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论文题目： The Sweet Solution: Enhancing the Biodegradable Plastics by Shaping Sugar Ring into Eco-friendly Plastics

The Sweet Solution: Enhancing the Biodegradable Plastics by Shaping Sugar Ring into Eco-friendly Plastics

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Abstract:

This study aims to study the possibility of enhancing the mechanical properties and biodegradability of polylactic acid (PLA) using cyclodextrins (CD). Through melt-blending and solvent casting methods, PLA/CD composites with varying content of CD (0%, 1%, 3%, and 5%) were produced. Mechanical properties and biodegradation are assessed by precise instruments and compared with traditional PLA materials without CD.

The research reveals that CD concentration not only enhances the mechanical properties of the samples but also maintains their biodegradability at a constant rate. The 3% CD composition shows an optimal balance of strength, ductility, and toughness. Further increasing the CD content to 5% leads to an even higher tensile strength of 56.4 MPa.

It can be concluded from biodegradation studies that a relatively low amount of CD added into the composite makes it break down faster. It is notable that the samples all change a lot from what they initially appeared around day 30, indicating that their internal structure has undergone significant changes. Overall, CD content is crucial for the speeding up of breakdown especially in the initial stages.

The result emphasizes the significant impact of CD incorporation on PLA properties. It presents key information for adjusting materials based on PLA to achieve desired mechanical characteristics and biodegradability, creating opportunities for innovative use in both daily life and industry.

Keywords: Bioplastics, Cyclodextrin, Mechanical properties and biodegradation.

Content

1. Introduction

The rapid development of the global economy has led to increased attention to green packaging solutions that address carbon footprint reduction, efficient use of natural resources, life cycle assessment, and recyclability. These environmentally conscious approaches have gained widespread endorsement and are creating significant social impact worldwide.[1,2] Currently, approximately 400 million plastic packaging units are produced annually, with the majority made from non-recyclable and non-biodegradable materials such as polyethylene, polypropylene, and polyethylene terephthalate.[3] The overproduction and growing demand for these environmentally unfriendly materials are creating substantial environmental problems and societal pressure.[4]

To address these challenges, polylactic acid (PLA) has emerged as a promising alternative in the packaging and catering industries. PLA is a biodegradable and bioactive thermoplastic aliphatic polyester derived from renewable resources such as corn starch, sugarcane, and tapioca roots.[5] As an environmentally friendly alternative to traditional petroleum-based plastics, PLA has gained significant attention across various industries due to its sustainable production process and versatile properties(Figure 1).[6]
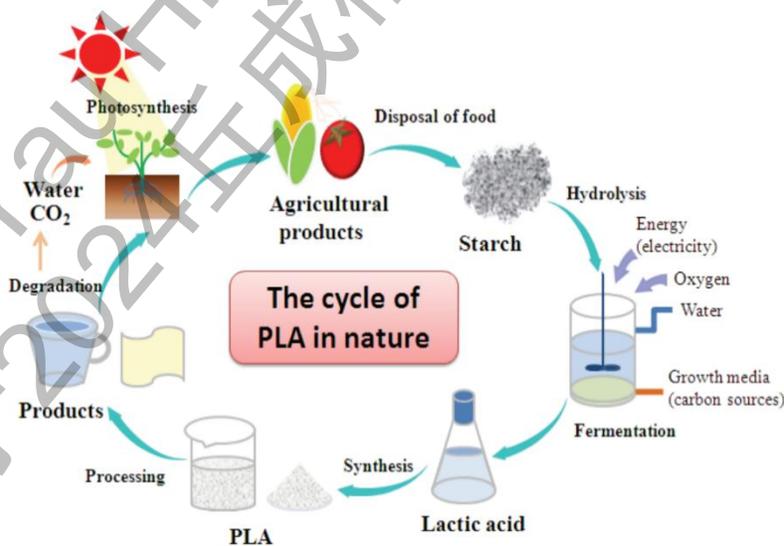

Figure 1. The cycle of PLA in nature

The environmental impact of PLA compared to traditional plastics is striking. While conventional plastics can persist in the environment for hundreds of years, accumulating in landfills and oceans, PLA can decompose within months under appropriate conditions.[7-9] However, the rapid degradability of PLA is often accompanied by inferior mechanical properties, which significantly limit its broader application. This limitation necessitates the development of new materials that combine high mechanical properties with high degradability.

Current methods to improve PLA's mechanical properties and biodegradability include blending with other polymers, incorporating nanomaterials, and adding plasticizers.[10] Each approach has its advantages and drawbacks: Polymer blending (e.g., with PBAT or PCL) can enhance flexibility and toughness but may compromise biodegradability. Nanomaterial incorporation (e.g., nano clay, graphene, cellulose nanocrystals) can improve mechanical strength and thermal stability but is often costly and poses potential environmental risks due to nanoparticle persistence and toxicity. Plasticizer addition (e.g., PEG, citrate esters) can improve flexibility but may lead to additive migration and property degradation over time.[11,12]

