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A Novel Barium Chloride-Catalyzed Approach for Direct Conversion of Waste Leaf Biomass to Methyl Butanoate

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Abstract. *The transition toward sustainable and circular bioeconomies requires environmentally benign and resource-efficient routes for producing value-added chemicals from renewable feedstocks. This study presents a rapid and novel approach for synthesising methyl butanoate via a barium chloride-catalyzed thermal hydrolytic process using Gmelina arborea leaf biomass as a low-cost lignocellulosic resource. Unlike conventional esterification methods that depend on refined substrates, corrosive catalysts, and extended reaction times, this process operates under mild conditions (80 °C, atmospheric pressure) with short reaction durations (10–50 minutes). Results show that the highest methyl butanoate yield (≈13.52% and ~900 mg/g) is achieved at 10 minutes, demonstrating good reproducibility (RSD ≈ 6%). ANOVA confirms that reaction time significantly influences yield ($p < 0.05$), with longer durations reducing performance due to competing reactions and equilibrium constraints. Gas Chromatography–Mass Spectrometry (GC–MS) analysis validates ester formation and composition. This work addresses a key research gap by enabling direct conversion of waste biomass into short-chain esters through a simple, rapid, and energy-efficient pathway. The approach aligns with Sustainable Development Goals: SDG 7 (Affordable and Clean Energy) through biofuel development, SDG 9 (Industry, Innovation and Infrastructure) via process innovation, SDG 12 (Responsible Consumption and Production) through biomass valorization, and SDG 13 (Climate Action) by promoting low-carbon processes. Overall, the study establishes a sustainable alternative for ester production with reduced environmental impact and operational complexity.*

Keywords: *Methyl Butanoate; Gmelina Arborea; Biomass Valorization; Thermal Hydrolytic Process; Barium Chloride Catalysis*

1. Introduction

Methyl butanoate (also known as methyl butyrate) is a colourless, volatile ester characterized by a strong fruity aroma reminiscent of apple and pineapple. Owing to this pleasant scent, it is widely employed in the flavour and fragrance industry for the formulation of artificial fruit essences, perfumes, and cosmetic products (Tofalo & Perpetuini, 2025; Sen, 2015). Beyond its sensory applications, methyl butanoate has notable industrial relevance, serving as an effective solvent for nitrocellulose in coatings, printing inks, and extraction processes due to its favourable solvency properties (Byrne, 2017). Additionally, it has attracted increasing attention as a relatively environmentally benign alternative to petroleum-derived solvents, being both biodegradable and less toxic (Prasad et al., 2022), thereby contributing to safer chemical practices aligned with United Nations SDG 12: Responsible Consumption and Production. In the energy and combustion field, methyl butanoate is extensively used as a surrogate compound for biodiesel. Its low molecular weight, good volatility, and well-defined chemical structure make it particularly suitable for studying ignition characteristics, reaction

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mechanisms, and emission profiles in combustion systems (Da Costa et al., 2022; Jiang et al., 2020). Consequently, it is frequently adopted as a model compound in combustion kinetics, cetane number evaluation, and biofuel formulation studies (Dooley et al., 2012; Diévart et al., 2012; Miller et al., 2013). These applications support the transition toward cleaner and more sustainable energy systems, in line with SDG 7: Affordable and Clean Energy. Furthermore, its role as a representative ester in catalytic biomass conversion research aids in optimizing renewable production pathways, advancing circular bioeconomy strategies, and reinforcing the objectives of SDG 13: Climate Action by reducing reliance on fossil-based resources and lowering greenhouse gas emissions.

