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DIRECT SYNTHESIS OF TRICONTANE FROM *GMELINA ARBOREA* LEAVES VIA MILD CALCIUM HYDROXIDE-CATALYSED ETHANOLYSIS

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Abstract

The sustainable production of wax-range hydrocarbons from renewable resources remains a key challenge in biomass valorization, as most reported routes rely on severe thermal conditions and exhibit low selectivity. In this study, a mild and energy-efficient pathway for the synthesis of long-chain saturated hydrocarbons (C_{30}) from *Gmelina arborea* leaf biomass is reported using calcium hydroxide-catalysed ethanolysis under atmospheric pressure. A Box-Behnken response surface methodology was employed to evaluate and optimize the effects of temperature (40-60 °C), reaction time (40-60 min), and catalyst loading (1.0-2.0 wt%). Product characterization by GC-MS confirmed the formation of five long-chain n-alkanes, tricontane, alongside other industrially relevant phytochemicals. Hydrocarbon yields varied significantly with operating conditions, ranging from 0.83 to 3.65% (41.25-101.56 mg g^{-1}), with the maximum yield achieved at 50 °C, 50 min, and 1.5% $Ca(OH)_2$. Statistical analysis revealed temperature as the most influential parameter, while reaction time and catalyst loading exhibited pronounced quadratic effects, indicating well-defined optima. Compared with previously reported

Keywords: *Gmelina arborea* leaves; long-chain hydrocarbons; tricontane calcium hydroxide catalyst; ethanolysis; biomass valorization; response surface methodology

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biomass-based routes requiring high temperatures, this process achieves competitive yields under markedly milder conditions. The findings demonstrate the effectiveness of calcium hydroxide as a low-cost, environmentally benign catalyst and highlight *Gmelina arborea* waste leaves as a promising feedstock for sustainable production of high-value wax-range hydrocarbons, supporting low-energy biorefinery and circular bioeconomy strategies.

Introduction

The growing demand for sustainable energy carriers and environmentally benign chemicals has intensified interest in biomass as a renewable feedstock for value-added hydrocarbon production (Akhtar et al., 2025; Zhang et al., 2023; Adeoye et al., 2023; Dutta, 2023). Long-chain saturated alkanes, particularly tricontane ($C_{30}H_{62}$), exhibit high energy density, thermal stability, and chemical inertness, making them attractive for lubricants, waxes, specialty fuels, and chemical feedstocks (Srivastava, 2014; Mortensen et al., 2011). However, environmental and economic challenges associated with fossil resources, including greenhouse gas emissions, climate change, and resource depletion, have accelerated the search for renewable alternatives (Wang & Azam, 2024). Tricontane (C_{30}) are long-chain saturated n-alkanes commonly found in natural waxes (Giuffrè, 2021; Mitra et al., 2017). In plants, they occur predominantly in cuticular wax layers, where they provide protection against water loss and environmental stress (Bhanot et al., 2025; Lewandowska et al., 2020; Dhanyalakshmi et al., 2019). These hydrocarbons are biosynthesized through fatty-acid elongation followed by decarboxylation or decarbonylation, yielding very high-molecular-weight paraffins (Adhikary et al., 2022; Singh & Jha, 2017). In geological and petroleum systems, C_{30} alkanes are characteristic of high-boiling fractions and paraffinic waxes and are widely used as biomarkers for terrestrial plant input and depositional environments (Ekejiuba, 2021; Gök, 2017; Saheed et al., 2023; Liu et al., 2022).