Given these limitations, our research focuses on using cyclodextrins (CDs) as an organic additive to enhance PLA's mechanical properties while maintaining its biodegradability. CDs are cyclic oligosaccharides derived from starch through enzymatic conversion. They are characterized by a cylindrical structure composed of glucose monomers linked by α-(1,4)-glycosidic bonds. The most common forms - α-CD, β-CD, and γ-CD - comprise 6, 7, and 8 glucose units, respectively (Figure 2). This unique structure results in a hydrophobic central cavity and a hydrophilic outer surface, enabling CDs to form inclusion complexes with various guest molecules.[15] Previous studies have demonstrated the potential of CDs in improving PLA properties. Researchers from Clemson University and the University of Illinois have shown that PLA-β-cyclodextrin inclusion complexes can enhance thermal stability.[13] Furthermore, a 2022 study from the University of Navarra demonstrated the practicality of β-cyclodextrin-TiO$_2$ and PLA bionanocomposites for food packaging

applications.[14] However, there is limited research on how CDs affect the mechanical properties and biodegradation behavior of PLA, which is the focus of our study.
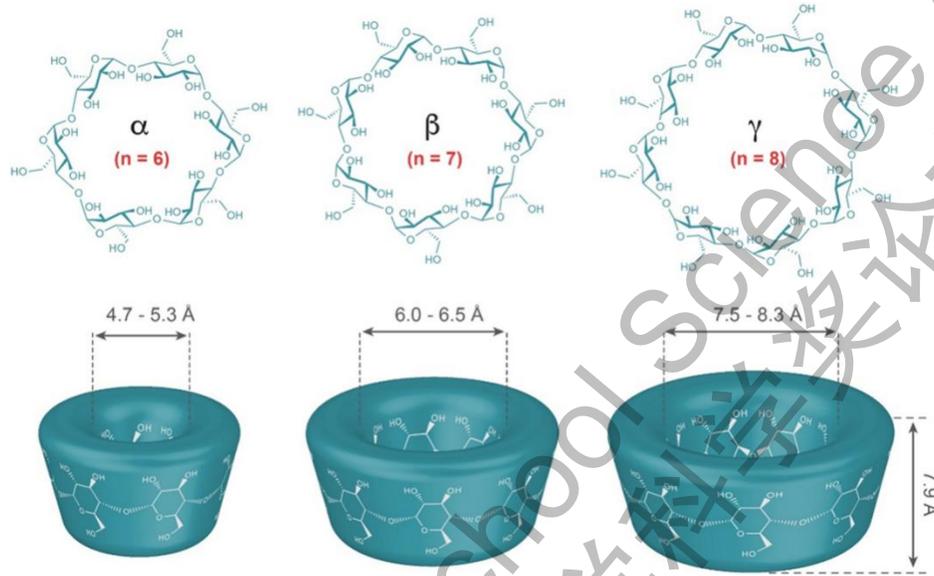

Figure 2. The three main cyclodextrins, namely α-cyclodextrin, β-cyclodextrin, and γ-cyclodextrin, contain six, seven, and eight glucose units in their structure, respectively, and their dimensions

Incorporating CDs into PLA offers several potential benefits: Enhanced mechanical properties through the formation of inclusion complexes that strengthen the material matrix. Improved biodegradability due to the cyclic structure facilitating enzymatic degradation. Compatibility with food packaging applications, as CDs are edible and composed of polysaccharides.

In this study, we prepare PLA/CD composites by melt-blending and solvent casting methods and investigate the effects of CD incorporation on PLA's mechanical properties and biodegradability. We employ various analytical techniques, including basic structural characterization, mechanical testing, and biodegradation assays, to elucidate the mechanisms by which CDs influence PLA's properties. Our goal is to develop PLA/CD composites suitable for advanced applications in food packaging and other fields requiring enhanced mechanical performance and biodegradability.
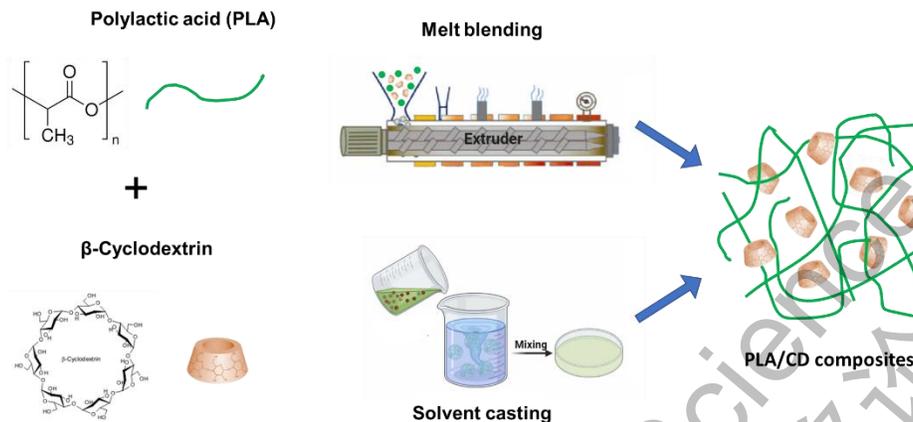
Figure 3. Schematics represent the preparation methods and structure of PLA/CD composites.

2. Methods

2.1 Materials

Chloroform (HCCl3, 99% purity, Shanghai Aladdin Biochemical Technology Co., Ltd. (China)), Cyclodextrin (β-CD, 99.9% purity, Shanghai Aladdin Biochemical Technology Co., Ltd. (China)), and PLA (Poly DL-lactic, average Mn 20,000, D/L ratio 50:50, Fengyuan Futailai (Anhui) company) were used as received.

2.2 Preparation of PLA/CD composites

Poly(lactic acid) (PLA) (2.5 g) was dissolved in 500ml chloroform ($CHCl_3$) under magnetic stirring at room temperature to obtain a PLA solution. Suitable amounts of β-CD powders were added to achieve PLA/CD solutions with concentrations of 1 wt%, 3wt%, and 5wt%. The mixtures were stirred for 5 hours at room temperature to ensure homogeneity. The resulting solutions were poured into petri dishes and left to evaporate the solvents at 120°C for 2 hours to form PLA/CD thin films. The films were further dried under vacuum at 40°C for 18 hours to remove any residual solvents.

