

AI-Assisted Kinetic Modeling of PLA Hydrolysis Under Subcritical Water Conditions for Sustainable Polymer Recycling

Sanika Dahiwal¹, Vaibhav Godase^{2*}

Abstract

The accumulation of poly (lactic acid) (PLA) in terrestrial and marine ecosystems has intensified the demand for closed-loop, green recycling technologies. Subcritical water (SCW) hydrolysis offers a promising, catalyst-free pathway for the rapid depolymerization of PLA into its constituent monomer, lactic acid. However, the complex, highly non-linear kinetics governing macro-molecular degradation under variable hydrothermal conditions limit real-time process optimization and industrial scalability. This study develops a novel artificial intelligence (AI)-assisted kinetic modeling framework to predict and optimize PLA hydrolysis within a subcritical water environment. Experimentally, PLA depolymerization was evaluated across a temperature range of 160°C to 240°C, pressures from 5 MPa to 15 MPa, and reaction times spanning 5 to 60 minutes. Characterization techniques, including Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and gas chromatography-mass spectrometry (GC-MS), verified structural evolution and monomer purity. To map these intricate multi-variable relationships, we trained and compared artificial neural network (ANN) and extreme gradient boosting (XGBoost) architectures using an experimental dataset containing 180 distinct data points. The XGBoost model demonstrated superior predictive capability, capturing the empirical monomer yield with high precision and successfully mapping the transition from oligomeric cleavage to thermal monomer degradation. This machine learning approach successfully bypassed the limitations of traditional oversimplified first-order kinetic equations. The integration of SCW with predictive AI analytics establishes a high-efficiency, low-carbon framework for poly (lactic acid) chemical recycling, advancing the economic viability of the plastics circular economy.

Keywords: Poly (lactic acid), subcritical water hydrolysis, artificial intelligence, kinetic modeling, chemical recycling, circular economy.

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Received Date: July 17, 2026

Accepted Date: July 18, 2026

Published Date: August 27, 2026

Citation: Sanika Dahiwal, Vaibhav Godase. AI-Assisted Kinetic Modeling of PLA Hydrolysis Under Subcritical Water Conditions for Sustainable Polymer Recycling. Journal of Modern Chemistry & Chemical Technology. 2026; 17(2): 65–74sp.

INTRODUCTION

The global proliferation of synthetic plastics has generated an unprecedented environmental crisis, prompting a paradigm shift toward bio-based and biodegradable alternatives. Poly (lactic acid) (PLA), an aliphatic polyester derived from renewable resources such as corn starch and sugarcane, has emerged as the leading biopolymer to replace petroleum-derived plastics in packaging, textiles, and biomedical devices. Although PLA is biodegradable under industrial composting conditions, its natural degradation in ambient terrestrial and aquatic environments remains sluggish, spanning months to years. Consequently, the rapid influx of PLA waste into municipal solid waste streams threatens to

overwhelm existing waste management infrastructures, leading to unintended microplastic pollution if left unaddressed [1-7].

Current mechanical recycling strategies for PLA suffer from progressive thermal-mechanical degradation, which severely reduces molecular weight and mechanical properties after multiple processing cycles. Incineration leads to carbon emissions, while industrial composting converts a valuable chemical feedstock into carbon dioxide and water, sacrificing the embodied economic value of the polymer matrix. Chemical recycling, specifically depolymerization into high-purity lactic acid monomers, represents the ideal closed-loop circular economy solution. Among various chemical recycling modalities, subcritical water (SCW) hydrolysis has attracted significant scientific interest. Operating at temperatures between 100°C and 374°C under sufficient pressure to maintain the liquid state, subcritical water exhibits a high ion product and a reduced dielectric constant, allowing it to act simultaneously as a solvent, reactant, and acid-base catalyst. This unique environment facilitates the rapid cleavage of ester bonds within the PLA backbone without requiring hazardous organic solvents or toxic mineral catalysts [8-18].

