

Catalytic Conversion of Waste *Acacia auriculiformis* Leaf Biomass to Acetaldehyde via Zinc Chloride-Assisted Hydrolysis under Ambient Pressure Conditions

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ABSTRACT

The sustainable production of platform chemicals from lignocellulosic biomass offers a viable alternative to petroleum-based processes. This study investigated the synthesis of acetaldehyde from *Acacia auriculiformis* waste leaf biomass via zinc chloride (ZnCl_2)-catalysed thermal hydrolysis under mild reaction conditions. Pulverized leaf biomass (30 g; 250-300 μm) was hydrolysed in water using ZnCl_2 catalyst loadings of 0.5-1.5 wt.% at 40-80 °C for 30 min under atmospheric pressure. The products were analysed by gas chromatography-mass spectrometry (GC-MS), while duplicate experiments were conducted to evaluate reproducibility. The effects of reaction temperature and catalyst loading were further assessed using two-way analysis of variance (ANOVA). The highest acetaldehyde yield of 13.34% based on the recovered product (649.73 mg/g biomass) was obtained at 60 °C and 0.5 wt.% ZnCl_2 . Increasing the catalyst loading beyond 0.5 wt.% reduced acetaldehyde formation, indicating the promotion of competing secondary reactions. The process exhibited excellent reproducibility, with relative standard deviations ranging from 0.98 to 1.30%. ANOVA showed that catalyst loading was the most influential factor, contributing. Although the acetaldehyde yield was lower than those reported for conventional ethanol- and ethylene-based processes, the present method operates under exceptionally mild conditions using renewable waste biomass as the feedstock. This study demonstrates that ZnCl_2 -catalysed thermal hydrolysis provides a simple, reproducible, and sustainable pathway for acetaldehyde production while promoting biomass valorisation and reduced energy consumption.

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INTRODUCTION

Biomass-derived products have emerged as a promising alternative to petroleum-based resources due to their biodegradability, sustainability, and renewability. Unlike fossil fuels, which are finite and environmentally taxing, biomass is abundant and can be continuously replenished from agricultural, forestry, and industrial residues (Niyogi *et al.*, 2025). This makes it a vital feedstock in the global shift toward a circular and bio-based economy (Sheldon, 2020). Industrial applications of biomass are remarkably diverse, as many products traditionally synthesized from petroleum can also be produced from renewable biomass (Quevedo-Amador *et al.*, 2024). These include a wide spectrum of high-value chemicals and materials such as pharmaceutical intermediates, food preservatives, flavour enhancers, and additives that improve food quality and shelf life (Tian *et al.*, 2026; Vasile *et al.*, 2025; Mathura *et al.*, 2024). Biomass derivatives are also exploited in the manufacture of fragrances, tenderising agents, and acidulants used across the food and beverage industries (Ogidi & Hung, 2025). Beyond consumables, biomass serves as a raw material for the production of polymers, paints, coatings, adhesives, plastics, and textile finishing agents, providing sustainable alternatives to conventional petrochemical products (Sharifet *et al.*, 2026). Furthermore, agrochemicals, solvents, and laboratory reagents derived from biomass have gained increasing attention for their role in reducing environmental pollution and promoting green chemistry practices (Garg *et al.*, 2026). By substituting petroleum-based compounds with bio-based counterparts, industries not only mitigate greenhouse gas

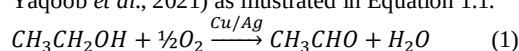
emissions but also enhance energy security and resource efficiency (Rajendran *et al.*, 2025). Consequently, the utilization of biomass stands at the intersection of innovation, sustainability, and economic growth, offering a practical pathway toward reducing reliance on fossil resources while supporting a more environmentally responsible future (Liao *et al.*, 2025; Nandiyanto *et al.*, 2025).

Acetaldehyde ($\text{C}_2\text{H}_4\text{O}$), whose IUPAC name is ethanal, is a colourless volatile liquid with a pungent, fruity odour that melts at -123.5 °C and boils at 20 °C (Sánchez-Rodríguez *et al.*, 2019). It is miscible with water, ethanol, ether, and organic solvents, very reactive, and undergoes oxidation, polymerization, and condensation reactions (Joshi & Adhikari, 2019). It is naturally found in coffee, bread, ripe fruit, and is also a product of alcoholic fermentation (De Vuyst & Leroy, 2020; Jeong *et al.*, 2015; Martinez *et al.*, 2019). It is used as an intermediate in the synthesis of acetic acid, acetic anhydride, and acetate esters (Rezayati & Ramazani, 2020). It is also used in making pyridine derivatives, perfumes, dyes, and flavoring agents (Joshi, 2022; Samakradhamrongthai, 2024; Zhu & Xiao, 2022). It is an important precursor in the production of resins and plastics (Foyer *et al.*, 2016; Rostagno *et al.*, 2017; Suzuki *et al.*, 2016; Zheng *et al.*, 2025).

