



DIRECT CONVERSION OF *Gmelina arborea* BIOMASS TO 2,5-DIMETHYLFURAN VIA BARIUM CHLORIDE-CATALYZED THERMAL HYDROLYSIS UNDER MILD CONDITIONS

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Introduction

Biomass-derived chemicals are increasingly recognized as sustainable alternatives to petrochemical-based products, offering applications in fuels, pharmaceuticals, cosmetics, food, and agrochemicals (Ali & Ibrahim, 2023; Espro et al., 2021; Ibrahim & Ali, 2025; Ibrahim et al., 2024b; Ibrahim et al., 2025; Kohli et al., 2019; Muilu-Mäkelä et al., 2024). Unlike fossil fuel-derived compounds, which are non-renewable and contribute to greenhouse gas emissions and environmental degradation,

Abstract

The synthesis of 2,5-dimethylfuran (DMF) from biomass-derived carbohydrates typically requires multi-step processes involving noble-metal catalysts, high temperatures, and pressurized systems, limiting industrial scalability. This study introduces a simplified, cost-effective, and sustainable approach for directly converting *Gmelina arborea* leaves into DMF through barium chloride-catalyzed thermal hydrolysis conducted at 80 °C under atmospheric pressure. The influence of reaction time (10–50 min) on DMF yield was evaluated, with an optimal yield of 13.22% (652.39 mg DMF/g biomass) achieved at 20 min. Prolonged heating beyond this point led to product degradation, emphasizing the need for precise process control. Gas chromatography-mass spectrometry (GC-MS) confirmed DMF formation along with minor by-products.

Mechanistic insights revealed barium chloride's dual catalytic role in promoting polysaccharide hydrolysis and subsequent sugar conversion to DMF via isomerization, dehydration, and hydrodeoxygenation steps. Compared with conventional glucose- or fructose-based routes, this method eliminates the need for intermediate isolation, noble metals, and hydrogenation under high pressure—significantly reducing both cost and energy demand. Utilizing non-food lignocellulosic biomass enhances environmental sustainability. The resulting DMF, with an energy density of approximately 33 MJ/L, shows potential as a renewable biofuel and a valuable intermediate in pharmaceutical, fragrance, and agrochemical applications. This study provides an eco-efficient and scalable pathway for green DMF synthesis, supporting biomass valorization and circular bioeconomy objectives.

Keywords: 2,5-dimethylfuran, *Gmelina arborea*, barium chloride, biomass conversion, green chemistry, sustainable biofuels

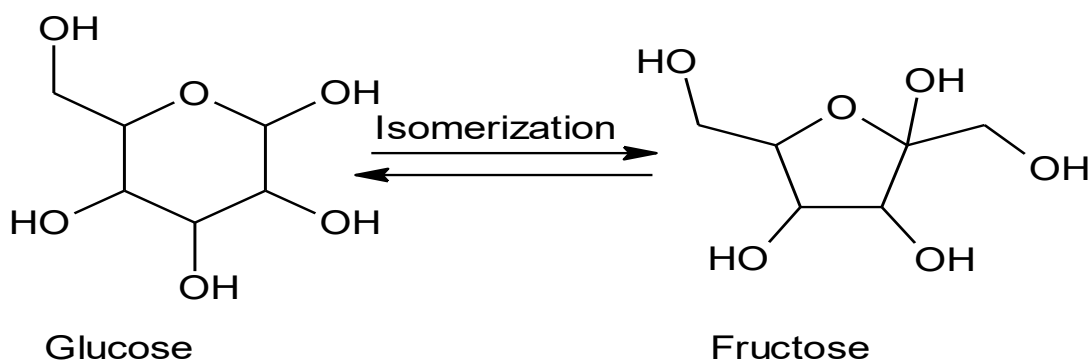
biomass-based chemicals are biodegradable, renewable, and environmentally benign (Khandaker et al., 2025; Mori, 2023). Emphasizing renewable feedstocks is central to sustainable development and the circular bioeconomy (Bozell & Petersen, 2010; Clark et al., 2009).

