

MULTI-RESPONSE OPTIMIZATION OF AMMONIUM OXALATE EXTRACTION OF PECTIC MATERIAL FROM LIBERICA COFFEE (*COFFEA LIBERICA* VAR. *LIBERICA*) HUSK USING RSM-CCD

Reta Reta ^{1,*}, Andi Lutfiah Husna², Zaimar Zaimar ¹, Muhammad Fitri ¹ and Sri Udayana Tartar ¹

¹Agroindustry Study Program, Department of Agricultural Technology, Pangkep State Polytechnic of Agriculture, Pangkajene Kepulauan, South Sulawesi, Indonesia

²Undergraduate student of Agroindustry Study Program, Department of Agricultural Technology, Pangkep State Polytechnic of Agriculture, Pangkajene Kepulauan, South Sulawesi, Indonesia

*Corresponding author: retariskinapolitani@gmail.com

ABSTRACT

Liberica coffee husk is an underutilized agro-industrial by-product with potential as a source of pectic material. This study investigated how ammonium oxalate concentration and extraction temperature affect gravimetric recovery and the physicochemical properties of pectic material extracted from Liberica coffee husk. A two-factor rotatable circumscribed central composite design (CCD) was employed within the framework of response surface methodology (RSM). We evaluated gravimetric recovery, moisture content, ash content, galacturonic acid (GalA) content, and instrumental total color difference (ΔE_{ab}) and subjected them to multi-response optimization using a desirability function. Gravimetric recovery ranged from 10.0 to 37.3%, while GalA content ranged from 52.31 to 79.32%, indicating differences in pectic enrichment rather than directly establishing product purity. Ammonium oxalate concentration was the most influential factor affecting both responses. Multi-response optimization identified 3% (w/v) ammonium oxalate at 80°C as the model-predicted preferred extraction condition, with a composite desirability of 0.858. Under these conditions, the models predicted a gravimetric recovery of 33.16%, moisture content of 13.20%, ash content of 1.44%, GalA content of 75.70%, and ΔE_{ab} of 19.64. The corresponding experimental run included in the original CCD yielded a gravimetric recovery of 37.30%, moisture content of 13.90%, ash content of 1.57%, GalA content of 78.45%, and ΔE_{ab} of 20.36. However, because this run was used to develop the predictive models, it cannot be considered an independent validation experiment. Therefore, the selected extraction condition should be regarded as a model-predicted preferred condition rather than an experimentally validated optimum. These findings demonstrate the potential of Liberica coffee husk as a source of pectic material and provide a basis for further process development. Independent experimental validation, along with structural characterization, functional evaluation, safety assessment, and scale-up studies, is needed to establish the recovered material's suitability for practical applications.

Keywords: Ammonium oxalate, Coffee by-products, Galacturonic acid, Liberica coffee, Multi-response optimization, pectin.

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1. INTRODUCTION

Coffee is one of the most widely cultivated and processed agricultural commodities worldwide. However, its economic importance is accompanied by the generation of substantial quantities of processing residues. These residues include pulp, husk, parchment, mucilage, and silverskin, which originate from different stages of coffee processing and differ in their anatomical and chemical characteristics (Iriondo-DeHond et al., 2020; Klingel et al., 2020; Moordiani et al., 2026). Coffee pulp generally consists of the outer skin and fleshy mesocarp removed during wet processing, whereas coffee husk produced during dry processing typically contains dried outer fruit tissues, including skin, pulp, mucilage, and parchment (Heeger et al., 2017; Klingel et al., 2020). The term *cascara* commonly refers to dried coffee fruit tissues or products derived from them, although its composition varies by processing method (Heeger et al., 2017; Iriondo-DeHond et al., 2020). In the present study, the extraction material consisted of dried outer tissues manually separated from fresh *Coffea liberica* cherries.

The coffee industry is estimated to generate more than 10 million tonnes of solid residues annually, although reported quantities vary depending on the types of residues considered (Hoseini et al., 2021; Soumya et al., 2022; Bairwan et al., 2024; Hamedani et al., 2025). Collectively, coffee pulp, husk, parchment, mucilage, and silverskin

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can account for a substantial proportion of the original fruit mass (Amran et al., 2021; Hoseini et al., 2021). Inadequate management and disposal of these residues may contribute to unpleasant odors, acidic leachate formation, soil degradation, eutrophication, and contamination of surface and groundwater (Takala, 2021; Mak et al., 2023; Rebollo-Hernanz et al., 2023). These environmental concerns are also linked to caffeine, tannins, chlorogenic acids, and other phenolic compounds, which may adversely affect microbial activity and plant growth when coffee residues are improperly managed (Takala, 2021; Pandey et al., 2025).

Despite these environmental challenges, coffee-processing residues contain valuable components that can be converted into useful products. Previous studies have identified cellulose, hemicelluloses, lignin, pectic polysaccharides, minerals, caffeine, chlorogenic acids, tannins, and other bioactive compounds in coffee-derived residues (Dadi et al., 2019; Hoseini et al., 2021; Tabassum et al., 2025). However, much of the available compositional information relates to Arabica, Robusta, mixed coffee residues, or biomass for which the coffee species was not clearly identified. These findings provide useful background information but may not apply directly to *Coffea liberica*. Differences in species, fruit maturity, processing methods, and anatomical fractions may substantially influence the chemical composition of coffee residues. This distinction is particularly important for Liberica coffee, as detailed information on the composition and recovery of its pectic fractions remains limited.

Among the potentially valuable components of coffee residues, pectin has attracted considerable interest because of its gelling, thickening, stabilizing, emulsifying, and film-forming properties. Nevertheless, studies specifically addressing pectin recovery from Liberica coffee residues remain scarce. It is also important to distinguish findings obtained from Liberica coffee pulp from those involving coffee husk. For example, Wong & Nillian (2023) investigated the microwave-assisted recovery of phenolic compounds, flavonoids, and carbohydrates from Sarawak Liberica coffee pulp. Although their findings demonstrate the potential of Liberica fruit residues as sources of valuable compounds, they do not directly establish the extraction behavior or pectic composition of the husk material investigated in the present study.