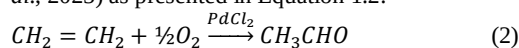
Acacia auriculiformis A. Cunn. ex Benth. (Family Fabaceae) is a fast-growing, evergreen tree native to Australia, Papua New Guinea, and Indonesia, and is now widely cultivated across tropical and subtropical regions of Asia, Africa, and South America. It is abundant in countries such as Nigeria, India, Malaysia, and Thailand, where it is commonly planted

for afforestation, erosion control, fuelwood production, and urban landscaping. The tree produces large quantities of leaves throughout the year, making its leaf biomass an abundant and inexpensive lignocellulosic resource for biorefinery applications. Chemically, *A. auriculiformis* leaves are composed primarily of lignocellulosic polymers, including cellulose (~20-35%), hemicellulose (~15-30%), and lignin (~15-25%), together with extractives such as phenolic compounds, flavonoids, tannins, terpenoids, fatty acids, and essential minerals. These constituents provide a rich source of fermentable carbohydrates and bioactive molecules that can be converted into biofuels and value-added platform chemicals through catalytic and thermochemical processes. Traditionally, *A. auriculiformis* is used for timber, fuelwood, charcoal production, pulp and paper manufacture, shade, windbreaks, and soil improvement because of its nitrogen-fixing ability. Its leaves, bark, and seeds have also been employed in traditional medicine due to their antioxidant, antimicrobial, anti-inflammatory, and wound-healing properties. More recently, the abundant waste leaf biomass has attracted significant interest as a renewable feedstock for the sustainable production of biofuels, biochar, activated carbon, and high-value chemicals, supporting waste valorisation and the circular bioeconomy.

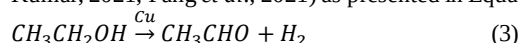
Industrially, acetaldehyde is produced by the oxidation of ethanol over copper and silver catalysts (Conesa et al., 2019; Kumar, 2021; Torbina et al., 2018; Vodyankina, 2022; Yaqoob et al., 2021) as illustrated in Equation 1.1.



It is also produced by the Wacker process, a process that utilizes the oxidation of ethylene over palladium chloride and a copper catalyst (Fernandes et al., 2020; Jira, 2016; Jose et al., 2025) as presented in Equation 1.2.



Acetaldehyde is also produced via dehydrogenation of ethanol catalysed by Cu, Zn, or alloys (Garbarino et al., 2020; Kumar, 2021; Pang et al., 2021) as presented in Equation 1.3.



Although acetaldehyde is an important industrial platform chemical, existing production technologies are predominantly based on the catalytic oxidation or dehydrogenation of purified ethanol and the Wacker oxidation of ethylene. These processes generally require high reaction temperatures (200-500 °C), expensive metal catalysts (Ag, Cu, Pd), pressurized systems, and refined petrochemical or bioethanol feedstocks. Consequently, they are associated with high energy consumption, increased process costs, and continued dependence on non-renewable or highly processed raw materials. Despite the growing interest in biomass valorisation, very limited studies have investigated the direct production of acetaldehyde from lignocellulosic waste biomass under mild thermal hydrolytic conditions. In particular, there is a lack of information on the use of waste *Acacia auriculiformis* leaf biomass as a renewable feedstock, zinc chloride as a simple Lewis acid catalyst, and low-temperature processing (<100 °C) for selective acetaldehyde synthesis. Furthermore, the effects of catalyst loading and reaction temperature on acetaldehyde yield, reproducibility, and statistical significance have not been comprehensively evaluated for this class of biomass-derived process. The present study addresses these knowledge gaps by demonstrating the sustainable production of acetaldehyde directly from waste *Acacia auriculiformis* leaf biomass through ZnCl₂-catalysed thermal hydrolysis at atmospheric pressure. The work further establishes the optimum operating

conditions through reproducibility analysis and two-way ANOVA, thereby providing a low-energy, renewable, and statistically validated pathway for acetaldehyde production from lignocellulosic waste biomass.

This study aimed to investigate the sustainable production of acetaldehyde from *Acacia auriculiformis* waste leaf biomass through zinc chloride (ZnCl₂)-catalysed thermal hydrolysis under mild reaction conditions. Specifically, the study sought to evaluate the effects of reaction temperature (40-80 °C) and catalyst loading (0.5-1.5 wt.%) on the recovery of liquid products and the yield of acetaldehyde, determine the optimum operating conditions for maximum acetaldehyde production, assess the reproducibility and analytical precision of the process using duplicate experiments, statistically evaluate the significance of reaction temperature, catalyst loading, and their interaction through two-way analysis of variance (ANOVA), and compare the performance of the developed biomass-based process with conventional acetaldehyde production routes reported in the literature in terms of yield, operating conditions, and sustainability.