One such compound, 2,5-dimethylfuran (DMF), has attracted attention as both a versatile platform chemical and a biofuel (Anchan & Dutta, 2021; Dutta, 2024; He et al., 2024; Hoang et al., 2022; Mehta et al., 2022). A furan derivative with methyl groups at the 2 and 5 positions (Carrasco, 2019; Iroegbu et al., 2020; Li et al., 2022; Parrish, 2022), DMF is useful in pharmaceuticals, bioactive molecules, and antioxidant therapies (Amalraj et al., 2024; Grosso, 2023; Książkiewicz et al., 2025; Makiso et al., 2024).

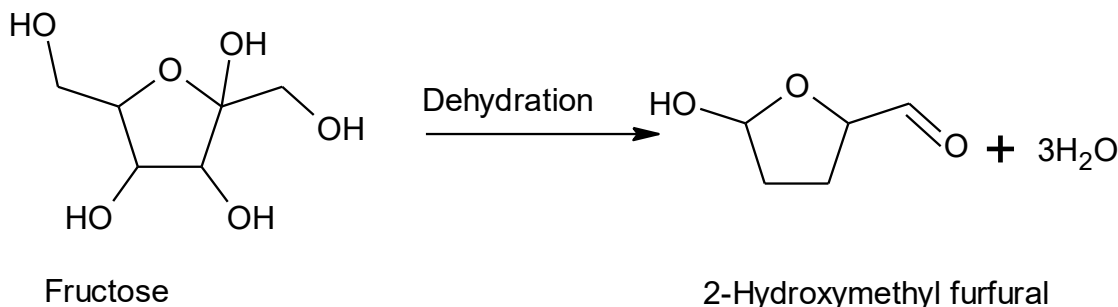
As a biofuel, DMF exhibits favourable properties: a high energy density (~33 MJ/L) comparable to gasoline (~34 MJ/L) and higher than ethanol (~21 MJ/L) (Feng et al., 2022; Nguyen et al., 2021). It shows high octane (~119), efficient combustion, and hydrophobicity—ensuring storage stability and compatibility with existing fuel infrastructure (Dhamodaran et al., 2023; Jin et al., 2024; Le et al., 2021; Lei et al., 2025; Liu et al., 2019; Xiao et al., 2017). Blends of DMF with gasoline reduce particulate emissions, making it a strong candidate for cleaner energy systems (Nguyen et al., 2021; Xu & Wang, 2016).

Beyond fuels, DMF's sweet, caramel-like aroma makes it relevant to the fragrance and flavour industries (Montgomery & Crompton, 2017; Oklu et al., 2020; Zhu & Xiao, 2022). It can serve as an intermediate in aroma compound synthesis and contributes to roasted or baked flavour profiles in food chemistry (Batool et al., 2023; Obando & Figueroa, 2024; Samakradhamrongthai, 2024). In agriculture, it acts as an intermediate in pesticides, fungicides, and herbicides, with potential for bio-based fumigation and allelopathic applications (Jeschke, 2022; Ma et al., 2024; Pavlović et al., 2021; Rathour et al., 2023).

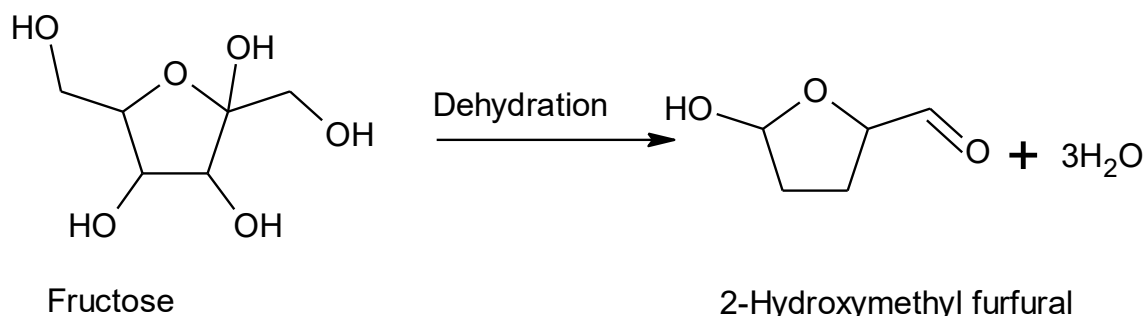
Historically studied in organic chemistry, DMF has gained renewed importance for its role in green chemistry and biorefinery models (Chauhan et al., 2024; Soukup-Carne et al., 2024). Conventionally, DMF is synthesized from carbohydrates through a three-step pathway: isomerization of glucose to fructose (Scheme 1), dehydration to 5-HMF (Scheme 2), and hydrodeoxygenation to DMF (Scheme 3) (Aranha & Gogate, 2023; Chen et al., 2024; Ji et al., 2021; Jiang et al., 2023; Turkin et al., 2022). Although effective, this conventional route is costly, energy-intensive, and reliant on noble-metal catalysts and expensive solvents (Grant, 2022; Gupta et al., 2018; Portillo Pérez, 2021). These challenges hinder large-scale deployment and limit sustainability.