Methyl butanoate ($C_5H_{10}O_2$), also referred to as methyl butyrate, is a short-chain fatty acid methyl ester (FAME) produced through the esterification of butyric (butanoic) acid with methanol (Zheng et al., 2021; Plácido & Zhang, 2018). Conventionally, it is synthesized via acid-catalyzed esterification, in which butanoic acid reacts with methanol in the presence of a strong mineral acid catalyst, typically sulfuric acid (H_2SO_4), yielding methyl butanoate and a small amount of water as a by-product (Dange & Rathod, 2017; Severini et al., 2016). This reaction is reversible, with the backward reaction involving hydrolysis of the ester to regenerate the parent acid and alcohol. The equilibrium nature of the process, combined with the corrosive characteristics of strong acid catalysts, presents operational and material challenges in industrial applications. Alternatively, methyl butanoate can be synthesized via transesterification of triglycerides such as glyceryl tributanoate (tributyrin) with methanol. This process typically employs strong base catalysts, such as sodium hydroxide (NaOH) or potassium hydroxide (KOH), and produces glycerol as a co-product. Although acid catalysts (e.g., H_2SO_4 or HCl) can also facilitate transesterification, base-catalyzed systems are significantly more efficient, with reaction rates reported to be up to 4000 times faster (Mishra & Goswami, 2018). The esterification and transesterification pathways are commonly represented in Equations 1 and 2, respectively.

$$C_4H_{10}O_2 + CH_3OH \overset{H_2SO_4}{\Longleftrightarrow} CH_3O_2C_4H_9 + H_2O \tag{1}$$

$$C_{15}H_{26}O_6 + 3CH_3OH \overset{Catalyst}{\Longleftrightarrow} 3CH_3O_2C_4H_9 + C_3H_8O_3 \tag{2}$$

This transesterification reaction does not produce glycerol as a co-product if a solid base catalyst such as CaO or MgO is used, a small quantity of water is produced (Ibrahim & Bugaje, 2018; Ibrahim et al.,2018; Ibrahim et al., 2016a; Ibrahim et al., 2016b), and sometimes hydrogen, depending on the size of the triglyceride. The reaction is as presented in Equation 3.

$$C_{15}H_{32}O_6 + 3CH_3OH \overset{Catalyst}{\Longleftrightarrow} 2CH_3O_2C_4H_9 + CH_3O_2C_5H_{11} + O_3 \tag{3}$$

Despite extensive studies on the synthesis of methyl butanoate, existing production routes are largely dominated by conventional esterification, transesterification, and biocatalytic processes, which often rely on pure chemical feedstocks such as butyric acid, methanol, or triglycerides and employ strong acid/base catalysts or specialized enzymes. While these methods can achieve high yields, they are typically associated with drawbacks such as corrosive reaction environments, high energy consumption, long reaction times, catalyst recovery challenges, and dependence on refined substrates. Furthermore, although recent advancements have explored heterogeneous and enzymatic catalysis, there remains limited research on the direct conversion of low-cost lignocellulosic biomass, particularly waste leaf materials, into short-chain esters such as methyl butanoate. Existing biomass-based studies primarily focus on bioethanol, biodiesel, or platform chemicals, with little emphasis on rapid ester formation via mild thermal hydrolytic pathways.

In addition, the use of unconventional catalysts such as barium chloride in biomass-to-ester conversion systems is scarcely reported, and the mechanistic role of such catalysts in promoting ester formation under aqueous thermal conditions is not well understood. There is also a lack of systematic investigation into the influence of short reaction times on ester yield, selectivity, and reproducibility within such systems. Therefore, a clear research gap exists in the development of a simple, rapid, and sustainable process for methyl butanoate production directly from biomass using mild conditions and inexpensive catalysts. This study addresses this gap by exploring a barium chloride-catalyzed thermal hydrolytic route using *Gmelina arborea* leaf biomass, aiming to provide an alternative pathway that minimizes processing complexity, reduces reaction time, and utilizes renewable feedstock.

This study aims to develop a simple, rapid, and sustainable method for the production of methyl butanoate from *Gmelina arborea* leaf biomass via a barium chloride-catalyzed thermal hydrolytic process. To achieve this, the study seeks to (i) utilize waste *Gmelina arborea* leaves as a renewable feedstock for ester production, (ii) investigate the effect of reaction time on the yield and selectivity of methyl butanoate under mild thermal conditions, (iii) evaluate the reproducibility and statistical significance of the process using appropriate analytical tools, and (iv) characterize the chemical composition of the products using Gas Chromatography-Mass Spectrometry (GC-MS). Ultimately, the work intends to establish an efficient and environmentally benign alternative route for methyl butanoate synthesis with reduced processing time and operational complexity.