The efficiency of pectic material extraction depends on several factors, including extractant type and concentration, extraction temperature, duration, and solid-to-liquid ratio. Increasing extraction severity may improve solvent penetration and facilitate the release of polysaccharides associated with plant cell walls. However, studies on other botanical materials show that excessive chemical or thermal treatment may promote hydrolysis, demethylation, or reductions in polymer molecular weight (Muñoz-Almagro et al., 2018; Peng et al., 2020; Ma et al., 2021; Jarrín-Chacón et al., 2023). These processes may influence the recovery and functional characteristics of the extracted material. Nevertheless, polymer degradation is considered only a possible explanation in the present study because molecular weight, degree of depolymerization, and degradation products were not directly measured. Therefore, differences in gravimetric recovery cannot independently establish whether polymer degradation occurred. Since extraction variables may interact and produce nonlinear responses, response surface methodology (RSM) offers a suitable approach for evaluating their individual and combined effects within a defined experimental range (Ma et al., 2021; Sarah et al., 2022).

Researchers have investigated ammonium oxalate as an extractant for recovering pectic fractions from various plant materials. Its effectiveness has been associated with its ability to facilitate the release of pectic polysaccharides linked to calcium within plant cell walls. In particular, low-methyl-esterified regions of homogalacturonan can interact with calcium ions through structures commonly described by the “egg-box” model. The calcium-binding properties of oxalate may contribute to solubilizing these calcium-associated pectic fractions (Ognyanov et al., 2018; Peng et al., 2020; Banjanac et al., 2026). However, this mechanism is regarded as a literature-supported explanation rather than a finding directly demonstrated in the present study, as calcium release, calcium binding, and oxalate–calcium complex formation were not measured. Previous studies have used ammonium oxalate to recover pectic materials from sunflower heads, *Fumaria officinalis* cultures, *Premna microphylla* leaves, and rapeseed meal, reporting considerable differences in recovery and composition among plant materials (Ognyanov et al., 2018; Ma et al., 2021; Li et al., 2025; Dharmawan & Masithoh, 2025; Banjanac et al., 2026). These findings highlight the importance of developing extraction conditions suited to each botanical source.

Previous investigations have also demonstrated that ammonium oxalate concentration and extraction temperature can substantially influence pectic material recovery and composition. Ma et al. (2021) reported that ammonium oxalate concentration was a key factor in extracting pectin from sunflower heads, yielding a product containing 76.2% galacturonic acid under their optimized conditions. Similarly, Peng et al. (2020) found that increasing oxalic acid extraction severity could enhance polysaccharide solubilization, although excessive treatment increased the risk of hydrolysis. Muñoz-Almagro et al. (2018) further showed that milder extraction conditions better preserve polymer molecular weight and certain technological properties than more severe acid treatment. In addition, RSM has been successfully applied to the extraction of pectic materials from cocoa residues and other agricultural by-products, allowing researchers to identify interactions among extraction variables and evaluate their combined effects on recovery and product characteristics (Sarah et al., 2022; Jarrín-Chacón et al., 2023; Mounya & Chowdary, 2024).

Although these studies demonstrate the usefulness of multivariable optimization, their findings cannot be directly extrapolated to Liberica coffee husk because plant cell-wall composition and extraction behavior differ.

Despite growing interest in valorizing coffee-processing residues, information on ammonium oxalate extraction of pectic material from Liberica coffee husk remains limited. Previous research on Liberica residues has largely focused on recovering bioactive compounds from coffee pulp, whereas studies applying ammonium oxalate extraction and response surface optimization have mainly examined other botanical materials. Consequently, the combined effects of ammonium oxalate concentration and extraction temperature on the recovery and physicochemical characteristics of Liberica coffee-husk pectic material remain poorly established. Addressing this gap is important for identifying suitable extraction conditions and assessing this underutilized residue's potential as a source of pectic material.

Therefore, this study aimed to evaluate the effects of ammonium oxalate concentration and extraction temperature on the gravimetric recovery and physicochemical characteristics of pectic material from Liberica coffee husk using a two-factor central composite design (CCD) within RSM. The study focused on identifying a suitable combination of extraction conditions through multi-response optimization rather than maximizing gravimetric recovery alone. Specifically, the optimization considered gravimetric recovery, galacturonic acid content, moisture content, ash content, and instrumental total color difference (ΔE^*ab). Because the study did not comprehensively evaluate pectin purity and functional properties, the resulting condition is a model-predicted preferred extraction condition rather than a validated optimum for pectin quality or functionality. The findings are expected to provide a foundation for subsequent experimental validation, structural characterization, functional evaluation, and further development of Liberica coffee husk as a potential source of value-added pectic material.

2. MATERIALS AND METHODS

2.1. Raw Material and Reagents

Liberica coffee cherries (*Coffea liberica* var. *liberica*) were harvested between 23 and 30 July 2025 from a coffee farm managed by Kelompok Tani Kopi Desa Harapan, Barru Regency, South Sulawesi, Indonesia. Fresh cherries were washed with clean water to remove soil and foreign material adhering to the fruit. The beans were manually separated from the outer fruit tissues; therefore, this study used dried outer fruit tissue removed from fresh cherries rather than husk obtained by dry hulling. In this manuscript, we refer to this material consistently as dried Liberica coffee outer fruit tissue (cascara). The separated material was initially dried to approximately 12% moisture and stored in airtight packaging until further preparation.

Before extraction, the dried cascara was manually inspected to remove remaining beans, twigs, stones, and other visible contaminants, milled using herb grinder, and passed through a 120-mesh sieve. The prepared powder was re-dried before extraction. Its moisture content was determined gravimetrically by oven drying at 45°C for 15 h and was 8%. Thus, the approximately 12% moisture value refers to the dried outer fruit tissue before milling/storage, whereas 8% refers to the 120-mesh powder actually used for extraction. The prepared powder was kept in a sealed container until use.

Ammonium oxalate was used as the extraction agent, while 96% ethanol was used for pectin precipitation and washing. Distilled water was used to prepare the extraction medium and for other analytical procedures. The extraction and physicochemical analyses were conducted from April to May 2026 at the Microbiology Laboratory, Pangkep State Polytechnic of Agriculture, South Sulawesi, Indonesia.

