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Sun Min Ji & Tai Gyu Lee

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A Fast and Accurate UV-Vis Method for the Quantification of Polylactic Acid in Biodegradable Plastics

Sun Min Ji^a and Tai Gyu Lee^{a, b, *}

^a *Department of Chemical and Biomolecular Engineering, Yonsei University,
50 Yonsei-ro, Seodaemun-gu, Seoul 03722, Korea*

^b *Korean Association of Biodegradable Plastic Ecosystem,
25 Sinchon-ro, Seodaemun-gu, Seoul 03785, Korea*

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* To whom correspondence should be addressed

Tel.: 82-2-2123-5751

E-mail: teddy.lee@yonsei.ac.kr (Lee, T.G.)

Abstract

Polymer blends are used to achieve tailored properties, but the quantification of their components remains difficult. We developed an ultraviolet-visible spectrophotometric method with a two-step pretreatment that involves the conversion of polylactic acid (PLA) into methyl lactate via alcoholysis and then into sodium lactate through alkaline hydrolysis, followed by detection with ferric chloride at wavelengths from 400 to 410 nm. The method demonstrated linearity, with a correlation coefficient above 0.99. For PLA and polypropylene blends with proportions that ranged from 10 to 100 wt%, the bias was less than $\pm 4\%$, and the relative standard deviation was less than 7%. Conversion and identity were confirmed via Fourier transform infrared and proton nuclear magnetic resonance spectroscopies. Selectivity was validated in blends with polybutylene adipate-coterephthalate, polyhydroxybutyrate, and cellulose acetate, with absorbance deviations within ± 0.03 . This cost-effective method enables reliable quantification for biodegradable plastic certification.

Keyword: Biodegradable plastics, Polylactic acid, Quantification, UV-vis spectroscopy, Certification, Pollution prevention

Introduction

Global plastic production has been continuously increasing, leading to severe environmental issues associated with plastic waste accumulation [1,2]. The production of plastics increased from 2 million tons in 1950 to 460 million tons in 2019 and is projected to triple by 2060, reaching approximately 1.23 billion tons [3,4]. In response to this critical issue, the development and use of biodegradable plastics have attracted much attention as potential solutions. Biodegradable plastics can be decomposed by microorganisms under specific conditions, thereby reducing their negative effects on the environment [5,6,7,8]. Among biodegradable polymers, polylactic acid (PLA) is widely recognized for its eco-friendly characteristics [9]. It is produced from renewable biomass sources such as corn starch and other plant-based raw materials [10,11]. Compared with petroleum-based plastics, PLA significantly reduces greenhouse gas emissions and is highly biodegradable, achieving more than 90% degradation under composting conditions within 180 days [12,13,14,15].

Owing to these advantages, PLA has been adopted in various applications, including packaging, disposable products, and agricultural films. The PLA market accounts for 31% (217,000 tons) of the biodegradable plastics sector in 2023 and is expected to grow to 43.6% (676,000 tons) by 2028, with a compound annual growth rate (CAGR) from 2021 to 2030 of 15.9%, reaching an estimated market value of 4 billion USD by 2030 [16]. However, PLA is often blended with other polymers or additives to improve its mechanical properties and

processability, which may complicate its degradation behavior.

Therefore, accurate determination of the PLA content in final products is essential for quality control and the certification of biodegradability [17,18].

Currently, a range of analytical techniques are employed for PLA characterization, including Fourier transform infrared (FT-IR) spectroscopy, nuclear magnetic resonance (NMR), pyrolysis-gas chromatography/mass spectrometry (Py-GC/MS), and high-performance liquid chromatography (HPLC) [19,20,21,22]. FT-IR spectroscopy provides structural and compositional information on PLA by identifying characteristic functional groups [19]. The accurate quantification of monomeric units through peak integration of the proton NMR spectrum has also been reported as a reliable method for determining the lactide composition of PLA [20]. Py-GC/MS can be used to identify and quantify the thermal degradation products of PLA, offering high sensitivity even for small sample amounts [21]. HPLC is also practical for analyzing lactide purity or monomer content through hydrolysis-based kinetics [22].