2. Methods

The materials employed in this study included dried *Gmelina arborea* leaves, a ceramic mortar and pestle, 250 and 300 µm sieves, a 1000 mL conical flask, a 1000 mL beaker, a 500 mL separating funnel, a thermometer, distilled water, a Gallenkamp hot plate equipped with a magnetic stirrer, filter cloth, and filter paper. The analytical reagents used were barium chloride and sodium sulphate.

Dried *Gmelina arborea* leaves were obtained from the Kaduna Polytechnic premises. The leaves were manually sorted to remove extraneous materials, washed with distilled water to remove impurities, and air-dried at ambient conditions, following the procedures previously reported by Ibrahim et al. (2025) & Ibrahim et al., (2022). After drying, they were ground using a ceramic mortar and pestle. A catalytic solution was prepared by dissolving 0.5% (w/w) barium chloride (0.25 g) in 500 mL of distilled water in a 1000 mL conical flask. Subsequently, 50 g of the pulverized leaf material was introduced into the solution. The mixture was heated to 80 °C and stirred continuously at 2000 rpm using a magnetic stirrer for 10 minutes in line with the method described by (Ibrahim et al., 2026; Ali et al., 2026).

Upon completion of the reaction, the mixture was first filtered through cotton wool into a 1000 mL beaker, then further filtered through filter paper. The resulting filtrate was transferred into a separating funnel, and 0.2% (w/w) sodium sulphate (based on the filtrate mass) was added following the procedure of Ali and Ibrahim (2023). The mixture was allowed to stand for several hours to enable phase separation (Ibrahim and Ali, 2023a). The organic phase was subsequently dried over sodium sulphate, collected, and weighed.

The experiment was repeated at reaction times of 20, 30, 40, and 50 minutes, with each condition conducted in duplicate. Samples obtained from each run were analysed using Gas

Chromatography-Mass Spectrometry (GC-MS) to determine the chemical composition and identify individual constituents of the products. A process flow diagram illustrating the production of methyl butanoate via barium chloride-catalysed thermal hydrolysis of *Gmelina arborea* leaves is presented in Figure 1.