2.2. Experimental Design

The effects of ammonium oxalate concentration and extraction temperature on the physicochemical characteristics of the recovered pectic material were evaluated using Response Surface Methodology with a two-factor rotatable circumscribed Central Composite Design (CCD). Ammonium oxalate concentration was designated factor A and extraction temperature factor B. The factorial levels were 2 and 3% (w/v) ammonium oxalate and 80 and 90°C, with a center point at 2.5% and 85°C. For two factors ($k = 2$), the axial distance was selected as $\alpha = (2^k)^{1/4} = \sqrt{2} = 1.4142$ to provide rotatability. The axial points therefore extended the design to 1.7929 and 3.2071% ammonium oxalate and 77.9289 and 92.0711°C. The design comprised 13 runs: four factorial points, four axial points, and five replicated center points. The treatment combinations were executed in randomized run order. Center-point replicates were included to estimate pure experimental error and support evaluation of model curvature. The coded factors were calculated as follows:

$$A = (C - 2.5) / 0.5$$

$$B = (T - 85) / 5$$

Where C represents ammonium oxalate concentration (%) and T represents extraction temperature (°C). This experimental configuration follows the conventional structure of a two-factor Response Surface Methodology-Central

Composite Design (Myers et al., 2016). The experimental conditions and measured responses are presented in Table 1.

Table 1: Central Composite Design and measured responses for ammonium oxalate extraction of Liberica coffee-husk pectic material

Run	Ammonium oxalate (%)	Temperature (°C)	Recovery (%)	Moisture (%)	Total ash (%)	GalA (%)	ΔE*ab
1	3.207	85	26.60	11.88	1.66	79.31	19.47
2	2.5	85	16.50	12.50	0.20	64.17	18.56
3	2.5	85	21.00	12.80	0.24	69.74	19.12
4	2.5	85	19.10	13.50	0.52	68.25	18.17
5	2.5	77.93	15.40	13.80	0.19	61.55	17.34
6	1.793	85	11.80	13.30	0.15	54.25	17.81
7	3	90	14.70	14.10	0.13	64.38	19.08
8	2.5	92.07	16.10	13.69	0.17	63.82	18.70
9	2.5	85	18.40	12.40	0.12	65.31	18.04
10	2	90	10.00	14.30	0.11	52.31	18.74
11	2.5	85	21.40	13.20	0.89	71.58	18.16
12	2	80	10.80	14.90	0.21	53.62	17.42
13	3	80	37.30	13.90	1.57	78.45	20.36

Note: GalA, galacturonic acid content; ΔE*ab, total color difference in the CIELAB color system relative to the selected reference ($L^* = 25.55$, $a^* = 4.08$, $b^* = 9.81$). Gravimetric recovery was calculated on an as-used 25-g sample-mass basis.

2.3. Pectin Extraction

For each experimental run, 25 g of 120-mesh Liberica coffee cascara powder (8% moisture) was transferred into an extraction vessel and mixed with 500 mL of aqueous extraction medium (solid-to-liquid ratio 1:20, w/v). Ammonium oxalate concentration was expressed as % w/v (g per 100 mL final extraction medium); accordingly, we prepared 2.0, 2.5, and 3.0% using 10.0, 12.5, and 15.0 g ammonium oxalate, respectively, to a final volume of 500 mL. The extraction mixture was heated to the assigned experimental temperature and agitated continuously at 350 rpm for 60 min. The measured pH was 2.6 before extraction and 4.6 after the 60-min extraction period for all experimental runs; pH was not an experimental factor. After extraction, the slurry was cooled to room temperature and filtered to separate the insoluble residue from the pectin-containing liquid extract.

The recovered pectic material was precipitated by mixing the filtrate with 96% ethanol at a fixed 1:1 (v/v) filtrate-to-ethanol ratio. This ratio was used as the laboratory precipitation condition throughout the study and was not included as an optimization factor. After homogenization, the mixture was left undisturbed for approximately 18 h at room temperature. The precipitate was separated by filtration and washed once with approximately 100 mL of 96% ethanol to remove soluble sugars, salts, pigments, residual extractant, and other low-molecular-weight compounds. Residual ammonium oxalate/oxalate in the final precipitate was not quantified analytically; therefore, interpret ethanol washing as an operational purification step rather than evidence of complete oxalate removal. The washed precipitate was dried at 45°C for 15 h, ground to a homogeneous powder, and stored in a tightly closed container until analysis.

2.4. Response Analyses

Gravimetric recovery was determined as the mass of dried recovered solid relative to the actual mass of cascara powder used for extraction. Each run used 25.0 g of prepared powder, and the denominator was not corrected from 25.0 g to an oven-dry equivalent despite the 8% initial powder moisture. Gravimetric recovery (%) was therefore calculated as:

$$\text{Gravimetric recovery (\%)} = \frac{\text{recovery dry solid mass (g)}}{25 \text{ g}} \times 100$$

Moisture content of the recovered material was measured gravimetrically by oven drying a known mass and expressing mass loss as a percentage of the initial sample mass. Total ash was determined by incinerating a known mass of dried recovered material until a stable inorganic residue remained, and expressed as a percentage of the original sample mass. GalA was calculated from laboratory-determined equivalent-weight and methoxyl titration data. For each run, the GalA calculation used the NaOH volume associated with the equivalent-weight determination (Z) and the NaOH volume associated with the methoxyl determination (Y), according to the study equation:

$$\text{GalA (\%)} = \frac{176 \times 0.1 \times Z \times 100}{m} + \frac{31 \times 0.1 \times Y \times 100}{m}$$

Where m is sample mass (mg). Equivalent-weight and methoxyl results were recorded for all 13 runs.

Color was measured using a Colorimeter AMT-501 (AMTAST) in the CIELAB system. Before measurement,

we calibrated the instrument using its white and black calibration plates. Instrument specifications for the AMT-501 indicate 8/d illumination/measurement geometry, D65 measurement condition, and a CIE 10° observer. Powder samples were presented to cover the measurement aperture uniformly. The reference color used for calculating total color difference was $L^* = 25.55$, $a^* = 4.08$, and $b^* = 9.81$. The response used for statistical modeling was ΔE^*ab , calculated from the coordinate differences relative to this reference:

$$\Delta E^*ab = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

Accordingly, a larger ΔE^*ab indicates a greater total color difference from the reference and does not represent greater brightness. Lightness is specifically described by L^* . The laboratory worksheet retained the reference coordinates and signed coordinate differences used to obtain ΔE^*ab ; the total color-difference response was therefore interpreted only as distance from the reference color.

