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Simplified Thermal Catalytic Pathway for 2-Methyltetrahydrofuran from Non-Food Biomass

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Abstract. *The growing demand for sustainable solvents and bio-based chemicals has heightened interest in renewable alternatives to petroleum-derived compounds. This study demonstrates an efficient and eco-friendly route for producing 2-methyltetrahydrofuran (2-MTHF) from Gmelina arborea leaves, a widely available lignocellulosic biomass. Thermal hydrolysis was conducted at 80 °C and atmospheric pressure using barium chloride as a low-cost catalyst. The reaction pathway proceeds through hemicellulose depolymerization to pentoses, dehydration to furfural, and subsequent hydrogenation–cyclization to yield 2-MTHF. Reaction time was optimized between 10–50 minutes, with Gas Chromatography-Mass Spectrometry (GC–MS) confirming a maximum yield of 19.47% (951.95 mg/g) at 50 minutes. The yield profile exhibited two distinct maxima, reflecting a balance between efficient conversion and secondary degradation reactions. Compared to conventional noble-metal-based hydrogenation processes, this method eliminates the need for high-pressure hydrogen and costly catalysts, thereby lowering energy intensity and production costs. The approach valorizes an underutilized agricultural residue, reduces environmental impact, and aligns with green chemistry principles. These findings highlight the potential of Gmelina arborea leaves as a sustainable feedstock for scalable 2-MTHF production, supporting its application as a green solvent, biofuel additive, and versatile platform chemical. Future work will focus on catalyst optimization, kinetic modeling, and techno-economic evaluation to advance industrial applicability.*

Keywords: *2-Methyltetrahydrofuran, Gmelina arborea, thermal hydrolysis, barium chloride catalyst, biomass valorization*

1. Introduction

The Industrial Revolution, which heavily depended on fossil resources, has significantly impacted the ecosystem, contributing to climate change and posing a serious threat to human life. However, transitioning to biomass as a primary source of industrial materials presents an opportunity to restore environmental balance. Biomass-based materials offer a viable alternative, producing equivalent products that currently rely on fossil resources (Ali & Ibrahim, 2023a; Ibrahim & Ali, 2023a; Ibrahim *et al*., 2025). Moreover, biomass-derived products provide several advantages. Pharmaceutical chemicals from biomass tend to be less harmful to human health. Biodegradable polymer products help reduce environmental pollution, promoting eco-friendly alternatives. Additionally, agrochemicals derived from biomass are environmentally benign, supporting sustainable agricultural practices. By adopting biomass as a fundamental industrial resource, we can mitigate the adverse effects of fossil fuel exploitation and work toward a more sustainable future.

This work builds upon our group's ongoing research into the valorization of *Gmelina arborea* leaves, an abundant and underutilized non-food biomass source in Nigeria. Our previous studies have demonstrated its potential as a rich source of valuable platform

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chemicals. For instance, we have successfully extracted and identified essential products, including furfural and levulinic acid precursors, from *Gmelina arborea* leaves via thermal processing (Ibrahim et al., 2024). Furthermore, we have established effective catalytic pathways using barium chloride catalyst for the conversion of this biomass into furan-based compounds, highlighting the catalyst's efficacy in facilitating hydrolysis and dehydration reactions (Ali & Ibrahim, 2023b). The present study directly extends this research by targeting the synthesis of 2-methyltetrahydrofuran ($C_5H_{10}O$), a higher-value hydrogenated derivative, from the same feedstock using a simplified, catalytic thermal hydrolysis process.

2-Methyltetrahydrofuran (2-MTHF) is a five-membered cyclic ether, characterized as a volatile, colourless, and flammable organic liquid with a distinct ether-like odour. It has a molecular weight of 86.13 g/mol, a boiling point of 80.3°C, a melting point of -136.4°C, and a density of 0.854 g/mL. It is partially miscible with water and highly soluble in organic solvents (Zhang *et al*., 2022; Monticelli *et al*., 2017). Its structural formula is depicted in Figure 1.

