Research Article

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Structure–property Relationships of Eco-friendly Spin-coated Poly(ε-caprolactone) (PCL) Thin Films

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ABSTRACT

The increasing demand for sustainable polymer processing requires the replacement of conventional toxic solvents. However, limited information is available on how environmentally friendly solvents, particularly ethyl acetate (EA) as a sustainable alternative to chloroform (H), influence the structural, morphological, surface, and optical properties of spin-coated poly(ε-caprolactone) (PCL) thin films. In this study, the effect of solvent selection on the properties of PCL thin films prepared by spin coating was investigated, highlighting the potential of EA as a more environmentally friendly alternative for PCL film fabrication. The films were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), contact angle measurements, and UV–Vis spectroscopy. XRD analysis confirmed the preservation of the orthorhombic crystalline structure of PCL, with similar crystallite size values indicating that the choice of solvent had no significant effect on the crystallization process or the size of the crystalline domains. SEM analysis showed that both films have a characteristic spherulite morphology, with the PCL-EA film forming larger spherulites than the PCL-H film. At the same time, the PCL-EA film exhibited a higher surface free energy and a more pronounced polar component, with a measured surface free energy of 54.70 mN/m compared with 41.62 mN/m for the PCL-H film, indicating better wettability and more favorable interfacial interactions. UV–Vis analysis showed higher optical band gap values for both direct and indirect electronic transitions in the PCL-EA film compared to the PCL-H film, with direct and indirect band gap values of 5.0 eV and 4.8 eV for PCL-EA, and 4.8 eV and 4.1 eV for PCL-H, respectively, which may indicate a reduced number of localized energy states and greater structural order, with both films retaining the optical properties characteristic of PCL as an electrically insulating polymer. The results confirm that ethyl acetate is a suitable, more sustainable alternative to chloroform for preparing PCL thin films, thereby improving surface and optical properties while preserving the structural characteristics of the biodegradable polymer. Such thin films have potential applications in sustainable polymer coatings, packaging materials, and other environmentally friendly technologies, contributing to the development of greener polymer fabrication approaches.

Keywords: Poly(ε-caprolactone) (PCL); Thin films; Spin coating; Surface properties; Biodegradable polymers.

1. INTRODUCTION

Growing concerns about environmental pollution and the accumulation of non-biodegradable plastic waste have spurred intensive research to develop sustainable, environmentally friendly polymeric materials (Tong *et al.* 2022; Park *et al.* 2024). In this context, biodegradable polymers represent an important alternative to conventional synthetic plastics, as they help reduce negative environmental impacts while maintaining suitable functional properties (Fiandra *et al.* 2023). Among various biodegradable polymers, poly(ε-caprolactone) (PCL) occupies a special position due to its biodegradability, biocompatibility, chemical stability, and ability to form different structures tailored to specific applications (Woodruff and Hutmacher, 2010). PCL is a semicrystalline polyester with a melting temperature ranging from 59 to 64°C, while its glass transition temperature is approximately −60°C (Woodruff and Hutmacher, 2010). These characteristics enable the material to have high flexibility, toughness, and mechanical stability. PCL is susceptible to degradation through hydrolytic processes and microbial activity, which makes it an interesting material for the development of more sustainable polymer systems (Yoon *et al.* 2023). Although it exhibits a slower degradation rate than some other biodegradable polyesters, its long-term stability is an advantage in applications that require preserving material structure and functionality over extended periods.

PCL-based polymer thin films represent an important class of functional materials due to their potential applications in various technological fields, including protective coatings, packaging systems, surface modifications, and the development of advanced sustainable materials (Bujok *et al.* 2021; Ahmadi *et al.* 2024). The performance of thin films largely depends on their structure, morphology, crystallinity, optical characteristics, and surface properties. Recent studies have demonstrated that structural parameters, including polymer chain organization and crystallization behavior,

