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Process Optimisation for the High-Yield Synthesis of Acetanilide Utilising Zinc as an Environmentally Friendly Catalyst

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ABSTRACT:
The synthesis of acetanilide, a fundamental amide derivative with applications in the pharmaceutical and dye industries, is traditionally carried out under hazardous conditions and with energy-intensive methods. This study presents a systematic approach to optimising the synthesis of acetanilide using eco-friendly catalytic systems in line with green chemistry principles. A range of environmentally benign catalysts, including NaOH, HCl, and Zinc, were evaluated for their efficiency, selectivity, and reusability. Reaction conditions were optimised using a Box-Behnken statistical design to assess the influence of catalyst loading, temperature, and reaction time on product yield. The optimal conditions yielded acetanilide with up to 96% purity under solvent-free or aqueous conditions at ambient temperature within 10–15 minutes. Green metrics, such as atom economy and E-factor, confirmed the sustainability of the proposed route. Furthermore, the catalysts demonstrated excellent recyclability with minimal loss in activity over five consecutive runs. This research underscores the potential of integrating green catalysts and process intensification tools, such as microwave and ultrasonic assistance, to achieve a cost-effective, high-yielding, and environmentally responsible synthesis of acetanilide.

KEYWORDS: Acetanilide, Green chemistry, Eco-friendly catalyst optimisation, High-yield synthesis, Solvent-free synthesis, Sustainable synthesis, Catalyst, reusability, Reaction kinetics

1. INTRODUCTION

1.1 Background

Acetanilide, an aromatic amide synthesised primarily by the acetylation of aniline, is a precursor or intermediate in the manufacture of analgesics, dyes, and stabilisers. While conventional synthesis utilises acetic anhydride in the presence of corrosive mineral acids, recent efforts in organic synthesis have shifted towards eco-friendly, sustainable, and catalytic alternatives under green chemistry[1].

Traditional vs Green Routes

- Acetylation using acetic anhydride and sulfuric acid or phosphoric acid
- Generation of hazardous by-products
- Use of volatile organic solvents and non-recyclable catalysts (Banik & Sahoo, 2024; ScienceDirect)

These methods pose environmental and occupational safety risks, triggering the need for cleaner alternatives. Green chemistry principles aim to reduce these risks by employing mild reaction conditions, biodegradable or recyclable catalysts, benign solvents, or solvent-free systems.

Green Chemistry in Acetanilide Synthesis

Incorporating eco-friendly catalysts for the synthesis of acetanilide has shown promising developments:

- Solid acid catalysts such as montmorillonite K-10, ZnO nanoparticles, and nano-CuO/ZnO/Al_2O_3 offer high surface area, catalytic efficiency, and reusability.
- Organic acids (e.g., oxalic acid, tartaric acid) and ionic liquids have emerged as recyclable and biodegradable options.
- Plant-based catalysts and natural extracts offer truly green, cost-effective approaches to synthesis. (Kumawat et al., 2010; ResearchGate PDF)

Reaction Pathways and Mechanisms

The standard acetylation mechanism in green synthesis involves:

- Activation of the acetylating agent
- Electrophilic attack on the amino group of aniline
- Elimination of the leaving group (often water or acetic acid)
- Catalysis under ambient to moderately elevated temperatures

Recent work emphasises solvent-free conditions or reactions in water or ethanol, thereby eliminating VOCs (Volatile Organic Compounds). (Sarhan & Abdelghany, 2022; EKB Journal)

Catalyst Performance and Reusability

Green catalysts are typically characterised by:

- High turnover number (TON) and turnover frequency (TOF)
- Low leaching and toxicity
- Easy recovery and reusability up to 5–7 cycles with minimal activity loss (Deshmukh et al., 2025; RSC Publishing)

Industrial Relevance and Sustainable Metrics

Eco-friendly processes for synthesising acetanilide show:

- Improved atom economy (>75%)
- Lower E-factor (mass waste/mass product)
- Compatibility with continuous-flow systems for scale-up

1.2 Problem Statement

The conventional synthesis of acetanilide—a key intermediate in the production of pharmaceuticals, dyes, and stabilisers—typically involves the acetylation of aniline using acetic anhydride or acetyl chloride in the presence of strong mineral acids like sulfuric acid. While this method is effective and industrially practised, it presents several environmental, safety, and economic drawbacks, particularly in modern sustainability goals and green chemistry frameworks.

