

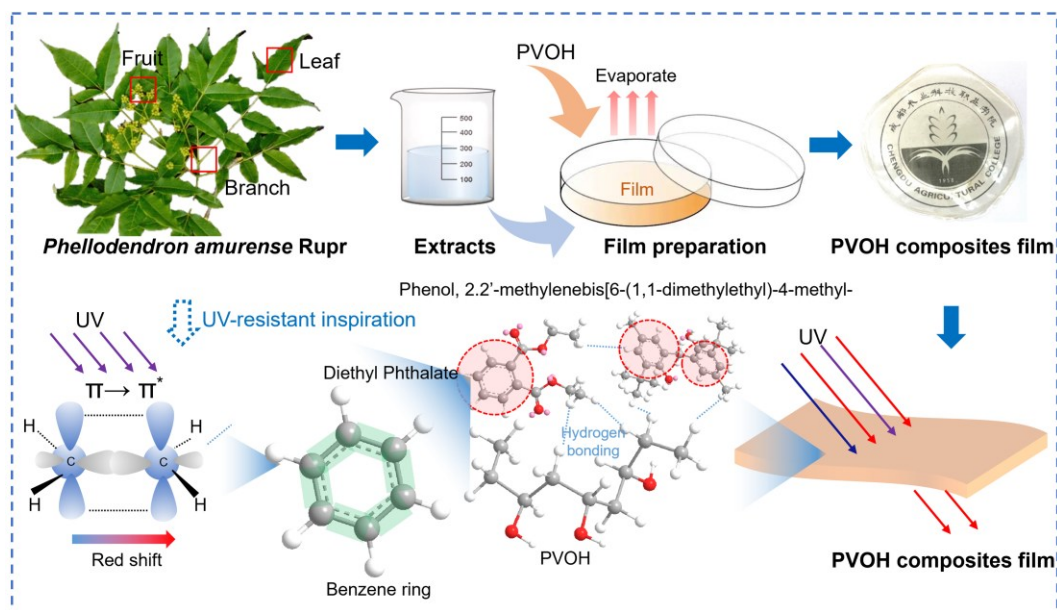
High-Strength UV-Resistant Polyvinyl Alcohol Composite Films Based on *Phellodendron amurense* Rupr Extract

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GRAPHICAL ABSTRACT



High-Strength UV-Resistant Polyvinyl Alcohol Composite Films Based on *Phellodendron amurense* Rupr Extract

Feng Li,^a Hongpeng Li,^a Guo Li,^a Yujia Zhang,^b Haoyu Ma,^c and Min Tang^{a,*}

Environmentally friendly and biodegradable polyvinyl alcohol (PVOH) film combined with *Phellodendron amurense* extract was used to prepare an anti-UV composite film through thermal flow molding technology. The prepared PVOH composite film containing *Phellodendron amurense* extract exhibited UV-resistant properties, with the composite film made from leaf extract showing the highest UV resistance. Additionally, the tensile strength and toughness of the PVOH composite film with added *Phellodendron amurense* extract significantly increased compared to pure PVOH film. Studies on film-forming and UV-resistant mechanisms have revealed that extract particles can act as nucleating agents to promote the local ordered arrangement of PVOH molecular chains, forming microcrystalline regions. This enhances the tensile strength of composite films while maintaining their toughness. During the film-forming process, the extract forms hydrogen bonds with PVOH, and the benzene ring conjugated double bonds in the extract can absorb ultraviolet light, contributing to the UV resistance of the PVOH composite film. The composite film prepared from *Phellodendron amurense* extract and PVOH with UV resistance and high strength can be applied in fields such as packaging, food, and sunscreen products, which is of great significance for promoting the efficient development and utilization of biomass resources.

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Keywords: Composite film; *Phellodendron amurense* Rupr; Extract; UV-resistant

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INTRODUCTION

Environmental pollution and human health issues caused by social development are receiving increasing attention (Xu *et al.* 2022). Using effective protective measures can mitigate the harm caused by environmental factors such as ultraviolet radiation to the human body (Khan *et al.* 2020). Traditional UV protection methods mostly rely on chemically synthesized materials, which suffer from poor environmental friendliness and high costs (Egambaram *et al.* 2020). However, under the influence of environmental hazards such as ultraviolet radiation, natural plants still maintain good growth conditions while thriving in their natural habitats (Ueda and Nakamura 2011). As a result, the inherent UV-resistant properties of plants have attracted widespread attention.