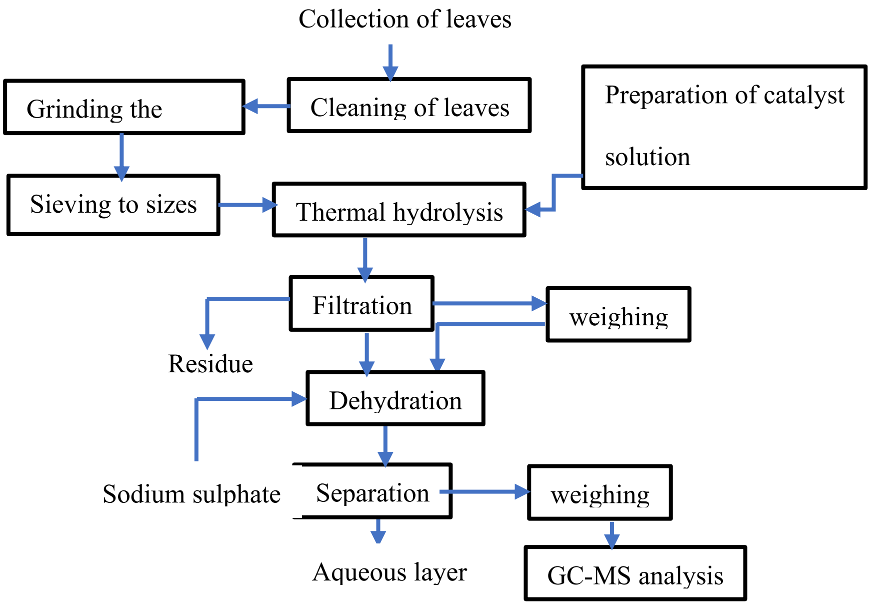

Figure 1. Flow process diagram for synthesis of methyl butanoate

For derivatization, 200 µL of the sample solution (analyte) was combined with 100 µL of trimethyl sulfonium hydroxide (TMSH) and 20 µL of triethylamine (TEA), and the mixture was heated at 70 °C for 1 hour in sealed vials before GC-MS analysis following the procedure of (Ibrahim et al., 2021). The analysis was performed using a Varian 3800/4000 gas chromatograph-mass spectrometer equipped with a DB-5 capillary column (30 m × 0.25 mm × 0.25 µm). Nitrogen was used as the carrier gas at a column head pressure of 10 psi. The oven temperature program started at 100 °C with a 3-minute hold, followed by a temperature ramp of 8 °C min^{-1} to 300 °C. The transfer line temperature was maintained at 290 °C, while the VG 7070E magnetic sector mass spectrometer operated under electron impact ionization. Mass spectra were acquired over an m/z range of 40-800 at a scan rate of 20 scans per second, with a solvent delay of 330 seconds to prevent detector saturation. Continuous signal monitoring ensured accurate and reliable detection.

3. Results and Discussion

Table 1 presents the weights of the dehydrated products (filtrates) obtained from two experimental runs, alongside the corresponding yields of methyl butanoate (MB, %) as determined by GC-MS analysis. The table also includes the average percentage yield, providing a basis for assessing reproducibility and the influence of reaction time on product formation. From the results, reaction time exhibits a significant effect on both filtrate weight and methyl butanoate yield. At 10 minutes, the system recorded the highest average MB yield (13.52%), accompanied by relatively high filtrate masses in both runs (343.51 g and 296.55 g). This suggests that the formation of methyl butanoate is favoured at shorter reaction times, possibly due to minimal secondary reactions or degradation pathways.

Table 1. The Yields of methyl butanoate from Gmelina. arborea leaf

Time (min)	1ST Run Filtrate (g)	2nd Run Filtrate (g)	1st Run MB (%)	2nd Run MB (%)	Ave (%)
10	343.51	296.55	13.10	13.94	13.52
20	375.00	278.90	2.00	2.46	2.23
30	153.85	105.87	5.20	6.08	5.64
40	150.00	103.11	4.00	4.70	4.35
50	96.49	72.01	5.70	6.38	6.04

However, at 20 minutes, a sharp decline in MB yield is observed (average 2.23%) despite the highest filtrate weight in the first run (375.00 g). This indicates that increased product mass does not necessarily correspond to higher methyl butanoate formation, implying the presence of other non-target compounds in the filtrate. The drastic reduction in MB yield may be attributed to equilibrium limitations or competing side reactions. Between 30 and 50 minutes, the MB yield shows moderate recovery and stabilization, ranging from 4.35% to 6.04%. Although the filtrate weights decrease progressively over this interval, the MB yield becomes relatively more consistent, suggesting improved selectivity towards methyl butanoate at longer reaction times compared to the 20-minute condition.

In terms of reproducibility, the two runs show reasonable agreement across all time intervals, with only slight variations in both filtrate weights and MB yields. This consistency indicates good experimental reliability. Overall, the results suggest that optimal methyl butanoate production occurs at shorter reaction times (around 10 minutes), while prolonged reaction times may lead to reduced yields due to side reactions, product degradation, or equilibrium effects.