Despite their utility, these methods have notable limitations. First, they require expensive instruments and skilled operation. Second, in multicomponent polymer systems, additives and polymer blends complicate the identification of PLA and the quantification of its content [23,24]. Third, conventional biodegradability tests such as ISO 14855 and ASTM D6400 demand extended durations (up to 180 days) and incur substantial costs [24,25]. Given that these standards do not quantify the

PLA content, rapid prescreening measurements are warranted.

Consequently, these approaches are often time-consuming, technically complex, and cost intensive [23,26].

To address these challenges, rapid, simple, and cost-effective methods that can accurately quantify the PLA content even in complex mixtures are needed. Ultraviolet-visible (UV-vis) spectroscopy can be used to determine the concentration of an analyte by measuring light absorption within the UV-vis range, which offers the advantages of a short analysis time, operational simplicity, and relatively low equipment cost [27]. In this study, to quantify the PLA content in complex mixtures, we propose a UV-vis spectrophotometric approach. We develop and validate a calibration-based method via UV-vis spectroscopy for the precise quantification of the PLA content in polymer blends. This approach involves a two-step pretreatment encompassing alcoholysis and alkaline hydrolysis, followed by colorimetric detection with ferric chloride at 400–410 nm. FT-IR, ^1H NMR, and UV-vis spectroscopy were employed to confirm the compositional identity and structural conversion. The developed technique demonstrated high sensitivity, notable linearity ($R^2 > 0.99$), and high precision, with bias within $\pm 4\%$ and relative standard deviations below 7%. These findings highlight its practical utility for biodegradable plastic certification and quality control.

Results and discussion

Characterization of the polymer blend standards. To determine whether UV-vis spectroscopy can be used to accurately quantify PLA in

polymer blends, we first characterized the chemical interactions and compatibility of representative blend standards via FT-IR spectroscopy. PLA was blended with polypropylene (PP), poly(butylene adipate-coterephthalate) (PBAT), polyhydroxybutyrate (PHB), and cellulose acetate (CA), and each blend was analyzed to identify characteristic vibrational modes and potential intermolecular interactions (Fig. 1). All the blend spectra for FT-IR fingerprinting were recorded at a fixed ratio of 50:50 (w/w) for PLA/PBAT, PLA/PHB, and PLA/CA.

In the PLA/PP blend, characteristic absorption bands were observed at $2950\text{--}2838\text{ cm}^{-1}$, $1455\text{--}1453\text{ cm}^{-1}$, and 1376 cm^{-1} , corresponding to the C-H stretching and CH_3 and C-H bending vibrations of PP. Similarly, prominent bands corresponding to PLA appeared at 1749 cm^{-1} (C=O stretching) and at 1181 and 1080 cm^{-1} (C-O-C stretching and asymmetric CH_3), confirming the coexistence of both components without notable chemical interactions [28,29].

For the PLA/PBAT blend, overlapping carbonyl stretching bands were detected in the $1720\text{--}1750\text{ cm}^{-1}$ region, indicating limited miscibility between PLA and PBAT. Furthermore, distinctive aromatic vibration bands in the $1000\text{--}1210\text{ cm}^{-1}$ region, characteristic of the benzene ring structure in PBAT, were clearly observed [30,31].

Compared with that of pure PLA, the crystallinity of the PLA/PHB blend was notably different, reflecting the influence of PHB crystallinity. Upon the addition of PHB, new or intensified bands emerged at 1722 cm^{-1} (C=O stretch), 1278 cm^{-1} (C-O-C stretch), and 1382 cm^{-1} (CH_3 symmetric deformation), indicating increased crystallinity and potential

transesterification interactions [32].

In the PLA/CA blend, an increase in the broad O-H stretching band in the 3200–3700 cm^{-1} region was observed, indicating strong hydrogen bonding between the hydroxyl groups of CA and the ester groups of PLA. Notably, the emergence of a new ester carbonyl band at 1739 cm^{-1} was attributed to esterification between the anhydride groups of grafted PLA and the hydroxyl groups of CA [33].

Overall, these FT-IR analysis results confirmed the structural characteristics and interactions within each blend system, validating their use as standard references for subsequent quantification via UV–vis spectroscopy.