2.5. Statistical Analysis and Multi-Response Optimization

Experimental data were analyzed using Design-Expert software (version 13). Separate response surface models were fitted for gravimetric recovery, moisture content, total ash content, galacturonic acid (GalA) content, and instrumental total color difference (ΔE^*ab). Model adequacy was assessed using analysis of variance (ANOVA) to evaluate overall model significance, the significance of individual model terms, and lack of fit. Additional measures of model performance included the coefficient of determination (R^2), adjusted R^2 , predicted R^2 , and adequate precision (Adeq Precision). Statistical significance was established at $P < 0.05$.

Model assumptions were evaluated by examining diagnostic plots for each response, including normal probability plots of externally studentized residuals, residuals versus predicted values, and residuals versus experimental run order. Normal probability plots were examined for approximate linearity, whereas residuals-versus-predicted plots were assessed for systematic patterns and changes in residual dispersion. Residuals-versus-run-order plots were inspected for trends that might indicate temporal or sequential effects. Across all five responses, the residuals generally followed the reference lines in the normal probability plots, with no pronounced funnel-shaped patterns or systematic trends in the residuals-versus-predicted or residuals-versus-run-order plots. These observations suggested no major departures from the assumptions of residual normality, homogeneity of variance, or independence within the experimental design. The individual and interactive effects of ammonium oxalate concentration and extraction temperature were further examined using contour plots and three-dimensional response surface plots. The fitted-model statistics for all responses are summarized in Table 2.

Table 2: Summary of fitted-model statistics for all responses

Response	Model	R^2	Adjusted R^2	Predicted R^2	Adeq Precision	Model p	Lack-of-fit p
Gravimetric recovery	2FI	0.8092	0.7456	0.3805	11.7008	0.0014	0.0647
Moisture	Quadratic	0.5897	0.2966	-1.3564	3.7724	0.1938	0.1040
Total ash	2FI	0.6328	0.5103	0.1422	7.4499	0.0238	0.2799
GalA	Linear	0.7624	0.7149	0.6515	11.6282	0.0008	0.1501
ΔE^*ab	2FI	0.7104	0.6139	0.1487	9.2606	0.0085	0.3036

We performed numerical multi-response optimization using the desirability-function approach (Derringer & Suich, 1980). We constrained ammonium oxalate concentration and extraction temperature to 2–3% (w/v) and 80–90°C, respectively. Gravimetric recovery and GalA were set to maximize, whereas moisture content, total ash, and ΔE^*ab were constrained within their observed experimental ranges. For all factors and responses, lower weight = 1, upper weight = 1, and importance = 3. Thus, equal desirability-shape weights and equal importance were used, and no response received preferential weighting. The factor combination with the highest composite desirability was selected as the model-predicted preferred condition. Table 3 summarizes the numerical optimization criteria and desirability-function settings.

Table 3: Numerical optimization criteria and desirability-function settings

Variable	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
Ammonium oxalate (%)	In range	2.00	3.00	1	1	3
Extraction temperature (°C)	In range	80	90	1	1	3
Gravimetric recovery (%)	Maximize	10.00	37.30	1	1	3
Moisture (%)	In range	11.88	14.90	1	1	3
Total ash (%)	In range	0.11	1.66	1	1	3
GalA (%)	Maximize	52.31	79.31	1	1	3
ΔE^*ab	In range	17.34	20.36	1	1	3

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3. RESULTS & DISCUSSION

3.1. Gravimetric Recovery

Gravimetric recovery from Liberica coffee cascara varied substantially across combinations of ammonium oxalate concentration and extraction temperature. Recovery ranged from 10.0 to 37.3%, with the highest observed value at 3% ammonium oxalate and 80°C and the lowest at 2% and 90°C. Because the response was based on the mass of dried precipitated solid rather than a direct purity measurement, these values are interpreted as gravimetric recovery, not confirmed pure-pectin yield. At the five replicated center points (2.5% ammonium oxalate, 85°C), gravimetric recovery was $19.28 \pm 2.00\%$ (mean $\pm$ SD, $n = 5$), estimating experimental variability under nominally identical conditions. The response surface in Fig. 1 indicated that increasing ammonium oxalate concentration generally increased recovered mass, whereas the effect of temperature depended on concentration.

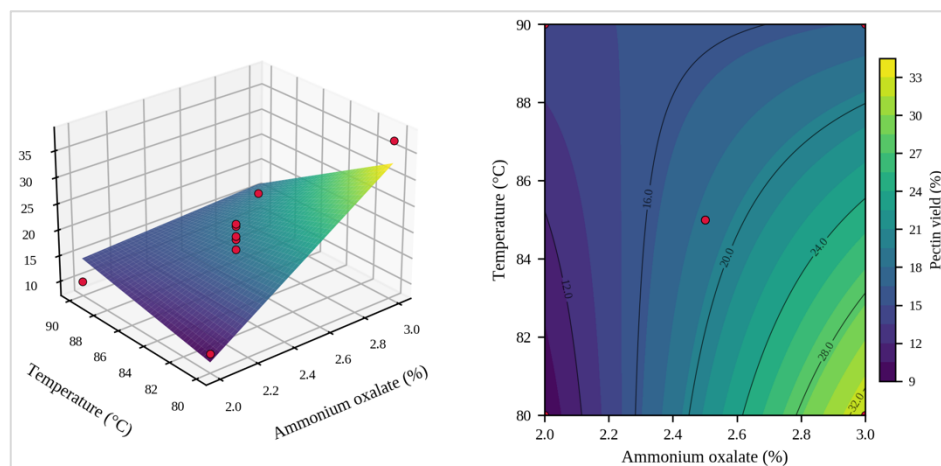


Fig. 1: Three-dimensional response surface and contour plot showing the effects of ammonium oxalate concentration and extraction temperature on gravimetric recovery of dried Liberica coffee-cascara extract.

The 2FI model fitted to gravimetric recovery was statistically significant ($F=12.73$, $P=0.0014$), with $R^2 = 0.8092$, adjusted $R^2 = 0.7456$, and predicted $R^2 = 0.3805$. Ammonium oxalate concentration significantly affected recovery ($P=0.0008$), and the concentration $\times$ temperature interaction was also significant ($P=0.0162$), whereas the temperature main effect did not reach $P<0.05$ ($P=0.0606$). Lack of fit was non-significant ($P=0.0647$), and Adeq Precision was 11.70. The lower predicted R^2 relative to adjusted R^2 indicates limited out-of-sample predictive strength and reinforces the need to treat the selected condition as model-predicted until independent confirmation is available.