The Amur cork tree (*Phellodendron amurense* Rupr) is a deciduous tree belonging to the Rutaceae family and is distributed in the northeastern and northern provinces of China (Zhang *et al.* 2023). Its wood is widely used in furniture and decoration, and its

extracts can be processed into soap, lubricants, insecticides, and dyes (Ji *et al.* 2023). The cork layer of *Phellodendron amurense* bark can be used to make cork stoppers. The alkaloid components such as berberine, palmatine, and jatrorrhizine contained in its leaf and fruit extracts have significant antibacterial, antioxidant, antitumor, anti-inflammatory, hypoglycemic, and immunosuppressive activities. Also, the extracts from different parts of *P. amurense* have demonstrated excellent activity and broad applications (Akihisa *et al.* 2017). However, the use of pure *Phellodendron* extracts is limited in scope, particularly as there have been no reported studies on utilizing its components for UV protection research. Therefore, the potential and applications of *Phellodendron* extracts await further exploration.

Composite materials combine the advantages of more than one material, enabling the integration of materials with different functionalities (Gibson 2010). Polymer-based biomass film composite materials are widely used in various fields such as food packaging, agricultural settings, and outdoor protection (Bhardwaj *et al.* 2020). Film composite materials have broad application prospects in areas such as UV-resistant functionalization (Dai *et al.* 2025). By incorporating strong ultraviolet absorbers such as benzotriazoles, light stabilizers, and nanofillers (TiO₂, ZnO) into the film composites, the materials' UV resistance and shielding capabilities can be enhanced (Zhang *et al.* 2024). However, these additives face issues of high economic and environmental costs such as photodegradation, migration, and environmental persistence. While benzotriazole derivatives typically exhibit strong UV absorption in the range of 290 to 360 nm, their compatibility with polymer matrices and long-term stability can be problematic. Newly developed natural UV-resistant substances based on lignin, tannins, and plant extracts provide fresh perspectives for the development of UV-resistant film composites. For example, bio-based films were found to suppress UV transmittance very well below 400 nm, along with improved transparency and environmental friendliness (Wang *et al.* 2023). In particular, plant extracts have garnered widespread attention in UV-resistant film composites due to their advantages of low cost, abundant resources, and easy accessibility. Plant extracts have gained increasing attention as natural UV-shielding agents due to their abundance of bioactive compounds, such as flavonoids, polyphenols, and alkaloids, which possess strong UV-absorbing capabilities. These compounds typically contain conjugated double bonds and aromatic ring structures that enable them to absorb and dissipate harmful ultraviolet radiation, particularly in the UVA and UVB ranges (Chandran *et al.* 2024). However, the poor mechanical properties caused by the compatibility issues between the polymer matrix and plant extracts have become a major obstacle in the preparation of UV-resistant composite films. Therefore, it is imperative to develop a plant extract-based UV-resistant film composite with excellent compatibility and superior anti-UV performance.

To address the efficient development and utilization of *Phellodendron amurense* extracts and the preparation of plant extract-based UV-resistant film composites, this study combined extracts from different parts of *P. amurense* with PVOH. Using thermal flow molding technology, UV-resistant composite films were successfully prepared, and they demonstrated excellent UV-blocking performance in the UVB range as well as high tensile strength. Meanwhile, based on the compositional characteristics of *P. amurense* extract and its binding properties with PVOH, the mechanical reinforcement and UV resistance mechanisms of the composite film were investigated. The preparation and mechanism analysis of PVOH-based UV-resistant composite films derived from *P. amurense* extracts not only provides a foundation for the refined and high-value utilization of *P. amurense* but also offers new perspectives for the development of UV-resistant composite materials.

EXPERIMENTAL

Materials

The *Phellodendron amurense* tree was harvested from Laoban Mountain in Yucheng District, Ya'an City, Sichuan Province, China. A total of 10 g of dried and crushed fruit, branch, and leaf powder were wrapped separately with qualitative filter paper and placed in a 250 mL Soxhlet extractor. Ethanol (250 mL) was used for extraction in a 500 mL round-bottom flask for 8 h to obtain the extract. The three extracts were concentrated to remove the remain ethanol by rotary evaporation.

Polyvinyl alcohol (PVOH) was commercially sourced with a polymerization degree of 1700 to 1800, molecular weight of 84,000 to 89,000, viscosity of 21 to 26 Pa·s, pH 5 to 7, and mesh size of 120 mesh.