SCHEME 1: ISOMERIZATION OF GLUCOSE TO FRUCTOSE



SCHEME 2: DEHYDRATION OF FRUCTOSE TO 5-HMF

**Scheme 3:** Hydrodeoxygenation of 5-HMF to DMF

A major research gap remains in exploring direct conversion of lignocellulosic biomass such as *Gmelina arborea* leaves into DMF under mild conditions using low-cost, eco-friendly catalysts. Current methods primarily depend on carbohydrate-rich feedstocks, leaving leaf biomass underutilized. This study aims to develop a simplified, cost-effective, and sustainable approach for DMF synthesis from *Gmelina arborea* leaves via barium chloride-catalyzed thermal hydrolysis. By optimising reaction time, assessing catalytic efficiency, and characterizing products via GC-MS, the study evaluates a greener pathway that reduces energy demands, avoids noble metals, and utilizes non-food biomass. This contributes to biomass valorization and the circular bioeconomy.

Materials and Methods

Dried *Gmelina arborea* leaves were collected from Kaduna Polytechnic premises and ground into a fine powder using a ceramic mortar and pestle. The powder was then sieved using 250 μm and 300 μm mesh sifters and stored in an airtight container until use. The catalyst solution was prepared in accordance with Ibrahim and Ali (2023a) by dissolving 0.25 g of barium chloride (equivalent to 0.5% w/w of the biomass feed) in 500 mL of distilled water in a 1000 mL conical flask, with continuous stirring to ensure complete dissolution. Subsequently, 50 g of the sieved leaf powder was added to the catalyst solution and heated on a Gallenkamp hot plate equipped with a magnetic stirrer at 80 $^{\circ}\text{C}$ for 10 minutes. After the reaction, the mixture was filtered first through a filter cloth, and then, through Whatman filter paper, following the method of Ibrahim and Ali (2023b). The resulting filtrate was dehydrated, following the method of Ali *et al.* (2024), by adding anhydrous sodium sulphate (0.2% w/w of the filtrate) into the filtrate and

allowed to stand for phase separation. The aqueous phase was discarded, and the organic layer was collected and weighed. This procedure was repeated for residence times of 20, 30, 40, and 50 minutes. Two experimental replicates were performed for each reaction time. From each reaction, a 5 mL aliquot of the organic layer was withdrawn and analyzed for chemical composition using gas chromatography-mass spectrometry (GC-MS).

Results and Discussion

Gas chromatography-mass spectrometry (GC-MS) analysis confirmed the presence of DMF in all five product samples, alongside various other phytochemicals generated during the thermal hydrolytic conversion process. The quantified average yields of DMF at reaction times of 10, 20, 30, 40, and 50 minutes were 5.78%, 13.22%, 10.13%, 6.35%, and 6.56% of the total product weight, respectively. These percentage yields were further converted to milligrams of DMF produced per gram of the initial *Gmelina arborea* leaf powder. A yield-time profile was subsequently developed by plotting these values against the corresponding reaction residence times, as depicted in Figure 1.