The dominant effect of ammonium oxalate concentration can be explained by its ability to disrupt calcium-associated structures within the plant cell wall. Pectic homogalacturonan contains non-esterified galacturonic acid residues that may be cross-linked by Ca^{2+} ions. These ionic junctions contribute to the insolubility of protopectin within the middle lamella and primary cell wall. Oxalate ions can chelate calcium and weaken these junction zones, thereby promoting the release of calcium-bound pectic fractions into the extraction medium (Li et al., 2025; Pattarapisitporn & Noma, 2025). Therefore, increasing ammonium oxalate concentration within the experimental range is expected to expose a greater proportion of accessible pectin to solubilization.

A similar concentration-dependent effect has been reported for ammonium-oxalate extraction from other plant materials. Ma et al. (2021) found that ammonium oxalate concentration contributed significantly to pectic-material recovery from sunflower heads. Reported gravimetric yields from other agro-industrial residues vary substantially with botanical source and extraction method. Therefore, the maximum gravimetric recovery of 37.3% in the present study should not be interpreted as direct evidence of unusually efficient pure-pectin extraction. Coffee-derived outer fruit tissues contain hemicelluloses, proteins, phenolics, minerals, and other carbohydrates that may solubilize or co-precipitate during ethanol recovery. The recovered mass may thus contain both pectic and non-pectic solids, making GalA and other compositional indicators essential for interpreting pectic enrichment.

Accordingly, we interpreted gravimetric recovery alongside GalA and ash rather than as a stand-alone measure of pectin purity. The GalA results discussed below show that the recovered fraction was enriched in galacturonic material, while the ash data indicate inorganic residue; neither measurement, however, establishes complete chemical purity.

Temperature had a less straightforward effect on gravimetric recovery. Although moderate heating promotes diffusion, solvent penetration, and cell-wall softening, temperatures beyond the optimal range may accelerate pectin degradation. Thermal treatment facilitates pectin release by weakening non-covalent interactions within the cell-wall matrix, but overly severe conditions can lead to depolymerization, demethylation, and chain cleavage (Muñoz-Almagro et al., 2018; Pattarapisitporn & Noma, 2025). Short-chain fragments generated during

degradation may remain soluble after ethanol addition and consequently fail to contribute to the gravimetric pectin yield. This provides a plausible explanation for the higher recovery at 80°C than in some treatments performed at higher temperatures.

3.2. Moisture Content

Moisture content varied from 11.88 to 14.90% across the experimental treatments. The lowest value, 11.88%, was obtained at the axial ammonium oxalate concentration of 3.2071% and 85°C, whereas the highest moisture content was recorded at 2% ammonium oxalate and 80°C. Unlike pectin yield, moisture content did not exhibit a clear linear response to either independent variable. Instead, the response surface in Fig. 2 showed curvature, with comparatively lower moisture values around the central temperature region.

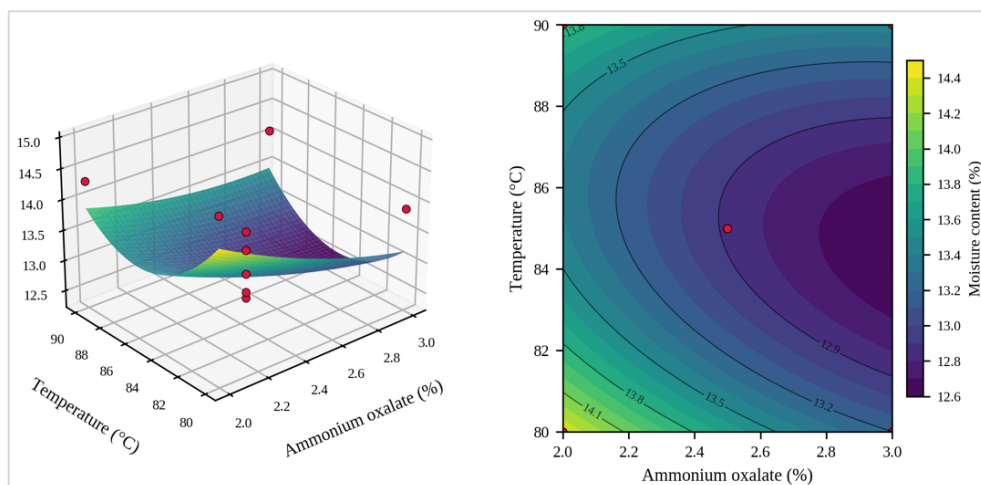


Fig. 2: Three-dimensional response surface and contour plot showing the effects of ammonium oxalate concentration and extraction temperature on pectin moisture content.

The quadratic model for moisture content was not statistically significant overall ($P=0.1938$) and had an R^2 of 0.5897, indicating that the model explained approximately 58.97% of the variability in moisture content. The quadratic temperature term was significant ($P=0.0324$), however, supporting the curved response observed across the experimental domain. Lack of fit was non-significant ($P=0.1040$). The relatively modest R^2 compared with the yield and galacturonic acid models suggests that factors other than ammonium oxalate concentration and extraction temperature contributed substantially to the final water content of the pectin.

This finding is reasonable because moisture content depends not only on extraction conditions but also on the recovered polymer's structural characteristics and the effectiveness of post-extraction drying. Pectin is strongly hydrophilic because of its numerous polar functional groups, and different molecular structures may retain different amounts of bound or associated water. Ma et al. (2021) showed that drying procedures can significantly alter the physical characteristics of ammonium-oxalate-extracted pectin, while Li et al. (2025) reported a high water-holding capacity for pectin extracted with ammonium oxalate. The final moisture value may therefore partly reflect the hydration properties of the recovered material rather than the extraction variables alone.

For comparison, Sarah et al. (2022) reported approximately 6% moisture in optimized cocoa-pod-husk pectin, whereas Sontakke et al. (2024) reported approximately 7.758% for pectin derived from jackfruit waste. The values obtained from Liberica coffee husk were higher, particularly at the selected multi-response condition, which contained 13.90% moisture. This represents a main quality limitation of the present product.

The elevated moisture content does not necessarily indicate unsuitable extraction chemistry. Rather, it suggests that the post-extraction drying protocol requires further refinement. The present study used oven drying at 45°C for 15 h, a relatively mild treatment intended to limit thermal damage to the polymer. Such mild drying may preserve pectin structure but may not remove sufficient residual moisture. Longer drying, controlled-air drying, vacuum drying, freeze-drying, or optimizing sample thickness and particle size could reduce final moisture without causing excessive thermal degradation.