From alcoholysis to sodium lactate: Optimization and ^1H NMR confirmation of conversion. To perform FT-IR-based characterization of polymer blends and ensure accurate quantification of PLA, we next focused on optimizing the alcoholysis-based pretreatment. This pretreatment was introduced as part of the overall sample preparation process, aiming to selectively degrade PLA into a quantifiable product. To identify the optimal reaction conditions, various parameters, such as temperature and time, were screened on the basis of visual dissolution behavior and reaction completeness. A temperature of 70 °C and a reaction time of 3 hours were selected as optimal conditions, as they consistently resulted in rapid and complete PLA dissolution under stirring. The degradation products were analyzed via NMR (^1H NMR) spectroscopy (Fig. 2a). The spectrum exhibited three distinct signals: a

doublet at approximately δ 1.30 ppm ($J \approx 7$ Hz), which corresponded to methyl protons ($-\text{CH}_3$), and a singlet at δ 3.70–3.75 ppm, which corresponded to the methoxy group ($-\text{OCH}_3$) [34,35]. These chemical shifts and splitting patterns greatly agreed with the reported ^1H NMR data for methyl lactate in CDCl_3 , confirming that the primary product of the alcoholysis reaction is methyl lactate. Building upon this foundation, to confirm the complete conversion of the methyl lactate generated during alcoholysis into sodium lactate, ^1H NMR analysis of hydrolyzed PLA/PP blend samples was conducted. The reference ^1H NMR spectrum of pure sodium lactate exhibits a characteristic methyl doublet at $\delta \approx 1.33$ ppm (Fig. 2b). The enlarged inset shows representative ^1H NMR spectra of hydrolyzed PLA/PP blends with varying PLA contents (10–100 wt%) [36]. The methyl doublet region ($\delta \approx 1.32$ – 1.36 ppm) is highlighted and shows a stepwise increase in the signal intensity with increasing PLA content. This systematic increase in the methyl proton peak area indicates a direct quantitative relationship between the amount of sodium lactate formed and the initial PLA content in the blend. These results support the feasibility of applying this method to quantitatively determine the PLA content on the basis of ^1H NMR analysis of hydrolysis products. Overall, these results demonstrate that during the optimized alcoholysis pretreatment, PLA is effectively and selectively converted into methyl lactate, whereas the subsequent hydrolysis step enables the quantitative determination of PLA on the basis of ^1H NMR analysis of the hydrolysis products, thus validating its suitability for downstream quantitative UV-vis analysis.

Optimization of UV–vis colorimetric detection. To evaluate the applicability of the developed method for quantifying the PLA content in polymer blends, we next conducted UV–vis colorimetric detection experiments. To obtain a yellow–brown Fe^{3+} –carboxylate complex, sodium lactate was reacted (from alcoholysis and hydrolysis) with 0.2% (w/v) $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. The resulting complex exhibited an absorbance proportional to the PLA content (Fig. 3a). The UV–vis spectra of the PLA/PP blends with varying PLA contents each showed a distinct peak from 400–410 nm [37]. For quantification, we used the absorbance at λ_{max} within this window, which is denoted as $\lambda_{\text{max}}(400\text{--}410 \text{ nm})$, for each spectrum. This absorbance response results from Fe^{3+} –carboxylate complexation between ferric ions and the –COO^- groups of sodium lactate, which is consistent with known iron(III)–organic acid mechanisms [38]. The absorbance at $\lambda_{\text{max}}(400\text{--}410 \text{ nm})$ linearly increases with the PLA mass fraction, which demonstrates the suitability of the method for quantitative analysis (Fig. 3b). The calibration curve was fitted as follows (Equation (1)):

$$\text{Absorbance} = 0.859 \times x + 0.005 \quad (R^2 = 0.9978) \quad (1)$$

These results demonstrate that the iron(III) complex formed with sodium lactate is highly sensitive to UV–vis light. The steep calibration slope (0.859) and excellent coefficient of determination ($R^2 = 0.9978$)

confirm the accuracy and reliability of this quantification method.

Calibration curve performance and verification. To validate the accuracy and generalizability of the developed PLA quantification method, we determined whether the calibration curve established with PLA/PP blends could be applied to other biodegradable polymer systems containing PHB, PBAT, and CA. We prepared PLA blends with PHB, PBAT, and CA and then applied the same pretreatment and UV-vis analysis procedures. Validation covered PLA/PBAT (60–80 wt%), PLA/PHB (25–75 wt%), and PLA/CA (70–90 wt%).