strongly influence the final morphology and functional properties of PCL-based materials (Fernández-Tena *et al.* 2023). Therefore, understanding the relationship between preparation conditions and the film's final properties is key to controlling its functionality. Among various thin-film fabrication techniques, the spin-coating method stands out for its simplicity, high reproducibility, and ability to control the formation of thin, homogeneous layers (Modrić-Šahbazović *et al.* 2025). However, the properties of the obtained films depend significantly on the deposition parameters, especially on the choice of solvent. The solvent plays a key role in polymer dissolution, evaporation rate, polymer chain organization, and the formation of the final microstructure, thereby directly affecting the material's surface, optical, and structural properties (Pandey *et al.* 2023). In accordance with the principles of green chemistry, increasing attention has been paid in recent years to replacing conventional organic solvents with more environmentally friendly alternatives (Byrne *et al.* 2016). Although chloroform is a frequently used solvent for preparing PCL thin films due to its excellent polymer-dissolving ability, its toxicity and adverse environmental impact pose limitations to more sustainable production processes. As a potential green alternative, ethyl acetate represents a solvent with more favorable environmental characteristics, but it is necessary to understand its influence on the formation and properties of PCL thin films.

Although PCL has been widely investigated, the influence of solvent selection on the structural organization, surface characteristics, and optical properties of spin-coated PCL thin films remains insufficiently understood. In particular, limited attention has been given to the use of environmentally preferable solvents, such as ethyl acetate, as alternatives to commonly used toxic solvents like chloroform, and to their influence on the final properties of PCL thin films. Therefore, establishing a direct relationship between solvent characteristics, film morphology, surface properties, and optical behavior represents an important research challenge for the development of more sustainable polymer processing approaches. The novelty of this study lies in the systematic comparison of PCL thin films prepared with chloroform and ethyl acetate as solvents, with particular emphasis on assessing whether ethyl acetate offers a more sustainable alternative while maintaining or improving the functional properties of PCL films. Therefore, the aim of this study is to investigate the relationship between the structure and properties of spin-coated PCL thin films prepared with different solvents, with particular emphasis on the potential application of environmentally friendly alternatives in their fabrication. The results provide new insights into the solvent-structure-property relationship of spin-coated biodegradable polymer films and contribute to the development of environmentally friendly polymer coatings for potential applications in sustainable packaging and surface modification.

2. EXPERIMENTAL

2.1 Preparation Of PCL Thin Films

PCL thin films were prepared using the spin-coating technique on a spin coater (Model P-6708D, Specialty Coating Systems, Indianapolis, IN, USA) with a maximum rotational speed of 8000 rpm. Poly(ε-caprolactone) (PCL, Thermo Scientific) was used as the polymer material. To investigate the influence of solvent selection on the structural and functional properties of the resulting thin films, PCL was dissolved in two solvents: chloroform (Lach:ner) and ethyl acetate (Gram-Mol).

PCL solutions were prepared by dissolving 4 g of poly(ε-caprolactone) in 20 mL of the corresponding solvent (chloroform or ethyl acetate), resulting in a polymer concentration of 20% (w/v). The dissolution process was carried out under continuous stirring for 3 h at 50 °C until homogeneous polymer solutions were obtained.

For the fabrication of PCL thin films, the prepared polymer solutions were deposited onto n-type silicon substrates by spin coating. Prior to deposition, the silicon substrates were cleaned in an ultrasonic bath for 10 min each in distilled water, acetone, and ethanol, then dried under an air stream. Homogeneous PCL thin films were obtained using optimized spin-coating parameters, with a rotation speed of 3000 rpm and a spinning time of 50 s. The prepared films were subsequently used for structural, morphological, surface, and optical characterization. A schematic representation of the methodology used to fabricate and characterize PCL thin films is presented in Figure 1.

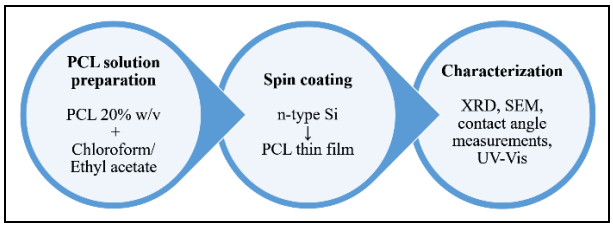

Fig. 1: Schematic representation of PCL thin film fabrication and characterization

2.2 Characterization of PCL Thin Films

The crystal structure of the prepared PCL thin films was analyzed by X-ray diffraction (XRD) using a Bruker D8 Advance diffractometer with CuK_α radiation ($\lambda = 0.154$ nm).

The morphology and surface structure of the PCL thin films were examined using scanning electron microscopy (SEM) with a JEOL JSM-IT200 microscope.