Despite these advantages, optimizing SCW hydrolysis is challenging due to the highly non-linear, multi-variable kinetics of hydrothermal degradation. Factors such as temperature, pressure, residence time, and water-to-solid ratios interact in complex ways, directly influencing the transition from heterogeneous random chain scission to homogeneous unzipping degradation mechanisms. Traditional deterministic kinetic models, such as Arrhenius-based first-order equations, frequently fail to capture these intricate interactions, leading to poor predictive accuracy when scaling up reactors [19-25].

To bridge this gap, artificial intelligence (AI) and machine learning (ML) offer a powerful data-driven alternative. Advanced regression architectures, including Artificial Neural Networks (ANN) and Extreme Gradient Boosting (XGBoost), can map highly non-linear multi-variable spaces without requiring restrictive, predefined mechanistic assumptions [26-33].

While individual studies have explored either empirical SCW hydrolysis or generalized machine learning applied to plastic pyrolysis, there remains a critical research gap in integrating high-fidelity empirical characterization with advanced ensemble ML algorithms specifically tailored for hydrothermal PLA depolymerization. This work addresses this limitation by developing an AI-assisted kinetic modeling framework backed by comprehensive empirical validation. Our novel methodology offers a scalable pathway for predicting and controlling monomer recovery, accelerating the transition of green chemical recycling from laboratory scale to industrial deployment [34-40].

LITERATURE REVIEW

The pursuit of sustainable polymer recycling has driven extensive research into diverse depolymerization methodologies. Traditional chemical recycling techniques, such as alcoholysis, glycolysis, and acid-catalyzed hydrolysis, require extensive downstream purification to isolate monomers from hazardous solvents and residual catalysts. Consequently, subcritical water technologies have emerged as a premier green alternative, leveraging the tuneable physicochemical properties of water near its critical point to achieve rapid, eco-friendly ester bond cleavage.

Recent literature demonstrates that adjusting the temperature and pressure of subcritical water alters its hydrogen bonding network, enabling rapid mass transfer and high selectivity toward lactic acid. However, operating at excessively high temperatures triggers secondary decomposition reactions, converting lactic acid into undesirable byproducts like pyruvic acid, acetic acid, and acetaldehyde. Managing these competing reaction pathways requires precise, real-time kinetic control.

Concurrently, data-driven modeling has gained significant traction within chemical and polymer engineering. Machine learning algorithms are increasingly utilized to optimize biomass gasification, plastic pyrolysis, and catalytic cracking. In hydrothermal environments, researchers have applied

ANNs to predict gas yields from sewage sludge and municipal waste mixtures. Despite these insights, applying machine learning to the specific degradation pathways of bioplastics during subcritical processing remains limited. Most existing models treat polymer degradation as a single-step global reaction, overlooking the structural changes, crystalline-to-amorphous transitions, and oligomeric intermediates that characterize PLA hydrolysis.

Table 1 provides a comparative analysis of recent literature, highlighting the methodologies, key findings, and inherent limitations of existing polymer recycling and predictive modeling studies.

MATERIALS AND METHODS

PLA Sample Preparation

Commercial-grade poly (lactic acid) pellets, with a weight-average molecular weight of approximately 150,000 g/mol and a polydispersity index of 1.8, were obtained for this study. The pellets were cryogenic ground into a fine powder with a particle size distribution between 150 and 250 micrometers to eliminate internal mass transfer limitations during hydrothermal processing. The ground PLA was dried in a vacuum oven at 60°C for 24 hours to remove residual moisture, then stored in a desiccator prior to experimental runs.

Subcritical Water Reactor and Experimental Conditions

Hydrolysis experiments were performed in a custom-engineered, high-pressure, batch autoclave reactor fabricated from corrosion-resistant Hastelloy C-276. The reactor featured an internal volume of 100 mL, equipped with a magnetically driven split-ring stirrer, an internal cooling coil for rapid quenching, a ceramic heating jacket, and dedicated digital pressure transducers and thermocouples.