The absorbance values of these blends were compared with the PLA/PP calibration curve ($y = 0.859x + 0.005$) (Fig. 4). All the validation points closely aligned with the calibration line, with absorbance deviations less than ± 0.03 , demonstrating high quantitative reliability.

To further evaluate method selectivity and robustness against common carboxylic-acid degradation products, tests comparing UV-vis spectra and spike recovery were performed for terephthalic acid (TPA), sodium 3-hydroxybutyrate (3-HB), and succinic acid (SA) (Supplementary Fig. S5 and S6). These compounds showed minimal absorbance in the 400–410 nm window, and the PLA recoveries remained within 98–102% across spike levels up to 2.0 g L^{-1} , indicating negligible interference under the tested conditions.

Commercial PLA-based materials often contain additives plasticizers, fillers, and stabilizers that may carry over and alter the absorbance at λ_{max} (400–410 nm). When such matrix effects are anticipated, they can be

mitigated by simple sample clarification (e.g., centrifugation and/or filtration to remove suspended particulates prior to the colorimetric reaction) and a matrix-appropriate calibration strategy.

These results confirm that the developed method is not only effective for PLA/PP blends but also broadly applicable to other biodegradable polymer matrices, supporting its versatility and analytical accuracy.

Limit of detection (LOD) and limit of quantification (LOQ). To assess the sensitivity of our UV-vis colorimetric method for PLA quantification, we calculated the LODs and LOQs according to the ICH guidelines. On the basis of these guidelines, the LOD and LOQ were determined using the standard deviation of the response (σ) and the slope of the calibration curve (S) as follows (Equation (2)) [39]:

$$LOD = \frac{3.3 \times \sigma}{S}, \quad LOQ = \frac{10 \times \sigma}{S} \quad (2)$$

As shown in Fig. 3b, the value of S was 0.859, and the value of σ , which was determined from ten replicate measurements of the 10 wt% PLA sample, was 0.0064. Substituting these values yielded an LOD of 2.5 wt% and an LOQ of 7.5 wt%. These results demonstrate that PLA can be reliably detected above 2.5 wt% and quantified above 7.5 wt%, confirming the sensitivity and reliability of the method.

Compared with conventional UV-vis quantification methods, which typically require ≥ 5 –10 wt% PLA for reliable detection, the developed method offers greater sensitivity and lower detection thresholds,

enabling more accurate analysis in trace-level applications.

One limitation of this study is that the LOD/LOQ values were derived from controlled PLA/PP blends. Potential interference from common carboxylic-acid degradation products (TPA, 3-HB, and SA) was evaluated through comparative spectra and spike-recovery experiments, the results of which showed negligible impact from these degradation products on the absorbance at 400–410 nm (Supplementary Fig. S5 and S6).

However, the influence of more complex real-world matrices (e.g., highly weathered plastics and additive-rich commercial packing materials or environmental samples) should be systematically assessed in future work.

Conclusions. In this study, we developed a two-step pretreatment and UV–vis colorimetric method for rapid, accurate quantification of PLA in polymer blends. PLA was first converted into methyl lactate via alcoholysis and then hydrolyzed to sodium lactate, forming a yellow–brown Fe^{3+} –carboxylate complex with a distinct absorbance at 400–410 nm. We observed a strong linear correlation between the PLA content and absorbance ($R^2 = 0.9978$), with an LOD of 2.5 wt% and an LOQ of 7.5 wt%.

The method showed high selectivity toward PLA in blends with PBAT, PHB, and CA. Compared with conventional techniques, this approach requires minimal instrumentation, a shorter analysis time, and lower reagent costs.

These findings indicate the suitability of this method for routine PLA

certification and wider applications requiring rapid, cost-effective quantification of biodegradable plastics. This approach can enable more accessible, widespread monitoring of PLA materials, thus contributing to plastic pollution mitigation and environmental sustainability. Therefore, extending the workflow to additional aliphatic and aromatic polyesters (e.g., PET, PBT, PBS, and PCL) via tailored pretreatments and complexation chemistries would increase applicability across diverse material streams.