Industrially, C_{30} alkanes are valued for their high melting points, hydrophobicity, and chemical inertness, enabling applications in lubricants, waxes, coatings, cosmetics, phase-change materials, and specialty fuels (Pal et al., 2025; Zhu et al., 2023). Consequently, increasing sustainability concerns have shifted attention toward renewable production routes using lignocellulosic biomass, plant residues, and waste leaves as alternatives to petroleum-derived wax fractions. Within biomass conversion research, the detection of C_{30} hydrocarbons indicates effective deoxygenation and carbon-chain preservation or reconstruction during thermal or catalytic processing (Li et al., 2024; Maity et al., 2021). Biomass-based routes typically involve mild thermal processing or catalytic treatment, under which biogenic lipids, wax esters, and long-chain fatty compounds undergo

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deoxygenation, hydrogen transfer, and carbon rearrangement reactions (Mascal & Dutta, 2020). The use of Lewis or alkaline catalysts can further promote chlorophyll hydrolysis, lipid transformation, and alkane stabilization under atmospheric conditions (Summers et al., 2025; Wang et al., 2020; Zhou & Hu, 2020). Compared with fossil-derived routes, biomass-to-tricontane pathways offer a sustainable strategy for producing high-value wax-range hydrocarbons.

In this context, *Gmelina arborea*, a fast-growing deciduous tree widely distributed in tropical and subtropical regions, represents a promising biomass resource. Although primarily cultivated for timber, its leaves are often discarded despite their rich organic composition (Akinyemi et al., 2021). Thermochemical pathways such as pyrolysis, hydrothermal liquefaction, and catalytic upgrading can convert the lignocellulosic matrix of *Gmelina arborea* leaves into long-chain hydrocarbons, supporting waste valorization and advancing circular bioeconomy principles (Bridgwater, 2012; Mohan et al., 2006; Cherubini & Strømman, 2011).

Despite extensive advances in biomass valorization, direct synthesis of long-chain saturated hydrocarbons (C_{30}) from lignocellulosic biomass remains sparsely reported, with most existing studies relying on high-temperature carbonization, pyrolysis, or fossil-derived upgrading routes. Reported biomass-based hydrocarbon productions typically require severe thermal conditions (>500 °C) or yield hydrocarbons as minor fractions within complex product matrices, limiting process selectivity, energy efficiency, and scalability. Moreover, systematic studies exploring mild, low-temperature catalytic pathways for selectively producing wax-range alkanes from waste leaf biomass are notably absent. In particular, the role of alkaline catalysts under atmospheric-pressure ethanolysis conditions, as well as the optimization of operating variables governing C_{30} alkane formation, has not been adequately investigated. This gap underscores the need for a controlled, low-energy, and catalyst-assisted strategy capable of converting underutilized biomass resources into high-value long-chain hydrocarbons, which the present study addresses through calcium hydroxide-catalysed ethanolysis of *Gmelina arborea* leaves under mild conditions

This study aims to develop a sustainable route for producing long-chain hydrocarbons and value-added chemicals from *Gmelina arborea* leaf biomass using mild thermal catalysis with calcium hydroxide. The objectives are to (i) optimize reaction conditions for maximum hydrocarbon yield, (ii) characterize the product mixture via GC-MS, (iii) evaluate the catalytic efficiency of calcium hydroxide, and (iv) assess the industrial applicability of the derived hydrocarbons.

Materials and Methods

A Box-Behnken design within the framework of Response Surface Methodology (RSM) was employed to generate 17 experimental runs, as presented in Plate 1. Dry *Gmelina*

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arborea leaves were collected from the Kaduna Polytechnic campus, thoroughly washed, air-dried, milled, and sieved to obtain a uniform particle size, following previously reported procedures (Ibrahim et al., 2025a; Ali & Ibrahim, 2023a). The catalyst solution was prepared by dissolving 1.0 g of calcium hydroxide $Ca(OH)_2$, corresponding to 2.0% (w/w) of the biomass feed, in 500 mL of ethanol in accordance with Ibrahim & Ali (2023). Subsequently, 50 g of the processed *Gmelina arborea* leaf powder was added to the solution. The reaction mixture was heated at 60 °C for 50 min on a Gallenkamp hot plate equipped with a magnetic stirrer to ensure uniform mixing, followed by the method of Ibrahim *et al.* (2022).