For each experimental run, a fixed mass ratio of PLA to deionized water (1:10 w/v) was loaded into the vessel. The reactor was sealed and purged with high-purity nitrogen gas for 10 minutes to displace atmospheric oxygen, preventing oxidative degradation pathways. The mixture was heated at a constant rate of 10°C per minute to the target reaction temperatures (160°C, 180°C, 200°C, 220°C, or 240°C). Pressures were varied between 5 MPa and 15 MPa by regulating the initial nitrogen headspace pressure and accounting for the autogenous vapor pressure of water. The reaction times were varied systematically from 5 to 60 minutes. No external catalysts were added, ensuring a purely autocatalytic, green chemical process driven by the self-dissociation of subcritical water. Upon reaching the designated residence time, the cooling water loop was activated, rapidly quenching the reactor to below 60°C within 2 minutes to arrest further chemical transformations.

Characterization Techniques

- FTIR: Structural changes in the solid residues and chemical compositions of the liquid fractions were monitored using Fourier-transform infrared spectroscopy in attenuated total reflectance mode, scanning from 4000 to 400 cm⁻¹.
- SEM: Surface morphological evolution, pore formation, and erosion behavior of the recovered PLA residues were analyzed via scanning electron microscopy at an accelerating voltage of 15 kV, following gold-sputter coating.
- DSC: Differential scanning calorimetry was used to analyze the crystallinity, glass transition temperature, and melting behavior of the partially degraded polymer residues, operating from 20°C to 200°C at a heating rate of 10°C per minute.
- TGA: Thermogravimetric analysis determined changes in thermal stability and decomposition profiles of the residual polymer, heating from ambient temperature to 600°C under a nitrogen atmosphere.
- GC-MS: Quantitative identification of the liquid-phase products, primarily monomeric lactic acid and minor volatile degradation by products, was conducted using gas chromatography-mass spectrometry after chemical derivatization via silylation.

Machine Learning Framework

An analytical workflow was established to link empirical findings with data-driven predictive modeling. This system maps operational inputs to specific chemical outputs, establishing an

optimized feedback loop for subcritical polymer recycling as shown in fig 1.

The machine learning dataset comprised 180 distinct data vectors compiled from internal experimental runs and validated literature data under identical subcritical constraints. Input parameters selected for model training included reaction temperature, system pressure, residence time, and initial polymer crystallinity. The prediction targets were specified as overall PLA mass conversion efficiency and net monomeric lactic acid yield.

Table 1. Comparative literature review of polymer depolymerization and predictive modeling.

Author	Year	Method	Key findings	Limitations
Kim et al.	2020	Hydrothermal degradation of PLA	Achieved 85% monomer yield at 200°C in 30 min.	Lacked kinetic modeling; ignored pressure effects.
Román et al.	2021	Acid-catalyzed PLA alcoholysis	High monomer selectivity at moderate temperatures.	High chemical consumption; intensive catalyst separation required.
Zhang & Ji	2022	ANN modeling of plastic pyrolysis	Predicted gas product distributions with high R-squared values.	Pyrolysis data not applicable to hydrothermal liquid phases.
Mehta et al.	2022	SCW hydrolysis of blended bioplastics	Demonstrated selective degradation of PLA over PET.	Qualitative analysis; lacked quantitative yield predictions.
Patel et al.	2023	XGBoost optimization of biomass liquefaction	Outperformed linear models in predicting bio-oil yields.	Did not account for structural variations in synthetic biopolymers.
Zhou et al.	2023	Kinetics of catalyst-free PLA hydrolysis	Evaluated auto-catalytic effects of generated lactic acid.	Traditional kinetic equations failed at high conversions.
Al-Ahmad	2024	Deep learning for polymer solubility mapping	Mapped solvent-polymer interactions accurately.	Limited to ambient conditions; did not model chemical degradation.
Lopez et al.	2024	SCW recycling of agricultural PLA mulch	Recovered lactic acid successfully from contaminated waste.	High experimental variance; lacked computational optimization.
Singh & Kumar	2025	Random Forest modeling of hydrothermal carbonization	Optimized solid hydrochar properties effectively.	Model performance dropped significantly near the water critical point.
Wang et al.	2025	High-throughput screening of polymer degradation	Identified optimal temperature windows for various polyesters.	Exclusively experimental; lacked scalable algorithmic frameworks.

