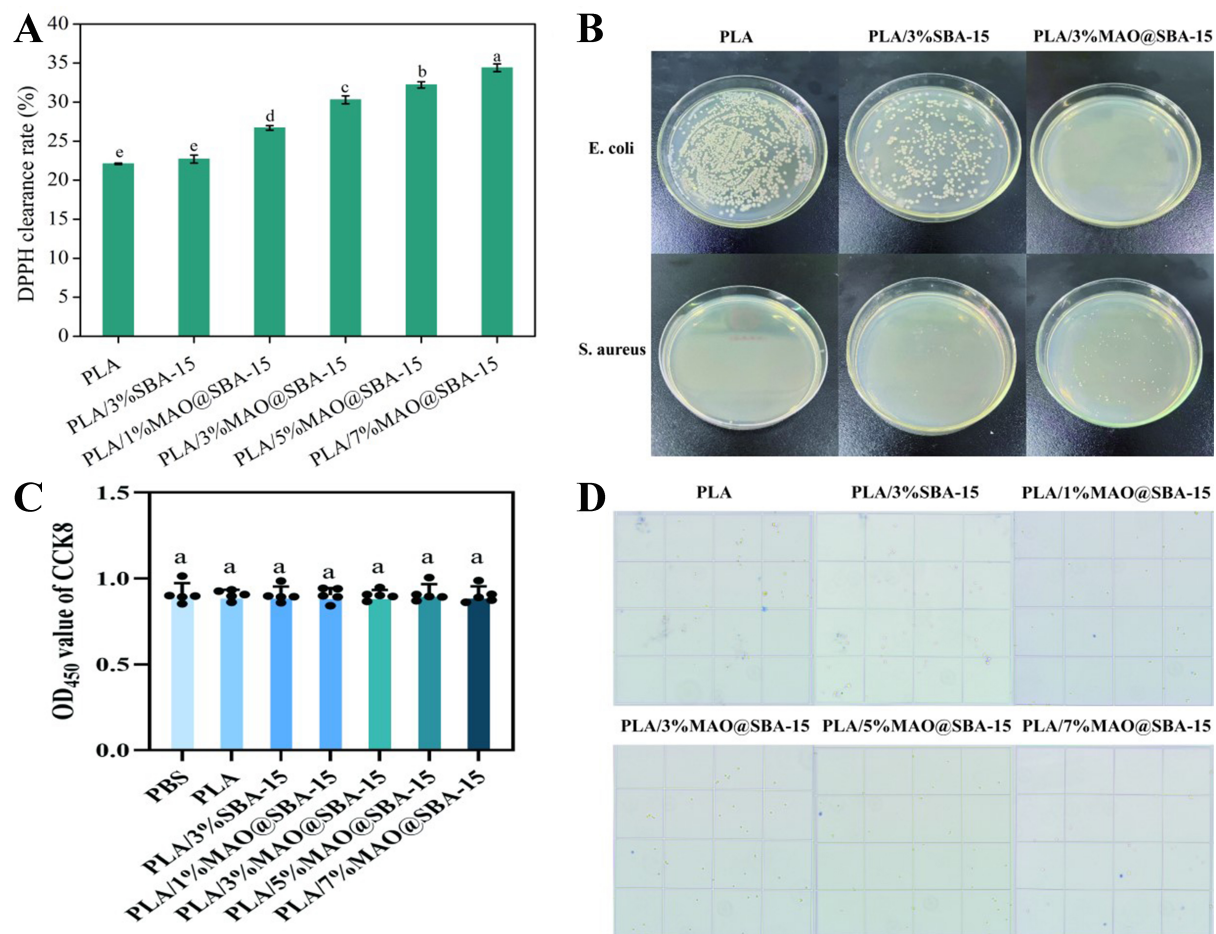


Figure 6. Activity and toxicity of the PLA-based packaging films. (A) Antioxidant activity; (B) Antibacterial activity; (C) Cell viability assay; and (D) Trypan blue staining. Different letters represent statistically significant differences ($P < 0.05$). PLA: Poly(lactic acid).

15 achieves a scavenging rate of $(34.4 \pm 0.5)\%$, representing a substantial 55.7% increase compared to the PLA film. This enhanced antioxidant activity was ascribed to the redox properties of the aldehydes in MAO, which act as free radical acceptors^[36].