2.3 Characterization

Fourier Transform Infrared Spectroscopy (FTIR) spectra were obtained using a Nicolet 6700 spectrometer with an accuracy of 4 cm^{-1}. Spectra were collected in the range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1}. Samples were prepared by mixing the composite material with potassium bromide (KBr) and pressing into pellets.

The morphology of the composites was initially examined using an XSP-3CA optical microscope to observe the dispersion of β-CD in the PLA matrix at magnifications of 100x, 200x, and 500x. Scanning Electron Microscopy (SEM) was used to further observe the surface morphology of PLA/CD composites using an SU8010 (Hitachi LTD., Japan). Samples were coated with a thin layer of gold (Au) using a sputter coater before imaging, and images were captured at an accelerating voltage of 10 kV.

Crystallinity and phase composition of the composites were determined using a Bruker D8 X-ray diffractometer at room temperature using Cu Kα radiation. Samples were scanned at a rate of 3°/min over the range of 5° to 60° 2θ.

Thermal stability and decomposition behavior of the composites were analyzed using a TGA2-8860 (NETZSCH, Germany). Samples (5-10 mg) were heated from room temperature to 600°C at a rate of 20°C/min under a nitrogen atmosphere.

Tensile properties were measured using a universal testing machine (Instron E44). Specimens were cut into rectangular shapes with a length of 20 mm, width of 10 mm, and thickness of 0.2mm. The tension speed was 5 mm/min.

Biodegradation behavior.

3. Results and Discussion

3.1 Morphological Analysis

The optical microscopy (OM) images reveal distinct morphological differences in PLA/CD composites prepared by melt blending and solvent casting methods. At 0 wt% CD, both

methods produce clean and uniform surfaces. With 1 wt% CD, small particles appear, more uniformly distributed in the solvent casting method compared to melt blending. At 3 wt%, both methods show increased particle density, but the solvent casting method results in a more even distribution with less aggregation. At 5 wt%, significant aggregation occurs in the melt blending method; however, the solvent casting method yields smaller, more uniformly distributed aggregates.

Overall, the solvent casting method demonstrates a superior ability to uniformly disperse CD within the PLA matrix, particularly at lower concentrations, and produces smaller aggregates at higher concentrations compared to melt blending. These findings suggest that solvent casting is a more effective method for achieving a homogeneous dispersion of CD in PLA, which is crucial for optimizing the material properties of the composites.
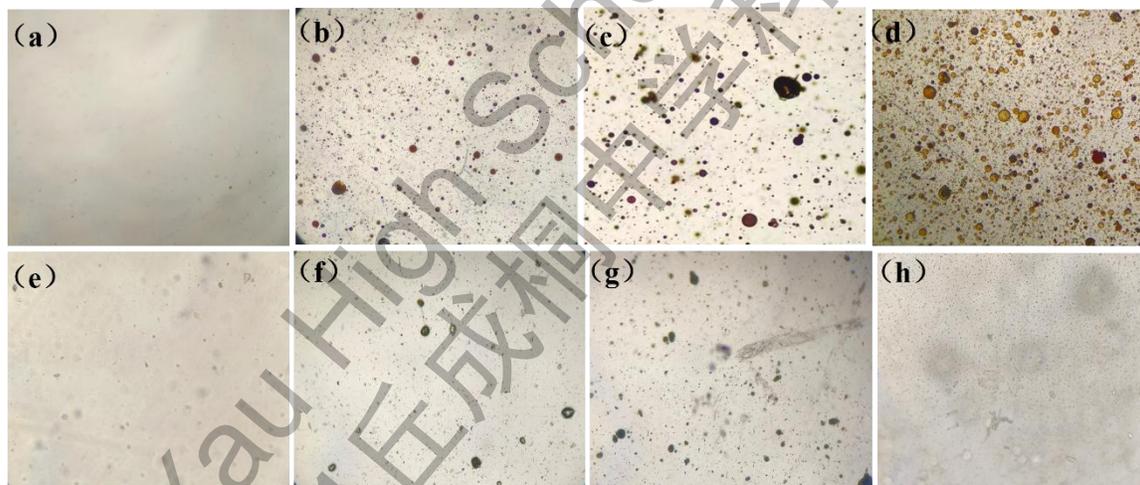

Figure 4. OM images of PLA/CD prepared by melt-blending with different concentration. 0,1,3,5wt% CD respectively(a-d); OM images of PLA/CD prepared by solvent casting with different concentrations. 0,1,3,5wt% CD respectively(e-h)

3.2 Chemical Structure and Crystallinity

The FTIR spectra of pure PLA and PLA containing different concentrations of cyclodextrin (CD) verify the chemical structure and purity of the materials. For pure PLA, key peaks