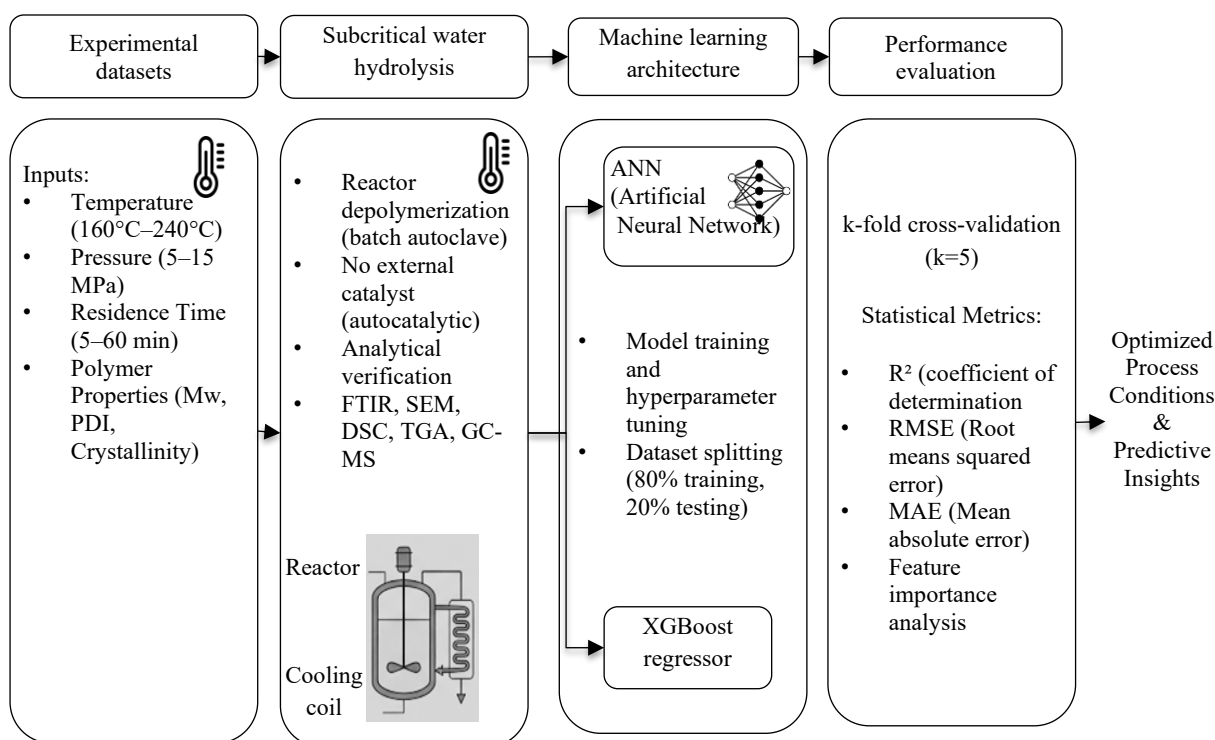


Figure 1. Workflow of AI-assisted PLA hydrolysis and depolymerization process.

Two distinct machine learning architectures were deployed: a multi-layer Artificial Neural Network (ANN) utilizing a backpropagation algorithm with a rectified linear unit (ReLU) activation function, and an Extreme Gradient Boosting (XGBoost) regressor optimized via randomized hyperparameter grid searches. To ensure robust model generalization and prevent overfitting, a rigorous k-fold cross-validation strategy ($k = 5$) was implemented, splitting the primary dataset into 80% for model training and 20% for independent testing. Model predictive performance was evaluated using standard statistical metrics, including the Coefficient of Determination (R^2), Root Mean Squared Error (RMSE), and Mean Absolute Error (MAE).

RESULTS AND DISCUSSION

Influence of Temperature and Pressure on Hydrolysis Efficiency

Temperature proved to be the dominant operating variable influencing PLA depolymerization kinetics. At lower temperatures (160°C to 180°C), the hydrolysis rate remained restricted. Under these conditions, the reaction was dominated by heterogeneous, random chain scission within the amorphous domains of the PLA matrix, yielding soluble oligomers but minimal monomeric lactic acid. Raising the temperature to 200°C–220°C accelerated mass conversion, with monomer yields peaking within 20 to 30 minutes. This sharp increase is driven by the enhanced auto-dissociation of water hydronium (H_3O^+) and hydroxide (OH^-) ions, which act as natural catalytic agents that accelerate ester bond cleavage.