The surface properties and wettability of the PCL thin films were evaluated by contact angle measurements. A droplet of distilled water was placed on the film surface, and the contact angle between the droplet and the film surface was measured. Image analysis and contact angle determination were performed using ImageJ software. The same procedure was repeated using ethylene glycol as the test liquid.

The optical properties of the PCL thin films were analyzed by UV–Vis spectroscopy using a PerkinElmer Lambda 365 UV–Vis spectrophotometer over the wavelength range 190-1100 nm, with a spectral resolution of 0.5 nm.

3. RESULTS AND DISCUSSION

The phase composition and crystal structure of the PCL thin films deposited on n-type silicon substrates were investigated by X-ray diffraction (XRD). Fig. 2 shows the XRD patterns of the PCL thin films prepared from solutions using chloroform (PCL-H) and ethyl acetate (PCL-EA) as solvents. The diffractograms exhibit characteristic PCL diffraction peaks in the 2θ range of 22°–24°, corresponding to the (110) and (200) crystallographic planes (ICDD No. 00-062-1286). The presence of these signals confirms the formation of the orthorhombic crystalline structure of PCL, consistent with data reported in the literature (Tenorio-Alfonso *et al.* 2023; Abdelrazek *et al.* 2016). The XRD analysis shows differences in the intensities of the crystalline reflections depending on the solvent used. The PCL-H film shows higher reflection intensities than the PCL-EA film, suggesting differences in polymer chain organization that may be related to variations in the crystalline structure induced by the solvent used during film preparation (Kamath *et al.* 2020). In addition to the main reflections, the PCL-H sample also shows a weak additional diffraction peak between the (110) and (200) reflections at approximately 2θ ≈ 23°. This feature may be attributed to differences in crystallite size, the distribution of crystalline domains, and the degree of polymer chain ordering induced by the solvent during film formation.

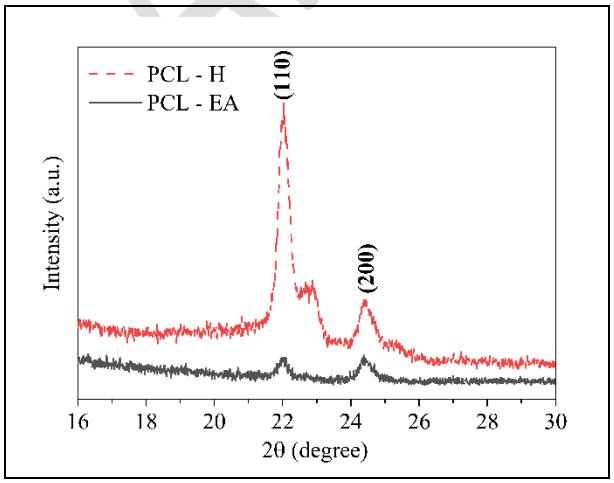

Fig. 2: XRD patterns of PCL thin films prepared using chloroform (PCL-H) and ethyl acetate (PCL-EA) as solvents

The average crystallite size of the PCL thin films was determined using the Debye–Scherrer equation based on the most intense diffraction peaks corresponding to the (110) and (200) crystallographic planes (Monshi *et al.* 2012):

$$D = \frac{k\lambda}{\beta \cos\theta} \quad \ldots (1)$$

where D is the average crystallite size, k = 0.9 is the Scherrer constant, which depends on the crystallite shape, λ is the wavelength of the CuK_α radiation (0.15406 nm), θ is the Bragg angle of the corresponding diffraction peak, and β is the full width at half maximum (FWHM) of the diffraction peak, expressed in radians. The calculated average crystallite sizes were very similar for both analyzed samples. The average crystallite size of the PCL-H film was 17.63 nm, whereas the PCL-EA film exhibited an average crystallite size of 17.56 nm (Table 1). This negligible difference indicates that the use of different solvents did not significantly affect the size of the crystalline domains in the PCL thin films.

Table 1. XRD peak positions, FWHM values, and crystallite sizes of PCL thin films prepared using chloroform (PCL-H) and ethyl acetate (PCL-EA)

Sample	2θ(°)	FWHM(°)	D(nm)	D_{avg}(nm)
PCL-H	22.05	0.51	15.89	17.63
	24.45	0.42	19.38	
PCL-EA	22.01	0.41	19.76	17.56
	24.45	0.53	15.36	

Based on SEM micrographs (Fig. 3), both PCL films prepared from ethyl acetate and chloroform exhibit similar surface morphologies, characterized by radially arranged flower-like or fluffy (spherulitic) aggregates (Simon *et al.* 2007). Such morphology indicates polymer crystallization during solvent evaporation and film formation. Although the overall morphological pattern is similar for both samples, a noticeable difference in the dimensions of the formed structures is observed. The PCL-EA thin film exhibits larger and more pronounced flower-like (spherulitic) structures compared to the PCL-H thin film, where the same structures are smaller and more densely distributed.