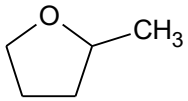


Figure 1. The structural formula of 2-methyltetrahydrofuran

2-MTHF finds extensive applications across diverse industries. In the pharmaceutical sector, it is widely used as a solvent, particularly in producing antibiotics and antivirals (Castiello et al., 2023; Albarrán-Velo et al., 2018; Lapponi et al., 2016). It also serves as a key intermediate in the synthesis of agrochemicals such as pesticides and herbicides (Gundekari et al., 2024; Morón-Ortiz et al., 2024; Dhanulkar et al., 2021; Pileidis et al., 2016). In polymer manufacturing, 2-MTHF is a solvent for producing materials like polyurethane and polyethylene (Lyu et al., 2024; Wu et al., 2023; Stadler, 2020). It is further employed as a mobile phase in chromatography for separating complex mixtures (Erdoğan et al., 2025; Ribas et al., 2024; de Gonzalo et al., 2019) and as an extraction solvent for isolating natural products such as essential oils and alkaloids (Saha et al., 2023; Prasad et al., 2022; Nehmeh et al., 2022).

In organic synthesis, 2-MTHF is preferred for reactions involving sensitive or moisture-sensitive compounds (Chen et al., 2024; Leitch et al., 2021). As a biofuel, it acts as a gasoline additive to boost octane ratings, with reported values including a Research Octane Number (RON) of approximately 87–90, a Motor Octane Number (MON) of 80–83, and an Anti-Knock Index (AKI or (R+M)/2) of 84–87 (Nagrimanov et al., 2024; Jiménez-Gómez et al., 2021; Kashyap et al., 2024). Additionally, 2-MTHF is employed as a solvent in cleaning agents such as paint strippers and degreasers (Samorì et al., 2023; Iftikhar et al., 2021; Jordan et al., 2020), as well as in adhesives, including epoxy and acrylic formulations (Stadler et al., 2020; Stadler, 2020). It also serves as an electrolyte solvent in lithium-ion batteries due to its favorable electrochemical stability (Li et al., 2024; Zhou et al., 2023).

2-MTHF offers several advantages over petroleum-based solvents. It exhibits lower toxicity than many conventional organic solvents, contributing to its approval for use in pharmaceutical chemical synthesis (Shah et al., 2022). Its high boiling point makes it well-suited for high-temperature reactions, supporting its classification as a green solvent (Lui et al., 2024), while its low viscosity facilitates easier handling and efficient mixing (Zhang et al., 2022; Jiménez-Gómez et al., 2021).

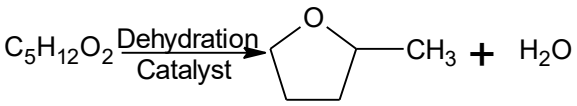
Despite these benefits, 2-MTHF must be handled with care due to its high flammability and volatility (Shen et al., 2022). Although it is biodegradable and can be decomposed by environmental microorganisms, its release into the atmosphere may still pose hazards to aquatic life and ecosystems (Usman et al., 2023; Rapinel et al., 2020). The compound can cause skin and eye irritation; thus, the use of protective gloves and eyewear is recommended during handling (Rodriguez Moreno et al., 2024). Proper ventilation is also essential to avoid inhalation of vapors, as prolonged exposure may result in symptoms such as dizziness, headaches, and nausea (Li et al., 2023).