Method

First, 1 g of polyvinyl alcohol (PVOH) was dissolved in 20 mL of ultrapure water at 85 °C with continuous magnetic stirring to prepare a 5 wt% PVOH solution. The ethanol extracts of branches, fruits, and leaves were added into the PVOH solution, which was stirred slowly for 15 min. The mixture was poured into a plastic Petri dish with a diameter of 8.5 cm (ensuring uniform thickness of the PVOH composite film by adding the same mass of the mixed solution). After solvent evaporation, the film was peeled from the dish and cured at room temperature for 24 h before characterization. The preparation process for the pure PVOH film was the same, except without the addition of ethanol extracts from the plant parts. A total of 3 sample groups were prepared, each with 4 mass gradients (0.05 wt%, 0.15 wt%, 0.25 wt%, and 0.35 wt%, respectively). Each group of specimens with 4 mass gradients was tested 3 times for the follow properties test.

Character

UV-resistant properties

The prepared composite films were cut into 10 mm × 20 mm pieces, then placed in cuvettes and pressed against the sidewall near the light source. A spectrophotometer was used to perform wavelength scanning within the range of 190 to 800 nm.

The color of the film surface

The prepared PVOH composite film was subjected to color testing using a colorimeter. The prepared composite film was placed under the colorimeter, and D65 standard illuminant with TAPPI/ANSI T 524 om-20 (2020). The CIE LAB color space was used for color specification. In that system, L^* represents the value on the white/black axis, a^* represents the value on the red/green axis, and b^* represents the value on the blue/yellow axis. The a^* value best reflects the variation between green and red, where a lower a^* value indicates a greener color of the sample. The b^* value reflects yellow/blue, with a lower b^* value indicating a bluer sample.

Mechanical properties

The prepared film was cut into dimensions of $50 \times 5 \times 0.12$ mm for mechanical property testing. The testing was performed using a DZW-103A micro-mechanical testing machine from Sichuan Ruili Zhongheng Technology Co., Ltd. (Chengdu, China).

Film formation and UV resistance mechanism

The prepared composite film was observed for surface morphology using SEM (ZEISS Sigma 360, Germany). FTIR (Thermo Fisher Scientific Nicolet iS20, USA) testing was performed with a scanning range of 800 to 4000 cm^{-1} and a resolution of 4 cm^{-1} .

RESULTS AND DISCUSSION

UV Resistance of the Film

The PVOH film composite with *Phellodendron amurense* extract exhibited excellent UV resistance, while films incorporating extracts from leaves, fruits, and tree branches showed varying UV resistance properties. Additionally, films with different extract addition amounts also demonstrated distinct UV resistance capabilities (Fig. 1a, b, c). The film using *P. amurense* leaf extract exhibited maximum UV absorbance in the UVB range (280 to 315 nm). Among them, the composite film with a 0.35 wt% addition showed the highest UV absorbance. However, the highest UV absorbance of the composite film with 0.05 wt% addition was lower than that of the PVOH film without extract (Fig. 1a, b), indicating that the composite film only was able to demonstrate UV resistance when the concentration of *P. amurense* leaf extract exceeded a certain threshold.

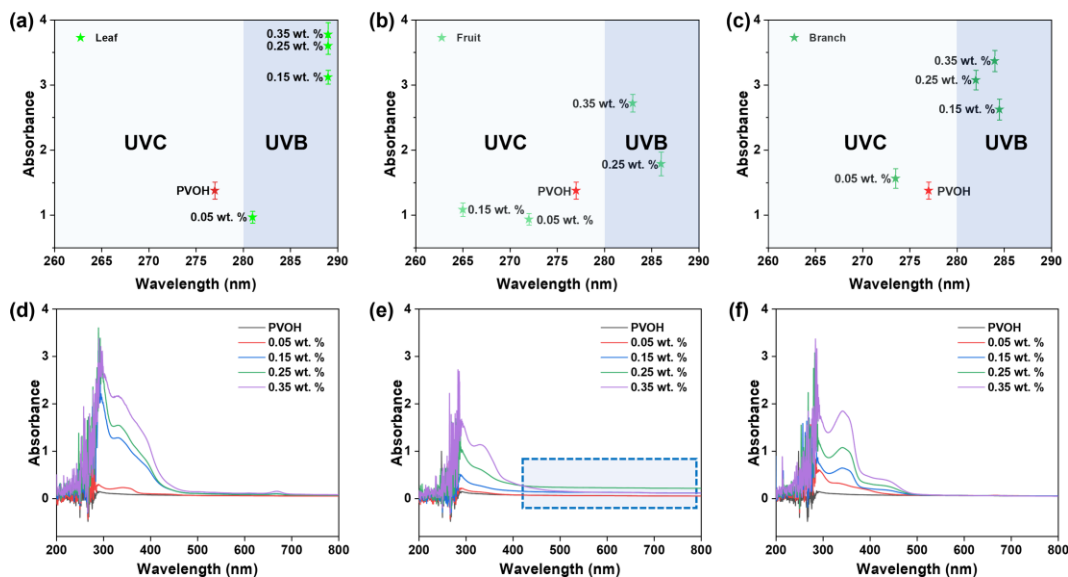


Fig. 1. UV resistance of composite film. UV resistance of leaves (a), fruits (b), and branches (c) with different addition amounts; Absorption curves of leaves (d), fruits (e), and branches (f) with different addition amounts under 200-800 nm spectrum