The plot in Figure 1 illustrates the variation of specific yield of methyl butanoate (mg/g) with reaction time for two experimental runs under barium chloride-catalysed thermal hydrolytic conditions. Overall, the trends from both runs are closely aligned, indicating good reproducibility of the process. At 10 minutes, the highest specific yield is observed in both runs (≈900 mg/g for the 1st run and ≈830 mg/g for the 2nd run). This clearly indicates that methyl butanoate formation is highly favoured at shorter reaction times, suggesting rapid initial conversion of reactive intermediates into the target ester. A sharp decline in yield occurs at 20 minutes, dropping to approximately 150 mg/g (1st run) and 140 mg/g (2nd run). This significant reduction implies that methyl butanoate is either being consumed in secondary reactions, undergoing hydrolysis, or being transformed into other products as the reaction progresses. It may also reflect the reversible nature of ester formation, where prolonged exposure promotes backward reactions. At 30 minutes, a slight recovery in yield is evident (≈165 mg/g and ≈130 mg/g), suggesting a temporary shift in reaction dynamics, possibly due to intermediate reformation or changes in reaction equilibrium. However, this increase is not sustained. From 40 to 50 minutes, the yield gradually declines and stabilizes around 100-120 mg/g. This plateau indicates that the system is approaching equilibrium or that most of the reactive species have been depleted, limiting further formation of methyl butanoate. In terms of reproducibility, the close proximity of the two curves across all time points demonstrates consistent experimental performance, with only minor deviations.

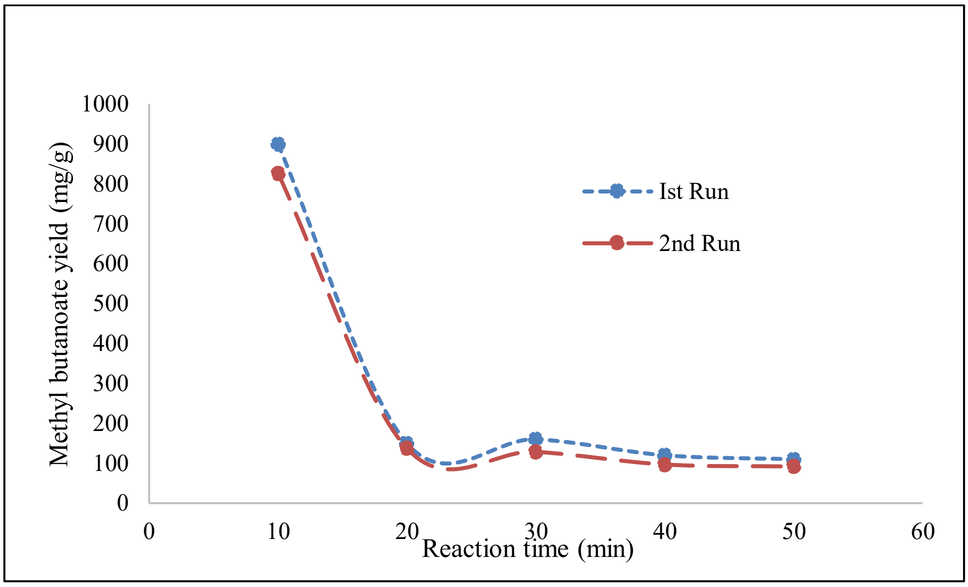

Figure 2.Methyl butanoate yield vs reaction time

Overall, the results indicate that the optimal reaction time for maximizing methyl butanoate yield is around 10 minutes. Prolonged reaction times are detrimental to yield, likely due to competing side reactions, product degradation, or equilibrium limitations inherent in the hydrolytic system.

3.1. Reproducibility Analysis of Methyl Butanoate

To assess reproducibility, the following statistical parameters were evaluated: Mean (Average), Absolute difference (AD), Standard deviation (SD), and Relative standard deviation (RSD, %). The reproducibility of methyl butanoate yield varies significantly with reaction time. At 10 and 20 minutes, the process exhibits good reproducibility, with low RSD values (~6%) as presented in Table 2. This indicates strong agreement between replicate experiments despite the large absolute yield at 10 min. The consistency suggests stable reaction kinetics at the early stage of the thermal hydrolytic process. At 30 and 40 minutes, reproducibility decreases noticeably, with RSD values rising to about 15%. This higher variability may be attributed to: increased contribution of secondary reactions, possible decomposition or transformation of methyl butanoate, and Sensitivity of intermediate species to slight experimental variations. At 50 minutes, reproducibility improves slightly (RSD ≈ 12.69%) but remains poorer than at shorter reaction times. This suggests that although the system begins to stabilize, competing reactions or equilibrium limitations still affect consistency.