include the C-H stretching vibration at approximately 2995 cm^{-1}, the C=O stretching vibration near 1748 cm^{-1} CH3 bending vibrations around 1455 cm^{-1} and 1360 cm^{-1}, and C-O-C stretching vibrations at 1180 cm^{-1} and 1128 cm^{-1}. In contrast, the FTIR spectrum of pure CD shows a broad O-H stretching band around 3400-3200 cm^{-1}, indicative of hydroxyl groups, and C-H stretching vibrations near 2930 cm^{-1}. Peaks around 1155 cm-1 and 1025 cm^{-1} correspond to C-O-C and C-O stretching vibrations, respectively, characteristic of the glucopyranose units in CD. The results show that the characteristic peaks of PLA remain largely unchanged with the addition of CD, suggesting that the low loading of CD may be the reason for minimal interaction and the preservation of PLA's chemical structure. Discussion: CD dispersion, intensity.
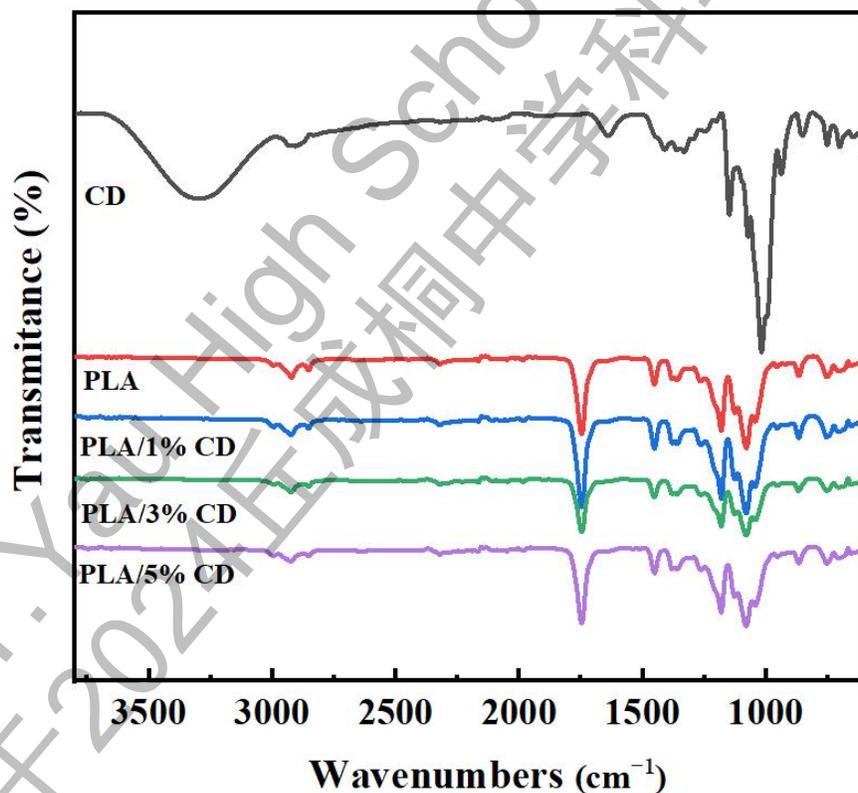

Figure 5. FTIR spectra of PLA/CD

The X-ray diffraction (XRD) profiles of pure PLA, pure CD, and PLA containing different concentrations of CD confirm the chemical structure and purity of the materials. The XRD pattern of pure CD exhibits numerous sharp and intense peaks, indicative of its highly crystalline nature. In contrast, the XRD pattern of pure PLA shows a broad diffraction peak around 16° to 19° 2θ, suggesting that PLA is primarily amorphous with some degree of crystallinity. For the PLA/CD composites (1% CD, 3% CD, and 5% CD), the patterns show a similar broad peak around 16° to 19° 2θ, characteristic of PLA's amorphous nature. The intensity of this peak remains consistent across all samples, indicating that the addition of CD does not significantly affect the crystallinity of PLA at these low concentrations. Additionally, the absence of sharp peaks corresponding to CD in the PLA/CD composite patterns suggests that CD is well-dispersed within the PLA matrix and does not form large crystalline domains.
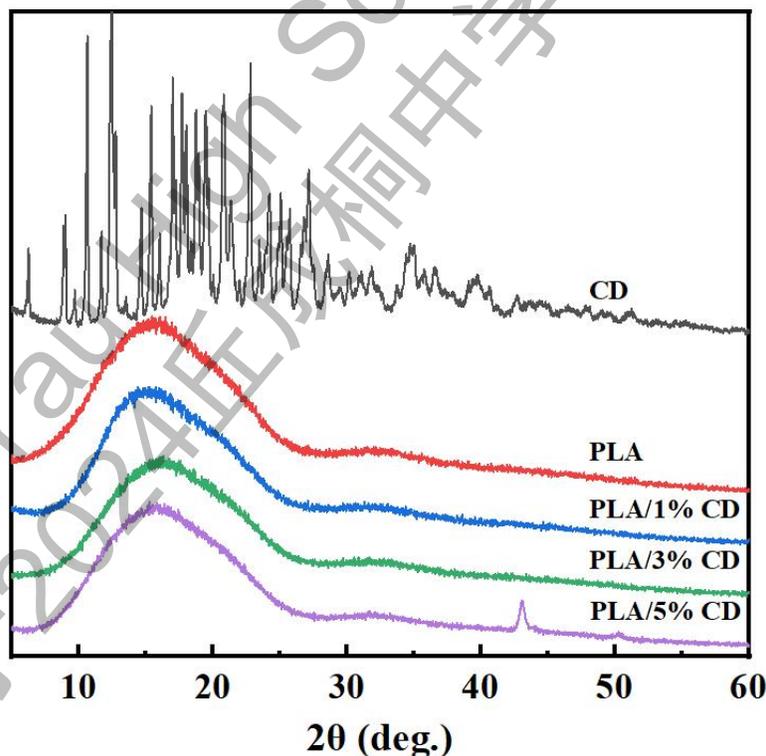

Figure 6. XRD profiles of PLA/CD

3.3 Mechanical properties

The stress-strain curves and mechanical property data for PLA and PLA/CD composites reveal significant changes in the material's behavior with the addition of CD. Pure PLA exhibits typical brittle behavior, with a relatively high tensile strength (45.8 MPa) but limited elongation at break (8.5%). The addition of CD modifies these properties in a concentration-dependent manner.