Similar to the leaf extract, the maximum UV absorbance of the *Phellodendron* fruit extract also occurred in the UVB range, with the PVOH film containing 0.35 wt% of the extract showing the highest UV absorbance. The UV resistance performance of adding *P. amurense* branch extract to PVOH film showed similar characteristics to leaf extract and fruit extract, with the optimal UV resistance achieved at the same addition amount of 0.35 wt%.

To verify the full-spectrum absorption capability of the composite film, the absorption spectrum from 200 to 800 nm was observed. The leaf and branch extract composite film exhibited certain light absorbance in the wavelength range of 300 to 450 nm, while showing extremely low absorbance in the range of 500 to 800 nm (Fig. 1d). For the fruit extract composite film, there was a certain absorbance in the wavelength range of 500 to 800 nm, with the film containing 0.25 wt. % of extract exhibiting the highest absorbance in this range (Fig. 1e, f). The above results indicate that the addition of *P. amurense* extract not only altered the UV resistance of PVOH films but also affected their absorbance in other wavelength ranges. In particular, the addition of fruit extract influences the absorbance of PVOH films in the 500 to 800 nm range, leading to changes in the light absorbance spectrum of the films, thereby impacting their applications.

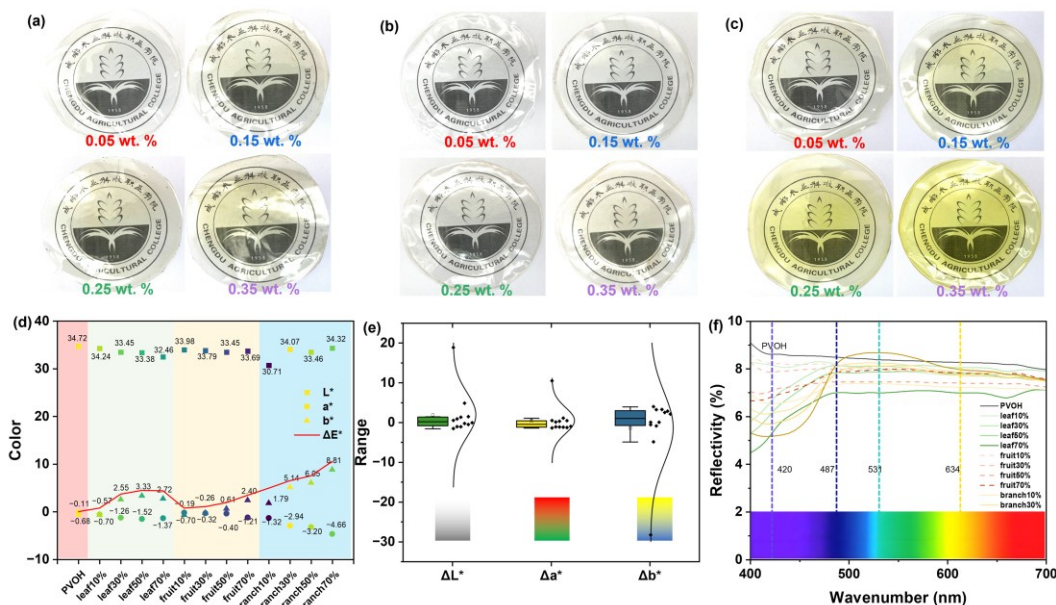


Fig. 2. Color characteristics of composite films. Physical images of PVOH composite films prepared from leaf (a), fruit (b), and branch (c) extracts; (d) Lab values of the color of composite films with different parts and extract addition levels; (e) Color difference values between different parts and addition amounts compared to pure PVOH film; (f) Reflectance of composite films with different parts and extract addition amounts in the 400 to 700 nm spectrum