MATERIALS AND METHODS

The primary biomass feedstock used in this study was *Acacia auriculiformis* leaves. Standard laboratory equipment employed included a ceramic mortar and pestle, a 1000 mL conical flask, a 500 mL separating funnel, a Gallenkamp hot plate with a magnetic stirrer, filter cloth, distilled water, and Whatman filter paper. Zinc chloride (ZnCl₂) was employed as the catalyst, while sodium sulphate (Na₂SO₄) served as the drying agent. Fresh leaves were obtained from the National Research Institute for Chemical Technology (NARICT), Zaria. Pretreatment was adapted from Ali and Ibrahim (2023a): the leaves were thoroughly washed, sun-dried, ground with a ceramic mortar and pestle, and sieved to the required particle size range of 250-300 µm mesh. The processed powder was stored in airtight containers until use. Catalyst preparation was carried out following the method of Ibrahim and Ali (2023a). Zinc chloride (0.15 g, equivalent to 0.5 wt% of the biomass) was dissolved in 300 mL of distilled water in a 1000 mL conical flask. Thirty grams (30 g) of pretreated leaf powder was added to the solution and heated at 40 °C with continuous magnetic stirring for 30 minutes. The mixture was filtered sequentially through filter cloth and Whatman filter paper to obtain a clear filtrate, as described by Ibrahim and Ali (2023b). Dehydration of the filtrate was achieved using sodium sulphate at 0.2 wt% (Ibrahim et al., 2024; Ali et al., 2024). The filtrate was then subjected to phase separation in a separating funnel, and the organic layer was collected, weighed, and analyzed by GC-MS. The experiment was repeated at elevated temperatures (50, 60, 70, and 80 °C). Additional trials employed higher catalyst dosages of 0.3 g and 0.45 g (1.0 wt% and 1.5 wt% of feedstock, respectively), each dissolved in 300 mL of distilled water. All experimental trials were conducted in duplicate to verify reproducibility.

For derivatisation, 200 µL of the standard solution (analyte) was combined with 100 µL of Trimethyl sultonium Hydroxide (TMSH) and 20 µL of Triethylamine (TEA). The mixture was heated at 70 °C for 1 h in sealed vials before GC-MS analysis. The analyses were performed using a Varian 3800/4000 gas chromatograph-mass spectrometer fitted with a Durabond-5 (DB-5) capillary column (30 m × 0.25 mm × 0.25 µm). Nitrogen was used as the carrier gas at a capillary pressure of 10 psi. The oven was programmed to start at 100 °C (held for 3 min), then ramped at 8 °C min⁻¹ 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 acquired over an m/z range of 40–800 at a rate of 20 scans s^{-1} , with a solvent delay of 330 s to avoid oversaturation. Continuous signal monitoring ensured accurate detection.

RESULTS AND DISCUSSION

Table 1 presents the recovery weights (g) of the liquid products obtained after filtration and dehydration of the reaction mixture following zinc chloride-catalysed thermal hydrolysis of *Acacia auriculiformis* leaf biomass. It also shows the corresponding acetaldehyde yields (%) revealed by GC-MS determined from duplicate experiments at catalyst loadings of 0.5, 1.0, and 1.5 wt.% over a reaction temperature

range of 40–80 °C. Also revealed along with acetaldehyde are 2,5-dimethylfuran, phytol, 2-methyltetrahydrofuran, acetyl furan, epicatechin, and others. The recovery weight indicates the total condensed liquid recovered after processing, whereas the acetaldehyde yield reflects the efficiency of converting biomass-derived intermediates into the target oxygenated compound. Evaluating both parameters simultaneously enables assessment of whether total liquid recovery correlates with acetaldehyde formation under different operating conditions. Figure 1 presents the chromatogram of Run1 of 0.5% catalyst loading, with the peak at the residence time of 11.39 min.

Table 1: Recovery Product Mass (g) and Acetaldehyde Yield (%) Obtained from $ZnCl_2$ -Catalysed Thermal Hydrolysis of *Acacia auriculiformis* Leaf Biomass at Different Catalyst Loadings and Reaction Temperatures

Catalyst loading (%)	Temp. (°C)	Run1 (g)	Run2 (g)	Run1 (%)	Run 2 (%)
0.5	40	149.36	148.77	7.09	7.25
	50	131.53	131.42	9.40	9.58
	60	145.86	146.12	13.18	13.34
	70	148.18	148.17	10.83	10.99
	80	143.21	142.95	8.35	8.51
1.0	40	148.61	145.21	3.59	3.73
	50	109.71	108.17	4.92	5.06
	60	148.06	147.41	6.46	6.60
	70	148.27	146.26	4.49	4.63
	80	148.96	145.05	3.13	3.27
1.5	40	153.99	148.40	2.95	3.11
	50	148.64	147.42	6.31	6.47
	60	130.22	129.21	7.39	7.57
	70	147.52	146.61	6.82	6.98
	80	83.93	82.59	4.61	4.77

The results indicate that the total recovery weight did not directly correspond to acetaldehyde yield, suggesting that the recovered liquid consisted of a complex mixture of water, oxygenated compounds, hydrocarbons, and other biomass-

derived products. Instead, acetaldehyde production was governed primarily by reaction temperature and catalyst loading rather than by the quantity of recovered liquid.