Table 2. Reproducibility results

Time (min)	1st Run	2nd Run(mg/g)	Mean	AD	SD	RSD (%)
10	900.00	826.78	863.39	73.22	51.78	6.00
20	150.00	137.22	143.61	12.78	9.03	6.29
30	160.00	128.74	144.37	31.26	22.11	15.31
40	120.00	96.92	108.46	23.08	16.32	15.04
50	110.00	91.88	100.94	18.12	12.81	12.69

Overall, the best reproducibility is 10-20 min; the least reproducibility is 30-40 min. Reproducibility decreases with increasing reaction time, then slightly stabilizes. This behaviour indicates that the optimal reaction window for both high yield and reproducibility is at shorter reaction times, particularly around 10-20 minutes. Prolonged reaction time introduces variability likely due to competing reaction pathways and product instability under sustained thermal conditions.

3.2 ANOVA Analysis of Methyl Butanoate Yield vs Reaction Time

To evaluate whether reaction time significantly affects the yield, a one-way ANOVA was conducted using the replicate data presented in Table 3. The calculated F-value (137.43) shown in Table 4 is exceptionally large, indicating substantial variation between group means relative to the within-group variability. At a typical significance level ($\alpha = 0.05$), the critical F-value ($F_{4,5} \approx 5.19$) is far lower than the calculated F-value; therefore, the result is statistically significant.

Table 3. Summary Statistics

Time (min)	n	Mean (mg/g)	Variance (mg/g)^2
10	2	863.39	2681.17
20	2	143.61	81.54
30	2	144.37	488.87
40	2	108.46	266.37
50	2	100.94	164.10

The ANOVA results clearly demonstrate that reaction time has a highly significant effect on the yield of methyl butanoate. The extremely high between-group sum of squares (SS) reflects pronounced differences in mean yields, particularly the sharp contrast between the 10-minute condition (very high yield: 863.39 mg/g) and longer reaction times (100–150 mg/g range). This indicates that the reaction is strongly time-dependent, with maximum yield achieved at a very short reaction time. Prolonged heating likely promotes product degradation, secondary reactions, or volatilization losses, leading to a substantial decline in yield. Furthermore, the relatively small within-group variation confirms that experimental error is minimal compared to the effect of reaction time, thereby reinforcing the reliability of the observed trend. Reaction time significantly influences methyl butanoate yield ($p < 0.05$). The process exhibits a sharp decline in yield after 10 minutes. Optimal production occurs at a short reaction time (≈10 min). ANOVA confirms that observed differences are not due to random experimental variation, but are process-driven.

Table 4. ANOVA Table

Source of Variation	SS	df	MS	F
Between Groups	1,011,061	4	252,765	137.43
Within Groups	9,200	5	1,840	
Total	1,020,261	9		

Worden et al. (1989) reported that the CO-adapted strain of *Butyribacterium methylotrophicum* was successfully manipulated to produce methyl butanoate at a concentration of 6 g/L in a batch culture system, facilitated by the continuous supply of 100%