2-MTHF is primarily produced from lignocellulosic biomass through a multi-step hydrogenation process. The most common route involves the hydrogenation of furfural, obtained from lignocellulosic feedstocks, into furfuryl alcohol, which is subsequently hydrogenated to yield 2-MTHF (Gundekari et al., 2024; Jiménez-Gómez et al., 2021). Alternatively, 2-MTHF can be synthesized via the hydrogenation of levulinic acid or γ-valerolactone, both of which are also derived from biomass sources (Gundekari et al., 2024; Xu et al., 2021), as illustrated in Eqs. 1 and 2. The first step involves the hydrogenation of levulinic acid to γ-valerolactone (Hijazi et al., 2022) as shown in Equation 1. This is followed by the hydrogenation of γ-valerolactone to 1,4-pentanediol (Huang et al., 2020), as shown in Equation 2.

$$C_5H_8O_3 + H_2 \xrightarrow{Catalyst/Heat} C_5H_8O_2 + H_2O \quad (1)$$

$$C_5H_8O_2 + 2H_2 \xrightarrow{Catalyst/Heat} C_5H_{12}O_2 \quad (2)$$

These reactions typically require costly catalysts such as ruthenium (Ru), palladium (Pd), and nickel (Ni). In the final stage, 1,4-pentanediol undergoes dehydration followed by cyclization to form 2-MTHF (Yan et al., 2023; England, 2025), as illustrated in Equation 3. The hydrogenation steps typically require base metal catalysts such as Ru, Pd, Ni, or Cu, whereas the subsequent dehydration and cyclization steps rely on acid catalysts, including zeolites, sulfuric acid (H_2SO_4), and heteropoly acids (Kochepka et al., 2020). However, these conventional processes are often costly and resource-intensive. Therefore, developing greener alternatives is essential for achieving a more sustainable and environmentally friendly production of 2-MTHF.



(3)

Despite the extensive studies on 2-MTHF production from biomass-derived feedstocks, existing methods primarily rely on expensive and noble metal catalysts such as Ru, Pd, and Ni. These conventional approaches demand high energy input, involve costly reagents, and often utilize fossil-based precursors, making them economically and environmentally unsustainable (Shen *et al.*, 2022). Unlike most conventional studies that begin with purified intermediates such as furfural or levulinic acid, this work employs raw, unprocessed Gmelina arborea leaves as a direct biomass feedstock. The process adopts a one-pot strategy, where hydrolysis of hemicellulose, dehydration of released sugars, and subsequent hydrogenation–cyclization of furfural intermediates all occur within the same reaction system without external hydrogen or multiple steps. Remarkably, the transformation proceeds under exceptionally mild conditions—80 °C and atmospheric pressure—contrasting

sharply with the high-temperature, high-pressure noble-metal-catalyzed protocols commonly reported. Together, these features establish a simplified, low-cost, and environmentally sustainable pathway for 2-MTHF production. Specifically, the study explores the catalytic potential of barium chloride and examines the effect of reaction time on the yield of 2-MTHF through the thermal hydrolysis of *Gmelina arborea* leaves.

2. Methods

The materials used in this study include 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 magnetic stirrer, filter cloth, and filter paper. The analytical reagents were barium chloride and sodium sulphate. Dried *Gmelina arborea* leaves were collected from the premises of Kaduna Polytechnic. The leaves were manually cleaned to remove unwanted materials, rinsed with distilled water to eliminate dirt, and air-dried at room temperature. After drying, they were pulverized using a ceramic mortar and pestle. A catalyst 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. Then, 50 g of the pulverized leaves was added to the catalyst solution. The mixture was heated to 80°C with continuous stirring at 2000 rpm on a magnetic stirrer for 10 minutes. Following the reaction, the product was filtered through cotton wool into a 1,000 mL beaker, followed by filtration through filter paper. The resulting filtrate was transferred into a separating funnel, and 0.2% (w/w) sodium sulfate (relative to the filtrate weight) was added, following the procedure of Ibrahim & Ali (2023c). The mixture was left overnight to facilitate phase separation. The organic layer was then collected, weighed, and analyzed using GC-MS. The experiment was repeated with reaction times of 20, 30, 40, and 50 minutes. Each set of readings was repeated twice. Samples from each experiment were collected and analyzed using Gas Chromatography-Mass Spectrometry (GC-MS) to determine the chemical composition and individual components of the products. The process flow diagram illustrating the production route of 2-MTHF through the thermal hydrolysis of *Gmelina arborea* leaves, catalyzed by barium chloride, is presented in Figure 2.