At 1% CD content, there is a slight decrease in tensile strength (42.0 MPa) and no significant change in elongation at break. However, as the CD content increases to 3%, we observe a marked improvement in mechanical properties. The PLA/3% CD composite shows an increase in tensile strength to 48.2 MPa, a significant enhancement in elongation at break to 10.6%, and a remarkable increase in toughness to 27.8 MJ/m³ - nearly double that of pure PLA. This suggests that at this concentration, CD acts as an effective reinforcing agent while also improving the material's ductility.

Further increasing the CD content to 5% leads to an even higher tensile strength of 56.4 MPa, representing a 23% increase compared to pure PLA. However, this comes at the cost of reduced elongation at break (6.3%) and a slight decrease in toughness compared to the 3% CD composite. This trend indicates that higher CD concentrations may lead to a more brittle material, despite the increased strength.

The stress-strain curves corroborate these findings, showing that the 3% CD composite maintains a good balance between strength and ductility, while the 5% CD composite exhibits higher strength but fails at lower strain. The Young's modulus data, although not provided for all compositions, would likely show an increasing trend with CD content, reflecting the material's increasing stiffness.

These results demonstrate that cyclodextrin can effectively modulate the mechanical properties of PLA, with the 3% CD composition offering an optimal balance of strength, ductility, and toughness. This enhancement can be attributed to several mechanisms. At lower concentrations (1% CD), the cyclodextrin particles may act as stress concentrators

within the PLA matrix. This could lead to localized debonding between the CD particles and the PLA, potentially promoting plastic deformation and increasing elongation. However, at 1% concentration, the amount of CD may be insufficient to fully trigger this toughening mechanism, explaining the lack of significant improvement in mechanical properties. As the CD content increases to 3%, we likely reach a threshold where there are enough CD particles to effectively initiate and propagate these toughening mechanisms throughout the material, resulting in the observed optimal balance of strength, ductility, and toughness. The further increase to 5% CD introduces a new dynamic. At this higher loading, CD particles may begin to form a more extensive network within the PLA matrix, constraining the movement of surrounding polymer chains. This network formation could explain the observed increase in tensile strength and apparent stiffness, as it enhances the material's resistance to deformation. However, this same network may also restrict the ability of the polymer chains to undergo extensive plastic deformation, leading to the decreased elongation at break seen in the 5% CD composite. This analysis underscores the complex interplay between CD concentration, stress distribution, and polymer chain mobility in determining the overall mechanical properties of the PLA/CD composites.

The ability of CD to simultaneously increase strength and ductility at certain concentrations is particularly noteworthy, as many traditional fillers tend to improve strength at the expense of ductility. This unique characteristic of CD as an additive for PLA opens up new possibilities for tailoring the mechanical properties of PLA-based materials for specific applications, ranging from packaging that requires both strength and flexibility to biomedical devices that need to withstand dynamic loads.

The mechanical properties of PLA/CD composites prepared by melt-blending reveal a distinct trend compared to those prepared by solvent casting, highlighting the crucial role of processing methods in determining material performance. Pure PLA exhibits a tensile strength of 67.7 MPa and an elongation at a break of 15.2%. With increasing CD content, we observe a gradual decrease in tensile strength, reaching 60.2 MPa at 5% CD. Interestingly, the elongation at break shows a non-monotonic trend, peaking at 16.75% for

1% CD before decreasing to 12.8% at 5% CD. Toughness follows a similar pattern, with the 1% CD composite showing the highest value (8.0 MJ/m³) before declining at higher CD concentrations. These results suggest that in melt-blended composites, CD particles may act as stress concentrators due to poor dispersion, leading to earlier failure at higher loadings. The improved performance at 1% CD indicates a delicate balance where minimal CD content can enhance ductility without significantly compromising strength, possibly due to localized plasticization effects. However, the overall decline in mechanical properties with increasing CD content contrasts sharply with the solvent-cast samples, underscoring the critical importance of processing techniques in achieving optimal dispersion and particle size.