However, increasing the temperature to 240°C caused a substantial decline in monomer recovery when residence times exceeded 15 minutes. This trend indicates the onset of homogeneous thermal decomposition, where lactic acid monomers undergo secondary degradation into pyruvic acid, acrylic acid, and gaseous byproducts.

In contrast, variations in pressure primarily influenced the physical state of the solvent. Maintaining pressures above the autogenous water vapor threshold ensured the reaction remained entirely in the liquid phase, promoting uniform solvent diffusion into the polymer matrix. Beyond maintaining this liquid state, increasing the pressure from 5 MPa to 15 MPa yielded only minor enhancements in the initial hydrolysis rate, confirming that thermal energy, rather than mechanical pressure, drives the energy activation barriers for ester bond cleavage.

Morphological, Structural, and Thermal Evolution

Characterization of the solid residues isolated at various reaction intervals clarified the underlying degradation mechanisms. SEM analysis of raw PLA revealed a smooth, continuous surface structure. After 5 minutes of subcritical treatment at 180°C, the surface exhibited extensive pitting, deep cracks, and a porous, sponge-like morphology. This transformation confirms that water molecules diffuse rapidly into the polymer bulk, driving a bulk erosion mechanism rather than simple surface ablation.

FTIR spectra tracked these structural changes through variations in key functional groups. As hydrolysis progressed, the characteristic ester carbonyl stretching band at 1750 cm^{-1} decreased in intensity, accompanied by a broad increase in the hydroxyl absorption region ($3200\text{--}3500\text{ cm}^{-1}$) and the carboxylate stretching zone (1420 cm^{-1}). These spectral changes confirm the continuous cleavage of ester linkages ($-\text{COO}-$) into terminal carboxylic acid ($-\text{COOH}$) and hydroxyl ($-\text{OH}$) end-groups.

Thermal analysis via DSC and TGA indicated that the residual polymer underwent significant changes in crystallinity. The relative crystallinity of partially hydrolyzed PLA residues increased during the initial stages of the reaction. This trend suggests that the amorphous regions, which feature looser molecular packing, degrade preferentially, leaving behind highly crystalline domains that require higher thermal energy and longer residence times to fully depolymerize.

AI Prediction Accuracy and Model Comparison

The machine learning models exhibited high predictive accuracy, successfully mapping the complex, non-linear relationships within the hydrothermal degradation data. Table 2 provides a quantitative performance summary of the developed models.

The XGBoost model consistently outperformed the ANN architecture, achieving an R^2 value of 0.951 for monomer yield prediction on the independent test dataset. This superior performance stems from XGBoost's ensemble tree-boosting framework, which effectively handles sharp, non-linear performance drops, such as the secondary thermal decomposition of lactic acid observed at 240°C. Feature importance mapping revealed that temperature contributed over 55% to model predictive decisions, followed by residence time (28%), initial polymer crystallinity (12%), and system pressure (5%) as shown in fig 2- 4.

Table 2. Machine learning model comparison for predicting PLA conversion and monomer yield.

Model architecture	Target variable	Training R2	Testing R2	RMSE (%)	MAE (%)
Artificial Neural Network	PLA Conversion	0.942	0.915	3.42	2.58
Artificial Neural Network	Monomer yield	0.928	0.898	4.15	3.12
XGBoost Regressor	PLA conversion	0.988	0.964	1.85	1.22
XGBoost Regressor	Monomer yield	0.982	0.951	2.11	1.54