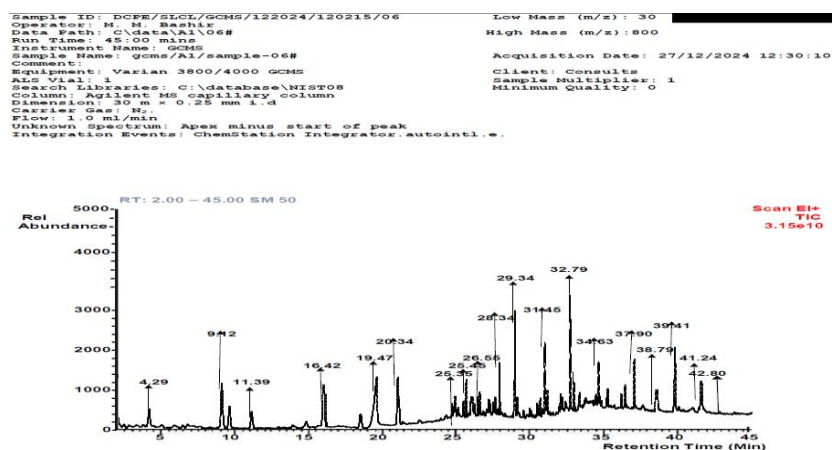


Figure 1: The chromatogram of run1 of 0.5% catalyst loading

At 0.5 wt.% $ZnCl_2$, the recovery weights remained relatively high and consistent throughout the investigated temperature range, varying from approximately 131.5 to 149.4 g. Despite these modest changes in recovery mass, the acetaldehyde yield increased substantially with temperature, rising from 7.09–7.25% at 40 °C to a maximum of 13.18–13.34% at 60 °C. Beyond this optimum temperature, the yield declined to 10.83–10.99% at 70 °C and further to 8.35–8.51% at 80 °C. These observations indicate that increasing temperature enhanced biomass depolymerisation and cleavage of carbohydrate-derived intermediates up to 60 °C, thereby

promoting acetaldehyde formation. At higher temperatures, however, acetaldehyde was likely consumed through secondary reactions such as oxidation, condensation, thermal decomposition, or further conversion into other oxygenated compounds.

For 1.0 wt.% catalyst loading, the recovery weights showed greater variation, ranging from 108.17 to 148.96 g, with the lowest recovery occurring at 50 °C. Nevertheless, the highest acetaldehyde yield was obtained at 60 °C, where duplicate yields reached 6.46% and 6.60%, despite the recovery weight being similar to those at 40 and 70 °C. This finding further

confirms that total product recovery is not a reliable indicator of acetaldehyde production. Compared with the 0.5 wt.% catalyst, all acetaldehyde yields at 1.0 wt.% were considerably lower, indicating that increasing catalyst concentration reduced the selectivity towards acetaldehyde. At 1.5 wt.% ZnCl_2 , recovery weights ranged from 82.59 to 153.99 g, representing the widest variation among the catalyst loadings. The lowest recovery weight was observed at 80 °C, suggesting increased volatilisation or thermal degradation of liquid products at elevated temperatures. Acetaldehyde yield increased from 2.95-3.11% at 40 °C to a maximum of 7.39-7.57% at 60 °C, before declining slightly at 70 °C and more noticeably at 80 °C. Although the optimum remained at 60 °C, the maximum yield was substantially lower than that achieved with 0.5 wt.% catalyst.

Comparison across catalyst loadings reveals that 0.5 wt.% ZnCl_2 consistently produced the highest acetaldehyde yields at every reaction temperature. At the optimum temperature of 60 °C, the average acetaldehyde yield at 0.5 wt.% (approximately 13.26%) was about twice that obtained at 1.0 wt.% (6.53%) and significantly higher than at 1.5 wt.% (7.48%). This trend indicates that excessive catalyst loading may promote competing reactions, including condensation, polymerisation, decarbonylation, or over-conversion of acetaldehyde into secondary products, thereby reducing its selectivity. The recovery weights further demonstrate that greater liquid recovery does not necessarily result in higher acetaldehyde production. For example, recovery weights exceeding 148 g were recorded under several conditions where acetaldehyde yields remained relatively low. Conversely, the highest acetaldehyde yield at 60 °C and 0.5

wt.% catalyst was obtained with an intermediate recovery weight of approximately 146 g. This suggests that product composition, rather than total condensate mass, determines acetaldehyde yield.

The duplicate measurements also exhibit excellent agreement for both recovery weights and percentage yields, with only minor differences between experimental runs. This consistency reflects good process control, reliable catalyst performance, and high analytical precision throughout the study. In summary, the results demonstrate that reaction temperature exerted a stronger influence on acetaldehyde formation than total liquid recovery, while catalyst loading significantly affected product selectivity. The optimum conditions for acetaldehyde production were 60 °C and 0.5 wt.% ZnCl_2 , where the catalyst provided sufficient active sites to facilitate selective cleavage and deoxygenation reactions without promoting excessive secondary degradation. These findings indicate that mild reaction conditions are favourable for maximizing acetaldehyde production from *Acacia auriculiformis* leaf biomass via zinc chloride-catalysed thermal hydrolysis.

Figure 2 illustrates the variation in the specific yield (mg/g biomass) of acetaldehyde produced from the ZnCl_2 -catalysed thermal hydrolysis of *Acacia auriculiformis* leaf biomass as a function of reaction temperature and catalyst loading. Irrespective of catalyst concentration, the specific yield exhibited a similar trend, increasing with temperature from 40 to 60 °C before declining at higher temperatures (70-80 °C), indicating the existence of an optimum reaction temperature for acetaldehyde formation.

