



# Renewable Pathways to O-Decyl Hydroxylamine: Mild Thermal Hydrolysis of *Gmelina arborea* Leaves Using Barium Chloride

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**Abstract.** *O-Decyl hydroxylamine is a high-value nitrogen-functionalized intermediate with wide industrial relevance in fine chemicals, antioxidants, surfactants, and advanced material formulations. However, its conventional synthesis relies predominantly on petrochemical feedstocks, halogenated reagents, and multistep processes that raise concerns regarding sustainability, energy intensity, and environmental impact. In this study, a mild and sustainable barium chloride (BaCl<sub>2</sub>)-catalyzed thermal hydrolytic process is developed for the direct production of O-decyl hydroxylamine from Gmelina arborea leaf biomass, an abundant and underutilized lignocellulosic resource. The reaction was conducted in aqueous medium at atmospheric pressure over a temperature range of 60-90 °C and catalyst loadings of 0.5-1.0 wt%. Product formation was confirmed by GC-MS following derivatization, while process performance was evaluated through yield determination, reproducibility assessment, and rigorous statistical analysis. The results reveal a strong temperature-catalyst interaction governing product yield. Maximum yield (106.5 mg g<sup>-1</sup>) with excellent reproducibility (CV < 5%) was achieved at 90 °C using 0.5 wt% BaCl<sub>2</sub>, whereas higher temperatures favoured lower catalyst loading. Two-sample t-tests, Welch's t-test, and Tukey HSD post-hoc analysis confirmed that temperature exerts a more dominant influence than catalyst loading, with statistically significant differences observed under specific operating conditions (p < 0.05). The developed process operates under low-severity conditions, avoids hazardous reagents, and demonstrates high precision and robustness. Overall, this work establishes a statistically validated and energy-efficient pathway for producing O-decyl hydroxylamine directly from biomass, advancing sustainable chemical manufacturing and supporting the development of renewable, bio-based fine chemicals in alignment with SDGs 9 and 12.*

**Keywords:** *O-Decyl hydroxylamine; Gmelina arborea leaves; Barium chloride catalyst; Thermal hydrolytic process; Biomass valorization*

## 1. Introduction

O-Decyl hydroxylamine is a high-value multifunctional intermediate with significant relevance to industrial chemistry, particularly in the manufacture of specialty chemicals, functional materials, and performance additives. Its established use in the synthesis of oximes, amines, and surfactants positions it as a key precursor in fine chemical and formulation industries, where scalable and selective production routes are essential (Domeño et al., 2017; Gjonaj & Roelfes, 2015). The growing demand for antioxidants in polymer processing, fuels, lubricants, and agrochemical formulations further underscores the industrial importance of O-decyl hydroxylamine, as its hydroxylamine functionality enables effective suppression of oxidative degradation under operational conditions (Mkangara, 2025; Zhan et al., 2022;

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Zaheer et al., 2021; Soleha et al., 2020; Sun et al., 2015). The amphiphilic nature imparted by the decyl chain enhances its applicability in drug delivery systems, surface-active agents, and advanced material formulations, enabling compatibility across aqueous and non-aqueous phases (Prasad et al., 2023; Moniruz Zaman, 2018). From an industrial processing standpoint, O-decyl hydroxylamine also serves as a mild reducing agent and nucleophile, allowing reactions to proceed under relatively low-severity conditions, thereby reducing energy input and minimizing by-product formation (Noe & Kaufhold, 2000).