The yield of DMF increases significantly from approximately 370 mg/g at 10 minutes to a maximum of about 660 mg/g at 20 minutes. This sharp rise indicates that the thermal hydrolytic process is highly active during the early stages. The reactants, including the leaf biomass and catalyst (i.e., BaCl_2), efficiently convert precursor compounds (such as carbohydrates) into DMF. The maximum yield is observed at 20 minutes, suggesting this is the optimal reaction time for maximum DMF production under the given conditions. At this point, the conversion rate is at its peak before degradation or side reactions dominate. The yield drops steeply from about 660 mg/g to 340 mg/g. After reaching the optimal point, prolonged heating likely causes thermal degradation of DMF or secondary reactions that consume the product, reducing the yield. From 30 to 50 minutes, the yield continues to fall gradually, reaching about 110 mg/g. Most of the DMF may have either degraded, volatilized, or been converted into other products. The precursor materials may also be nearly exhausted by this stage. The profile is unimodal, showing a clear optimum at 20 minutes. This behaviour emphasises the importance of precise reaction time control to maximise DMF yield. Longer reaction times are not beneficial and may even be detrimental due to product instability or side reactions.

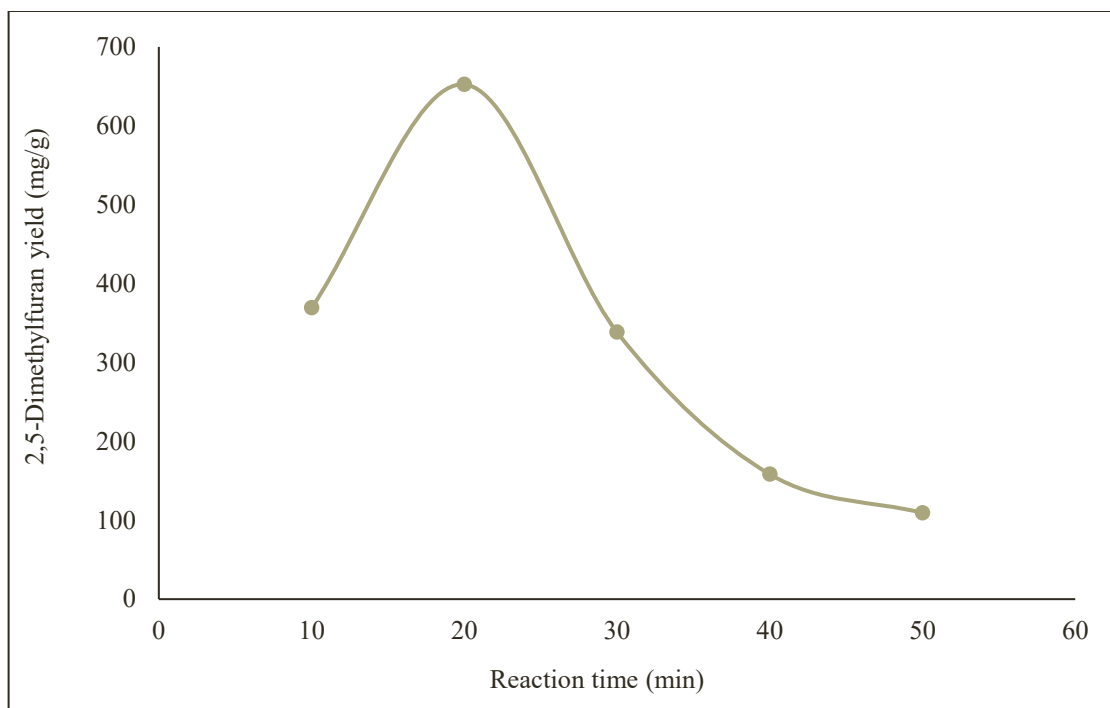
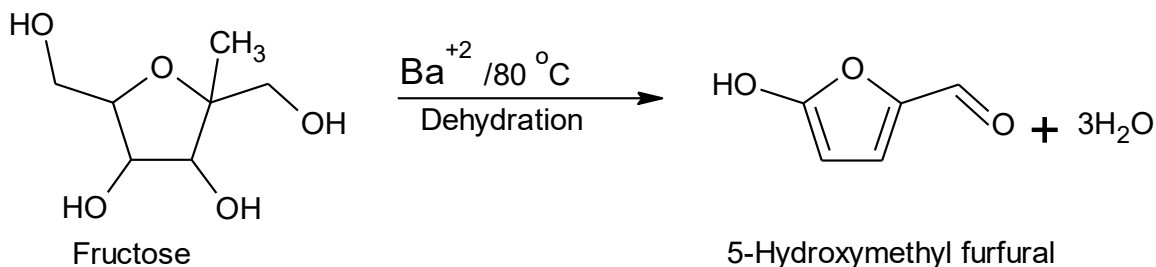
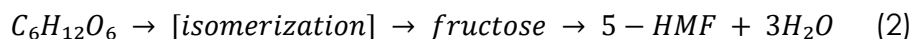
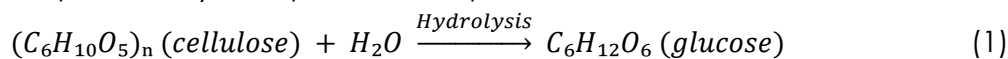