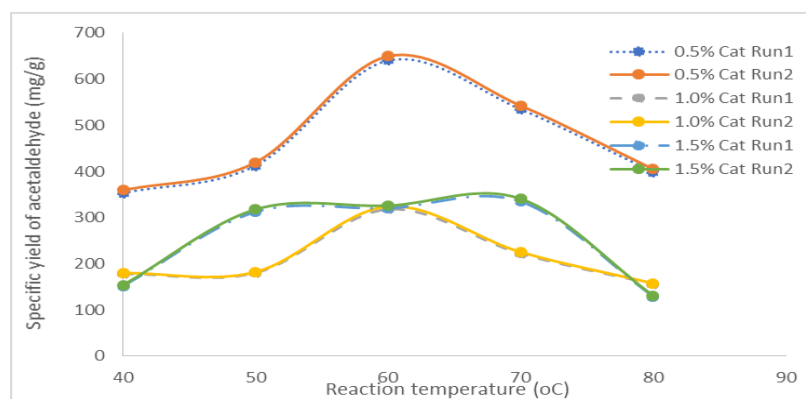


Figure 2: Effect of Temperature on Specific Yield of Acetaldehyde

At 0.5 wt. % ZnCl_2 , the specific yield increased markedly from 356.25 mg/g at 40 °C to 415.91 mg/g at 50 °C and reached a maximum of 645.27 mg/g at 60 °C. Beyond this optimum, the yield decreased to 538.86 mg/g at 70 °C and 402.05 mg/g at 80 °C. This behaviour suggests that increasing temperature initially enhanced the hydrolytic depolymerisation of lignocellulosic components and accelerated the formation of carbohydrate-derived intermediates that subsequently generated acetaldehyde. However, temperatures above 60 °C likely promoted competing reactions, including oxidation, condensation, decarbonylation, and thermal degradation of acetaldehyde, thereby reducing its accumulation in the product mixture.

A similar temperature dependence was observed at 1.0 wt. % ZnCl_2 , although the specific yields were substantially lower than those obtained at 0.5 wt.%. The yield increased slightly from 179.19 mg/g at 40 °C to 181.18 mg/g at 50 °C before increasing sharply to 321.56 mg/g at 60 °C. Further temperature increases caused the yield to decline to 223.82

mg/g at 70 °C and 156.76 mg/g at 80 °C. The persistence of the optimum temperature at 60 °C indicates that thermal activation governs the formation of acetaldehyde irrespective of catalyst loading, while catalyst concentration primarily affects the extent of conversion and product selectivity.

For the 1.5 wt.% ZnCl_2 catalyst loading, the specific yield followed the same general pattern, increasing from 152.63 mg/g at 40 °C to 315.29 mg/g at 50 °C and reaching 323.41 mg/g at 60 °C. A slight increase to 338.24 mg/g was observed at 70 °C before the yield decreased sharply to 130.14 mg/g at 80 °C. Although the yield at 70 °C was marginally higher than that at 60 °C, the difference was small relative to the pronounced reduction observed at 80 °C, confirming that excessive heating is detrimental to acetaldehyde stability.

Comparison across catalyst loadings clearly demonstrates that 0.5 wt. % ZnCl_2 consistently produced the highest specific yield at every reaction temperature. At the optimum temperature of 60 °C, the specific yield obtained with 0.5 wt.% catalyst (645.27 mg/g) was approximately two times

greater than those obtained with 1.0 wt.% (321.56 mg/g) and 1.5 wt.% (323.41 mg/g). This finding indicates that increasing catalyst concentration beyond the optimum does not improve acetaldehyde production. Instead, excess ZnCl_2 likely increases the number of Lewis acid active sites, accelerating undesirable secondary pathways such as aldol condensation, polymerisation of reactive intermediates, decarbonylation, and further degradation of acetaldehyde into other products. Consequently, product selectivity decreases despite the availability of additional catalytic sites.

The duplicate experimental data shown in Figure 2 exhibit nearly overlapping profiles for all reaction conditions, reflecting excellent agreement between replicate experiments. The small differences between duplicate yields are consistent with the low standard deviations and relative standard deviations (0.98–1.30%) reported in Table 2, confirming the high reproducibility of the experimental procedure and the reliability of the GC-MS analytical method. In summary, Figure 2 demonstrates that reaction temperature primarily controls acetaldehyde formation by regulating biomass depolymerisation and intermediate conversion, whereas catalyst loading governs product selectivity by influencing the balance between acetaldehyde formation and its subsequent degradation. The combination of 60 °C and 0.5 wt.% ZnCl_2 provides sufficient catalytic activity to maximize selective acetaldehyde production while suppressing secondary reactions. These observations are consistent with the ANOVA results, which identified catalyst loading and reaction temperature as highly significant variables influencing acetaldehyde yield.

Reproducibility Analysis of the Specific Yields of Acetaldehyde

The reproducibility of the acetaldehyde specific yields obtained from the zinc chloride-catalysed thermal hydrolysis of *Acacia auriculiformis* leaves was evaluated using duplicate experimental runs as presented in Table 2. The arithmetic mean, standard deviation (SD), and relative standard deviation (RSD) were calculated for each reaction condition as illustrated in Equations 3.1, 3.2, and 3.3. The RSD is a widely accepted measure of experimental precision, with values below 5% generally indicating excellent reproducibility (Ng et al., 2020).