The UV-resistant properties of PVOH films prepared with *P. amurense* extract enable their application in packaging, clothing, and other scenarios. However, the apparent color of the film also influences its potential applications (Ran *et al.* 2023). Therefore, this study characterized the color of composite films with different extraction parts and varying additive amounts. The composite films made from extracts of different parts exhibited varying colors. The extract from the branch displayed the darkest color, appearing yellow, while the composite film from the fruit extract showed the lightest color with almost no noticeable hue (Fig. 2a, b, c). As the concentration of the extract increased, the color of the

film consistently shifted from light to dark. Compared to pure PVOH films, the addition of leaf extract significantly increased the b^* -value of the films, and the b^* -value increased with the amount added. The addition of leaf extract caused the PVOH film to shift from colorless to reddish. The addition of fruit extract significantly increased the b^* -value and decreased the a^* -value of the PVOH composite film. The b^* -value increased with higher additive amounts, while the a^* -value decreased with higher additive amounts. The incorporation of the fruit extract shifted the PVOH composite film toward blue and red hues. The addition of branch extracts had the most significant impact on the color of the composite film. As the branch extracts were added, the b^* -value of the composite film increased with the increasing amount of addition, while the a^* -value decreased, and the L^* -value also decreased with the increasing amount of addition. This indicates that the incorporation of branch extracts caused the PVOH composite film to gradually shift toward reddish and bluish tones (Fig. 2d). Compared to pure PVOH films, the composite films with added extracts showed a color difference b^* -value greater than 0, indicating a yellowish shift in the film's color (Fig. 2e). Observation of the total reflection spectrum of the composite film revealed that when the addition amount of leaf and branch extracts was 0.35 wt. %, the reflectance of the composite film in the blue light range of 400 to 450 nm was lower than that of the pure PVOH film, indicating that the composite film has a certain reflection effect on violet light (Fig. 2f).

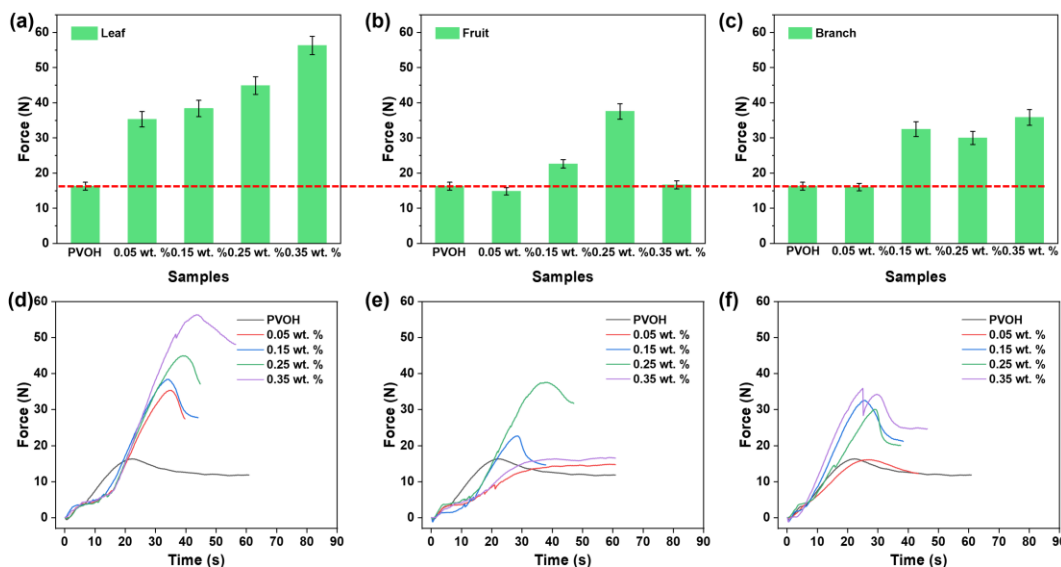


Fig. 3. Mechanical characteristics of composite films. Tensile strength of composite films with varying amounts of leaf(a), fruit(b), and branch(c) extracts; Time and force curves of composite films with different addition amounts of leaf(d), fruit(e), and branch(f) extracts

The mechanical properties of composite films affect their application as UV-resistant materials. Tensile performance tests indicated that the addition of leaf extracts enhanced the tensile strength of the composite film. The film's strength increased with the amount of extract added. Compared to fruit and branch extracts, the addition of leaf extracts showed the most significant improvement in the tensile strength of the film. At the same time, when the addition amount of fruit and branch extracts was 0.05 wt. %, it did not increase the tensile strength of the film (Fig. 3a, b, c). From the tensile curve, it can be observed that the leaf extract enhanced the endurance time and maximum force value of the composite film (Fig. 3d), while the increase in the slope of the curve also indicated that

the extract significantly improved the tensile modulus of the composite film (Homthawornchoo *et al.* 2022). The addition of fruit and branch extracts also was able to increase the tensile strength and tensile modulus of the film. However, when the amount of branch extract added was 0.35 wt%, the tensile curve shows obvious fluctuations (Fig. 3e, f). This phenomenon may be caused by film defects resulting from poor compatibility between the PVOH matrix and the extracts.