In conclusion, the mechanical property data underscores the potential of cyclodextrin as a versatile additive for PLA. By carefully controlling the CD concentration, it is possible to engineer PLA composites with enhanced mechanical performance, potentially expanding the range of applications for this biodegradable polymer.
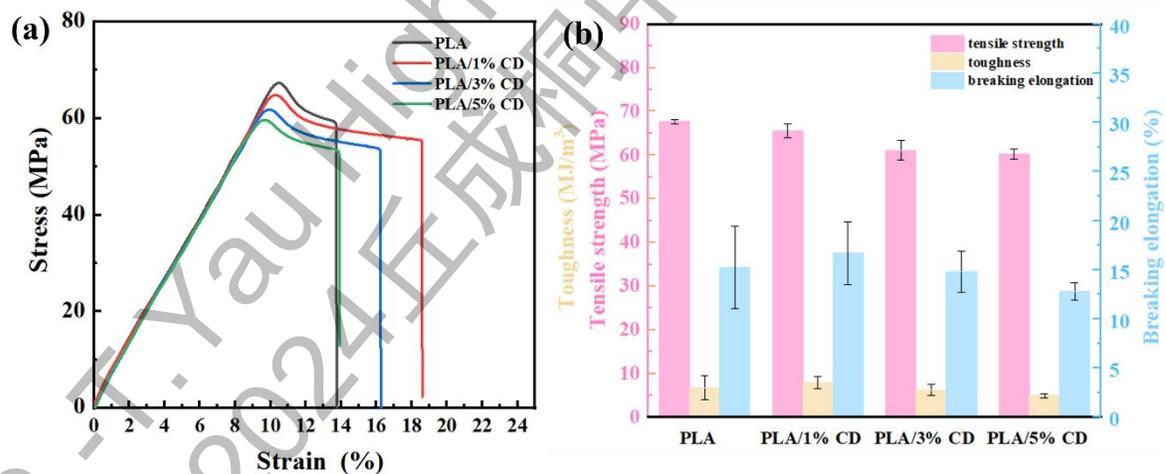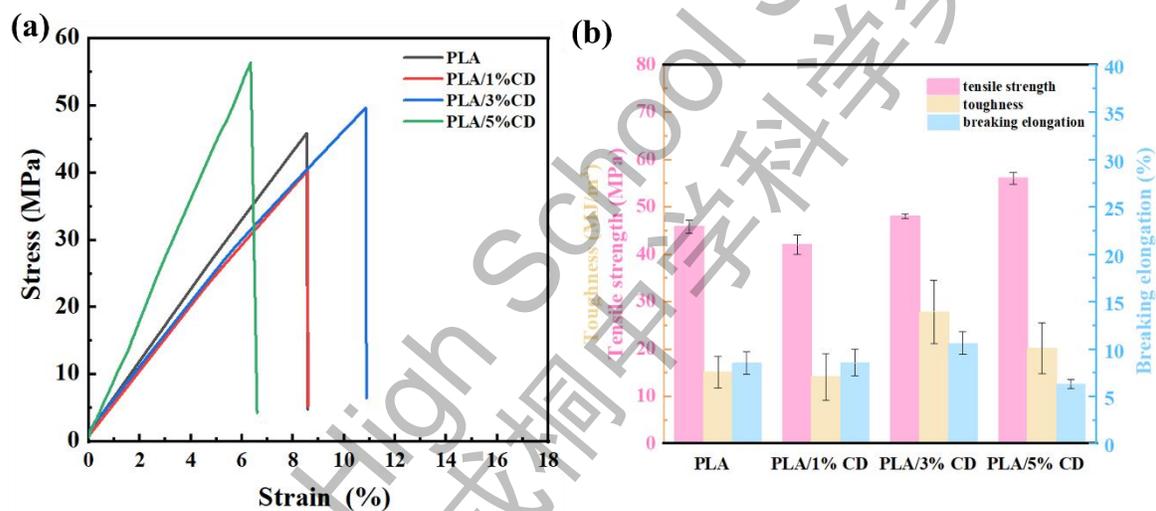

Figure 7. (a) Stress−strain curves of PLA and PLA/CD prepared by melt-blending method and (b) Tensile strength, elongation at break, toughness values with deviation bar

.Table 1. Tensile strength, elongation at break, and toughness of PLA and PLA/CD prepared by melt-blending method

Samples	Tensile strength (MPa)	Elongation at break (%)	Toughness (MJ/m3)
PLA	67.7±0.5	15.2±4.2	6.8±2.6
PLA/1% CD	65.5±.1.6	16.75±3.2	8.0±1.4
PLA/3% CD	61.2±2.3	14.8±2.1	6.2±1.3
PLA/5% CD	60.2±1.2	12.8±1.0	4.9±0.5

Figure 8. (a) Stress−strain curves of PLA and PLA/CD prepared by solvent casting method and (b) Tensile strength, elongation at break and toughness values

Table 2. Tensile strength, elongation at break, and toughness of PLA and PLA/CD prepared by solvent casting method

Samples	Tensile strength (MPa)	Elongation at break (%)	Toughness (MJ/m3)
PLA	45.8±1.3	8.5±1.2	15.1±3.3
PLA/1% CD	42.0±2.0	8.5±1.4	14.1±4.9
PLA/3% CD	48.2±0.5	10.6±1.2	27.8±6.7
PLA/5% CD	56.4±1.2	6.3±0.5	20.1±5.4

3.4. Biodegradation:

According to the naked eye observation, it is clear that the samples for each group on Day 30, in comparison with Day 0, have changed significantly.

On Day 10, observing from the image captured by the photograph, there are no significant differences among the 4 groups. All of them maintain their shapes uniformly. Compared with the samples from the first day, however, a slight wrinkle can be observed, which makes the materials translucence.

Following that, on Day 20, the difference became distinct. For pure PLA, the sample could not maintain its integrity and started to tatter and split out tiny debris. As for the PLA/CD, for 1wt% and 3wt%, the wrinkles on the surface increased, but although some tiny debris could be observed around the material, the integrity of the sample was still maintained. For the PLA/CD sample containing 5wt% CD, the sample only showed an increasing number and density of wrinkles on the surface.