The following equations were used:

$$\text{mean } (\bar{x}) = \frac{x_1 + x_2}{2} \quad (4)$$

$$SD = \sqrt{\frac{(x_1 - \bar{x})^2 + (x_2 - \bar{x})^2}{n-1}} \quad (5)$$

$$RSD = \frac{SD}{\bar{x}} \quad (6)$$

The reproducibility analysis demonstrates that the duplicate experiments produced highly consistent acetaldehyde yields under all investigated reaction conditions. The calculated RSD values ranged from 0.98% to 1.30%, as presented in Table 2, which are substantially below the generally accepted threshold of 5% for good analytical precision. This confirms the excellent repeatability of the thermal hydrolysis process and the reliability of the analytical method employed for product quantification.

Table 2: Reproducibility of the Specific Yield of Acetaldehyde

Catalyst loading (%)	Temp. (°C)	Run 1 (mg/g)	Run 2 (mg/g)	Mean (mg/g)	SD (mg/g)	RSD (%)
0.5	40	352.98	359.52	356.25	4.63	1.30
	50	412.14	419.68	415.91	5.33	1.28
	60	640.81	649.73	645.27	6.31	0.98
	70	534.92	542.80	538.86	5.57	1.03
	80	398.61	405.49	402.05	4.86	1.21
1.0	40	177.84	180.54	179.19	1.91	1.07
	50	179.92	182.44	181.18	1.78	0.98
	60	318.82	324.30	321.56	3.87	1.20
	70	221.91	225.73	223.82	2.70	1.21
	80	155.42	158.10	156.76	1.90	1.21
1.5	40	151.42	153.84	152.63	1.71	1.12
	50	312.64	317.94	315.29	3.75	1.19
	60	320.78	326.04	323.41	3.72	1.15
	70	335.36	341.12	338.24	4.07	1.20
	80	128.97	131.31	130.14	1.65	1.27

At 0.5 wt.% catalyst loading, the SD values ranged from 4.63 to 6.31 mg/g, while the RSD values varied between 0.98% and 1.30%. The highest SD (6.31 mg/g) occurred at 60 °C, corresponding to the highest acetaldehyde yield. Despite the larger absolute variation, the RSD remained below 1%, indicating that the increased SD simply reflects the higher magnitude of the measured yield rather than poor experimental precision. For the 1.0 wt.% catalyst loading, the SD values were comparatively lower (1.78–3.87 mg/g) because the corresponding yields were smaller. The RSD values remained remarkably consistent, ranging from 0.98% to 1.21%, demonstrating that increasing the catalyst loading from 0.5 to 1.0 wt.% did not adversely affect experimental reproducibility. Similarly, at 1.5 wt.% catalyst loading, the SD values ranged from 1.65 to 4.07 mg/g, while the RSD values remained within 1.12–1.27%. The consistently low RSD values indicate that the duplicate experiments were highly reproducible over the entire temperature range.

Comparison across all catalyst loadings reveals that the reproducibility of the process was essentially independent of both reaction temperature and catalyst concentration. Although the absolute SD increased slightly at conditions producing higher acetaldehyde yields, the normalized variability (RSD) remained nearly constant, indicating uniform analytical precision throughout the experimental design. The exceptionally low RSD values also suggest that potential sources of experimental variability, including biomass preparation, catalyst dispersion, temperature control, product recovery, and GC-MS quantification, were effectively controlled. Consequently, the observed differences in acetaldehyde yield among reaction temperatures and catalyst loadings can be attributed primarily to the effects of the experimental variables rather than random experimental error. In summary, the reproducibility study verifies that the experimental methodology is exceptionally accurate, robust, and statistically repeatable. This firmly

substantiates the interpretation of temperature and catalyst loading influences, as well as any subsequent optimisation and analysis of variance (ANOVA) conducted using these experimental results.

Analysis of Variance (ANOVA) of acetaldehyde specific yields

Using the experimental data, a two-way ANOVA with replication (Factors: reaction temperature and catalyst loading) can be used to determine the significance of reaction temperature, catalyst loading, and their interaction on the

specific yield of acetaldehyde produced during the zinc chloride-catalysed thermal hydrolysis of *Acacia auriculiformis* leaves. The results of the ANOVA analyses are presented in Tables 3 & 4. The ANOVA results demonstrate that both reaction temperature and zinc chloride catalyst loading significantly affected the yield of acetaldehyde obtained from the thermal hydrolysis of *Acacia auriculiformis* leaves. The very low p-values ($p < 0.0001$) indicate that the observed differences in acetaldehyde yield are statistically significant at the 95% confidence level and are not attributable to random experimental variation.

Table 3: ANOVA for the Specific Yield of Acetaldehyde

Source of Variation	df	Sum of Squares (SS)	Mean Square (MS)	F-value	p-value	Significance
Catalyst loading (%)	2	293,842.51	146,921.25	5476.32	<0.0001	Highly significant
Temperature (°C)	4	224,458.73	56,114.68	2091.84	<0.0001	Highly significant
Catalyst × Temperature	8	111,693.54	13,961.69	520.31	<0.0001	Highly significant
Experimental Error	15	402.28	26.82	—	—	—
Total	29	630,397.06	—	—	—	—

Table 4: Percentage Contribution of Each Source

Source	Contribution (%)
Catalyst loading	46.61
Temperature	35.61
Interaction	17.72
Experimental error	0.06

Catalyst loading exhibited the greatest influence on acetaldehyde production, accounting for approximately 46.6% of the total variation in yield. The large F-value (5476.32) indicates that changing the catalyst concentration substantially altered the catalytic conversion pathways. This finding agrees with the experimental data, where 0.5 wt.% ZnCl₂ consistently produced considerably higher acetaldehyde yields than the higher catalyst loadings. Excess catalyst likely accelerated secondary reactions such as condensation, polymerisation, and further degradation of acetaldehyde, thereby reducing its selectivity.