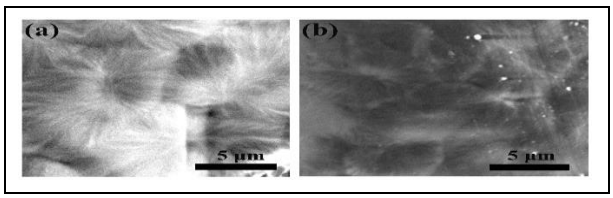

Fig. 3: SEM micrographs of PCL thin films prepared from ethyl acetate (PCL-EA) (a) and chloroform (PCL-H) (b)

The observed differences in film morphology can be attributed to the different evaporation rates of the solvents used (Chen and Woo, 2017). Chloroform, with a boiling point of approximately 61°C, evaporates more rapidly than ethyl acetate (approximately 77°C), promoting a higher nucleation rate and the formation of smaller, more densely packed spherulites in the PCL-H film. On the other hand, the slower evaporation of ethyl acetate provides greater mobility of the polymer chains and allows for prolonged crystal growth, resulting in larger spherulitic structures in the PCL-EA film.

The surface properties of the PCL thin films were evaluated by measuring the contact angle between a droplet of the test liquid and the film surface. The contact angle (θ) is an important parameter for evaluating the wettability of a surface, i.e., the interaction between a solid material and a liquid (Fig. 4). Lower contact angle values indicate better wettability and higher surface hydrophilicity, whereas higher contact angle values reflect weaker interactions between the liquid and the surface (Modrić-Šahbazović *et al.* 2016).

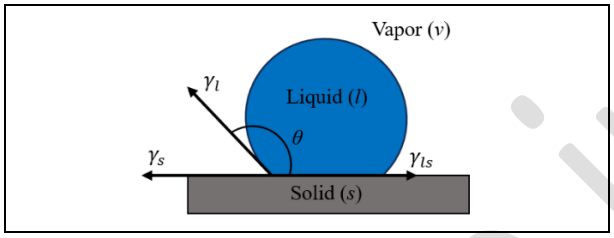

Fig. 4: Schematic illustration of the contact angle (θ) at the solid–liquid–gas interface

The contact angle was measured using distilled water and ethylene glycol as test liquids with known surface energy values. Based on the obtained contact angle values, the surface free energy of the PCL films was determined using the Owens–Wendt–Rabel–Kaelble (OWRK) method, which is based on separating the surface free energy into two components (Uba and Raush, 2025):

$$\gamma_s = \gamma_s^d + \gamma_s^p \quad \ldots (2)$$

where:

γ_s^d is dispersive (London dispersion forces) component of the surface free energy, and

γ_s^p is polar component of the surface free energy that includes dipole–dipole interactions, hydrogen bonds and other polar interaction forces.

The OWRK model relates the contact angle between a liquid and a solid surface to their respective surface energies according to the following equation (Modrić-Šahbazović and Gazdić, 2016):

$$\gamma_l(1+\cos\theta) = 2\sqrt{\gamma_s^d \gamma_l^d} + \sqrt{\gamma_s^p \gamma_l^p} \quad \ldots (3)$$

where:

θ is the measured contact angle between the liquid droplet and the film surface,

γ_l is the surface free energy of the test liquid used,

γ_l^d i γ_l^p are dispersive and polar components of the surface energy of the liquid,

γ_s^d i γ_s^p are unknown dispersive and polar components of the surface energy of the PCL film.

Fig. 5 shows droplets of ethylene glycol (left) and water (right) on the surfaces of PCL-EA (a) and PCL-H (b) thin films.