For derivatization, 200 µL of the standard solution was mixed with 100 µL of Trimethylsulfonium hydroxide (TMSH) and 20 µL of Triethylamine (TEA), then heated in sealed vials at 70 °C for 1 h before GC-MS analysis. The analyses were conducted using a Varian 3800/4000 gas chromatograph–mass spectrometer fitted with a DB-5 capillary column (30 m × 0.25 mm × 0.25 µm). Nitrogen was employed as the carrier gas at a column head pressure of 10 psi. The oven program was initiated at 100 °C with a 3 min hold, followed by a ramp of 8 °C min^{-1} up to 300 °C. The transfer line was maintained at 290 °C, and the VG 7070E magnetic sector MS operated in electron impact ionization mode. Mass spectra were recorded across m/z 40–800 at a rate of 20 scans s^{-1}, with a solvent delay of 330 s to avoid detector saturation. Continuous monitoring of the signal ensured reliable detection.

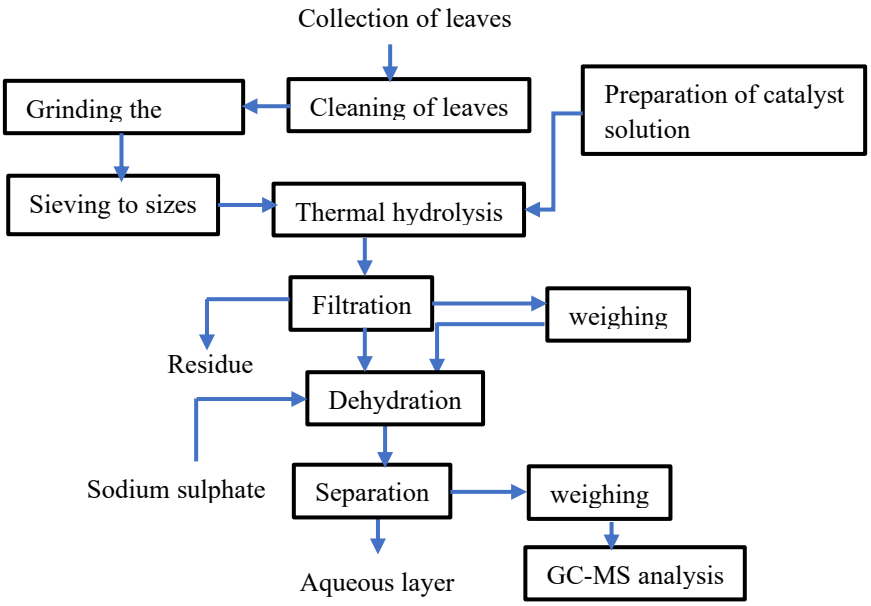


Figure 2. Flow process diagram for synthesis of 2MTHF

3. Results and Discussion

The weight of the dried products obtained after filtration and dehydration is shown in Table 1. The GC-MS analysis identified 2-MTHF among other phytochemicals, with percentage yields at reaction times of 10, 20, 30, 40, and 50 minutes, respectively, as presented in Table 2. The identification of 2-MTHF in the product stream confirms the successful hydrogenation-cyclization of furfural intermediates derived from the hemicellulose fraction of *Gmelina arborea* leaves. This aligns with our prior GC-MS analyses of thermally processed *Gmelina arborea* extracts, which consistently identified furfural as a major product (Ali & Ibrahim, 2023b; Ali et al., 2024; Ibrahim & Ali, 2025). The presence of barium chloride, which we have previously shown to catalyze the dehydration of pentoses to furfural, is therefore crucial in the initial stages of this reaction pathway. The subsequent in-situ conversion of this furfural to 2-MTHF under the applied conditions suggests a promising catalytic role for barium chloride beyond dehydration, potentially facilitating hydrogen transfer reactions.