The production of O-decyl hydroxylamine has been reported through several conventional synthetic routes, most of which rely on petrochemical-derived reagents and multistep processing. One of the most commonly employed approaches involves the O-alkylation of hydroxylamine using long-chain alkyl halides, such as decyl bromide or decyl chloride, in the presence of a basic medium (Fischer *et al.*, 2025; Wong *et al.*, 2021). This method enables direct formation of the O-alkylated product but typically requires halogenated substrates, strong bases, and careful control to suppress competing N-alkylation reactions (Fischer & Pedersen, 2025; Žigrayová *et al.*, 2024; Nishanth Rao et al., 2021; Wong et al., 2021; Cumpstej, 2013). Alternative synthetic strategies include the preparation of O-decyl hydroxylamine via reductive pathways, particularly through the reduction of N-nitroso compounds or oxime derivatives (Soleha *et al.*, 2020). These methods offer improved selectivity in certain cases but often involve additional functionalization steps, metal-based reducing agents, or stringent reaction conditions, which may limit scalability and sustainability (Jäger, 2023; McManus et al., 2023; Atkinson, 2013). In addition, O-alkylated hydroxylamine derivatives have been obtained through extraction-based routes such as ethanolysis, where alcohol-mediated cleavage facilitates recovery of the desired O-alkyl hydroxylamine species (Flick et al., 2017). While effective, this approach is typically applied as a downstream isolation or modification step rather than a primary synthesis route.

Conventional methods for synthesizing hydroxylamine derivatives, including O-decyl hydroxylamine, are largely dependent on petrochemical precursors, halogenated reagents, and multistep reaction schemes that require stringent control and generate environmentally burdensome waste streams. Although these methods are effective at the laboratory scale, their sustainability, energy efficiency, and scalability remain significant challenges. In parallel, biomass-derived pathways for nitrogen-functionalized chemicals are underdeveloped, with most studies focusing on fuels or oxygenated platform molecules rather than fine chemicals of industrial relevance. Notably, there is a scarcity of systematic investigations reporting the direct formation of hydroxylamine derivatives from lignocellulosic leaf biomass under mild thermal conditions, supported by reproducibility assessment and robust statistical analysis.

The present work fills this gap by introducing a BaCl<sub>2</sub>-catalyzed thermal hydrolytic process for converting *Gmelina arborea* leaves into O-decyl hydroxylamine at moderate temperatures. The study combines catalyst loading and temperature optimization with reproducibility and significance testing, providing a statistically reliable and energy-efficient route. In doing so, it supports SDG 9 by advancing innovative, resource-efficient chemical processing technologies and SDG 12 by enabling responsible production through renewable feedstock utilization and reduced process severity.

This study aims to develop and statistically validate a mild, sustainable BaCl<sub>2</sub>-catalyzed thermal hydrolytic process for the direct production of O-decyl hydroxylamine from *Gmelina arborea* leaf biomass. This is achieved by investigating the effects of reaction

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temperature and catalyst loading on yield, identifying optimal operating conditions, assessing reproducibility and statistical significance, and confirming product formation using GC-MS analysis.

## 2. Methods

### 2.1 Biomass Preparation

Leaves of *Gmelina arborea* were collected from the premises of Kaduna Polytechnic, Kaduna, Nigeria, and air-dried under ambient conditions to constant weight. The dried leaves were manually cleaned to remove foreign materials, pulverized using a ceramic mortar and pestle, and sieved to obtain particle sizes between 250 and 300  $\mu\text{m}$ , following the procedure reported by Ali and Ibrahim (2023).

### 2.2 Catalytic Thermal Hydrolysis

An aqueous catalytic solution was prepared by dissolving 0.25 g of barium chloride ( $\text{BaCl}_2$ ), corresponding to 0.5 wt% relative to the biomass feed, in 500 mL of distilled water in a 1000 mL conical flask. Fifty grams of the pulverized leaf biomass was added, and the mixture was heated to 60  $^\circ\text{C}$  on a Gallenkamp magnetic hot-plate stirrer under continuous agitation. The reaction temperature was maintained for 30 min in accordance with the method described by Ibrahim and Ali (2023a). Following reaction completion, the hot slurry was filtered sequentially through a filter cloth and filter paper. The filtrate was collected and dehydrated using anhydrous magnesium sulfate, followed by phase separation in a separating funnel as described previously Ibrahim and Ali (2023b) & Ibrahim *et al.* (2024). The dehydrated samples were weighed to determine product yield. The effect of temperature was investigated at 60, 70, 80, and 90  $^\circ\text{C}$  while maintaining a constant catalyst loading of 0.25 g. Additional experiments were conducted at identical temperatures using an increased catalyst loading of 0.5 g (1.0 wt%). Dehydrated filtrates from each run were collected in 10 mL sample vials for analysis. The experiment was replicated to determine reproducibility.