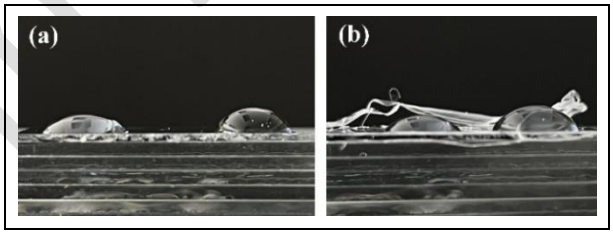

Fig. 5: Contact angle images of ethylene glycol (left) and distilled water (right) on the surface of PCL-EA (a) and PCL-H (b) thin films

The calculated surface free energy parameters of the investigated PCL thin films are summarized in Table 2. For the PCL-EA thin film, the polar component of the surface free energy was determined to be 50.70 mN/m, while the dispersive component was 4.00 mN/m, resulting in a total surface free energy of 54.70 mN/m. The high value of the polar component indicates stronger polar interactions between the PCL surface and the test liquids, leading to improved wettability and more effective contact between the liquid and the polymer. Consequently, these interactions can facilitate a more uniform organization of the polymer chains during film formation, resulting in a more homogeneous surface structure. In contrast, the PCL-H thin film shows lower values of the polar component (28.03 mN/m) and a dispersive component of 13.59 mN/m, with a total surface free energy of 41.62 mN/m. The lower total surface free energy indicates a reduced ability to establish polar interfacial interactions compared with the PCL-EA film. Although the PCL-H film exhibits a greater contribution from dispersive interactions, its overall

interaction with the test liquids is weaker, which may result in a larger contact angle and poorer surface wettability.

The band gap energy is an important parameter for describing the electronic structure of polymer materials, as it provides information about the energy required for electron transitions from the valence band to higher energy levels. To determine the optical band gap of the PCL thin films, UV–Vis optical reflectance measurements were performed. Reflectance measurements were used because the PCL films were deposited on n-type silicon substrates, rendering transmission measurements unsuitable for determining optical properties. Fig. 6 presents the UV–Vis diffuse reflectance spectroscopy (DRS) spectra of PCL thin films prepared from ethyl acetate (PCL-EA) and chloroform (PCL-H) solutions. It can be observed that the PCL-H film exhibits higher reflectance than the PCL-EA film, indicating differences in the films' optical properties due to the different solvents used during preparation. These differences may be related to the specific morphology of the PCL-H film, which is characterized by smaller, more densely distributed spherulitic structures. A higher number of crystalline domains and interfacial regions may enhance scattering of incident light, increasing diffuse reflectance. In contrast, the presence of larger spherulites in the PCL-EA film and a lower number of structural boundaries leads to weaker light scattering and lower reflectance (Nallapaneni *et al.* 2017).

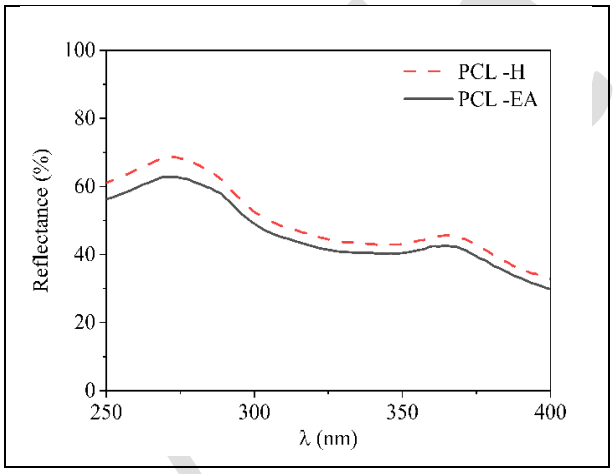

Fig. 6: UV–Vis DRS spectra of PCL thin films prepared from ethyl acetate (PCL-EA) and chloroform (PCL-H) solutions

Based on the measured reflectance values, the Kubelka–Munk function was applied (López and Gómez, 2012):

$$F(R) = \frac{(1-R)^2}{2R} \quad \ldots (4)$$

where R represents the reflectance of the sample. The obtained values of F(R) were used to generate Tauc plots to determine the optical band gap energy. The band gap energy was determined by extrapolating the linear region of the curves of $[F(R) \times E]^2$ for a direct transition and $[F(R) \times E]^{1/2}$ for an indirect transition as a function of E (eV) to to the intercept with the energy axis (Fig. 7). For the direct transition, the PCL-EA thin film shows a slightly higher band gap energy value (5.0 eV) compared with the PCL-H thin film (4.8 eV). Similarly, for the indirect transition, the band gap energy of PCL-EA is also higher (4.8 eV) than that of the PCL-H thin film (4.1 eV). The higher optical band gap values observed for PCL-EA may indicate a lower density of localized energy states and a more ordered structure, whereas the lower band gap values obtained for PCL-H may be associated with a higher presence of structural imperfections and defect states that influence optical transitions.