After completion of the reaction, the mixture was first filtered through a filter cloth and then clarified using Whatman filter paper, in accordance with the method of Ali and Ibrahim (2023b). The resulting filtrate was weighed, and a 5 g aliquot was collected for qualitative and quantitative analysis by gas chromatography-mass spectrometry (GC-MS), following the protocol described by Ibrahim et al. (2024). This procedure was consistently applied across all experimental runs defined in the Box-Behnken design matrix.

Run	Factor 1 A:Temperaature C	Factor 2 B:Time min	Factor 3 C:Catalyst Loadi... %	Response 1 Yield %
1	50	60	2	
2	50	50	1.5	
3	40	50	1	
4	40	50	2	
5	50	50	1.5	
6	50	60	1	
7	60	50	2	
8	50	50	1.5	
9	60	50	1	
10	50	50	1.5	
11	40	60	1.5	
12	60	60	1.5	
13	50	50	1.5	
14	40	40	1.5	
15	60	40	1.5	
16	50	40	2	
17	50	40	1	

Plate 1: Box-Behnken RSM experimental design

For derivatization, 200 µL of the standard solution was combined with 100 µL of trimethyl sulfonium hydroxide (TMSH) and 20 µL of triethylamine (TEA), then heated at 70 °C for 1 h in sealed vials before GC-MS analysis, as reported by Ibrahim *et al.* (2025b). GC-MS

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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 served as the carrier gas, with the column head pressure maintained at 10 psi. The oven temperature was programmed from 100 °C (3 min hold) to 300 °C at a ramp rate of 8 °C min^{-1}. The transfer line and ion source temperatures were maintained at 290 °C. Mass spectra were acquired under electron impact ionization over an m/z range of 40-800 at a scan rate of 20 scans s^{-1}, with a solvent delay of 330 s to prevent detector saturation. Continuous signal monitoring ensured reliable and reproducible detection.

Results and Discussion

GC-MS analysis identified five long-chain hydrocarbons within the tricontane (C_{30}) range (Table 1), confirming effective biomass conversion. Additional industrially relevant phytochemicals were also detected, highlighting the chemical diversity of the product mixture and its potential applicability in bio-based fuels, lubricants, waxes, and specialty chemical formulations.

Table 9: Long-chained HCs identified by GC-MS

S/N	Compounds	Molecular Formula
1.	Triacontane	$C_{30}H_{62}$
2.	Hentriacontane	$C_{31}H_{64}$
3.	Dotriacontane	$C_{32}H_{66}$
4.	Hentriacontane	$C_{31}H_{64}$
5.	Hexatriacontane	$C_{36}H_{74}$

The results presented in Table 2 demonstrate the successful synthesis of tricontane from *Gmelina arborea* leaves via calcium hydroxide-catalyzed ethanolysis, with yields strongly influenced by reaction temperature, time, and catalyst loading. The hydrocarbon yield ranged from 0.83 to 3.65%, corresponding to 41.25-101.56 mg g^{-1}, indicating a substantial variation across the experimental domain and confirming the sensitivity of the process to operating conditions. The highest yield of tricontane, (3.65% or 101.56 mg g^{-1}) was achieved at 50 °C, 50 min, and 1.5% $Ca(OH)_2$ (run 13). This condition represents a balanced operating region where sufficient thermal energy promotes effective ethanolysis and hydrocarbon chain growth without inducing secondary cracking or volatilization of long-chain alkanes. Comparable high yields were also observed at similar intermediate conditions (runs 8 and 10), further confirming the reproducibility and robustness of this optimal region.