Moisture becomes particularly important during storage. Higher residual moisture may facilitate microbial deterioration, enzyme activity, agglomeration, and changes in functional properties during storage. The minimum value of 11.88% approached the benchmark adopted in this study, whereas the optimum multi-response treatment remained above it. Thus, unlike yield or galacturonic acid content, moisture appears to be a parameter that could potentially be improved through post-extraction process modification without fundamentally changing the preferred chemical extraction conditions.

3.3. Ash Content

Ash content remained low throughout the experimental domain, ranging from 0.11 to 1.66%. The lowest ash content (0.11%) occurred at 2% ammonium oxalate and 90°C, while the maximum (1.66%) occurred at 3.2071% ammonium oxalate and 85°C. As shown by the response surface in Fig. 3, increasing ammonium oxalate concentration tended to increase ash content, whereas temperature produced a comparatively smaller influence.

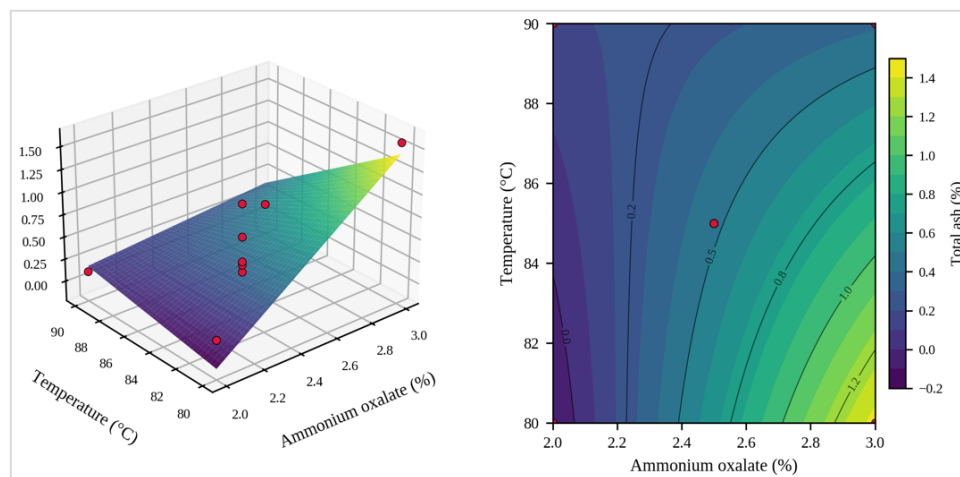


Fig. 3: Three-dimensional response surface and contour plot showing the effects of ammonium oxalate concentration and extraction temperature on pectin ash content.

The 2FI model was statistically significant ($F = 5.15$, $P = 0.0238$). Ammonium oxalate concentration was the only significant factor affecting ash content ($P = 0.0104$), while temperature and the concentration–temperature interaction were not significant. The model explained 63.28% of the experimental variation ($R^2 = 0.6328$). The lack of fit was non-significant ($P = 0.2799$), and Adeq Precision was 7.45, indicating sufficient discriminatory ability within the investigated region. The five center-point ash measurements (0.20, 0.24, 0.52, 0.12, and 0.89%) showed appreciable analytical dispersion despite identical nominal extraction settings. This variability is reflected in the pure-error component and was retained rather than excluded because no documented analytical error or procedural deviation was identified. Therefore, the center-point variation is acknowledged as a limitation of ash-response precision.

The increase in ash content at higher extractant concentrations may reflect a greater release or retention of inorganic components during extraction. Coffee husk naturally contains mineral matter, and increasing extraction intensity may facilitate the transfer of soluble inorganic constituents into the liquid phase. Some salts may subsequently remain associated with the precipitated material despite ethanol washing. At the same time, ammonium oxalate can bind calcium, and filtration may separate some calcium-containing material. The resulting ash level therefore reflects a balance between mineral solubilization, chelation, filtration, washing, and precipitation.

The ash concentrations measured in this study were low when compared with many pectins derived from agricultural residues. Sarah et al. (2022) reported approximately 8% ash in microwave-assisted cocoa-pod-husk pectin, while Valdivia-Rivera et al. (2021) obtained approximately 3.53% for Haden mango-peel pectin. Pectin recovered from rambutan peel through pulsed-electric-field-assisted extraction has been reported with an ash content of approximately 4.98% (Wicaksono et al., 2026). Han et al. (2024) also observed that ammonium oxalate could produce comparatively low-ash pectin from sweet potato residue.

At the model-predicted preferred condition, ash content was 1.57%. This relatively low ash value indicates that the high recovered mass was not accompanied by proportionally high inorganic residue, although it does not establish pectin purity on its own. This finding matters because a high gravimetric yield could otherwise be interpreted as substantial mineral co-precipitation. The combination of high yield, high galacturonic acid content, and relatively low ash therefore strengthens the interpretation that a considerable proportion of the recovered solid consisted of pectic material (Tesfaye et al., 2023).

The minimum ash value was obtained under a different treatment from the selected overall optimum. This illustrates a principal trade-off in multi-response optimization. Although 2% ammonium oxalate and 90°C minimized ash, that condition did not simultaneously provide the highest yield or galacturonic acid content. Minimizing ash alone would therefore have led to an extraction condition with less favorable overall performance.

3.4. Galacturonic Acid Content

Galacturonic acid content ranged from 52.31 to 79.32% and was strongly influenced by ammonium oxalate concentration. The maximum value of 79.32% occurred at 3.2071% ammonium oxalate and 85°C, whereas the

lowest value of 52.31% was obtained at 2% ammonium oxalate and 90°C. The response surface in Fig. 4 showed a pronounced increase in GalA content with increasing ammonium oxalate concentration, while changes associated with extraction temperature were comparatively limited.