On Day 30, the pure PLA had broken into several fragments; the situation was the same for PLA/CD with 1wt % and 3wt%. However, for PLA/CD 5wt%, the sample still remains in its original shape. Moreover, it can be observed that all the samples were covered by some red and brown colored matter. According to our speculation, the red and brown substances could probably be the product after the decomposing of the material.
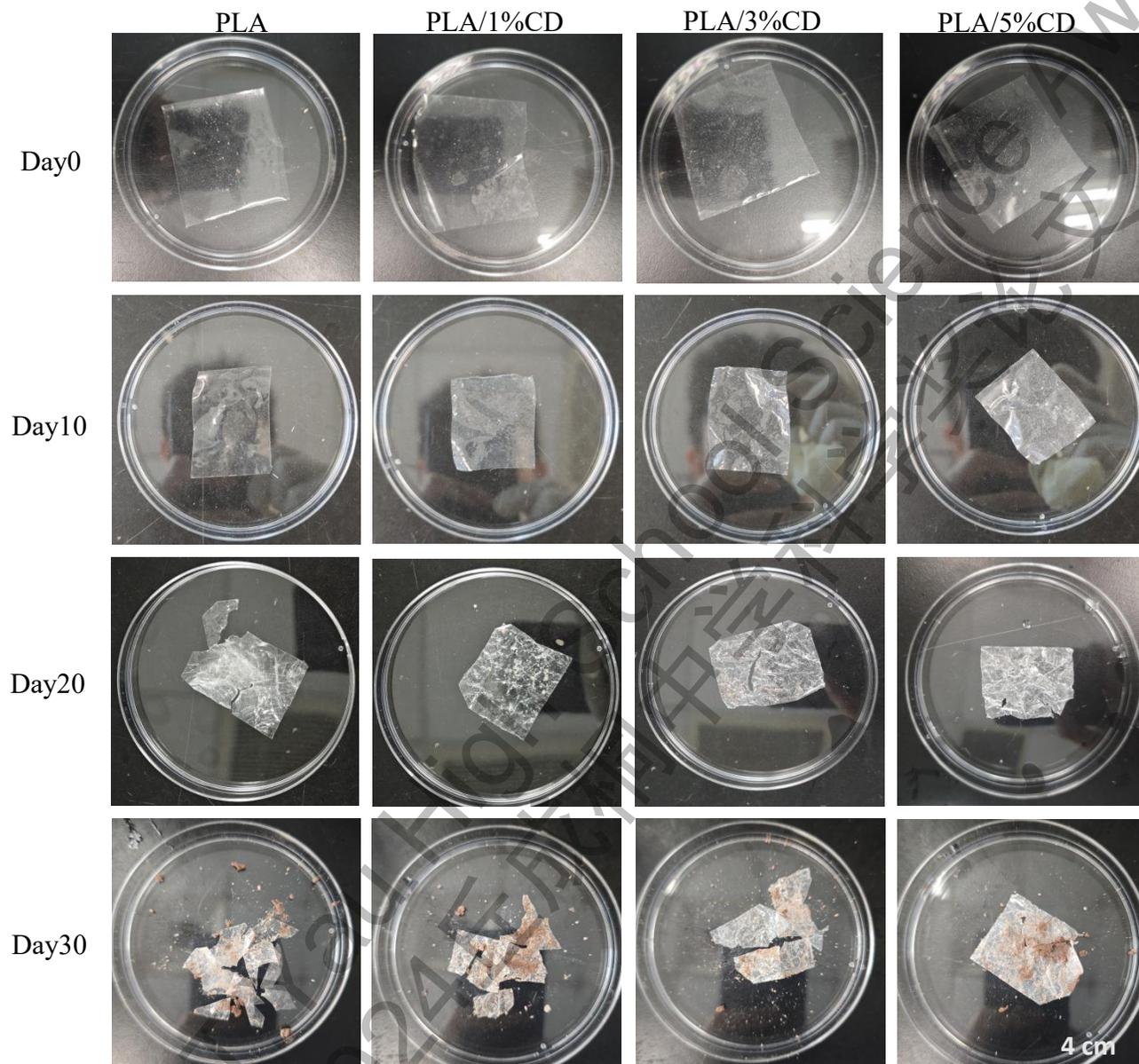
Figure 9. The change of PLA/CD materials (captured by camera)

Overall, from the observations, it seems that PLA contains more CDs and is less degradable compared with pure PLA. Interestingly, the PLA/CD composites appeared to become more opaque over time. To gain more detailed insights into these morphological changes, we employed OM. It's important to note that before OM analysis, all film samples were

sonicated in water for 10 minutes to remove any surface contaminants, ensuring clear and accurate observations of the degradation process.

The OM images show how PLA with different amounts of CD changes over 30 days. On Day 0, the pure PLA looks pretty clear. As we add more CD (1%, 3%, and 5%), we see more little dots in the material. These dots are probably the CD particles.

On day 10, there are no obvious and significant differences among each group. Observing the graph, we can observe that the black spots for each group increased a little bit, but it could also be caused by the reasons that different parts and regions of the material were observed.

By Day 10, things start to change a bit. The pure PLA has a few more spots than before. The samples with CD show even more dark spots, especially the ones with more CD. This might mean the material is starting to break down. More bacteria are attached to the surfaces of the plastics.