FIGURE 1: YIELD (MG/G)-TIME OF DMF FROM GMELINA ARBOREA LEAVES

Several studies on the processing of *Gmelina arborea* leaves have reported a characteristic unimodal yield–time relationship, in which product yield rises to an optimum at short reaction times before declining with prolonged treatment. For instance, Ibrahim and Ali (2023c) investigated the thermal methanolic processing of *Gmelina arborea* leaves for squalene production at reaction times of 10, 20, 30, 40, 50, and 60 minutes. They recorded the highest yield at 20 minutes (171.98 mg/g), with lower values at both shorter and longer durations (e.g., 162.39 mg/g at 10 minutes, 145.69 mg/g at 30 minutes, and a minimum at 40 minutes). In a related study, Ibrahim et al. (2024a) examined the methanolysis of *Gmelina arborea* leaves at 60 °C and assessed the yields of three bioactive metabolites—benzofuran-2-carboxaldehyde, 2,5-dimethyl-3(2H)-furanone (DMHF), and 9-octadecenamide. Their results similarly showed that yields peaked after a short residence time and then declined steadily with longer reaction periods.

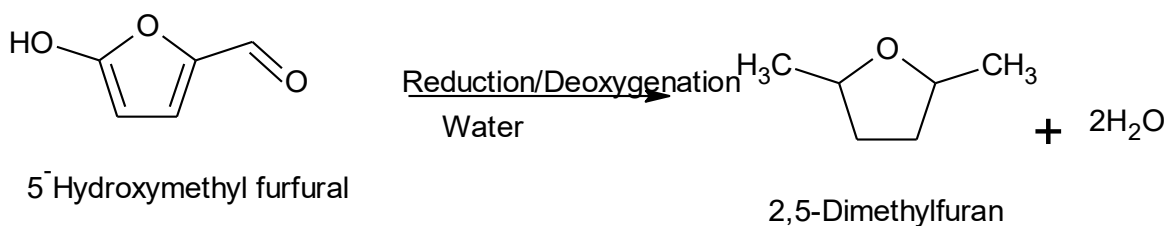
Gmelina arborea leaves contain carbohydrates (cellulose, hemicellulose, possibly starch) (Adebayo et al., 2019; Esteves et al., 2023; Oloche et al., 2021). Under mild heating (80 °C) and in the presence of water, BaCl₂ catalyzes the hydrolysis of polysaccharides into monomeric sugars, mainly hexoses (glucose, fructose) (Chaudhary et al., 2022; Zayed et al., 2020) as illustrated in Equation 1. Glucose undergoes isomerization (possibly assisted by Ba²⁺) to fructose, which

then dehydrates to 5-HMF through a triple dehydration reaction (Liu & Kerton, 2021; Zampieri, 2023) as expressed in Equation 2 and Scheme 4.



SCHEME 4: CONVERSION OF FRUCTOSE TO 5-HMF

5-HMF undergoes hydrogenation of the aldehyde group to form 2,5-bis(hydroxymethyl)furan (BHMF) and then hydrodeoxygenation (removal of -OH groups) to form DMF (Panigrahy *et al.*, 2022; Przydacz *et al.*, 2024) as presented in Scheme 5. Although molecular hydrogen is not externally introduced, biomass-derived hydrogen donors (like lignin degradation intermediates) under thermal conditions may act as reducing agents (Ashraf *et al.*, 2023; Mkumbuzi *et al.*, 2023; Zhao *et al.*, 2024). Ba^{2+} may assist in facilitating H-transfer and the reduction of the aldehyde group to alcohol, and Subsequent hydrogenolysis of the hydroxyl groups and aromatization (Cowan, 2007).