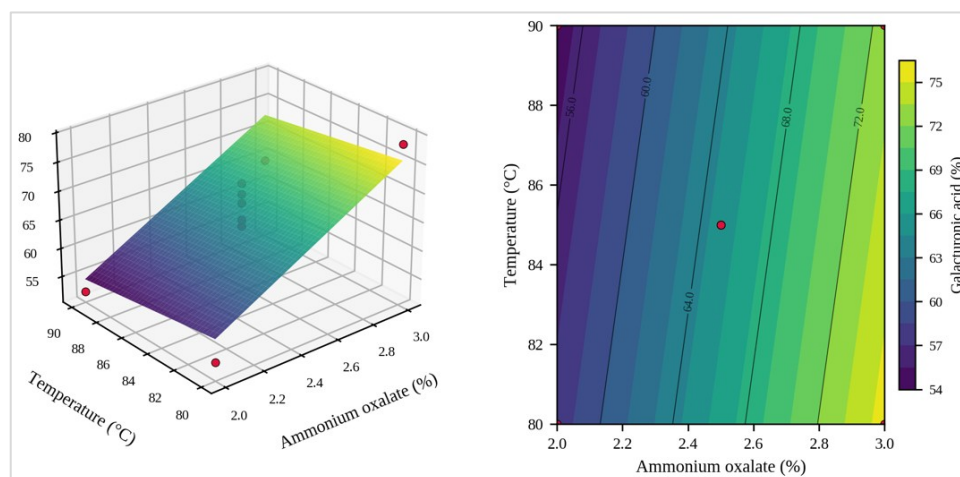


Fig. 4: Three-dimensional response surface and contour plot showing the effects of ammonium oxalate concentration and extraction temperature on galacturonic acid content.

The fitted model was statistically significant ($P=0.0008$) and explained 76.24% of the experimental variation ($R^2 = 0.7624$). Ammonium oxalate concentration exerted a highly significant effect ($P=0.0002$), while the temperature term was not significant ($P=0.3696$). The lack-of-fit value was also non-significant ($P=0.1501$). These results confirm that extractant concentration was the main factor controlling GalA enrichment in the recovered material.

The response can be understood from the structural organization of pectin. Homogalacturonan is composed mainly of α -(1 $\rightarrow$ 4)-linked D-galacturonic acid residues. Regions containing non-esterified carboxyl groups can interact with calcium ions, forming ionic junction zones. Because ammonium oxalate removes calcium from these structures, increasing the chelating-agent concentration can preferentially release homogalacturonan-rich material. The extraction process may therefore increase both the total amount of recovered pectin and the relative abundance of galacturonic acid in the recovered fraction.

Comparable results have been reported for other ammonium-oxalate-extracted materials. Ma et al. (2021) obtained sunflower-head pectin containing approximately 76.2% GalA. Pectic fractions from other botanical matrices using ammonium oxalate have shown greater compositional variation, showing that botanical source remains an important determinant of final polymer composition (Ognyanov et al., 2018; Banjanac et al., 2026). The maximum value obtained from Liberica coffee husk, 79.32%, was therefore within the upper range reported for several chelator-extracted pectins.

The selected multi-response treatment produced 78.45% GalA, only slightly below the maximum individual observation. This value was also above the commonly cited minimum level used to indicate a sufficiently high galacturonic component in commercial pectin (Alpizar-Reyes et al., 2022; Valdivia-Rivera et al., 2021). Compositionally, this is one of the study's strongest outcomes.

However, GalA content should not be considered a complete substitute for structural characterization. Two pectin samples with similar GalA concentrations may have very different molecular weight distributions, degrees of esterification, degrees of acetylation, neutral-sugar side chains, and functional properties. Therefore, the present result indicates that the recovered material was rich in galacturonic acid but does not, by itself, establish whether it behaved as high-methoxyl or low-methoxyl pectin or whether its gel-forming properties were comparable to commercial citrus or apple pectin.

3.5. Color Difference

The instrumental total color-difference response (ΔE^*_{ab}) ranged from 17.34 to 20.36. The highest ΔE^*_{ab} (20.36) was recorded at 3% ammonium oxalate and 80°C, whereas the lowest (17.34) occurred at 2.5% ammonium oxalate and approximately 77.93°C. These values represent the magnitude of total color difference from the reference ($L^* = 25.55$, $a^* = 4.08$, $b^* = 9.81$) and must not be interpreted as brightness. The color model was significant ($P = 0.0085$); ammonium oxalate concentration was significant ($P = 0.0044$); temperature was not significant ($P = 0.2203$); and the interaction was significant ($P = 0.0357$). The model explained 71.04% of the response variation ($R^2 = 0.7104$), with adjusted $R^2 = 0.6139$, predicted $R^2 = 0.1487$, a non-significant lack of fit ($P = 0.3036$), and Adeq Precision = 9.26. Fig. 5 shows response surface and contour plots of the effects of ammonium oxalate concentration and extraction temperature on the instrumental color response of Liberica coffee-husk pectin.

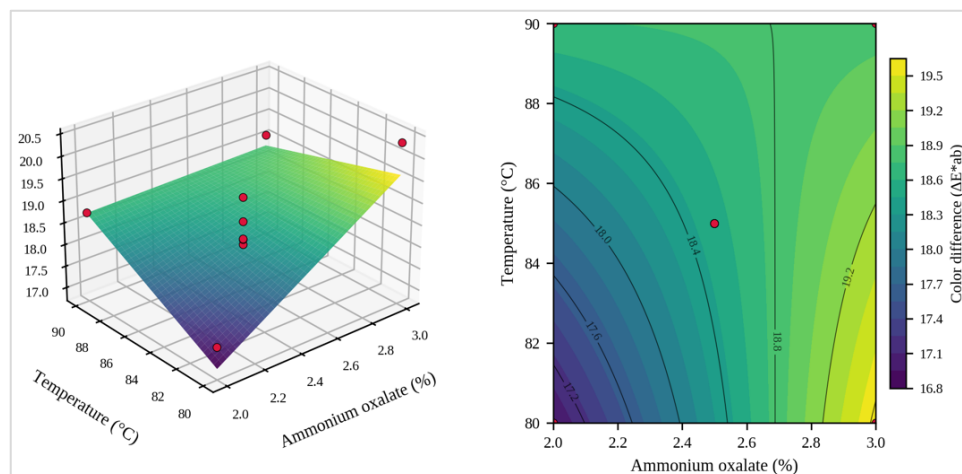


Fig. 5: Three-dimensional response surface and contour plot showing the effects of ammonium oxalate concentration and extraction temperature on the instrumental color response of Liberica coffee-husk pectin.