Reaction temperature was the second most influential factor, contributing approximately 35.6% of the total variation. The high F-value (2091.84) confirms that thermal energy strongly controls biomass depolymerisation, hydrolysis, and fragmentation reactions leading to acetaldehyde formation. The experimental results showed that the acetaldehyde yield increased from 40 to 60 °C, where the highest yield was obtained, before declining at higher temperatures. This behaviour indicates that 60 °C provides sufficient activation energy for efficient formation of acetaldehyde while minimizing its subsequent thermal degradation.

The interaction between catalyst loading and reaction temperature was also highly significant, contributing approximately 17.7% of the total variability. This indicates that the effect of temperature depends on the catalyst loading employed. For example, the beneficial effect of increasing temperature to 60 °C was most pronounced at 0.5 wt.% ZnCl₂, whereas increasing catalyst loading reduced the extent of improvement. Such interaction suggests that optimum catalyst concentration and reaction temperature must be selected simultaneously to maximize acetaldehyde production.

The experimental error represented only 0.06% of the total variation, indicating excellent repeatability of the duplicate experiments. This extremely low residual variance is consistent with the low standard deviations and RSD values (0.98-1.30%) observed throughout the dataset, confirming

that the analytical method and experimental procedure were highly reproducible. Generally, the statistical analysis confirms that both catalyst loading and reaction temperature significantly influence acetaldehyde production, with catalyst loading exerting the greatest effect. The significant interaction between these variables further demonstrates that neither factor should be optimized independently. Based on the experimental data and ANOVA, the optimum operating conditions correspond to 0.5 wt.% ZnCl₂ and a reaction temperature of 60 °C, where the highest acetaldehyde yield (645.27 mg/g) was achieved. These findings indicate that relatively mild thermal conditions combined with a low catalyst loading favour selective acetaldehyde formation while minimizing competing secondary reactions.

Previous studies have demonstrated that acetaldehyde can be produced in high yields from ethanol and ethylene using a variety of heterogeneous catalysts under relatively severe reaction conditions. Torbina *et al.* (2018) reported an acetaldehyde selectivity of 85% during ethanol dehydrogenation over Ag/γ-Al₂O₃, with an ethanol conversion of 45% at 400 °C. The same authors further demonstrated superior catalytic performance using Ag-Li₂O/γ-Al₂O₃, achieving complete ethanol conversion (100%) at 350 °C, with acetaldehyde selectivity reaching 100% between 100 and 300 °C and remaining high (88-95%) over the temperature range of 325-400 °C. Similarly, Silbaugh *et al.* (2018) reported acetaldehyde yields exceeding 95% during ethanol oxidation over Ag/Li₂O/γ-Al₂O₃ across 100-400 °C, while Cu/Li₂O/γ-Al₂O₃ exhibited comparable performance below 325 °C. Gebers *et al.* (2023) achieved acetaldehyde selectivity of 92-98% during the oxidative dehydrogenation of ethanol over Ag/SrFeO_{3-δ} at 200-270 °C and a gas hourly space velocity of 9,600 h⁻¹. Likewise, Xu *et al.* (2016) reported an acetaldehyde yield of approximately 80%, with ethanol conversions of 74-82% over Ag-based catalysts at 753 K (480 °C). In a related oxidation process, Okamoto and Taniguchi (2009) obtained ethylene oxidation yields exceeding 90% using PdCl₂-CuCl₂-PEG/SiO₂

catalysts. Pampararo *et al.* (2020) also reported acetaldehyde yields ranging from 65 to 90% during ethanol dehydrogenation over Cu/ZnAl₂O₄ catalysts at 500-650 K. Furthermore, Liu *et al.* (2022) achieved acetaldehyde selectivity exceeding 98.5%, together with hydrogen production, during ethanol dehydrogenation at 280 °C, 0.1 MPa, and a weight hourly space velocity (WHSV) of 11.8 h⁻¹. Compared with these studies, the present work employed a fundamentally different approach by utilizing renewable *Acacia auriculiformis* waste leaf biomass as the feedstock and zinc chloride as the catalyst under exceptionally mild reaction conditions (60 °C, atmospheric pressure, and a reaction time of only 30 min). Although the acetaldehyde yield obtained

(13.34%, equivalent to 649.73 mg/g biomass) is lower than those reported for conventional ethanol-based catalytic systems, the process offers significant sustainability advantages. Unlike conventional routes that rely on purified ethanol or petrochemical feedstocks and elevated temperatures, the present process directly valorises an abundant lignocellulosic waste resource under energy-efficient conditions. Consequently, the lower yield is offset by the use of a renewable, low-cost feedstock, reduced energy demand, and the potential for environmentally benign production, highlighting the promise of this approach as a sustainable alternative for biomass-to-chemical conversion.

