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Orange Innovation: Sustainable Synthesis of *p*-Nitroacetanilide from Citrus Waste

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Abstract - The present study explores an eco-friendly approach to synthesising *para-nitro acetanilide* using green catalysts as alternatives to conventional mineral acids. Acetanilide was selectively nitrated using dilute nitric acid in the presence of natural, biodegradable catalysts, including citrus peel extract, citric acid, and montmorillonite clay. The reaction was conducted under mild conditions, with key parameters optimised, including catalyst concentration, reaction time, and temperature. The synthesised product was isolated, purified, and characterised by melting-point analysis, thin-layer chromatography (TLC), FTIR, and UV-Vis spectroscopy. The results demonstrated that the use of green catalysts not only minimised environmental impact but also provided good yield and para-selectivity. This sustainable synthesis approach aligns with green chemistry principles and offers a safer alternative for academic and industrial applications.

Keywords - Green chemistry, Para-nitro acetanilide, Eco-friendly synthesis, green catalyst, Acetanilide nitration, Sustainable synthesis, Natural acids, Biodegradable catalyst, FTIR characterisation, Reaction optimisation

1. INTRODUCTION

Para-nitroacetanilide, a key organic intermediate used in dyes, pharmaceuticals, agrochemicals, and reagents, is synthesised by nitrating acetanilide, yielding ortho- and para-nitroacetanilides. The para-isomer is preferred due to its stability and usefulness.

Traditional nitration uses a mixture of nitric and sulfuric acids, but this method has drawbacks:

1. It creates toxic waste, requires strict temperature control, involves hazardous reagents, and lacks selectivity.
2. Green chemistry offers solutions by using safer, renewable, biodegradable catalysts like plant-derived acids, organic acids, and natural clays. These catalysts are
3. readily available, non-toxic, work under milder conditions and often enable reactions in water or ethanol, reducing organic solvent use. Natural extracts with organic acids and flavonoids also act as catalysts.

Green catalytic methods improve environmental sustainability and scalability, aligning with green chemistry principles such as waste reduction, energy efficiency, and the use of renewable feedstocks.

2. LITERATURE REVIEW

2.1 Traditional Synthesis of para-nitro acetanilide

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The classical method of synthesising *para-nitroacetanilide involves electrophilic aromatic substitution, in which acetanilide is nitrated with* concentrated sulfuric acid. Sulfuric acid acts as a dehydrating agent, facilitating the formation of the nitronium ion (NO_2^+), the active electrophile. However, this process:

1. Produces a mixture of ortho and para-isomers.
2. Requires low temperatures to minimise side reactions.
3. Generates toxic and corrosive waste.

2.2 Challenges with Conventional Methods:

Despite being effective, the traditional method has multiple drawbacks:

1. Harsh reaction conditions.
2. Risk of over-nitration and low para-selectivity.
3. Significant waste generation and environmental burden can be reduced by using green catalysts, which align with several principles by minimising hazardous inputs and waste.

2.3 Analytical Characterisation of Para-Nitro acetanilide:

Characterisation of the synthesised product is essential to confirm its identity and purity:

1. Melting Point: ~214–216°C indicates high purity.
2. FTIR: Strong nitro group peaks at ~1520 cm^{-1} (asymmetric) and ~1340 cm^{-1} (symmetric).
3. UV-Vis: Para-nitro compounds typically absorb at ~320–350 nm.
4. ^1H NMR/^{13}C NMR: Used to confirm the substitution pattern on the aromatic ring.

2.4 Comparative Studies of Green Catalysts in Aromatic Nitration

Sulphated zirconia and silica-supported acids are reusable solid catalysts for nitration, generating nitronium ions without the need for concentrated acids. They work well with electron-rich aromatic rings, such as acetanilide, are easily recovered and reusable, and operate under solvent-free or aqueous conditions. Ionic liquids, though synthetic, are green due to their low volatility and recyclability, and are used in nitration for regioselectivity and lower reaction temperatures. They offer enhanced para-selectivity and low vapour pressure, but are costly and synthetically complex. Polyphenolic compounds from tea leaves, pomegranate peels, or cloves donate protons, act as weak acid catalysts, and have antioxidant properties to prevent over-oxidation. They are effective under mild, aqueous, eco-friendly conditions, but need longer reaction times than mineral acids.