Materials and methods

Materials. PLA (4032D) was supplied by NatureWorks LLC. PP and CA were obtained from Sigma-Aldrich Co., Ltd., and DAEJUNG Chemicals & Metals Co., Ltd., respectively. PBAT (Ecoflex[®] C1200) was supplied by BASF SE. PHB was obtained from BOC Sciences. 4-Dimethylaminopyridine (DMAP, catalyst) was purchased from Tokyo Chemical Industry Co., Ltd. FeCl₃·6H₂O and dichloromethane (swelling co-solvent) were obtained from Wako Pure Chemical Industries, Ltd. Sodium hydroxide, acetic acid, and chloroform were supplied by DAEJUNG Chemicals & Metals Co., Ltd. Methanol (alcohol reagent) was obtained from J.T. Baker, and xylene was obtained from SAMCHUN Pure Chemical Co., Ltd.

Statistical analysis. All the statistical analyses were performed via Microsoft Excel. Calibration curve linearity was evaluated by calculating the coefficient of determination (R^2) on the basis of four replicates per

PLA concentration. Precision was assessed via the relative standard deviation (RSD), which was calculated as (standard deviation/mean) \times 100, from three replicate measurements per sample. The accuracy was evaluated by calculating the bias between the measured and theoretical PLA contents. Confidence intervals (CIs) were obtained at the 99% confidence level via the standard error of the mean. All the precision and accuracy assessments were performed on independent blends that were not used for calibration.

PLA blend preparation. PLA-based blends were prepared via a solvent casting method. We dissolved PLA in chloroform (4% w/v) at 300 rpm for 4 h at room temperature (23 ± 2 °C). PP was dissolved in xylene (4% w/v) at 300 rpm for 4 h at 140 °C. After the PP solution cooled to room temperature, it was combined with the PLA solution, and the mixture was homogenized (Mark II, Model 2.5; Tokushu Kika Kogyo Co., Ltd., Japan) at 4000 rpm for 10 min. The cast films were then dried at room temperature for 48 h to yield PLA/PP blend samples.

For the validation blends (PLA/PBAT, PLA/PHB, and PLA/CA), PLA, PBAT, and PHB were each dissolved in chloroform (10% w/v), whereas CA was dissolved in acetone (10% w/v); all the solutions were stirred at 300 rpm for 4 h at room temperature. After mixing the solutions at the specified compositions, the blends were homogenized using the same homogenizer at 4000 rpm for 10 min and dried at room temperature for 48 h to prepare the final test samples.

Two blend sets were prepared to support characterization and

validation. For FT-IR fingerprinting and band assignment, PLA/PBAT, PLA/PHB, and PLA/CA were prepared at a fixed ratio of 50:50 (w/w). For UV-vis calibration, the PLA/PP standards spanned 10–100 wt% PLA in 10 wt% increments. Cross-matrix validation was conducted using blends with PLA at 60, 70, and 80 wt% in PLA/PBAT; at 25, 50, and 75 wt% in PLA/PHB; and at 70, 80, and 90 wt% in PLA/CA. UV-vis quantification was performed using the absorbance at λ max(400–410 nm).

Sample preparation: alcoholysis and hydrolysis. We performed alcoholysis on each 1 g blend sample by adding 0.1 g of DMAP (10 wt% relative to the sample mass) and methanol and dichloromethane at a ratio of 1:1 (v/v) to form a total volume of 100 mL. The mixture was refluxed at 70 °C and stirred at 700 rpm for 3 hr. The solvents were removed under vacuum at 30 °C and 100 rpm using a rotary evaporator (HAHNVAPOR HS-2005 V-N; HAHN SHIN Scientific, Korea). The two-step pretreatment pathway (alcoholysis → hydrolysis) is summarized in Supplementary Fig. S2. Next, an aqueous 1% (w/v) NaOH solution (1.00 g NaOH in 100 mL DI water) was added. For a 1.00 g sample, we added 100 mL of the NaOH solution and hydrolyzed the mixture under vacuum at 40 °C for 2 h. After hydrolysis, the mixture volume was less than 30 mL; the pH was adjusted to 4.0 with acetic acid, and then the mixture was diluted to a final volume of 150 mL with DI water.

Colorimetric reaction and UV-vis spectroscopy. $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (0.3 g) was dissolved in 100 mL distilled water to prepare a 0.2% (w/v) FeCl_3

solution. For color development, 75 μL of the final 150 mL of diluted sodium lactate solution was mixed with 3.0 mL of FeCl_3 solution. The absorbance was recorded from 385 to 550 nm via a PerkinElmer Lambda 35 UV-vis spectrophotometer, and we defined λ_{max} (400–410 nm) as the spectrum-specific maximum within the 400–410 nm window for each sample and used the corresponding absorbance at λ_{max} (400–410 nm) to construct the calibration curve and calculate the sample concentrations. Each sample was measured in quadruplicate, and the mean absorbance was used for subsequent calculations.