The antibacterial activities of the PLA-based films are shown in Figure 6B. The PLA film displayed better efficacy against *S. aureus* than against *E. coli*. Compared to the PLA film, the inhibition rates of the PLA/3%SBA-15 film against *E. coli* and *S. aureus* are 55.3% and 79.8%, respectively. This significant enhancement in antibacterial activity can be attributed to the unique physicochemical properties of the SBA-15 material. Although the addition of SBA-15 alone to the PLA matrix inhibited both *E. coli* and *S. aureus* compared to the PLA film, a marked improvement was observed with the incorporation of 3 wt% MAO@SBA-15. The PLA/3%MAO@SBA-15 film showed 100% inhibition against *E. coli* and *S. aureus*. This significantly enhanced antibacterial effect is attributed to the active ingredients in MAO that disrupt bacterial cell membrane integrity, thereby inhibiting growth^[37]. These results demonstrate that the PLA-based films containing MAO@SBA-15 exhibited excellent antibacterial activity against both pathogens. Consequently, the incorporation of MAO@SBA-15 is an auspicious approach for developing effective PLA-based active packaging films.

After incubating LO2 cells with various types of PLA materials for 24 h, CCK-8 assay results, as shown in Figure 6C, revealed that none of the PLA materials significantly inhibited cell proliferation compared to the control group ($P > 0.05$). Consistent with this finding, trypan blue staining, a common method for detecting cell death, showed that viable cells remained unstained, while dead cells exhibited blue staining as shown in Figure 6D. Importantly, no notable increase in cell death was observed in any PLA-treated group, and there were no statistically significant differences among the experimental conditions. These results demonstrate that the PLA materials exhibit excellent biocompatibility and do not induce cytotoxic effects on LO2 cells under the tested conditions. The absence of both anti-proliferative and cell-death-inducing effects highlights the potential safety of these materials.

Preservation of PLA-based packaging films for mulberries

The weight loss rate and hardness of mulberries preserved for seven days using PLA-based packaging films are shown in Figure 7. Weight loss rates for mulberries packaged with PLA, PLA/3%SBA-15, PLA/1%MAO@SBA-15, PLA/3%MAO@SBA-15, PLA/5%MAO@SBA-15, and PLA/7%MAO@SBA-15 films are measured at $(5.8 \pm 0.1)\%$, $(4.0 \pm 0.2)\%$, $(5.4 \pm 0.2)\%$, $(4.5 \pm 0.1)\%$, $(3.8 \pm 0.2)\%$, and $(3.6 \pm 0.1)\%$, respectively. Hardness for mulberries packaged with PLA, PLA/3%SBA-15, PLA/1%MAO@SBA-15, PLA/3%MAO@SBA-15, PLA/5%MAO@SBA-15, and PLA/7%MAO@SBA-15 films is measured at (0.55 ± 0.05) N, (0.58 ± 0.04) N, (0.63 ± 0.05) N, (0.71 ± 0.06) N, (0.77 ± 0.08) N, and (0.82 ± 0.05) N, respectively. The mulberries encased in the PLA/7%MAO@SBA-15 film exhibited the lowest weight loss rate and the highest hardness. This significant reduction was directly attributable to the PLA-based film containing 7 wt% MAO@SBA-15, which showed the highest gas barrier and antioxidant activity. These findings unequivocally establish that PLA-based films incorporating MAO@SBA-15 possess a remarkable preservation efficacy for mulberries. Most importantly, they effectively minimize water loss and improve antioxidant activity, thereby substantially extending the shelf life and maintaining quality over extended periods.

Release of MAO in PLA-based packaging films

Figure 8A presents the standard curve for the MAO-ethanol solution. This curve exhibited a linear relationship, yielding the fitting equation $A = 0.0146 \times c + 0.1005$. Release ratios of the PLA/3%MAO and PLA/3%MAO@SBA-15 films are shown in Figure 8B. It can be seen from Figure 8B that the release ratio of MAO from the PLA/3%MAO film was higher than that from the PLA/3%MAO@SBA-15 film at the same time, indicating that MAO@SBA-15 exhibits slow-release characteristics.