Composite film formation and UV resistance mechanism

The chemical components of *Phellodendron amurense* extract are the main reason for improving the UV resistance and mechanical properties of PVOH composite films. By observing the GC-MS spectra of extracts from the leaves, fruits, and branches, it was found that the main components of the three extracts differed significantly (Fig. 4a, b, c). The main components of leaf extracts were primarily divided by their content as follows: diethyl phthalate; nordazepam, TMS derivative; cyclodecasiloxane, eicosamethyl-, neophytadiene and cyclononasiloxane, octadecamethyl- (Fig. 4d).

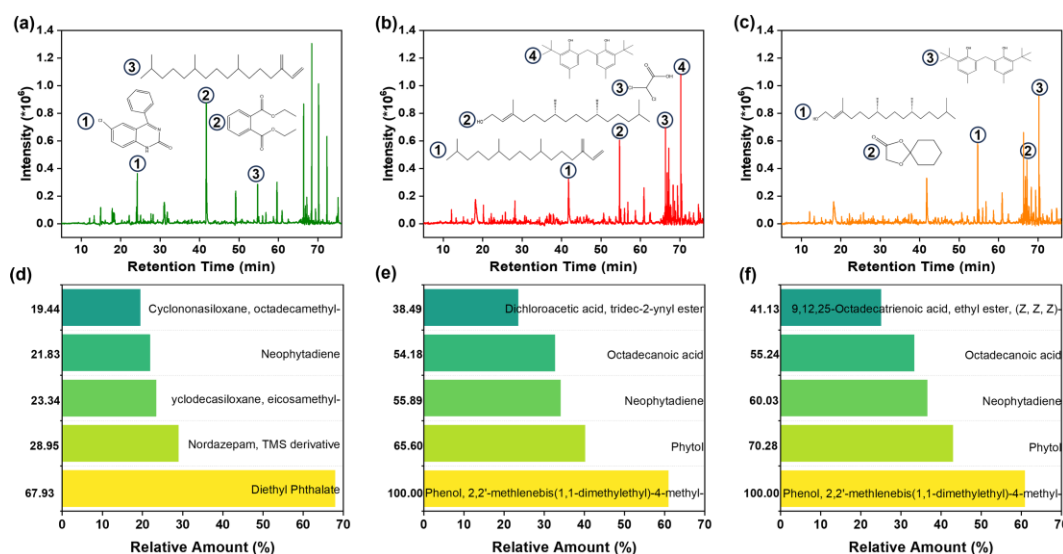


Fig. 4. Chemical composition of extracts. GC-MS chromatograms of leaf(a), fruit(b), and branch(c) extracts; Main components of leaf(d), fruit(e), and branch(f) extracts

Diethyl phthalate, as a plasticizer, can enhance the compatibility and ductility of the extract with PVOH composite films, and it is likely the main reason for the improvement in the mechanical properties of the films (Etuk and Inyang 2024). The extract of the fruit mainly contained: phenol; 2,2'-methylenebis[6-(1,1-dimethylethyl)-4-methyl]; phytol; neophytadiene; octadecanoic acid and dichloroacetic acid, tridec-2-ynyl ester (Fig. 4e). The branch extract had phenol; 2,2'-methylenebis[6-(1,1-dimethylethyl)-4-methyl]; phytol; neophytadiene; octadecanoic acid and 9,12,15-octadecatrienoic acid, ethyl ester, (Z, Z, Z) (Fig. 4f). The top four dominant components were consistent with those of the fruit extract. Therefore, there was a similarity in the enhancement effects of the film's UV resistance and mechanical properties. The extract components from different parts of *Phellodendron amurense* all contained benzene ring structures, which was the main reason why the PVOH composite film exhibited UV-resistant properties (Xu *et al.* 2023).

By observing the micro-morphology of PVOH composite films, the compatibility between the film and extractives as well as the mechanical reinforcement mechanism can be investigated. SEM observations indicated that the PVOH film without added extract

appeared smooth and flat with no apparent defects. The magnified image revealed particulate matter on the film surface, likely originating from incompletely dissolved PVOH particles (Fig. 5a).