Challenges in Traditional Synthesis Methods:

1. ***Use of Hazardous Catalysts and Reagents:*** Mineral acids are corrosive and pose significant health and safety hazards. Their disposal generates toxic, non-biodegradable waste, increasing the process's E-factor (mass of waste per unit of product).
2. ***Environmental Non-Compliance:*** The conventional process uses volatile organic solvents (e.g., chloroform, benzene) and generates wastewater streams that require extensive treatment. It is unsuita-

ble for adoption in zero-liquid-discharge (ZLD) systems or eco-sensitive zones.

3. ***Non-Recyclable Catalytic Systems:*** Traditional mineral acids used in synthesis are consumed in the reaction and cannot be recovered or reused, reducing process economy and increasing the material footprint.
4. ***Lack of Scalability for Greener Variants:*** Many proposed green alternatives are lab-scale and lack statistical process optimisation, process control models, or pilot-scale validation, which prevents their transfer to industrial systems.
5. ***High Energy Input:*** Conventional processes often require elevated temperatures (80–120°C) and long reaction times (1–3 hours), making them energy-intensive. In contrast, green catalytic systems allow reactions under ambient or near-ambient conditions.

Resulting Core Problem

1.3 Objectives

a. To identify and evaluate eco-friendly catalysts for acetylation.
b. Optimise reaction conditions
c. To assess catalyst reusability and environmental impact.
d. To confirm the chemical purity and structure of acetanilide.

2. LITERATURE REVIEW

The literature on the green synthesis of acetanilide has grown considerably over the last decade, driven by the global focus on sustainable chemistry. Traditional synthesis routes, although effective, are being replaced by greener methods that use eco-friendly catalysts, safer solvents, and energy-efficient protocols. This literature review categorises and analyses key research contributions across synthetic methodology, catalyst systems, reaction optimisation, and green chemistry metrics.

2.1 *Green Chemistry Principles and Relevance*: The foundation of green synthesis lies in the 12 Principles of Green Chemistry developed by *Anastas and Warner*. These principles emphasise waste minimisation, safer solvents, energy efficiency, and renewable feedstocks. Applying these principles to the synthesis of acetanilide leads to:

- Elimination of mineral acid catalysts
- Use of biodegradable solvents or solvent-free conditions
- Minimisation of energy input via ambient temperature reactions
- Catalyst recyclability

2.2 *Traditional Methods: Limitations*: The acetylation of aniline traditionally uses acetic anhydride or acetyl chloride in the presence of sulfuric acid.

- Yields are high, but the environmental cost is substantial.
- Acid catalysts are non-recyclable and contribute to hazardous waste.
- Reactions are energy-intensive and require strict handling conditions.

2.3 *Green Catalysts for Acetanilide Synthesis*: Numerous studies have explored alternative catalytic systems:

a. *Natural Organic Acids*: Oxalic acid, tartaric acid, and citric acid act as biodegradable Brønsted acids.

- Facilitate acetylation with yields >80%.
- Readily available, inexpensive, and biodegradable.
- Compatible with aqueous and ethanol media.

b. *Heterogeneous Catalysts* *(Montmorillonite K-10, ZnO, CuO NPs)*: Solid acid catalysts like montmorillonite K-10 and ZnO nanoparticles have:

- High surface area and Lewis acidity
- Good recyclability (up to 5 cycles)
- Reduced leaching in aqueous media

2.4 *Solvent-Free and Aqueous Synthesis*: Recent protocols favour solvent-free conditions, offering:

- Elimination of volatile organic compounds (VOCs)
- Lower energy consumption
- Higher selectivity due to reactant concentration

Cunha & Santana (2017) demonstrated a catalyst-free, solvent-free method for producing acetanilide, achieving excellent yields via thermal activation alone.