Table 1. Weight of dried product

Time (min)	Product 1 (g)	Product 2 (g)
10	245.66	244.08
20	249.31	251.11
30	247.52	249.18
40	246.23	243.75
50	240.92	242.30

Table 2: Yield of 2-methyl tetrahydrofuran

Time (min)	1st (%)	2nd (%)
10	8.00	8.48
20	10.50	10.94
30	14.70	15.28
40	12.60	12.88
50	19.20	19.74

3.1 Reproducibility

From the replicate data: The standard deviations (SD**)** are very small relative to the mean values. The %RSD values are all **<** 2%, as presented in Table 3, which is generally considered *excellent reproducibility* in experimental chemistry

Table 3: Reproducibility of 2-MTHF yields at different residence times

Time (min)	1st (mg/g)	2nd (mg/g)	Average (mg/g)	SD	%RSD
10	128.00	131.28	129.64	2.31	1.78
20	145.20	148.46	146.83	2.31	1.57
30	234.00	238.54	236.27	3.21	1.36
40	355.00	361.10	358.05	4.31	1.20
50	222.00	227.20	224.60	3.68	1.64

3.2. Statistical significance with respect to residence time

The replicates (1st vs 2nd) do not differ significantly (ANOVA, $p \approx 0.94$) as presented in Table 4. The mean yields change strongly with residence time, from ~130 mg/g at 10 min to a peak of ~358 mg/g at 40 min, then slightly decreasing at 50 min. These differences between time points are much larger than the SD values (by more than an order of magnitude). Therefore, the variation in yield is statistically significant with respect to residence time, showing a clear dependence of 2-MTHF formation on reaction duration. The data are reproducible (%RSD < 2%). The effect of residence time on yield is statistically significant, with yields increasing steadily to an optimum (40 min), then declining slightly at 50 min due to secondary degradation or side reactions.

Table 4: One-way ANOVA for replicate comparison (1st vs 2nd)

Source of Variation	**F-value**	**p-value**	**Interpretation**
Between replicates (1st vs 2nd)	0.0061	0.9397	Not significant ($p \gg 0.05$); replicates are statistically indistinguishable

Figure 3 shows how the average yield (mg/g) of 2-MTHF varies with reaction time during barium chloride-catalyzed thermal hydrolysis of *Gmelina arborea* leaves. The yield (in mg/g) was computed from the mass of the dried product (shown in Table 1) and the percentage yield of 2-MTHF (shown in Table 2) using Equation 1. The yield profile shows a non-monotonic trend with at least two local maxima (one around 30–40 min, and a larger maximum at 50 min). This behaviour can be rationalized by competing kinetics and secondary chemistry common to thermochemical biomass conversion: The yields in mg/g were calculated from the expression in Equation 1

$$Yield\ \left(\frac{mg}{g}\right) = \frac{Y \times w \times 1000}{F} \qquad (1)$$

where, Y is the average %yield, w is the average product weight (g), and F is the weight of feed (50 g).