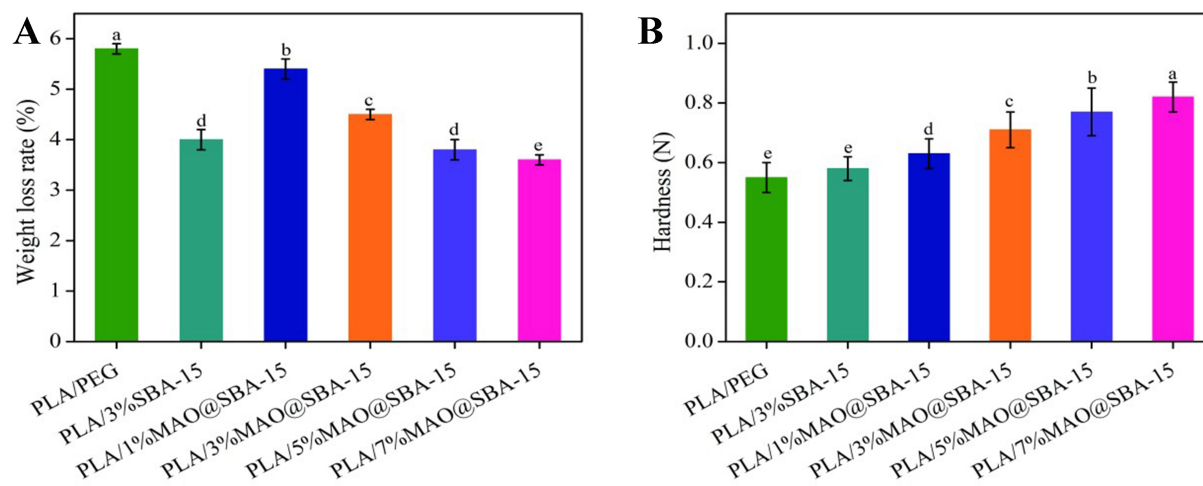


Figure 7. Preservation efficacy of mulberries preservation for 7 d by PLA-based packaging films. (A) Weight loss rate; and (B) Hardness. Different letters represent statistically significant differences ($P < 0.05$). PLA: Poly(lactic acid).