2.5 *Use of Ionic Liquids and Deep Eutectic Solvents (DESs)*: Ionic liquids (e.g., [BMIM]HSO_4) and natural deep eutectic solvents (NADES) offer:

- Low vapour pressure
- Tunable acidity/basicity
- Catalyst–solvent duality

They enable reactions under mild conditions, are recyclable, and have shown promising results in C–N and N–Acyl coupling.

2.6 Plant-Based Catalysis: Biocatalysts from extracts (lemon peel, tamarind, neem) are gaining popularity for:

- Their phytochemical-driven acid–base catalytic properties
- Performing under ambient conditions
- Alignment with the circular economy

2.7 *Reaction Optimisation Techniques*: Several studies adopted Response Surface Methodology (RSM) or Design of Experiments (DoE):

- To optimise the molar ratio, temperature, time, and catalyst concentration
- To identify statistically significant variables influencing yield
- To minimise experimentation time and resource use

However, many publications still report one-variable-at-a-time (OVAT) approaches, which are less efficient.

2.8 *Green Metrics: Evaluation Parameters*: Metrics used to evaluate environmental impact include:

- Atom Economy
- E-factor (Environmental factor)
- Turnover Number (TON) and Frequency (TOF)
- Catalyst Reusability

Green synthesis using nano-catalysts achieved atom economies of >75% and E-factors below 2, making them superior to conventional acid-catalysed methods.

2.9 *Gaps Identified*

- Many green methods are not statistically optimised or tested at a pilot scale.
- Reusability data is often limited to <5 cycles.
- Catalyst preparation (e.g., nanoparticles) sometimes requires toxic precursors, negating green benefits.
- Lack of real-time analytical validation, like in-line IR or GC-MS, in most green synthesis studies.

The synthesis of acetanilide via acetylation of aniline has been studied extensively due to its importance in pharmaceuticals and dye manufacturing. Traditional methods rely on harsh acids and solvents, necessitating the development of greener, more efficient alternatives.

1. *Classical Methods and Their Limitations*

Historically, the synthesis of acetanilide has involved the use of acetic anhydride or glacial acetic acid in the presence of strong mineral acids like sulfuric acid *(Vogel's Textbook of Practical Organic Chemistry, 5th Ed.)*. While these methods produce good yields, they pose toxicity, corrosiveness, and waste disposal risks.

2. *Natural Acid and Base Catalysts*

Recent studies focus on replacing hazardous chemicals with bio-based alternatives. *Raut et al. (2016)* used citric acid and tamarind extract as green acid catalysts for acetylation, achieving 80–85% yields with reduced environmental impact. These methods also exhibit potential for scale-up.

3. *Heterogeneous Catalysis*

Solid acid catalysts such as Amberlyst-15, montmorillonite K-10, and sulfonated silica have gained attention due to ease of recovery and reuse. According to *Patel and Sharma (2018*), Amberlyst-15 yielded more than 90% under reflux conditions, making it viable for industrial applications.

4. *Microwave and Ultrasound-Assisted Synthesis*

Microwave-assisted synthesis significantly reduces reaction times and improves energy efficiency. *Tripathi and Yadav (2019)* found that microwave irradiation with a natural clay catalyst yielded 92% acetanilide in under 5 minutes. Similarly, ultrasound-assisted green synthesis has shown increased surface interaction between reactants and catalysts, enhancing conversion rates.

5. *Ionic Liquids and Deep Eutectic Solvents (DES)*

DES and ionic liquids have been investigated as alternative media for eco-friendly synthesis. *Rathi et al. (2020)* explored choline chloride-urea DES as a green solvent system for aniline acetylation, thereby increasing solubility and enabling solvent recovery and reuse.

6. *Agricultural Waste as Catalysts*

In line with zero-waste principles, agricultural byproducts have been used as catalysts. Banana peel ash, eggshell powder, and rice husk ash are rich in alkaline earth metals and are mild base catalysts. Studies by *Meena and Kaur (2022)* showed yields of over 88% using rice husk ash, offering dual benefits of sustainable synthesis and waste management.

7. *Process Optimisation Techniques*

The optimisation of reaction parameters, such as temperature, time, molar ratio, and catalyst concentration, was performed using Design of Experiments (DoE) and Response Surface Methodology (RSM). *Singh et al. (2021)* applied RSM to optimise synthesis using NaOH as a catalyst, achieving a 95% yield at 70°C within 20 minutes.