1. Initial increase (10 → 30 min): This phase reflects depolymerization of hemicellulosic fractions and accumulation of dehydrated intermediates (pentoses → furfural). As furfural concentration rises, the downstream hydrogenation/cyclization to 2-MTHF proceeds more rapidly and yields increase.
2. Transient decline (around 30-40 min): Once intermediate concentrations become substantial and temperature/time exposure increases, side reactions become kinetically significant. Typical side processes include furfural polymerization to humins, condensation of reactive aldehydes with sugars/oligomers, and formation of heavier, non-volatile humic materials. These pathways consume furfural (and possibly nascent 2-MTHF), resulting in a temporary decrease in the measured 2-MTHF yield.
3. Secondary rise (to the largest maximum at 50 min): A renewed increase in 2-MTHF at longer times can arise if slower breakdown of oligomeric or condensed intermediates releases additional reactive monomers (e.g., bound furfural or furfuryl derivatives) that are subsequently converted to 2-MTHF before degradation dominates. Alternatively, catalytic restructuring of condensed material or slow hydrogen-transfer reactions, which require longer times, may account for the late rise. This two-peak behaviour (formation → partial loss to condensation → re-release/conversion of bound intermediates) has been reported qualitatively in related biomass dehydration/hydrogenation studies (Ali & Ibrahim, 2023b; Ali et al., 2024; Ibrahim & Ali, 2025; Ibrahim et al., 2024) and is consistent with the complex, multistep network indicated by our GC–MS speciation.

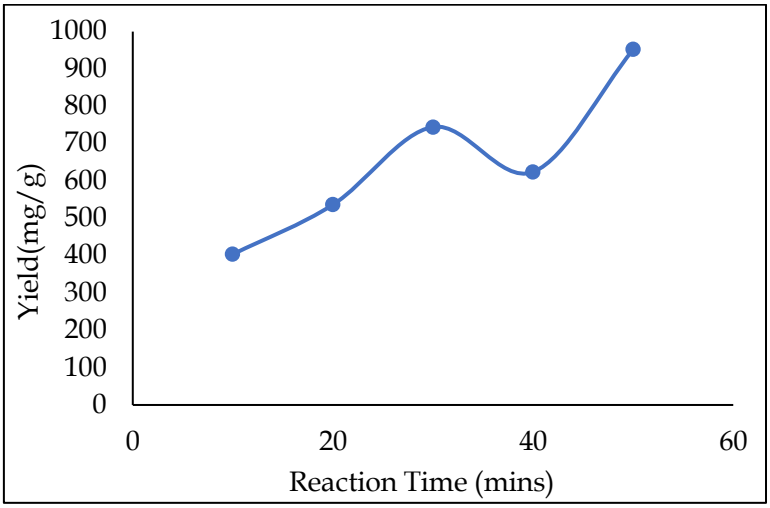


Figure 3: Average yield of 2-MHTF from G.aborea leaves

Biomass-derived polyols and carbohydrates: During thermal hydrolysis, hemicellulose and cellulose fragments release sugars, polyols, and organic acids (Vaidya *et al.*, 2022; Peng *et al.*, 2011). These oxygenated compounds can undergo dehydration and reforming reactions, liberating hydrogen in situ.

Aqueous phase reactions (water as a hydrogen donor): Under heating, water can act as a hydrogen donor via *water-gas shift-like chemistry* (Jiang *et al.*, 2024; Pavlova *et al.*, 2016) as illustrated in Equation 4.

$$CO + H_2O \rightarrow CO_2 + H_2 \qquad (4)$$

Furfural and other aldehydes from the leaves can react with water, generating formic acid or CO intermediates, which further decompose to release H_2 (Sun *et al.*, 2020).