FT-IR spectroscopy. We recorded the FT-IR spectra of the PLA/PP blends and validation samples (PLA/PBAT, PLA/PHB, and PLA/CA) via a Bruker Alpha II FT-IR spectrometer (Bruker Co., USA). Spectra were collected from 400 to 4000 cm^{-1} with 32 scans per sample.

NMR spectroscopy. ^1H NMR spectroscopy was used to monitor the progress of alcoholysis and hydrolysis reactions and confirm the molecular structure of the resulting products. All the spectra were acquired via a Bruker Avance III HD 400 NMR spectrometer (Bruker Co., USA) operated at 400 MHz. CDCl_3 was used as the solvent for methyl lactate (after alcoholysis), and D_2O was used for sodium lactate (after hydrolysis).

Data availability

The data that support the findings of this study are available from the

corresponding author upon request.

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Author contributions

T.G.L. managed the whole project, designed all the experiments, and jointly wrote the manuscript. S.M.J. conducted the experiments, analyzed the data, and wrote the manuscript.

Competing interests

The authors declare that they have no competing interests.

Additional information

Correspondence and requests for materials should be addressed to T.G.L.

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Figure Legends

Fig. 1. Coexisting and interacting chemical signatures in the FT-IR spectra of PLA-based blends. Neat PLA, PP, PBAT, PHB, and CA reveal their characteristic bands—such as PLA carbonyl stretching ($\sim 1750\text{ cm}^{-1}$), PP methylene bending ($\sim 2950\text{ cm}^{-1}$), PBAT aromatic C-O vibrations ($\sim 1720\text{ cm}^{-1}$), and PHB ester vibrations. In the PLA/CA and PLA/PHB blends, shifts and band broadening in the O-H and C-O regions suggest hydrogen bonding and increased crystallinity. All the FT-IR blend spectra were recorded at 50:50 (w/w).

Fig. 2. ^1H NMR confirmation of selective conversion and PLA-content-dependent response. (a) ^1H NMR spectrum of the alcoholysis product of neat PLA in CDCl_3 , showing characteristic resonances of methyl lactate at $\delta \approx 1.30\text{ ppm}$ (doublet, $-\text{CH}_3$) and $3.70\text{--}3.75\text{ ppm}$ (singlet, $-\text{OCH}_3$), confirming selective conversion of PLA into methyl lactate as the primary degradation product. (b) ^1H NMR spectra of sodium lactate in D_2O obtained after hydrolysis of PLA/PP blends. A characteristic methyl doublet at $\delta \approx 1.33\text{ ppm}$ is observed. The inset shows an expanded view of the methyl region for blends with varying PLA contents (10–100 wt%), revealing a systematic increase in peak intensity with increasing PLA content.

Fig. 3. Absorbance at 400–410 nm increases linearly with PLA content in the polymer blends. (a) UV-vis absorbance spectra of PLA/PP blend samples with varying PLA contents (mass fractions ranging from 0.1 to 1.0 g g^{-1}), showing an increase in absorbance with increasing PLA content. (b) Calibration curve of absorbance from λ_{max} (400–

410 nm) versus the PLA mass fraction, fitted with the linear regression equation $y = 0.859x + 0.005$ ($R^2 = 0.9978$), demonstrating the high linearity and quantitative sensitivity of the developed method.

Fig. 4. Cross-validation of PLA quantification across different polymer blends. Validation of the PLA/PP calibration using PLA/PBAT (PLA = 60, 70, and 80 wt%), PLA/PHB (PLA = 25, 50, and 75 wt%), and PLA/CA (PLA = 70, 80, and 90 wt%) blends. Absorbance at λ_{\max} (400–410 nm) was plotted against PLA content (wt%), showing close agreement with the calibration curve ($y = 0.859x + 0.005$). Symbols: ● PBAT, ■ PHB, and ◆ CA.

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- * -CH₃ (doublet)
- ** -OCH₃ (singlet)