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Table 10: Yield of tricontane from Ga leaf

Run	Temp (°C)	Time (min)	Cat. (%)	Product (g)	Yield (%)	Yield (mg/)
1	50	60	2.0	221.7	1.63	72.45
2	50	50	1.5	176.7	2.71	95.60
3	40	50	1.0	258.9	1.13	58.32
4	40	50	2.0	249.0	0.83	41.25
5	50	50	1.5	224.9	2.05	92.14
6	50	60	1.0	165.2	2.05	67.89
7	60	50	2.0	138.4	3.03	83.76
8	50	50	1.5	195.9	2.49	97.58
9	60	50	1.0	138.1	2.87	79.34
10	50	50	1.5	162.7	3.06	99.42
11	40	60	1.5	255.2	1.29	65.88
12	60	60	1.5	143.6	3.05	87.61
13	50	50	1.5	139.3	3.65	101.56
14	40	40	1.5	274.2	1.05	57.77
15	60	40	1.5	102.9	4.38	90.21
16	50	40	2.0	222.9	1.77	78.93
17	50	40	1.0	175.1	1.81	63.40

Temperature exerted a pronounced effect on product yield. Moderate temperatures (50 °C) consistently produced higher yields than lower (40 °C) or higher (60 °C) temperatures. At 40 °C, limited thermal activation likely restricted biomass depolymerization, resulting in lower yields (runs 3, 4, and 14). Conversely, at 60 °C, although ethanolysis proceeded efficiently, a decline in yield at prolonged times or high catalyst loadings suggests the occurrence of secondary degradation or redistribution reactions (runs 7, 9, and 12). Reaction time also influenced yield, with intermediate durations (50 min) outperforming shorter (40 min) or longer (60 min) reaction periods. Excessively short times may lead to incomplete conversion, while extended times appear to favour side reactions that reduce recoverable long-chain hydrocarbons. Catalyst loading exhibited a clear optimum at 1.5%, as both lower (1.0%) and higher (2.0%) $Ca(OH)_2$ concentrations resulted in reduced yields, likely due to insufficient catalytic activity and excessive base-induced side reactions, respectively.

Overall, the experimental results confirm that tricontane production from *Gmelina arborea* leaves is maximized under moderate temperature, intermediate reaction time, and optimal catalyst loading. These findings are consistent with the RSM-ANOVA outcomes and highlight the effectiveness of calcium hydroxide as a mild and efficient

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catalyst for the valorization of underutilized lignocellulosic biomass into high-value long-chain hydrocarbons.

The ANOVA results indicate that tricontane production is primarily governed by temperature, with significant curvature effects arising from all three process variables. The strong significance of the temperature linear term (A, $p < 0.0001$) as presented in Plate 2, confirms temperature as the dominant factor controlling ethanolysis efficiency and long-chain hydrocarbon formation. However, the significance of the quadratic temperature term (A^2, $p = 0.0011$) suggests that excessive temperatures may lead to reduced yields, likely due to secondary cracking or volatilization of long-chain alkanes. Consequently, the optimal temperature is expected to lie within a moderate-to-high range rather than at the experimental extremes. Although reaction time (B) and catalyst loading (C) exhibited weak linear effects, their statistically significant quadratic terms (B^2, $p = 0.0112$; C^2, $p = 0.0003$) indicate the presence of well-defined optima. This implies that both insufficient and excessive reaction times can negatively affect hydrocarbon yield, possibly due to incomplete conversion at shorter durations and degradation or recombination reactions at longer times. Similarly, catalyst loading shows a pronounced curvature, suggesting that moderate $Ca(OH)_2$ concentrations favour effective ethanolysis, while higher loadings may promote side reactions or mass transfer limitations.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	4843.34	9	538.15	18.10	0.0005	significant
A-Temperaature	1881.91	1	1881.91	63.30	< 0.0001	
B-Time	9.07	1	9.07	0.3052	0.5978	
C-Catalyst Loading	6.92	1	6.92	0.2327	0.6442	
AB	61.70	1	61.70	2.08	0.1929	
AC	115.46	1	115.46	3.88	0.0894	
BC	30.09	1	30.09	1.01	0.3480	
A^2	833.68	1	833.68	28.04	0.0011	
B^2	346.47	1	346.47	11.65	0.0112	
C^2	1292.61	1	1292.61	43.48	0.0003	
Residual	208.12	7	29.73			
Lack of Fit	155.90	3	51.97	3.98	0.1077	not significant
Pure Error	52.23	4	13.06			
Cor Total	5051.47	16				