On Day 20, it can be clearly observed that the surface of pure PLA did not change a lot, except for the increasing number of black spots on the surface. The phenomenon was the same for the PLA/CD 1wt%. However, on the PLA/CD with 3wt%, a quantity of black dots and defects. As for the 5wt%, the quantity of dots increases remarkably, and the distribution of black dots becomes much denser than before.
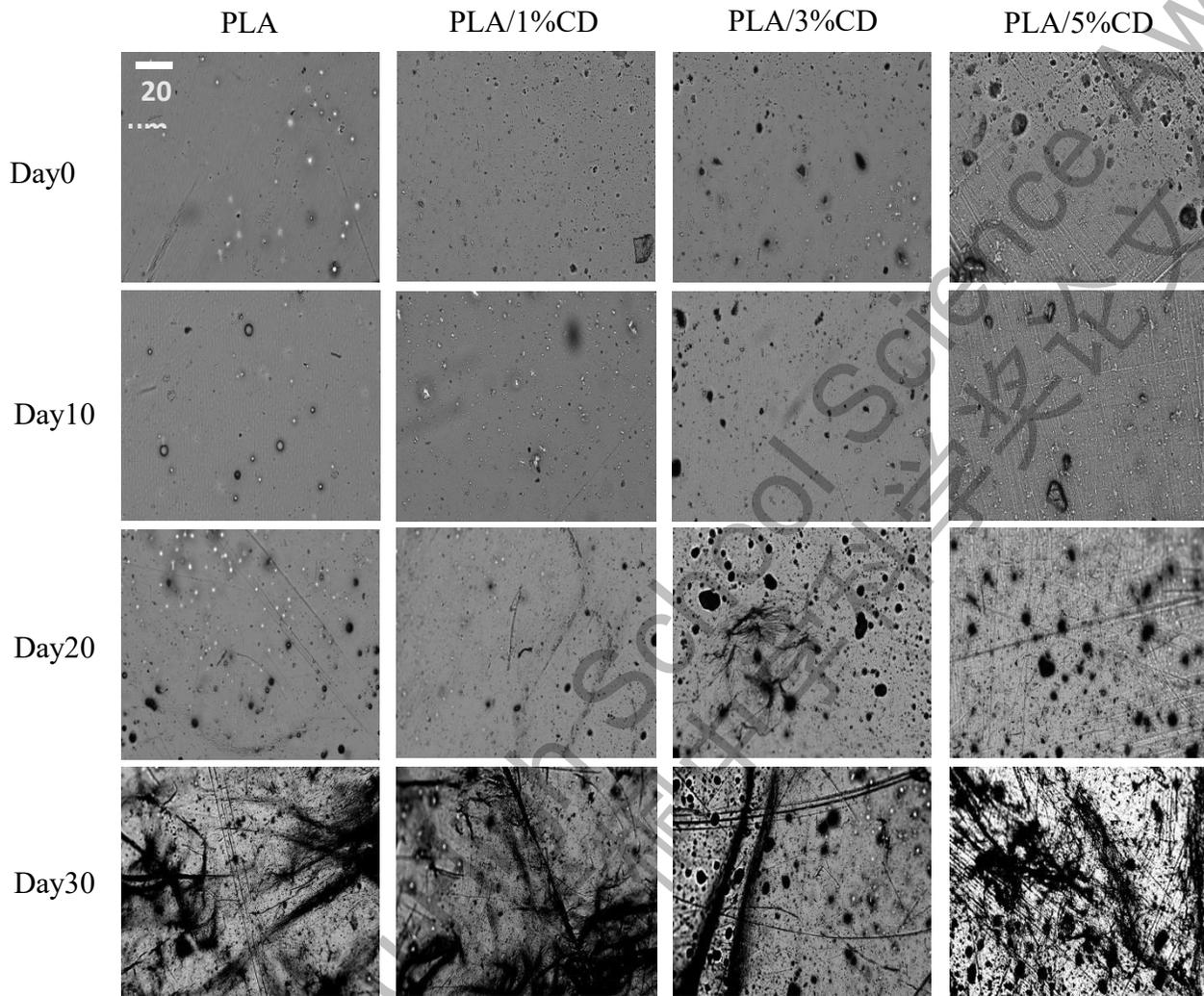
| | PLA | PLA/1%CD | PLA/3%CD | PLA/5%CD |

Figure 10. The change of PLA/CD materials (captured by OM)

On Day 30, almost every sample was covered with intensive dark and thread-like objects. From the images, we can figure out that all of the samples are covered with dark objects. We speculate that these dark objects are the opaque substances generated by bacteria when they are degrading and decomposing the samples.

Day 20 is when we really see big changes. All the samples have way more dark spots now. The ones with 3% and 5% CD look the most different, with lots of dark spots and weird shapes. It looks like having more CD makes the material break down faster at this point.

On Day 30, all the samples looked super different from how they started. They all have these dark lines all over, kind of like a spider web. This probably means the material has broken down a lot. Interestingly, the sample with 5% CD looks a bit lighter than the others. This could mean that a lot of CD might slow down the breakdown process after a while.

From what we see in these pictures, adding CD to PLA changes how it breaks down over time. A little bit of CD seems to make it break down faster. But when we add a lot of CD, it might speed up the breakdown at first but then slow it down later on.

This information could be really useful for making things like biodegradable plastic. By changing how much CD we add, we might be able to control how fast the plastic breaks down. This could be great for making packaging that needs to last for a certain amount of time before it starts to decompose.

Conclusion

This study demonstrates the significant impact of cyclodextrin incorporation on PLA properties. Optimal mechanical performance was achieved at 3% CD content, balancing increased strength and toughness. Biodegradation rates were generally accelerated by CD addition, with a potential stabilizing effect at higher concentrations. Structural and thermal analyses revealed good CD dispersion within the PLA matrix without compromising PLA's chemical structure or thermal stability. The processing method significantly influenced the final composite properties. These findings highlight the potential of CD as a versatile additive for tailoring PLA characteristics, opening avenues for developing high-performance, biodegradable materials with controlled properties. Future research should focus on optimizing PLA/CD composites for specific applications and scaling up production methods.

Acknowledgment

solve: we were not able to explain the erratic trend in the flexibility and toughness of PLA/CD composites based on the data we collected. Thanks to Dr. Liu we finally reached a consensus on the explanation that composites with different content of CD each formed distinct structures, resulting in an unexpected trend.

For the central part of the writing, we not only collaborated but also split the paper into small sections and divided our work. Specifically, for instance, Hongjie Shan focused on the analysis of biodegradation and chemical structure of the composites, while Yizeng Zheng concentrated on the characterization of thermal stability and mechanical properties.

All in all, we experienced moments of temporary depression and confusion throughout the writing process, but fortunately, we managed to overcome them. Thank the committee of competition for giving us the opportunity to present our work on a broader stage. We would like to extend our sincere gratitude once again to all those who contributed to the completion of this paper!

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