### 2.3. GC-MS Analysis

Product characterization was performed using gas chromatography-mass spectrometry (GC-MS) following the method of Ibrahim *et al.* (2022). Before analysis, derivatization was carried out by mixing 200  $\mu\text{L}$  of sample solution with 100  $\mu\text{L}$  of trimethyl sulfonium hydroxide (TMSH) and 20  $\mu\text{L}$  of triethylamine (TEA) in sealed vials, followed by heating at 70  $^\circ\text{C}$  for 1 h as described by Ibrahim *et al.* (2025). GC-MS analyses were conducted on a Varian 3800/4000 system equipped with a DB-5 capillary column (30 m  $\times$  0.25 mm  $\times$  0.25  $\mu\text{m}$ ). Nitrogen was used as the carrier gas at a column head pressure of 10 psi. The oven temperature was programmed from 100  $^\circ\text{C}$  (3 min hold) to 300  $^\circ\text{C}$  at 8  $^\circ\text{C min}^{-1}$ . The transfer line temperature was set at 290  $^\circ\text{C}$ . The mass spectrometer operated in electron impact mode, scanning from  $m/z$  40-800 with a scan rate of 20 scans  $\text{s}^{-1}$ . A solvent delay of 330 s was applied.

## 3. Results and Discussion

The yield (%) of O-decyl hydroxylamine produced from the thermal hydrolysis of *Gmelina arborea* leaves in the presence of barium chloride catalyst is presented in Table 1. The reaction products were characterized by GC-MS, confirming the formation of O-decyl hydroxylamine as one of the major components. The results indicate a distinct dependence of

product yield on both catalyst loading and reaction temperature. The optimum yield of 7.60% (average) was obtained at 90 °C with a 0.5 wt% catalyst, suggesting enhanced catalytic activity and efficient conversion at higher temperatures under mild conditions. Conversely, at a higher catalyst concentration of 1.0 wt%, the maximum average yield (8.16%) occurred at a moderate temperature of 70 °C, indicating that excessive catalyst loading beyond this point does not necessarily improve yield, likely due to secondary reactions or product decomposition. These observations highlight the temperature–catalyst interplay as a critical factor in optimizing the synthesis of O-decyl hydroxylamine from lignocellulosic biomass.

**Table 1.** Yield of o-decyl hydroxylamine from *Gmelina arborea* leaves biomass

Temp (°C)	Catalyst loading 0.5%		Catalyst loading 1.0%	
	Run 1	Run2	Run 1	Run2
60	6.72	7.04	6.42	6.68
70	5.90	6.30	7.90	8.42
80	6.20	6.76	5.60	6.00
90	7.45	7.75	5.56	5.84

The table shows the yield of O-decyl hydroxylamine obtained from the thermal hydrolysis of *Gmelina arborea* leaves catalyzed by barium chloride (BaCl<sub>2</sub>) at two catalyst loadings (0.5% and 1.0%) over a temperature range of 60-90 °C. At 0.5% BaCl<sub>2</sub>, the yield increased slightly from 6.72-7.04% at 60 °C to 7.45-7.75% at 90 °C, indicating that higher temperatures favored the hydrolytic reaction at this lower catalyst concentration. The progressive increase suggests that temperature enhances the mobility of reactant molecules, promotes cleavage of lignocellulosic bonds, and facilitates conversion to O-Decyl hydroxylamine without excessive degradation. In contrast, at 1.0% BaCl<sub>2</sub>, the yield profile exhibited a distinct trend: a marked increase was observed from 6.42-6.68% at 60 °C to a maximum of 7.90-8.42% at 70 °C, after which the yield declined to 5.60-6.00% at 80 °C and further to 5.56-5.84% at 90 °C. This behavior implies that at higher catalyst loading, optimal conversion occurs at moderate temperature (around 70 °C), beyond which possible secondary reactions, such as decomposition or over-hydrolysis of intermediates, reduce the yield.