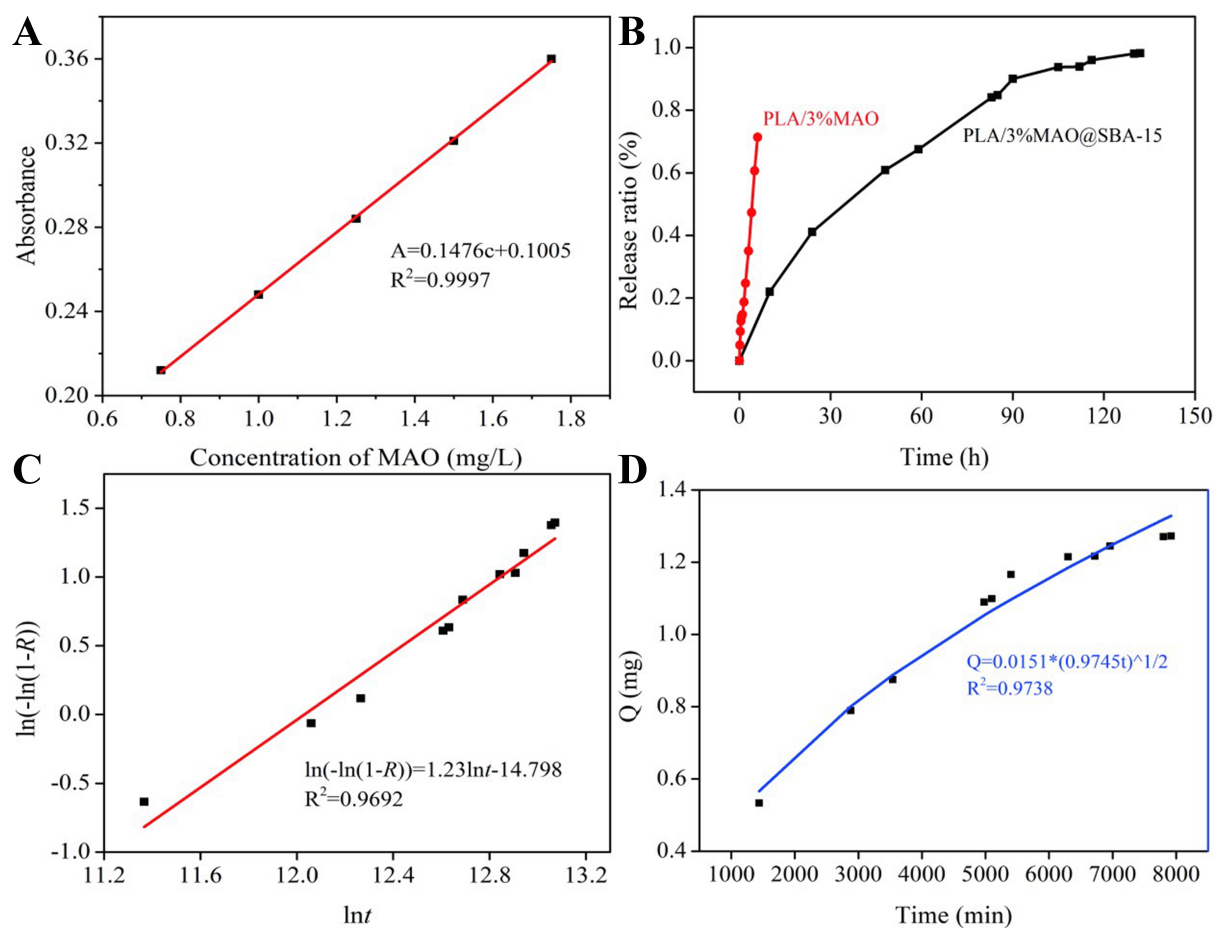


Figure 8. Release kinetics of MAO in the PLA/3%MAO@SBA-15 film. (A) Standard curve; (B) Release ratio; (C) Avrami model; and (D) Higuchi model. PLA: Poly(lactic acid); MAO: cinnamon oil; SBA-15: Santa Barbara Amorphous-15.

The release kinetics of MAO from the PLA/3%MAO@SBA-15 film, along with the fitting results, are shown in Figure 8C and D. The release data were effectively modeled by the Avrami equation^[38], as evidenced by the fitting equation $\ln(-\ln(1-R)) = 1.23 \times \ln t - 14.798$ and a high coefficient of determination ($R^2 = 0.9692$). The fitted parameters include a rate constant (k) of $5.96 \times 10^{-6} \text{ s}^{-1}$ and an Avrami exponent (n) of 1.23. Most significantly, the derived Avrami exponent ($n = 1.23$) and the sustained nature of the release kinetics implied a controlled-release effect. The fitting Higuchi equation was $Q = 0.0151 \times (0.9745t)^{1/2}$, and the R^2 reached 0.9738. Critically, the excellent fit strongly indicated that the Higuchi equation accurately described the release mechanism of MAO from the PLA-based packaging film containing MAO@SBA-15. The Peleg rate constant of TEO in SBA-15-TEO/potato starch films (2.07) was higher than that in MCM-41-TEO/potato starch films (1.77) and SBA-16-TEO/potato starch films (1.61)^[39]. This is because the pore diameter of SBA-15 (8.5 nm) exceeds those of MCM-41 (4.0 nm) and SBA-16 (5.1 nm)^[40]. This controlled release is pivotal because it suggests the potential of the PLA/3%MAO@SBA-15 film to provide sustained, long-term antibacterial and antioxidant activity in practical applications.

CONCLUSIONS

PLA-based active packaging films were fabricated by using SBA-15 loaded with MAO. The specific surface area and pore size of SBA-15 are 568.9 m²/g and 7.5 nm, respectively. The loading capacity of SBA-15 for MAO is 801.5 mg/g. MAO@SBA-15 enhanced the crystallinity, tensile strength, gas barrier, antioxidant activity, and antibacterial activity of the PLA-based composite films. The PLA-based composite films with 3 wt% MAO@SBA-15 exhibited the uniform dispersion, the highest crystallinity (32.4%), and the highest tensile strength (16.0 MPa). PLA-based composite films with 7 wt% MAO@SBA-15 had the superior DPPH scavenging rate (34.4%), the lowest WVP [$2.6 \times 10^{-13} \text{ g}\cdot\text{cm}/(\text{cm}^2\cdot\text{s}\cdot\text{Pa})$], the lowest OTR [$4.5 \times 10^{-13} \text{ cm}^3\cdot\text{cm}/(\text{cm}^2\cdot\text{s}\cdot\text{Pa})$], and the lowest weight loss rate (3.6%) and the highest hardness (0.82 N) of mulberries preservation for 7 days. The release kinetics of MAO from the PLA-based packaging films are in accordance with the Higuchi model. The PLA-based packaging films with MAO@SBA-15 exhibited a sustained-release effect. The absence of both anti-proliferative and cell-death-inducing effects highlights the potential safety of these materials. Long-term stability and degradation of the PLA-based packaging films in practical packaging applications will be further investigated.

DECLARATIONS

Authors' contributions

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Validation, investigation: Jin, D.

Formal analysis, validation: Han, X.

Software, formal analysis: Yuan, S.

Formal analysis, investigation: Hu, L.

Funding acquisition, writing - review and editing: Li, H.

Conceptualization, supervision, funding acquisition, writing - review and editing: Wang, Y.

Availability of data and materials

The datasets generated or analyzed during this study are available from the corresponding author upon reasonable request.

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Conflicts of interest

All authors declared that there are no conflicts of interest.

Ethical approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

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