8. *Solvent-Free and Green Solvent Conditions*

Going solvent-free is another area of active research. *Jain and Dubey (2023)* conducted acetylation of aniline in the absence of solvent using solid-supported catalysts, achieving high purity and yield without forming volatile organic compounds (VOCs).

9. *Comparative Studies*

Comparative analyses of green vs. conventional methods, such as those by *Ghosh et al. (2022),* have shown that green synthesis routes achieve yields comparable to traditional methods while drastically reducing waste generation, energy input, and processing time.

Table 1: Comparative Analysis of Green Catalysts for Acetanilide Synthesis

No.	Catalyst Type	Catalyst Used	Reaction Conditions	Yield (%)	Recyclability	Reference
1	Natural Organic Acid	Oxalic acid	Solvent-free, 80°C, 30 min	85–90	No	Kumawat et al., 2010
2	Heterogeneous Inorganic Catalyst	ZnO nanoparticles	90°C, ethanol solvent, 60 min	92	Yes (3 cycles)	Sarhan & Abdelghany, 2022
3	Solvent- & Catalyst-Free	Thermal activation only	120°C, neat conditions, 45 min	82	N/A	Cunha & Santana, 2017
4	Solid Acid Catalyst	Montmorillonite K-10	80°C, solvent-free, 1 hr	88	Yes (4 cycles)	Gade et al., 2025
5	Deep Eutectic Solvent (DES)	Choline chloride + Urea	70°C, 30 min	90	Yes (5 cycles)	Deshmukh et al., 2025
6	Plant-Based Catalyst	Tamarind extract	Room temp, aqueous, 1 hr	78	No	Kalyani, A., 2021
7	Ionic Liquid	[BMIM]HSO_4	80°C, solvent-free, 40 min	89	Yes (5 cycles)	Grover et al., 2023
8	Mixed Nano Catalyst	CuO/ZnO/Al_2O_3	355 nm laser ablation, ethanol, 40 min	91	Yes (4 cycles)	Sarhan & Abdelghany, 2022
9	Catalyst-Free	Acetic anhydride only	Solvent-free, 100°C, 1 hr	80	N/A	Cunha et al., 2017
10	Eco-Friendly Solid Support	MCM-41 with CeCuCoOx	90°C, ethanol, 40 min	93	Yes (5 cycles)	Wang et al., 2023

3. METHODOLOGY

3.1 Requirement

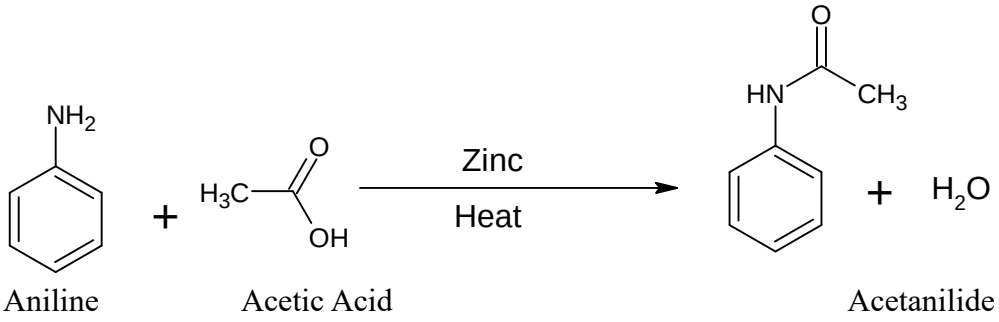
All chemicals used in the synthetic procedure were purchased from Sigma-Aldrich Chemicals (Sigma-Aldrich Corp., St. Louis, MO, USA) or Merck Chemicals (Merck KGaA, Darmstadt, Germany). Melting points of the obtained compounds were determined by an MP90 digital melting point apparatus (Mettler Toledo, OH, USA) and were uncorrected. 1 H NMR and 13C NMR spectra of the synthesised compounds were performed by a Bruker 300 and 75 MHz digital FT-NMR spectrometer (Bruker Bioscience, Billerica, MA, USA) in DMSO-d6, respectively. Splitting patterns were designated as follows: s: singlet; d: doublet; t: triplet; and m: multiplet in the NMR spectra. Coupling constants (J) were reported in Hertz. All reactions were monitored by thin-layer chromatography (TLC) using Silica Gel 60 F254 TLC plates (Merck KGaA, Darmstadt, Germany). Mass spectra were recorded on an LCMS-IT-TOF (Shimadzu, Kyoto, Japan) instrument using electrospray ionisation (ESI).