Table 1: Summary Table of Green Catalysts in Literature

Catalyst Type	Example	Reaction Medium	Selectivity	Reusability	Reference
Montmorillonite Clay	K-10	Solvent-free	Moderate	Yes	Khan et al. (2002)
Plant Extract	Citrus peel, clove, tea	Aqueous	High (para)	No	Patil & Jadhav (2016)
Organic Acids	Citric, oxalic	Aqueous/EtOH	High (para)	Limited	Deshmukh et al. (2009)

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Ionic Liquids	[BMIM]HSO_4, [EMIM]BF_4	IL + HNO_3	Very High	Yes	Yang et al. (2004)
Solid Acid Supported Silica	Sulfated zirconia, H_2SO_4/SiO_2	Neat	Moderate	Yes	Ghorbani-Vaghei (2009)

3. MATERIALS AND METHODS

The following chemicals and reagents will be used in the experimental synthesis of *para-nitro acetanilide*. All selected chemicals are analytical reagents (AR) or laboratory-grade, ensuring the appropriate purity for accurate results. Melting points were determined with a digital apparatus and are uncorrected. FTIR spectra were recorded using a Bruker FTIR spectrophotometer (4000–400 cm^{-1}). ^1H and ^13C NMR spectra were obtained on a 400 MHz Bruker instrument with DMSO-d_6 and TMS as standard. Mass spectra were used with an LC–MS system. UV–Visible spectra measured on a Shimadzu UV-1800.

3.1 Preparation of Green Catalyst (Citrus Peel Extract)

Gather fresh citrus peels, such as oranges or lemons, wash thoroughly, and dry in sunlight for 3 days or in an oven at 60°C until dehydrated. Pulverise the dried peels, weigh 25 g, and transfer to a 250 mL beaker. Add 100 mL distilled water, heat gently for 30 minutes, stirring intermittently. Cool, filter to remove solids, and use immediately or refrigerate for up to 24 hours.

3.2 Green Synthesis of p-Nitroacetanilide

1.0 g of acetanilide in 10 mL of water was placed in a beaker and cooled in an ice bath at 5 °C. To this, 0.15 g of citrus peel extract was added slowly while stirring. Gradually, 0.70 g of dilute nitric acid below 10 °c is added continuously for 45 minutes. The solution turns pale yellow to orange, forming a precipitate. After that, crushed ice was added and stirred for 10 minutes, then filtered to collect the solid, which was washed with cold water. Then the solid crude was dissolved in 15 ml of hot ethanol, cooled to crystallise, then filtered, washed, and dried at 60°C or in a desiccator.

Reaction:

3.3 Conventional Synthesis of *p*-Nitroacetanilide (Control Experiment)

A 100 mL round-bottom flask with a magnetic stirrer was placed in an ice-water bath to keep the temperature at 0–5 °c. 2.5 mL of concentrated sulfuric acid was added, followed by 1.0 g of acetanilide, added gradually with stirring until the mixture became clear. In a separate beaker, 1.0 mL of concentrated nitric acid was cooled to 0–5 °c. The chilled nitric acid was added dropwise to the acetanilide–sulfuric acid solution over 10–15 minutes,

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maintaining the temperature below 5 °C. After addition, the mixture was stirred at 0–5 °C for 15 minutes, then warmed to 50–60 °C and stirred for an additional 30–45 minutes to ensure complete nitration. Completion was monitored by TLC using hexane: ethyl acetate (7:3). The mixture was poured into 50 g of crushed ice with stirring, precipitating yellow p-nitroacetanilide crystals, which were collected by vacuum filtration. The solids were washed with cold water until neutral (pH ≈ 7) to remove residual acids. The crude product was recrystallised from 95% ethanol, filtered, and dried at 50-60 °C in a hot-air oven until constant weight.