carbon monoxide. This finding underscores the potential of gas-fermentation pathways for ester biosynthesis. In a related chemical approach, Battistel et al. (2011) achieved a 70% yield of methyl butanoate through the esterification of glycerol tributyrate (tributyrin) with methyl acetate at 60 °C using either acid or base catalysts, with the base-catalyzed system demonstrating faster reaction rates. This highlights the influence of catalyst type on reaction kinetics and efficiency. Furthermore, Dange *et al.* (2015) reported a maximum conversion of 91.64% within 120 minutes using an ultrasound-assisted, isothermal batch reactor. The process employed an acid ion-exchange resin (Amberlyst-15) as the catalyst for the esterification of butyric acid with methanol over a temperature range of 323-353 K, where ultrasonic irradiation significantly enhanced mass transfer and reaction performance. Similarly, Kaur et al. (2019) reported a maximum yield of 86% methyl butanoate via enzymatic esterification of vinyl butyrate with methanol. Optimal conditions involved incubation at 40 °C for 16 hours in the presence of 30 μg/mL purified lipase, demonstrating the effectiveness of biocatalysis under mild conditions. In contrast to these conventional and often time-intensive or catalyst-specific methods, the present study demonstrates a rapid and simplified approach. A maximum yield of 13.1%, corresponding to 900 mg/g of methyl butanoate, was achieved within just 10 minutes at 80 °C via a barium chloride-catalyzed thermal hydrolytic processing of waste *Gmelina arborea* leaf biomass. Although the yield is comparatively lower, the significantly reduced reaction time, moderate operating conditions, and utilization of low-cost biomass feedstock highlight the practical and sustainable advantages of this method as presented in Table 5.

Table 5. Comparison of methyl butanoate yields

Study	Substrate/System	Catalyst/Method	Conditions	Time	Yield/Conversion
Worden *et al.* (1989)	*Butyribacterium methylotrophicum* (CO strain)	Microbial fermentation (CO-fed system)	Continuous 100% CO supply (batch culture)	Not specified	6 g/L methyl butyrate
Battistel *et al.* (2011)	Tributyrin + methyl acetate	Acid/Base catalysis (faster with base)	60 °C	Not specified	70% methyl butanoate
Dange *et al.* (2015)	Butyric acid + methanol	Amberlyst-15 (acid ion-exchange resin), ultrasound-assisted	323–353 K	120 min	91.64% conversion
Kaur *et al.*	Vinyl butyrate + methanol	Lipase (30 μg/mL)	40 °C	16 h	86% methyl butanoate
Present study	*Gmelina arborea* leaf biomass (waste)	Barium chloride-catalyzed thermal hydrolytic process	80 °C	10 min	13.1% (≈900 mg/g) methyl butanoate

Conclusions

This study has successfully demonstrated a simple, rapid, and sustainable pathway for the production of methyl butanoate from *Gmelina arborea* leaf biomass via a barium chloride-catalyzed thermal hydrolytic process. The results confirm that reaction time plays a critical role in determining product yield, with an optimum performance observed at 10 minutes, yielding approximately 13.52% (≈900 mg/g) of methyl butanoate alongside excellent reproducibility (RSD ≈ 6%). Both statistical (ANOVA) and experimental analyses establish that prolonged reaction times adversely affect yield due to competing side reactions, product degradation, and equilibrium limitations. The method distinguishes itself from conventional approaches by eliminating the need for refined feedstocks, harsh reaction conditions, and extended processing durations, thereby offering a more efficient and environmentally friendly alternative.

Importantly, this work bridges a key research gap by demonstrating the direct conversion of low-value lignocellulosic waste into a value-added ester under mild conditions using an unconventional and low-cost catalyst. The process exhibits strong potential for scale-up due to its operational simplicity, reduced energy demand, and minimal processing complexity.

From a sustainability perspective, the findings align with the objectives of the United Nations Sustainable Development Goals. Specifically, the utilization of renewable biomass and the potential application of methyl butanoate as a biofuel surrogate contribute to SDG 7 (Affordable and Clean Energy). The development of an innovative catalytic route under mild conditions supports SDG 9 (Industry, Innovation and Infrastructure) by advancing cleaner production technologies. The valorisation of waste leaf biomass into useful chemical products promotes SDG 12 (Responsible Consumption and Production), while the reduction in reliance on fossil-derived chemicals and energy-intensive processes aligns with SDG 13 (Climate Action).

Overall, this study provides a compelling foundation for future research into biomass-derived ester production, highlighting the feasibility of integrating green chemistry principles with practical process innovation to achieve sustainable industrial development.

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Conflicts of Interest

No conflict of interest.

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