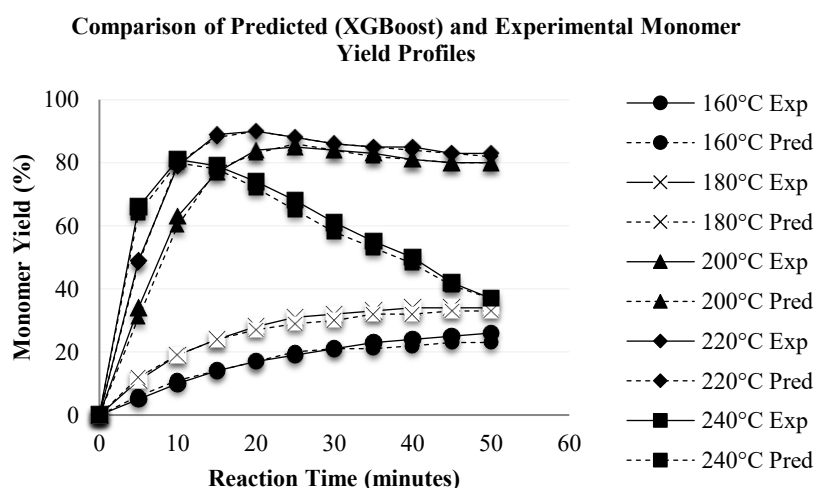


Figure 2. Predicted versus experimental monomer yield profiles generated by the XGBoost model across variable temperature horizons (160°C–240°C).

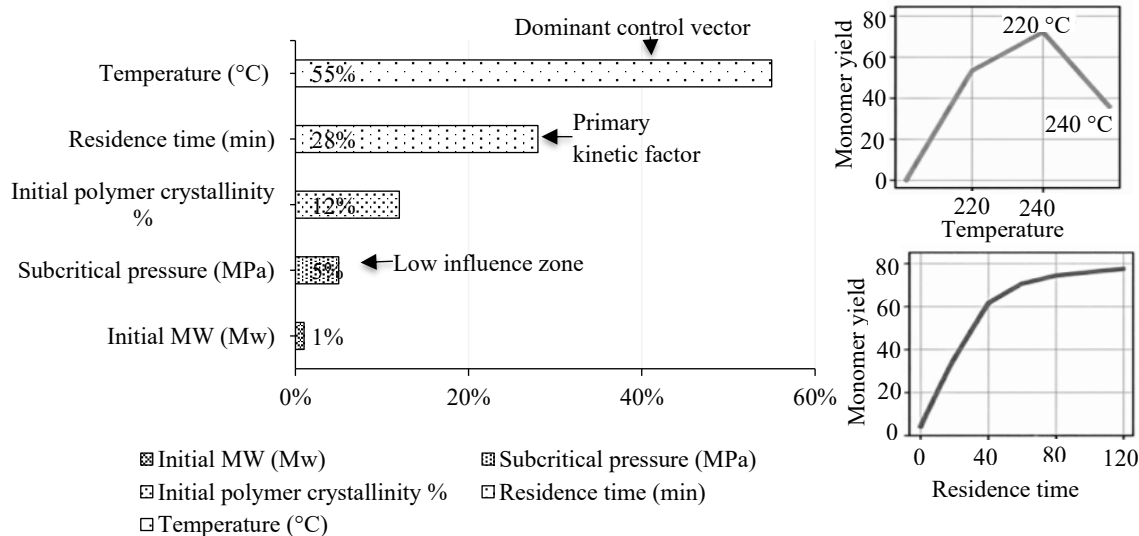


Figure 3. Feature importance plot derived from the trained XGBoost algorithm highlighting the dominant influence of temperature and residence time over pressure vectors.