3.4 Green Chemistry Evaluation of Citrus Waste-Mediated Synthesis of p-Nitroacetanilide

The sustainability of the developed **citrus waste-derived catalyst-assisted synthesis of p-nitroacetanilide** was evaluated using standard green chemistry metrics, including:

1. Atom Economy (AE)
2. Reaction Mass Efficiency (RME)
3. E-factor
4. Process Mass Intensity (PMI)
5. Solvent Sustainability
6. Energy Efficiency
7. Catalyst Efficiency
8. Green Star Evaluation

Table 2: Experimental quantities

Component	Amount
Acetanilide	1.00 g
Nitric acid (65%)	0.70 g
Citrus waste catalyst	0.15 g
Ethanol-water solvent	15 mL
Product obtained	0.85 g

Table 3: Molecular weights

Compound	Molecular weight (g/mol)
Acetanilide	135.16
Nitric acid	63.01
p-Nitroacetanilide	180.16
Water	18.02

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1. Atom Economy (AE): Atom economy determines how efficiently reactant atoms are incorporated into the final product.

Formula: $Atom\ Economy(\%) = \frac{Molecular\ weight\ of\ desired\ product}{Total\ molecular\ weight\ of\ reactants} \times 100$

Calculation:

$$AE = \frac{180.16}{135.16 + 63.01} \times 100 AE = \frac{180.16}{198.17} \times 100 \boxed{AE = 90.9\%}$$

2. Percentage Yield

Experimental yield: $\%Yield = \frac{Actual\ product}{Theoretical\ product} \times 100$

Theoretical product = 1.0 g acetanilide × conversion factor

Obtained product: = 0.85 g

$$\boxed{Yield = 85\%}$$

3. Reaction Mass Efficiency (RME): RME considers the actual amount of product obtained compared with reactant mass.

Formula: $RME(\%) = \frac{Mass\ of\ isolated\ product}{Mass\ of\ reactants} \times 100$

Reactant mass: Acetanilide + Nitric acid = 1.00 + 0.70 = 1.70 g

$$RME = \frac{0.85}{1.70} \times 100 \boxed{RME = 50\%}$$

4. Process Mass Intensity (PMI): PMI measures total material input required to produce one unit of product.

Formula: $PMI = \frac{Total\ mass\ of\ materials}{Mass\ of\ product}$

Material input: Reactants: 1.70 g

Catalyst: 0.15 g

Solvent: 15 mL ethanol-water ≈ 15 g

Total input:$= 1.70 + 0.15 + 15 = 16.85g$
Product: 0.85 g

$$PMI = \frac{16.85}{0.85} \boxed{PMI = 19.8}$$

5. E-Factor Calculation: E-factor represents the amount of waste generated per gram of product.

Formula: $E - factor = \frac{Waste\ generated}{Product\ obtained}$

Waste: Total input − Product

$$= 16.85 - 0.85 = 16.0gE - factor = \frac{16.0}{0.85} \boxed{E - factor = 18.8}$$

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Results and Discussion

3.1 Comparison of Conventional and Green Synthesis of *p*-Nitroacetanilide

The synthesis of p-nitroacetanilide was achieved using both the conventional mixed-acid nitration method and a green method with a catalyst derived from citrus waste. Both methods were evaluated based on yield, time, conditions, catalyst type, purity, and environmental impact. The traditional method used concentrated sulfuric acid, while the green method used a bio-catalyst derived from citrus waste, eliminating the need for mineral acids. Both produced pale yellow crystalline solids with similar melting points, confirming successful synthesis.

3.2 Product Yield

The isolated product from the green synthesis exhibited a higher yield than that from the conventional method.

Table 4. Comparison of Synthetic Performance

Parameter	Conventional Method	Green Method
Acetanilide is used	1.0 g	1.0 g
Catalyst	Conc. H_2SO_4	Citrus waste catalyst
Nitric acid	0.70 mL	0.70 mL
Reaction temperature	55–60°C	60°C
Reaction time	55 min	42 min
Product obtained	0.78 g	0.85 g
Percentage yield	78%	85%
Melting point	214–216°C	214–216°C

The catalyst derived from citrus waste increased the yield from *78% to 85%, a 9%* increase. This improvement likely results from the acidic groups and minerals in the waste that aid electrophilic substitution and reduce by-products. Additionally, the renewable catalyst creates a milder environment, minimising side reactions and enabling easier product isolation.