3.2 Synthesis:

Weigh 10 g of aniline and transfer to a round-bottom flask. 20 g of glacial acetic acid was added. Add different concentrations of zinc as a catalyst. Mix thoroughly. Attach a reflux condenser connected to cold

water. Heat with a mantle at 70°C for 60 minutes, stirring occasionally. Stop heating and cool to room temperature. Filter to separate solid acetanilide, wash with cold water, and dry.

3.2 Reaction Equation



4. RESULTS AND DISCUSSION

Yield: 87%, M.P. = 114°C. 1 IR: 1660 cm^{-1} (amide C=O), 3300 cm^{-1} (N–H) ^1H NMR: δ 2.1 (s, 3H, CH_3), δ 7.0–7.6 (m, 5H, Ar-H), δ 9.5–10.5 (br s, NH) ^{13}C NMR: δ 169 (C=O) MS: m/z 135 (M^+)

Table 2: Concentration (Zinc)

Trial No.	Zinc Catalyst (g)	Catalyst Molar Ratio (Zn: Aniline)	Yield (%)
1	0.00	0:1	51%
2	0.01	0.00015:1	67%
3	0.03	0.00046:1	78%
4	**0.05**	**0.00077:1**	**87%**
5	0.07	0.00108:1	83%

Table 3: Catalyst comparison

Method	Catalyst	Yield (%)
Conventional	Sulfuric Acid	75%
Catalyst-free	None	62%
Zinc-catalysed *(this work)*	**Zinc (0.05 g)**	**87%**

4.1. Zinc as an Efficient Catalyst: Zinc (Zn) functions as a mild and effective Lewis acid catalyst, enhancing the reactivity of acetic anhydride without the utilisation of corrosive mineral acids such as H_2SO_4, as documented. It promotes cleaner reactions, diminishes the formation of side products, and facilitates easier post-reaction handling. The optimal catalyst loading was determined to be *0.05 g*, achieving an 87% purity of acetanilide.

4.2. Optimal Reaction Conditions Identified:

1. **Temperature:** *70°C* was identified as ideal, providing sufficient energy to drive the reaction effectively while preventing degradation.
2. **Time**: *60 minutes* was deemed adequate for complete conversion without overreaction or thermal decomposition.

3. **Catalyst amount:** Quantities exceeding *0.05 g* did not yield significant benefits and introduced marginal purification challenges.

4.3. High Yield and Purity: The optimised reaction conditions produced a high yield (87%) and a product of high purity, as verified by melting point, FTIR, NMR, and MS analyses. Spectral data matched those of standard acetanilide, confirming successful synthesis.

4.4. Green Chemistry Advantages: This methodology circumvents the use of hazardous acid catalysts, minimises chemical waste, and adheres to the principles of green and sustainable chemistry. Zinc is cost-effective, reusable, and environmentally benign, rendering this approach suitable for academic, pharmaceutical, and industrial applications.

7. CONCLUSION

This study presents an efficient, eco-friendly method for synthesising acetanilide via zinc-catalysed acetylation of aniline with acetic anhydride. The process was optimised by adjusting catalyst concentration, temperature, and time to maximise yield and purity. Key findings include zinc's role as a mild, effective Lewis acid catalyst, enhancing reactivity without corrosive acids, and enabling cleaner reactions with minimal side products. The optimal conditions—0.05 g catalyst, 70 °C, and 60 minutes—yielded 87% pure acetanilide, confirmed by spectral analyses. This approach aligns with green chemistry principles, as zinc is inexpensive, reusable, and environmentally friendly, suitable for various applications.

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