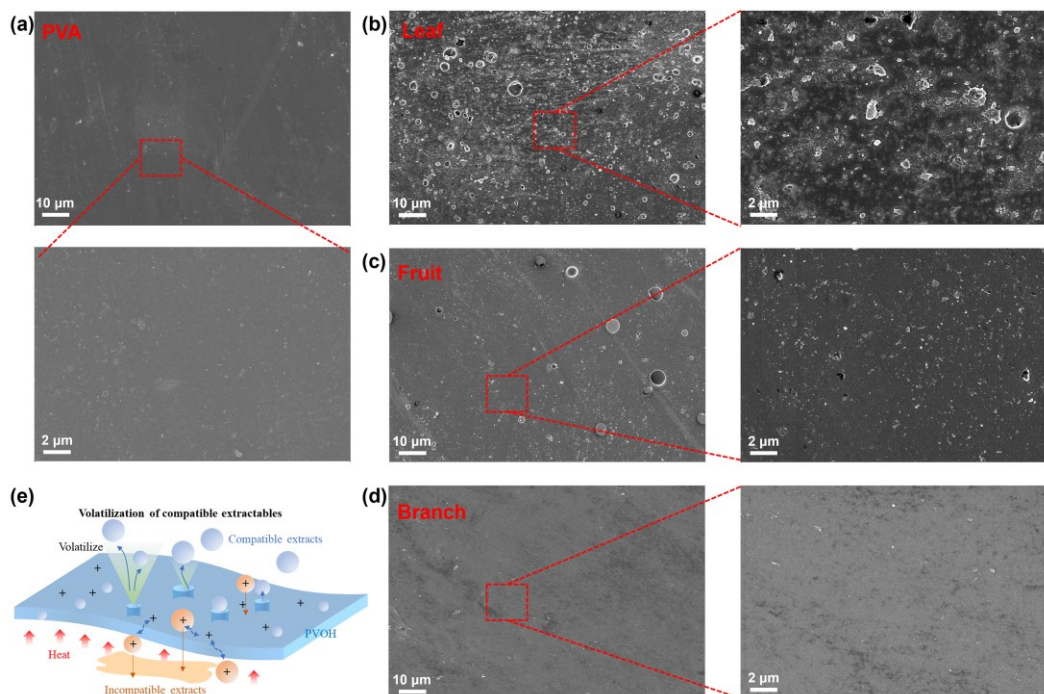


Fig. 5. Microscopic morphology and formation mechanism of composite films. SEM images of pure PVOH film (a), composite films with leaf(b), fruit(c), and branch(d) extracts; (e) Mechanism of composite film formation

The SEM image of the leaf extract-PVOH composite film revealed unevenly distributed pores of varying sizes, along with a granular and rough surface morphology (Fig. 5b). This may be due to the particles in the extract acting as nucleating agents for the film, which can promote the locally ordered arrangement of PVOH molecular chains, forming tiny crystalline regions. This enhances the tensile strength of the composite film while maintaining its toughness (Shahbazi *et al.* 2017). The surface smoothness of the fruit extract composite film was lower compared to that of leaves, while the extract film also had fewer holes (Fig. 5c). The surface of the composite film added with branch extract appeared smooth and flat, without obvious pore structure, similar to the composite film without extract (Fig. 5d). The extract of *P. amurense* was mostly composed of volatile substances. During the heating and film-forming process, the extract components compatible with PVOH volatilized under heat, resulting in uneven surfaces or pores in the film. In contrast, the extract components incompatible with the film remained in the film-forming vessel, thus having a limited impact on the surface morphology of the film (Fig. 5e).

FTIR was used to investigate the binding mechanism of extract addition to PVOH composite films. In the FTIR spectrum, the range of 3000 to 3600 cm^{-1} is attributed to the O-H stretching vibration of PVOH. The C-H stretching vibration peak near 2900 cm^{-1} indicates the presence of numerous carbon-hydrogen bonds in the PVOH molecule. The peak at 1730 cm^{-1} corresponds to C=O stretching vibration, while the C=O stretching vibration peak near 1650 cm^{-1} may originate from partial oxidation during the preparation

of PVOH. The absorption at 1455 cm^{-1} is due to CH_2 deformation vibration, and the C-O stretching vibration peaks appear in the range of 1000 to 1200 cm^{-1} . Compared to pure PVOH film, the PVOH film with added extracts from leaves, fruits, and branch only showed a slight decrease in the peak intensity attributed to OH, while the other characteristic peaks exhibited no significant changes (Fig. 6a, b, c).