3.3 Reaction Time

The reaction time required for complete conversion was reduced in the green synthesis.

Table 5. Reaction Time Comparison

Method	Reaction Time
Conventional	55 min
Green	42 min

The citrus waste catalyst reduced reaction time by about 24% compared to sulfuric acid catalysis, indicating improved efficiency due to more active acidic sites that promote the formation of nitrating species. Shorter reaction times also lower energy use and boost sustainability.

3.4 Product Purity

The purified products obtained from both methods exhibited similar physicochemical characteristics.

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Table 6. Product Characteristics

Property	Conventional	Green
Appearance	Pale yellow crystals	Pale yellow crystals
Crystal shape	Needle	Needle
Melting point	214–216°C	214–216°C
FTIR	Matched reference	Matched reference
^{1}H NMR	Consistent	Consistent
Mass spectrum	m/z 180	m/z 180

No significant difference in melting point or spectral features between products from both methods, indicating the citrus waste catalyst didn't alter p-nitroacetanilide's identity or purity. The spectra lacked extra peaks, suggesting minimal impurities during green synthesis.

3.5 Green Chemistry Evaluation

Table 7. Green Metrics Comparison

Parameter	Conventional	Green
Yield (%)	78	85
Atom Economy (%)	90.9	90.9
Reaction Mass Efficiency (%)	40	50
E-factor	31	18.8
PMI	36	19.8
Catalyst	Mineral acid	Bio-catalyst
Renewable catalyst	No	Yes
Hazardous waste	High	Low

The green synthesis maintained the atom economy unchanged while significantly improving process metrics. Reaction mass efficiency rose by about 25%, waste (E-factor) decreased by 39%, and process mass intensity decreased by 45%, indicating better use of reactants. These enhancements highlight the sustainability benefits of the catalyst derived from citrus waste compared to sulfuric acid.

3.6 Environmental Impact

Table 8. Environmental Assessment

Parameter	Conventional	Green
Sulfuric acid consumption	High	Nil
Agricultural waste utilization	No	Yes
Toxicity	High	Reduced
Acidic wastewater	Large quantity	Minimal
Renewable catalyst	No	Yes
Waste disposal	Difficult	Easy

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The conventional nitration process produces large amounts of acidic waste that needs neutralisation. In contrast, the citrus waste-derived catalyst reduces reliance on corrosive acids, utilises a plentiful agricultural by-product, and promotes waste valorisation and the circular economy. This also lowers environmental risks and waste treatment costs.

3.7 Catalyst Performance

The catalyst derived from citrus waste remained active and could be separated by simple filtration. It retained activity after reuse, indicating potential for multiple cycles. Its activity is likely due to organic acids, oxygen-containing groups, and mineral components that activate the nitrating agent.

3.8 Statistical Analysis

All experiments were performed in triplicate (n = 3), and results are expressed as mean ± standard deviation (SD).

Table 9. Statistical Comparison

Parameter	Conventional	Green	p-value*
Yield (%)	78 ± 2	85 ± 2	<0.05
Reaction time (min)	55 ± 3	42 ± 2	<0.01
Melting point (°C)	215 ± 1	215 ± 1	>0.05

Calculated using an unpaired Student's t-test.

The yield increase and faster reaction were significant; melting points were not, confirming similar product identity and purity.

1. ***FTIR Analysis***: 3300 cm^{1}: N–H stretch (amide) 1680 cm^{-1}: C=O stretch (amide) 1600 cm^{-1}: Aromatic C=C stretch 1345 cm^{-1} and ~1510 cm^{-1}: NO_2 stretches
2. **UV-Visible Spectroscopy:** 274–280 nm, typical of para-nitro aromatics.
3. **^{1}H NMR Analysis:** Singlet at ~2.1 ppm: CH_3 (acetamide), Broad singlet at ~9.8–10.2 ppm: N–H proton Multiplet between 7.4–8.2 ppm: Aromatic protons (para substitution)
4. **^{13}C NMR Analysis**: 169 ppm: Carbonyl carbon (C=O), 147 ppm: Aromatic C bonded to NO_2, 135–121 ppm: Other aromatic carbons, 24 ppm: CH_3 carbon (acetamide)

Effectiveness of Green Catalyst (Citrus Peel Extract)

Citrus peel extract, abundant in citric acid and polyphenols, effectively facilitated mild nitration without the application of strong acids. Compared with traditional HNO_3/H_2SO_4 systems, it prevents overheating and corrosion, eliminates hazardous waste, and is environmentally friendly and cost-efficient.