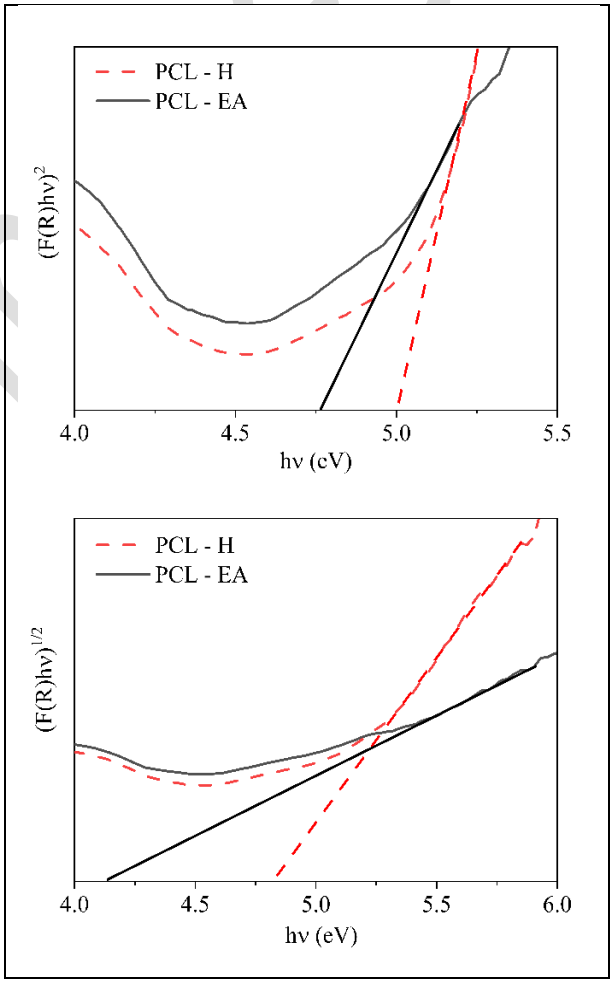

Fig. 7: Optical band gap energy of PCL-EA and PCL-H thin films for direct (a) aindirect (b) optical transitions

The band gap energies of the investigated PCL films are within the range characteristic of PCL as an electrically insulating polymer, with a band gap value of approximately 5 eV, reported in the literature (Abdelrazek *et al.* 2016). Such a high band gap energy

indicates limited electronic conductivity and a low concentration of free charge carriers. The optical band gap values for direct and indirect transitions are summarized in Table 2.

Table 2. Surface and optical properties of PCL thin films prepared using different solvents

Sample	γ_l (mN/m)	$E_{g,\ indirect}$(eV)	$E_{g,\ direct}$(eV)
PCL-H	41.62	4.8	4.1
PCL-EA	54.70	5.0	4.8

4. CONCLUSION

PCL thin films were successfully prepared by spin coating from solutions in chloroform and ethyl acetate, with the latter serving as a green alternative to conventional toxic solvents such as chloroform. The characterization results showed that the choice of solvent influences the surface and optical properties of PCL films, while the polymer's crystalline structure is preserved.

The XRD analysis confirmed the preservation of the orthorhombic crystal structure of PCL, while the similar crystallite sizes indicate that solvent selection has no significant influence on the crystalline domains. The SEM analysis revealed a characteristic spherulitic morphology in both films, with differences in spherulite size attributable to solvent evaporation behavior. The surface analysis demonstrated that the PCL-EA film exhibits improved wettability and more favorable surface interactions compared with the PCL-H film. The UV-Vis analysis revealed that both films maintain the characteristic insulating optical behavior of PCL, with the PCL-EA film showing slightly improved optical properties.

The obtained results demonstrate that ethyl acetate enables the formation of PCL films with more favorable surface and optical characteristics while maintaining the properties of this biodegradable polymer. Due to their biodegradability and the possibility of tailoring their properties by selecting an appropriate solvent, PCL thin films represent promising materials for environmentally sustainable applications, including sustainable packaging, protective coatings, and other approaches aimed at reducing the environmental impact of polymer waste.

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CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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