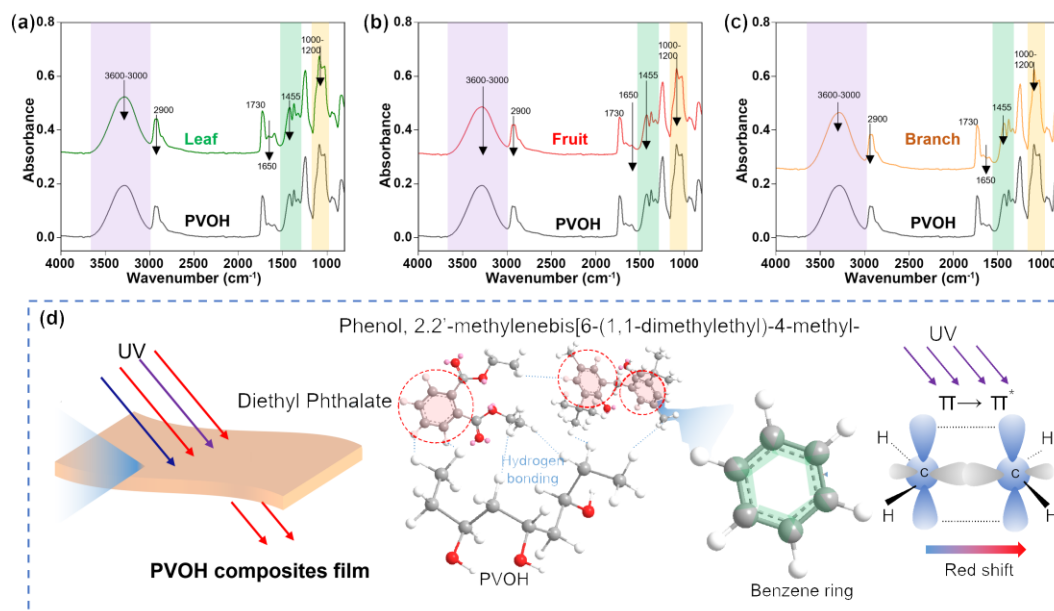


Fig. 6. Composite film formation and UV resistance mechanism. FTIR spectra of leaf(a), fruit(b), and branch(c) extracts with PVOH composite films; (d) UV resistance mechanism of composite films.

This indicates that the *Phellodendron amurense* extract combined with the PVOH film did not undergo a chemical reaction but instead formed a simple physical mixture (Liang and Wang 2018). The OH groups in PVOH and the OH groups in the extract form hydrogen bonds, enhancing the mechanical properties of the composite film. Pure PVOH films lack UV resistance. After compounding, the components from the *P. amurense* extract are dispersed within the film. The extract contains conjugated double bonds in its benzene rings, which can absorb UV light. This is primarily because the π electrons in the conjugated system undergo energy level transitions when exposed to UV radiation. When UV light irradiates the benzene rings, energy is transferred to the π electrons, causing them to transition to higher energy levels (Fang *et al.* 2024). This process absorbs the energy of the UV light, thereby endowing the composite film with UV resistance (Fig. 6e).

CONCLUSIONS

1. The polyvinyl alcohol (PVOH) composite film incorporated with *Phellodendron amurense* extract exhibited UV-resistant properties, and its UV-blocking performance improved with increasing additive amounts. The composite film prepared with leaf extracts demonstrated the highest UV resistance. The color of the composite film with added extract changed from transparent to slightly yellow, while reducing the reflectance of the pure PVOH film in the range of 500 to 700 nm. In addition, the tensile

strength of PVOH composite film with added *P. amurensis* extract significantly increased compared to pure PVOH film, while also enhancing the film's toughness.

2. Observation of the extract components from the leaves, fruits, and branch of *P. amurensis* revealed that the extracts from the branch and fruits have similar compositions, while the leaf extract differs significantly from both. All extracts contain chemical substances with benzene ring structures. During the film-forming process, the extract particles can act as nucleation agents for the film, promoting the local ordered arrangement of PVOH molecular chains and forming tiny crystalline regions. This enhances the tensile strength of the composite film while maintaining its toughness, a phenomenon particularly evident in leaf extract composite films.
3. The *P. amurensis* extract did not undergo a chemical reaction with PVOH during the film-forming process. Rather, it was uniformly distributed within it through hydrogen bonding. The presence of conjugated double bonds in the benzene ring within the extract enabled it to absorb ultraviolet light, contributing to the UV resistance of the PVOH composite film.

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