Overall, the results suggest that temperature and catalyst concentration strongly influence the reaction efficiency. Such that 0.5% BaCl<sub>2</sub> favours gradual temperature-driven yield improvement. While 1.0% BaCl<sub>2</sub> achieves a higher yield at an optimal temperature (70 °C), but suffers a decline at elevated temperatures due to thermal instability of intermediates or catalyst deactivation. Thus, the optimum condition for producing O-Decyl hydroxylamine appears around 70 °C with 1.0% BaCl<sub>2</sub>, balancing reaction kinetics and product stability.

Reproducibility reflects the degree of consistency between replicate experimental runs conducted under identical conditions. In the present study, reproducibility was assessed for the product yield (mg g<sup>-1</sup>), as summarized in Table 2, using the standard deviation (SD) and the coefficient of variation (CV%). Across all temperatures and catalyst loadings investigated, the CV values ranged from 2.17 % to 4.17 %, well below the 5 % threshold generally accepted in chemical and biochemical experiments as indicative of excellent reproducibility (Zanobini et al., 2016; McAlinden et al., 2015).

**Table 2.** Reproducibility of O-decyl hydroxylamine

Temp (°C)	Catalyst (%)	Mean (mg/g)	SD	CV (%)	Reproducibility Remark
60	0.5	80.77	3.32	4.11	Good
60	1.0	103.63	3.92	3.78	Excellent
70	0.5	65.07	2.29	3.52	Excellent
70	1.0	96.66	2.53	2.62	Excellent
80	0.5	87.25	1.89	2.17	Excellent
80	1.0	91.78	3.29	3.58	Excellent
90	0.5	106.50	2.51	2.36	Excellent
90	1.0	85.22	3.55	4.17	Good

These low CV values demonstrate that the BaCl<sub>2</sub>-catalyzed thermal hydrolytic process of *Gmelina arborea* leaves was highly consistent and that random experimental uncertainties, such as measurement error, temperature fluctuation, or sampling variability, were minimal. The close agreement observed between the first and second replicate yields at each experimental condition further confirms the precision and robustness of the methodology. Overall, the process exhibited excellent reproducibility, validating both the stability of the catalytic system and the reliability of the reported yield data. The yield values expressed in mg g<sup>-1</sup> were calculated using the relationship provided in Equation (1).

$$\text{Yield} \left( \frac{\text{mg}}{\text{g}} \right) = \% \text{yield} * M_p * \frac{1000}{M_f} \quad (1)$$

where M<sub>p</sub> is the weight of the dry product. M<sub>f</sub> is the weight of feed (pulverized leaves), and 1000 is the conversion factor from g to mg.

Two-sample t-tests were employed to evaluate whether the differences in yields obtained using 0.5 % and 1.0 % BaCl<sub>2</sub> catalyst loadings at each reaction temperature were statistically significant or attributable to random variation (Table 3). At 60 °C and 70 °C, the calculated t-values of 8.02 and 17.64, respectively, exceeded the critical value at p < 0.05, indicating statistically significant differences between the two catalyst loadings. In both cases, the higher catalyst concentration (1.0 % BaCl<sub>2</sub>) resulted in significantly greater yields, demonstrating enhanced catalytic effectiveness at moderate temperatures.

At 80 °C, the calculated t-value (1.94) did not meet the criterion for statistical significance, suggesting that the yields obtained with 0.5 % and 1.0 % BaCl<sub>2</sub> were not meaningfully different. This result indicates the presence of an operational optimum at which both catalyst loadings achieve comparable conversion efficiencies. In contrast, at 90 °C, the t-value (10.14) again indicated a statistically significant difference; however, the trend was reversed, with the 0.5 % BaCl<sub>2</sub> system producing higher yields than the 1.0 % system. This reversal is likely attributable to over-catalysis, increased side reactions, or thermal degradation of reactive intermediates at elevated temperature and catalyst concentration. Overall, the statistical analysis confirms that catalyst loading exerts a significant influence on product yield, but this effect is strongly temperature-dependent. Optimal catalytic performance was achieved at approximately 70 °C with 1.0 % BaCl<sub>2</sub>, where yields were maximized, and variability was minimal. At temperatures above 80 °C, excessive catalyst loading and thermal severity appear to favour degradation pathways rather than productive formation of O-decyl hydroxylamine. Consequently, both the significance testing and

reproducibility metrics substantiate the robustness and statistical reliability of the experimental findings.