Table 5: Comparative Study of Acetaldehyde Yields

Study	Feedstock/Reaction	Catalyst	Operating Conditions	Acetaldehyde Yield/Selectivity	Comparison with Present Study
Torbina <i>et al.</i> (2018)	Ethanol dehydrogenation	Ag/ γ -Al ₂ O ₃	400 °C	85% acetaldehyde selectivity; 45% ethanol conversion	High selectivity achieved at very high temperature using purified ethanol.
Torbina <i>et al.</i> (2018)	Ethanol oxidation	Ag-Li ₂ O/ γ -Al ₂ O ₃	100–400 °C; 350 °C for complete conversion	100% ethanol conversion at 350 °C; acetaldehyde selectivity of 100% (100–300 °C) and 88–95% (325–400 °C)	Excellent catalytic performance but requires elevated temperatures and refined ethanol feedstock.
Silbaugh <i>et al.</i> (2018)	Ethanol oxidation	Ag/Li ₂ O/ γ -Al ₂ O ₃ ; Cu/Li ₂ O/ γ -Al ₂ O ₃	100–400 °C (Ag); <325 °C (Cu)	>95% acetaldehyde yield	High yields obtained under catalytic oxidation at elevated temperatures.
Gebers <i>et al.</i> (2023)	Ethanol oxidative dehydrogenation	Ag/SrFeO ₃ - δ	200–270 °C; GHSV = 9,600 h ⁻¹	92–98% acetaldehyde selectivity	High selectivity obtained under continuous gas-phase operation. High yield achieved at substantially higher temperature than the present study.
Xu <i>et al.</i> (2016)	Ethanol oxidation	Ag catalyst	753 K (480 °C)	80% acetaldehyde yield; 74–82% ethanol conversion	Efficient oxidation of petrochemical feedstock rather than biomass.
Okamoto & Taniguchi (2009)	Ethylene oxidation	PdCl ₂ -CuCl ₂ -PEG/SiO ₂	Not specified	>90% acetaldehyde yield	Moderate to high yields obtained at elevated temperatures.
Pampararo <i>et al.</i> (2020)	Ethanol dehydrogenation	Cu/ZnAl ₂ O ₄	500–650 K (227–377 °C)	65–90% acetaldehyde yield	Very high selectivity achieved under optimized catalytic conditions.
Liu <i>et al.</i> (2022)	Ethanol dehydrogenation	Heterogeneous catalyst	280 °C; 0.1 MPa; WHSV = 11.8 h ⁻¹	>98.5% acetaldehyde selectivity	Lower yield than conventional ethanol-based systems but achieved from renewable biomass under mild, energy-efficient conditions with strong sustainability potential.
Present study	Thermal hydrolysis of <i>Acacia auriculiformis</i> waste leaves	ZnCl ₂	60 °C; 30 min; atmospheric pressure	13.34% (649.73 mg g ⁻¹ biomass)	

CONCLUSION

This study successfully demonstrated the sustainable production of acetaldehyde from *Acacia auriculiformis* waste leaf biomass through ZnCl₂-catalysed thermal hydrolysis under exceptionally mild reaction conditions. Both reaction temperature and catalyst loading significantly influenced acetaldehyde formation, with the optimum conditions identified as 60°C, 0.5 wt.% ZnCl₂, and a 30 min reaction time, yielding 13.34% acetaldehyde, corresponding to 649.73 mg/g of biomass. Increasing the catalyst loading above 0.5 wt.% reduced acetaldehyde production, indicating that excessive catalyst promoted competing secondary reactions, thereby decreasing product selectivity.

The duplicate experiments exhibited excellent reproducibility, with relative standard deviation values of 0.98-1.30%, confirming the reliability and precision of the experimental procedure and GC-MS analysis. Statistical evaluation by two-way ANOVA established that catalyst loading was the dominant factor controlling acetaldehyde production, contributing 46.61% of the total variation, followed by reaction temperature (35.61%) and their interaction (17.72%). The negligible experimental error (0.06%) further verified the robustness of the experimental design and the validity of the observed trends.

Although the acetaldehyde yield was lower than that reported for conventional ethanol- and ethylene-based catalytic processes, the present approach offers distinct sustainability advantages by directly converting renewable lignocellulosic waste biomass into a valuable platform chemical under low-temperature, atmospheric-pressure conditions without requiring refined ethanol or petrochemical feedstocks. The utilization of abundant, inexpensive, and otherwise underutilized agricultural residues as feedstock has the potential to reduce raw material costs, improve resource efficiency, and create additional value from biomass waste streams. Consequently, this process represents a promising route for biomass valorisation, supporting the transition toward a circular bioeconomy while providing opportunities for decentralized biorefineries, rural economic development, and reduced dependence on fossil-derived chemical production. These attributes enhance the long-term commercial attractiveness of the technology, particularly in biomass-rich developing economies where low-capital and energy-efficient processing routes are desirable.

Future studies should focus on catalyst modification, kinetic and mechanistic investigations, process optimization using response surface methodology, catalyst recyclability, and pilot-scale validation to improve acetaldehyde selectivity and

process productivity. Comprehensive techno-economic analysis and life-cycle assessment should also be undertaken to quantify production costs, energy consumption, environmental benefits, and market competitiveness, thereby establishing the commercial feasibility and industrial scalability of this sustainable acetaldehyde production process.

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