Scheme 5: Reduction and deoxygenation of 5-HMF to DMF

Reported yields of 5-hydroxymethylfurfural (5-HMF) from fructose, using acid catalysts such as HCl, H_2SO_4 , or solid acids typically range between 60–90% (Pande *et al.*, 2018; Rusanen, 2021; Tempelman *et al.*, 2021). Subsequent conversion of 5-HMF to DMF with metal catalysts such as Cu–Ru/C, Pd/C, or Ni-based systems generally affords 60–80% yields (Boro *et al.*, 2024; Vinod & Dutta, 2021). Consequently, the overall yield of DMF from fructose is reported to fall within 45–65% (). In comparison, the conversion of glucose to DMF via isomerization to fructose, followed by dehydration and hydrogenation, achieves an overall yield of only about 25% (Chheda *et al.*, 2007). In this study, a yield of

13.22% DMF was obtained. Although lower than conventional fructose- or glucose-derived processes, this result is economically more attractive because it employs waste biomass as feedstock, a low-cost BaCl_2 catalyst, and operates under mild conditions (80 °C, atmospheric pressure). This demonstrates the potential of the approach as a cost-effective and sustainable alternative to traditional methods.

Conclusion

This study demonstrates a novel, simplified, and sustainable method for synthesizing DMF directly from *Gmelina arborea* leaves via barium chloride-catalyzed thermal hydrolysis under mild conditions (80 °C, atmospheric pressure). The results validate the effectiveness of this method, with the highest yield of DMF (13.22%, or 652.39 mg/g biomass) achieved at a reaction time of 20 minutes. Beyond this optimal point, extended heating led to product degradation and reduced yields, underscoring the necessity for precise control of reaction duration.

The GC-MS analysis confirmed the presence of DMF and supported the proposed mechanistic pathway involving hydrolysis, isomerization, dehydration, and hydrodeoxygenation steps facilitated by the BaCl_2 catalyst. This pathway circumvents the need for noble-metal catalysts, high-pressure hydrogenation, and intermediate isolation (e.g., fructose or 5-HMF), marking a significant departure from conventional, multi-step DMF production processes.

By employing underutilized lignocellulosic biomass and an inexpensive, non-toxic catalyst, this approach aligns strongly with green chemistry principles and the goals of the circular bioeconomy. It offers a promising route for scalable and cost-effective production of DMF, with potential applications in biofuels, pharmaceuticals, fragrances, and agrochemicals. The study paves the way for future research into optimizing catalyst systems, expanding biomass feedstock options, and scaling up this process for industrial implementation.

Recommendations

Based on the promising results obtained in this study, it is recommended that future research should focus on the following areas to advance the practical application and scalability of the barium chloride-catalyzed thermal hydrolytic process for DMF synthesis:

1. **Process Optimization and Scale-Up:** Pilot-scale studies should be conducted to validate the process efficiency beyond laboratory conditions. Parameters such as catalyst concentration, biomass-to-

- solvent ratio, and continuous-flow configurations warrant further investigation for industrial viability.
2. **Catalyst Recovery and Reusability:** Investigating the recyclability and long-term stability of barium chloride in repeated batch or continuous operations will be essential to enhance process economics and environmental compatibility.
 3. **Mechanistic Studies:** Detailed kinetic and mechanistic studies using advanced spectroscopic and analytical techniques can help elucidate the exact role of Ba^{2+} ions in facilitating isomerization and hydrodeoxygenation, potentially revealing new catalytic insights.
 4. **Feedstock Diversification:** Exploring other lignocellulosic leaves and agricultural wastes with similar carbohydrate profiles may expand the applicability of this method and contribute to regional biomass valorization strategies.
 5. **Product Purification and Application Testing:** Further refinement of product separation methods and performance testing of the synthesized DMF in target applications (e.g., fuel blends, pharmaceutical intermediates, agrochemicals) will strengthen the commercial appeal of this green synthesis route.

Overall, this study lays the groundwork for a more sustainable and decentralized model of DMF production using non-food biomass under mild conditions, and future efforts should aim to integrate this method within broader biorefinery frameworks.

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