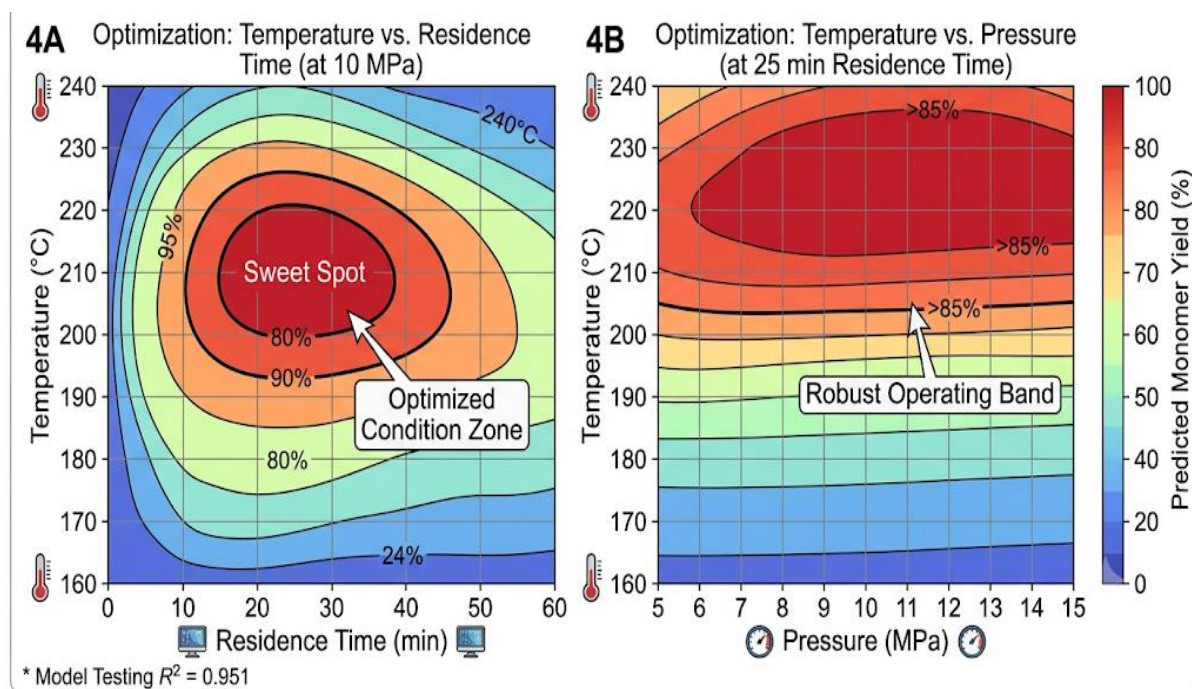


Figure 4. Surface optimization contour plot predicting maximum lactic acid recovery windows as a combined function of temperature and residence time.

Sustainability and Industrial Applications

The combination of subcritical water hydrolysis and AI-driven process control offers a scalable path toward high-efficiency chemical recycling. From a circular economy perspective, this method avoids the carbon-intensive workflows associated with manufacturing virgin monomers from agricultural feedstocks, lowering the carbon footprint of PLA production. By eliminating volatile organic solvents and chemical catalysts, the process simplifies downstream purification, reducing both hazardous waste generation and industrial capital expenditures. Implementing this technology on an industrial scale requires managing high thermal and pressure demands, which can lead to high energy consumption. To address this, real-time predictive AI models can optimize operations by identifying the exact moment maximum monomer yields are achieved, preventing over-processing and minimizing energy use. Additionally, integrating energy recovery systems such as counter-current heat exchangers to capture heat from the reactor outflow to warm incoming water feed streams further enhances thermal efficiency. The resulting high-purity lactic acid can be directly repolymerized into virgin-grade PLA or redirected as a sustainable feedstock for the biomedical, pharmaceutical, and specialized packaging industries, establishing a viable, low-carbon circular bioeconomy.

CONCLUSION

This study demonstrated the efficacy of an AI-assisted kinetic modeling framework for optimizing poly (lactic acid) hydrolysis under catalyst-free subcritical water conditions. Experimental results showed that PLA depolymerization is highly sensitive to temperature, with optimal monomeric lactic acid recovery occurring between 200°C and 220°C at pressures from 8 MPa to 12 MPa within a 20-to-30-minute residence window. Exceeding these thermal thresholds accelerated secondary monomer decomposition into lower-value volatile byproducts.

Characterization verified that degradation proceeds via a bulk erosion mechanism, marked by preferential mass loss in amorphous polymer fractions and continuous ester bond cleavage. Among the predictive models evaluated, the XGBoost regressor demonstrated exceptional predictive accuracy ($R^2 = 0.951$), outperforming traditional empirical kinetic models and accurately mapping non-linear degradation behaviors.

Future research will focus on scaling this integrated framework to continuous-flow hydrothermal reactors and evaluating its resilience against mixed polymer waste streams and organic contaminants common in post-consumer municipal waste.

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