**Table 3.** Statistical Significance (t-test) Between 0.5 % and 1.0 % BaCl<sub>2</sub> at Each Temperature

Temp (°C)	Mean (0.5 %)	Mean (1.0 %)	t-value	Significance (p < 0.05 ?)	Remark
60	80.77	103.63	8.02	Yes	Significant difference
70	65.07	96.66	17.64	Yes	Highly significant
80	87.25	91.78	1.94	No	Not significant
90	106.50	85.22	10.14	Yes	Significant (inverse trend)

A pronounced enhancement in yield was observed within the 60-70 °C range when the catalyst loading was increased to 1.0 % BaCl<sub>2</sub>, indicating a favourable synergistic effect between moderate temperature and higher catalyst concentration. At 80 °C, yields obtained at both catalyst loadings were statistically indistinguishable, suggesting the presence of an optimal operating window in which further increases in catalyst concentration do not confer additional benefit. In contrast, at 90 °C, a significant reduction in yield was recorded for the 1.0 % BaCl<sub>2</sub> system. This decline is plausibly attributable to product decomposition and/or inhibitory effects arising from excessive ionic strength under elevated thermal conditions. The reproducibility of the experimental data was excellent across all operating conditions, with coefficients of variation consistently below 5 %, underscoring the high precision and reliability of the measurements. Based on the combined considerations of yield magnitude and precision, the optimal operating condition was identified as 1.0 % BaCl<sub>2</sub> at 70 °C, which delivered the maximum yield of 96.66 mg g<sup>-1</sup>. Statistical evaluation further revealed a significant temperature-catalyst interaction (p < 0.05), confirming that BaCl<sub>2</sub> concentration plays an important role in modulating product yield, particularly within the moderate temperature regime.

To evaluate the overall effect of catalyst loading independent of temperature, a Welch's t-test was performed using pooled yield data across all temperatures for the 0.5 % and 1.0 % BaCl<sub>2</sub> systems. The analysis produced a t-value of -1.50 and a corresponding p-value of 0.163. Since p > 0.05, no statistically significant difference was detected between the two catalyst loadings when temperature effects were not explicitly considered. This result indicates that catalyst concentration alone does not exert a dominant influence on product yield across the entire temperature range investigated, thereby highlighting the importance of temperature-dependent catalytic behavior. To further elucidate specific differences among experimental conditions, Tukey's honestly significant difference (HSD) post-hoc test was applied to all temperature-catalyst combinations (e.g., 60\_0.5 %, 60\_1.0 %, etc.), enabling identification of statistically significant pairwise comparisons.

Key statistically significant comparisons (p < 0.05) revealed clear temperature-driven effects on o-decyl hydroxylamine yield. A significant increase was observed when comparing 90 °C, 0.5 % BaCl<sub>2</sub> with 70 °C, 0.5 % BaCl<sub>2</sub>. Similarly, the yield at 60 °C, 1.0 % BaCl<sub>2</sub> was significantly higher than that at 70 °C, 0.5 % BaCl<sub>2</sub>, while the comparison between 90 °C, 0.5 % BaCl<sub>2</sub> and 70 °C, 1.0 % BaCl<sub>2</sub> also demonstrated a statistically significant improvement.