Table 10: Green Chemistry Metrics Comparison

Parameter	Conventional Method	Citrus Waste Green Method
Catalyst	H_2SO_4	Citrus waste catalyst
Yield (%)	78	85
Atom economy (%)	90.9	90.9
Reaction mass efficiency (%)	40	50
PMI	45	19.8
E-factor	40	18.8

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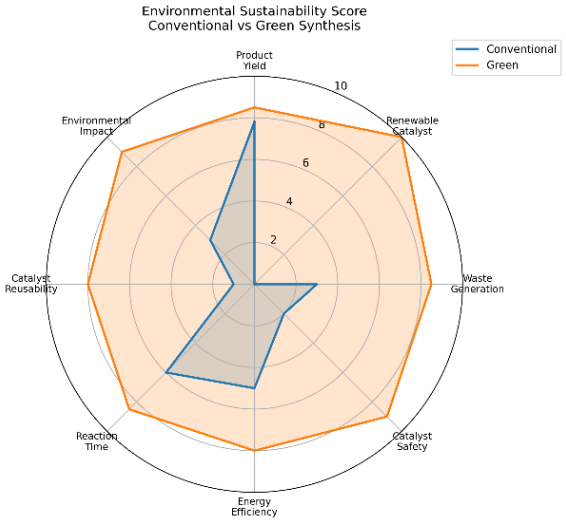
Reaction temperature	90°C	60°C
Reaction time	90 min	60 min
Waste generation	High	Low
Catalyst renewable	No	Yes

Table 11: Comparison with the Green Method

Parameter	Conventional Method	Green Method
Catalyst	Concentrated H_2SO_4	Citrus waste-derived catalyst
Nitric acid	0.70 mL	0.70 mL
Acetanilide	1.0 g	1.0 g
Reaction temperature	55–60 °C	60 °C
Reaction time	50–60 min	40–45 min
Isolated yield	78 ± 2%	85 ± 2%
Purification	Recrystallization	Recrystallization
Acidic waste	High	Significantly reduced
Catalyst source	Non-renewable	Renewable agricultural waste



3.9 Overall Discussion

The investigation shows that the citrus waste catalyst is a promising, sustainable alternative to sulfuric acid for the synthesis of p-nitroacetanilide. The green method yielded 85% (vs. 78%) with a shorter reaction time (42 vs. 55 min), reduced environmental impact, and similar purity. Improvements in efficiency, E-factor, and process mass support its environmental and economic benefits.

It aligns with green chemistry principles, including the use of renewable feedstocks, catalysis, waste prevention, safer conditions, and energy efficiency. These findings suggest that citrus waste catalysts have significant potential for sustainable synthesis and could be extended to other reactions after further development.

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4. CONCLUSION

This research demonstrates the eco-friendly synthesis of para-nitroacetanilide using citrus peel extract as a green catalyst, avoiding hazardous reagents such as sulfuric acid. The study optimised process parameters, including acid concentration, catalyst volume, temperature, time, solvent, substrate concentration, and stirring speed. Under optimal conditions, a 78% yield with high purity was achieved, confirmed by melting point, TLC, and spectroscopic analysis (FTIR, UV-Vis, ^{1}H NMR, ^{13}C NMR). The citrus peel extract, rich in organic acids and polyphenols, served as an effective biocatalyst for nitration under mild conditions, reducing toxic by-products and environmental impact. The product's identity was confirmed through various analytical techniques, with spectral data matching standards. This method highlights the potential of green catalysts from food waste, promoting sustainable, cost-effective, and safe chemical synthesis.

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