A comparative analysis of the product route and yield of 2-MTHF is shown in Table 5. Chen et al. (2015) demonstrated a 97% yield of 2-methyltetrahydrofuran (2-MTHF) from furfural using a Cu–Pd catalyst at 180 °C. Despite the high yield, the reliance on expensive feedstock and catalyst renders the process uneconomical compared with the present work, which achieved a 19.62% yield (951.95 mg/g) at just 80 °C within 50 minutes using low-cost Gmelina arborea waste leaves and an inexpensive $BaCl_2$ catalyst. Similarly, Ma et al. (2016) reported an 83.9% yield from furfural employing a Cu–Pd catalyst in 2-propanol at 220 °C for 4 h, while Sharma and Pant (2020) obtained 56–75% yield from levulinic acid using Ni–Cu/Al_2O_3 at 250 °C for 5–24 h. Both methods, however, remain energy- and cost-intensive relative to the simplified approach adopted in this study. In related work, Li et al. (2021) achieved an 87.6% yield from levulinic acid at 190 °C under 5 MPa H_2 for 5 h, and Zhang et al. (2025) attained a 93.8% yield from furfural at 200 °C and 10 bar H_2 over 8 h. Shen et al. (2025) reported 82% yield from furfural using a $Cu_2Cr_2O_x$-Pd/C catalyst at 240-300 °C, while Liu et al. (2024) observed only 33% yield with a W-Cu/γ-Al_2O_3 system. These processes are limited by their high energy requirements, long reaction times, and costly catalysts, which restrict their industrial feasibility. By contrast, the present study demonstrates a more economical and sustainable route to 2-MTHF production through the use of abundant, valueless leaf biomass and a low-cost $BaCl_2$ catalyst under mild conditions, highlighting its strong potential for large-scale commercialization.

Table 5: Comparative result analysis of production methods and yield of 2-MTHF

Study / Reference	Feedstock / Precursor	Catalyst	Conditions (T, P, time)	Yield (%)	Remarks
Chen et al. (2015)	Furfural	Cu–Pd	180 °C, ambient pressure	97	High-yield but costly feedstock and catalyst
Ma et al. (2016)	Furfural	Cu–Pd in 2-propanol	220 °C, 4 h	83.9	Energy-intensive, noble metal catalyst
Sharma & Pant (2020)	Levulinic acid	Ni–Cu/Al_2O_3	250 °C, 5–24 h	56–75	Long reaction times, high temperature
Li et al. (2021)	Levulinic acid	(metal catalyst)	190 °C, 5 MPa H_2, 5 h	87.6	Requires high-pressure H_2, energy-intensive
Zhang et al. (2025)	Furfural	(metal catalyst)	200 °C, 10 bar H_2, 8 h	93.8	High-pressure H_2, long reaction
Shen et al. (2025)	Furfural	$Cu_2Cr_2O_x$–Pd/C	240–300 °C	82	Harsh conditions, noble metal catalyst
Liu et al. (2024)	Furfural	W–Cu/γ-Al_2O_3	(elevated Temp.)	33	Low yield
This study	*Raw Gmelina arborea leaves*	$BaCl_2$ (0.5%)	80 °C, 1 atm, 50 min	19.6 (951.95 mg/g)	Mildest conditions, no external H_2, low-cost biomass & catalyst

The yield of 2-MTHF was limited to a maximum of 19.7% because the reaction simultaneously produced other phytochemicals of equal importance from the biomass. These

co-products, which compete with 2-MTHF formation, include 2-furylmethanol, furan-2-ylmethanamine, 2,5-dimethylfuran, Gmelinol, and methyl butanoate.

Conclusions

This study establishes a simplified and sustainable route for producing 2-MTHF from *Gmelina arborea* leaves through barium chloride-catalyzed thermal hydrolysis under mild conditions. The process demonstrated excellent reproducibility, with yields showing clear dependence on residence time. A maximum yield of ~19.47% (951.95 mg/g) was obtained at 50 minutes, highlighting the significance of optimizing reaction duration to balance efficient conversion with the onset of degradation reactions. The findings confirm that both catalyst presence and residence time critically influence product distribution, with distinct yield peaks reflecting competing formation and degradation pathways. Importantly, this approach avoids high-pressure hydrogenation and expensive noble-metal catalysts typically required in conventional processes, making it cost-effective and environmentally favorable. Overall, the valorization of *Gmelina arborea* leaves into 2-MTHF not only provides a renewable platform for green solvents and biofuel additives but also contributes to waste reduction and sustainable chemical production. Future investigations should focus on scaling up the process, refining catalyst performance, and developing kinetic and techno-economic models to support industrial applications.

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Conflicts of Interest

The authors declare no conflict of interest. No one outside the authors has any role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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