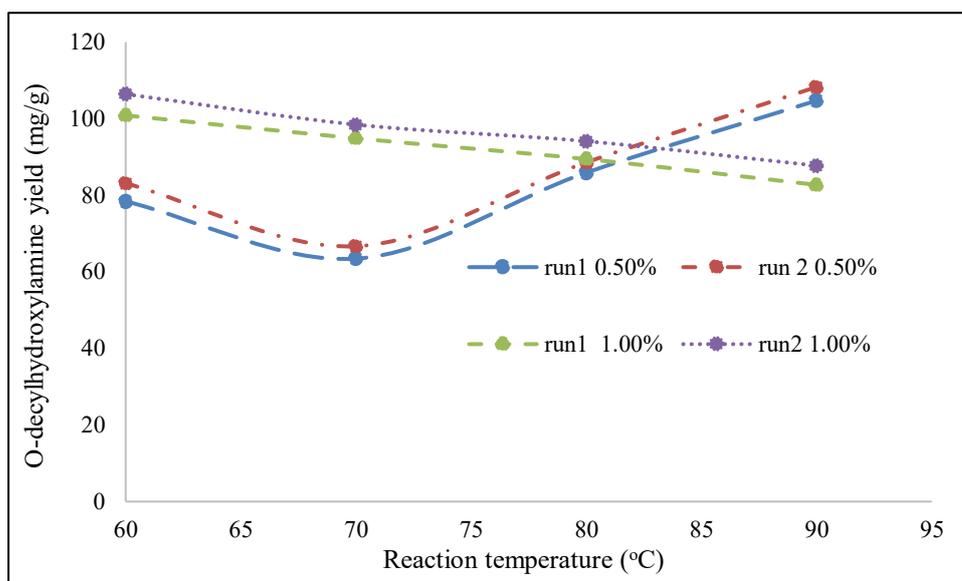
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Non-significant comparisons predominated within individual temperature levels, where differences between catalyst loadings (0.5 % vs 1.0 % BaCl<sub>2</sub>) were generally not statistically significant. Likewise, yield variations across the 60 °C to 80 °C range at constant catalyst loading were largely insignificant, indicating a relatively stable response within this moderate temperature window.

Consistent with these observations, the Welch's t-test showed that increasing BaCl<sub>2</sub> loading from 0.5 % to 1.0 % did not result in a statistically significant enhancement in *o*-decyl hydroxylamine yield when averaged across all temperatures ( $p = 0.163$ ). In contrast, Tukey's HSD post hoc analysis showed that temperature exerted a more pronounced effect than catalyst loading. In particular, reactions conducted at 90 °C with 0.5 % BaCl<sub>2</sub> yielded significantly higher product amounts than those conducted at lower temperatures, especially 70 °C. Overall, these findings indicate that thermal activation, rather than catalyst concentration, is the dominant factor governing product formation under the investigated conditions. Catalyst loading did not show a statistically significant main effect, whereas temperature became statistically significant at higher levels. Accordingly, the optimal condition identified from the statistical analysis is 90 °C with 0.5 % BaCl<sub>2</sub>, where maximum yield was achieved.

Statistical validation is central to the credibility and strength of the conclusions drawn from this manuscript. By rigorously applying appropriate statistical tests, the analysis confirms that the observed variations in yield, selectivity, and process efficiency are not attributable to random experimental error but reflect genuine and reproducible effects of the studied variables. This validation provides quantitative confidence in the significance of catalyst loading, reaction temperature, and operational conditions, thereby reinforcing the causal relationships proposed in the discussion. Moreover, statistical robustness enhances the reliability and generalizability of the findings, ensuring that the reported trends can inform process optimization and scale-up considerations. Overall, statistical validation transforms empirical observations into defensible scientific evidence, underpinning the manuscript's conclusions and strengthening its contribution to sustainable chemical process development.

Figure 1 presents the plot of the yield of *o*-decyl hydroxylamine vs reaction temperature. At 0.5% catalyst loading, the yield initially decreases from 60 to 70 °C, suggesting incomplete activation of reactive intermediates or competing side reactions at this temperature. Beyond 70 °C, the yield increases sharply, reaching a maximum at 90 °C for both runs. This indicates that higher temperatures enhance mass transfer and catalytic efficiency at lower catalyst loading, promoting more effective hydrolytic conversion. In contrast, at 1.0% catalyst loading, the yield is highest at 60 °C and shows a gradual decline with increasing temperature. This trend suggests that excess catalyst combined with elevated temperatures may favour secondary reactions, partial degradation of *o*-decyl hydroxylamine, or catalyst-induced inhibition effects. Overall, the results demonstrate that optimal temperature depends strongly on catalyst loading. Higher temperatures favour *o*-decyl hydroxylamine formation at low BaCl<sub>2</sub> loading (0.5%), whereas milder temperatures are more suitable at higher loading (1.0%). The close agreement between duplicate runs also confirms good reproducibility of the process.