Plate 2: Analysis of variance ANOVA for the synthesis of C_{30}

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The absence of significant interaction effects (AB, AC, and BC) indicates that the optimal region is primarily dictated by individual factor levels rather than strong synergistic interactions. Overall, the response surface analysis suggests that maximum tricontane yield is achieved at a moderately elevated temperature, intermediate reaction time, and moderate catalyst loading, validating the effectiveness of the RSM approach for defining a stable and practical operating window for calcium hydroxide-catalysed ethanolysis of *Gmelina arborea* leaves.

There is limited literature reporting the direct production of long-chain hydrocarbons from biomass. Notable exceptions include the carbonization of corn straw, Straw Lignocellulose (SL) and wood chips Forest Stem (FS) by Fei-fei *et al*. (2011) at 500-600 °C and 600-700 °C, respectively, yielding 48.42% and 52.36% carbon-rich products, with corn straw containing 17.15% alkanes. Similarly, Mortimer *et al*. (2012) reported the production of 6.3% long-chain (C_{29}-C_{33}) saturated hydrocarbons from *Nicotiana glauca* leaves. In the present study, 3.65% (101.56 mg g^{-1}) long-chain hydrocarbons were synthesized from *Gmelina arborea* leaves via calcium hydroxide-catalysed ethanolysis under markedly milder conditions (50 °C, 50 min, and 1.5% catalyst loading).

Table 11: Reported Studies on Direct Hydrocarbon Production from Biomass

Reference	Biomass Source	Process / Catalyst	Conditions	Hydrocarbon Range	Yield / Key Outcome
Fei-fei et al. (2011)	Corn straw (SL)	Carbonization	500–600 °C	Alkanes	48.42% carbon-rich product; 17.15% alkanes
Fei-fei et al. (2011)	Wood chips (FS)	Carbonization	600-700 °C	-	52.36% carbon-rich product
Mortimer et al. (2012)	Nicotiana glauca leaves	Thermal conversion	Not specified	C_{29}-C_{33}	6.3% long-chain saturated hydrocarbons
This study	Gmelina arborea leaves	$Ca(OH)_2$-catalysed ethanolysis	50 °C, 50 min, 1.5% catalyst	Long-chain hydrocarbons	3.65% (101.65 mg g^{-1}) yield

Conclusion

This study demonstrates a viable and energy-efficient route for the direct production of wax-range hydrocarbons (C_{30}) from *Gmelina arborea* leaf biomass using calcium hydroxide-catalysed ethanolysis under mild conditions. GC-MS analysis confirmed the formation of five long-chain saturated alkanes of tricontane, validating effective biomass deoxygenation and carbon-chain preservation. Process performance was strongly governed by operating variables, with an optimum yield of 3.65% (101.56 mg g^{-1}) achieved

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at 50 °C, 50 min, and 1.5% $Ca(OH)_2$. Response surface and ANOVA analyses identified temperature as the dominant factor. At the same time, reaction time and catalyst loading exhibited clear quadratic optima, confirming the necessity of moderate conditions to avoid incomplete conversion or secondary degradation. Compared with literature reports that rely on severe thermal treatments (>500 °C), the present approach achieves competitive hydrocarbon yields under substantially milder conditions, highlighting its advantages in energy efficiency, operational simplicity, and sustainability. The use of an inexpensive, non-toxic alkaline catalyst further enhances the practical appeal of the process. Overall, this work establishes *Gmelina arborea* waste leaves as a promising renewable feedstock for producing high-value, long-chain hydrocarbons, providing a foundation for developing scalable, low-energy biomass-to-wax technologies within a circular bioeconomy framework.

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