Color is particularly relevant for pectin recovered from coffee husk because the source material contains phenolic compounds and naturally colored constituents that may be extracted together with polysaccharides (Cangussu et al., 2021; Hoseini et al., 2021). Chlorogenic acids, tannins, and other chromophoric compounds may contribute to the final precipitate's color, while oxidation and thermal reactions during extraction can further alter the product's appearance. Tan et al. (2026) showed that thermal processing can significantly influence the physicochemical and color characteristics of coffee-husk-derived materials.

The largest ΔE^*ab coincided with the model-predicted preferred condition, indicating the greatest overall color distance from the selected reference among the observed runs; this is neither intrinsically favorable nor equivalent to greater lightness. In CIELAB, L^* specifically represents lightness, whereas a^* and b^* describe chromatic coordinates. The study therefore interprets ΔE^*ab only as total color difference. Future analyses should retain and report complete sample-specific L^* , a^* , and b^* coordinates whenever color appearance is a critical product-quality attribute.

The available laboratory worksheet retained the reference L^* , a^* , and b^* coordinates and the signed coordinate differences used to calculate ΔE^*ab , but the manuscript does not reconstruct sample coordinates beyond the directly recorded data. This limitation does not alter the ΔE^*ab modeling but restricts interpretation of whether color changes were driven primarily by lightness or chromaticity.

3.6. Multi-response Optimization

The individual response surfaces showed that no single extraction condition produced the most favorable value for every response. We therefore used numerical desirability optimization to identify a compromise among the predefined objectives. We maximized gravimetric recovery and GalA, while keeping moisture, ash, and ΔE^*ab within their observed ranges. Lower and upper desirability weights were 1, and importance was 3 for every factor and response, so all included criteria contributed equally to the composite desirability.

Two acceptable numerical solutions were generated. The selected model-predicted preferred condition was 3.00% (w/v) ammonium oxalate and 80.00°C, with composite desirability 0.858. At this condition, the models predicted 33.160% gravimetric recovery, 13.202% moisture, 1.444% ash, 75.700% GalA, and $\Delta E^*ab = 19.644$. The original CCD Run 13, conducted at the same factor combination and used in model construction, produced 37.30% recovery, 13.90% moisture, 1.57% ash, 78.45% GalA, and $\Delta E^*ab = 20.36$. These observed values are presented only as the original design observation at the selected factor combination and not as an independent validation experiment.

The desirability value of 0.858 indicates that 3% ammonium oxalate at 80°C provided a comparatively strong compromise among the specified objectives within the investigated factor space. We calculated the signed relative deviation in Table 4 as $[(\text{observed} - \text{predicted})/\text{predicted}] \times 100$; positive values therefore indicate that the original CCD observation exceeded the corresponding model prediction. The 95% prediction intervals quantify the uncertainty expected for a future single observation under the fitted models. All original Run 13 responses fell within their respective prediction intervals, but we do not treat this agreement as statistical validation because Run 13 contributed to model fitting. For a future independent confirmation study, a prespecified acceptance criterion would be that the mean response from replicated independent extractions at 3% and 80°C falls within the corresponding 95% prediction interval, together with acceptable repeatability (Sabater et al., 2018).

This distinction is important when translating laboratory optimization into application-specific process design. If purity were given the highest priority, the desirability function could assign greater weight to GalA and ash. If the intended use were a dark-colored food or coating, color might receive relatively little weight. Conversely, applications requiring nearly colorless pectin could treat lightness as a critical quality constraint. Similarly, production aimed

primarily at maximizing biomass utilization might assign greater importance to yield than would a process designed to produce a highly purified food hydrocolloid. Multi-response optimization therefore does not generate a universally optimal condition; rather, it identifies the most favorable condition for the priorities specified in the model (Podetti et al., 2023; John & Maharaj, 2025).

Table 4: Model predictions, 95% prediction intervals, and the corresponding original CCD observation at 3% ammonium oxalate and 80°C

Response	Model prediction	95% prediction interval	Original CCD Run 13	Signed relative deviation (%)
Yield (%)	33.160	22.665-43.657	37.30	+12.5
Moisture (%)	13.202	11.064-15.340	13.90	+5.3
Total ash (%)	1.444	0.350-2.539	1.57	+8.7
Galacturonic acid (%)	75.700	63.945-87.452	78.45	+3.6
ΔE^{*ab}	19.644	18.147-21.141	20.36	+3.6

Comparing predicted and experimental values also provides useful information about model performance. Although the observed responses followed the models' predicted pattern, the experimental yield and GalA content were somewhat higher than their predicted values. A separate replicated validation experiment at the predicted optimum would strengthen the evidence for predictive reliability. Response-surface studies commonly verify the optimized condition through additional experiments not used to construct the original regression models (Ma et al., 2021; John & Maharaj, 2025). Such validation would be particularly valuable before transferring the process to larger-scale operation (Riveros-Gomez et al., 2022).

No independent confirmation experiment was performed outside the original CCD. This is a limitation of the present exploratory optimization study. Accordingly, we describe the selected condition throughout as the model-predicted preferred condition rather than a validated optimum, and we do not use original CCD Run 13 as validation evidence. Independent replicated confirmation at 3% ammonium oxalate and 80°C is required in subsequent work before claims of predictive validation or process transfer are made.

4. CONCLUSION

Ammonium oxalate concentration and extraction temperature influenced the gravimetric recovery and physicochemical characteristics of pectic material obtained from Liberica coffee outer fruit tissues. Multi-response optimization identified 3% (w/v) ammonium oxalate at 80°C as the model-predicted preferred extraction condition within the investigated experimental range, with a composite desirability of 0.858. The corresponding experimental run included in the original central composite design yielded 37.30% gravimetric recovery, 78.45% galacturonic acid (GalA), 1.57% ash, and 13.90% moisture. However, gravimetric recovery may include co-precipitated non-pectic constituents, and the selected extraction condition has not been independently validated. Therefore, these findings demonstrate the potential of Liberica coffee outer fruit tissues as a source of GalA-rich pectic material and provide a preliminary basis for further extraction process development. Future studies should independently validate the model-predicted conditions, improve post-extraction drying procedures, determine residual oxalate levels, and evaluate the structural, functional, and safety characteristics of the recovered material. Further investigation of process scalability and economic feasibility is also needed to assess its potential for practical applications.

Declarations

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Data Availability: All data supporting the findings of this study are included within the article.

Ethics Statement: This study involved no experiments on humans or animals; therefore, ethical approval was not required.

Author's Contribution: Reta: Conceptualization, Methodology, Investigation, Data curation, Formal analysis,

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