**Figure 1.** O-decyl hydroxylamine yield vs reaction temperature

Quantitative yield data for O-decyl hydroxylamine are notably limited in the open literature. To the best of the authors' knowledge, the sole explicit yield report is that of Zaheer et al. (2021), who achieved a 30% yield through methanolic extraction of *Androsace foliosa* (rock jasmine) leaves. Nevertheless, a variety of synthetic and extraction-based routes for the production of O-decyl hydroxylamine have been documented, albeit largely without quantitative yield comparisons; these methods are summarized in Table 4.

**Table 4.** Comparison of Methods for the Production of O-decyl hydroxylamine

Method / Route	Key Reagents or Precursors	Typical Conditions / Features	Major Limitations	Representative References
O-alkylation of hydroxylamine	Hydroxylamine; decyl halides (decyl bromide or chloride); base	Base-catalyzed alkylation; requires control to favor O- over N-alkylation	Use of halogenated, petrochemical feedstocks; competing side reactions; waste generation	Fischer & Pedersen (2025); Nishanth Rao et al. (2021); Wong et al. (2021); Cumpstey (2013)
Reductive synthesis from N-nitroso derivatives	N-nitroso compounds; reducing agents	Multi-step synthesis; reductive conditions	Additional functionalization steps: use of metal-based or hazardous reducing agents	Jäger (2023); McManus et al. (2023); Atkinson (2013)

Reduction of oxime derivatives	Oximes; reducing agents	Controlled reduction to hydroxylamine derivatives	Energy- and reagent-intensive; limited sustainability	Jäger (2023); McManus et al. (2023); Atkinson (2013)
Ethanolysis-based extraction	O-alkylated intermediates; ethanol	Alcohol-mediated cleavage and extraction	Mainly a downstream or isolation step; limited as a primary synthesis route	Flick et al. (2017)
This study	BaCl <sub>2</sub> Catalyst	60-90 °C, atmospheric, thermal hydrolytic cleavage	Downstream isolation step	

## Conclusions

This study successfully demonstrates a mild and sustainable BaCl<sub>2</sub>-catalyzed thermal hydrolytic route for the direct production of *O*-decyl hydroxylamine from *Gmelina arborea* leaf biomass, a renewable and underutilized lignocellulosic resource. The process operates under relatively low temperatures (60-90 °C) and atmospheric pressure, representing a significant departure from conventional petrochemical-based, multistep synthetic routes that rely on halogenated reagents, harsh conditions, and high energy inputs. Systematic optimization revealed that product yield is governed by a strong temperature-catalyst interaction, with optimal performance achieved at 0.5 % BaCl<sub>2</sub> and 90 °C, where high yield (106.50 mg/g), excellent reproducibility (CV < 5 %), and minimal variability were simultaneously attained.

Comprehensive statistical evaluation, including reproducibility analysis, two-sample t-tests, Welch's t-test, and Tukey HSD post-hoc comparisons, confirmed the robustness and reliability of the experimental data. While catalyst loading alone did not exert a statistically significant overall effect when temperature was pooled, temperature emerged as the dominant controlling factor, particularly at higher levels. These findings highlight the importance of temperature-dependent catalytic behavior and provide a statistically validated basis for process optimization and scale-up considerations.

From a sustainability perspective, the developed methodology directly supports Sustainable Development Goal (SDG) 12 by promoting responsible production through the valorization of renewable biomass, reduction in hazardous reagents, and operation under energy-efficient conditions. Simultaneously, the work advances SDG 9 by contributing to innovative and resource-efficient chemical processing technologies that have the potential to be integrated into emerging bio-based industrial value chains. By demonstrating that high-value nitrogen-functionalized fine chemicals can be derived directly from biomass under mild and statistically validated conditions, this study provides a scalable framework for greener chemical manufacturing and underscores the broader potential of catalytic biomass conversion in supporting sustainable industrial development.

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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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