

# Investigating the Efficiency of Microwave-Enhanced Fenton Advanced Oxidation Process ( $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ ) in Degrading Emerging Organic Contaminants and Remediating Complex Industrial Effluents

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## Research Article

**Keywords:** Microwave-Fenton Process, Advanced Oxidation Processes (AOPs), Emerging Organic Contaminants, Pharmaceutical Effluent, Endocrine-Disrupting Chemicals (EDCs), Gas Chromatography-Mass Spectrometry (GC-MS), Wastewater Remediation

**Posted Date:** September 21st, 2026

**DOI:** <https://doi.org/10.21203/rs.3.rs-10704186/v1>

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# Abstract

This study investigated the efficiency of a microwave-enhanced Fenton Advanced Oxidation Process ( $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ , pH 2.5–3.5, 700 W, 70°C, 10 min) as a tertiary treatment strategy for degrading emerging organic contaminants in industrial effluents. The treatment involved a 30-minute pre-stirring phase (20 min equilibration with  $\text{Fe}^{2+}$ , 10 min mixing with  $\text{H}_2\text{O}_2$  prior to 10 minutes of microwave irradiation. Twenty complex wastewater samples were collected from pharmaceutical, healthcare, and simulated cosmetic systems across industrial point sources. Physicochemical parameters and Gas Chromatography-Mass Spectrometry (GC-MS) profiles were evaluated before and after treatment.

Untreated effluents exhibited heavy pollution, with baseline chemical oxygen demand (COD) ranging from 1000 to 5300 mg/L, high coliform counts, heavy metals, elevated hardness ( $\text{Ca}^{2+}/\text{Mg}^{2+}$ ), and phenolic activity. Post-treatment COD reductions reached 10.0% to 85.0%, with highest removal in Methylated Spirit (85.0%) and Boric Acid (82.4%) streams, while mineral-dense softener brine yielded 10.0%. Initial zero-BOD values were transformed into biodegradable fragments, coliforms were rendered undetectable, and initial heavy metals, hardness, and phenolic activity were remediated. GC-MS resolved 223 organic compounds in raw effluents, including Diphenhydramine (39.8%), DEHP (5.1–10.9%), DBP (8.0–20.0%), and Octocrylene. Post-treatment chromatography confirmed 55% to 100% removal for the majority of tracked pharmaceutically active compounds, personal care products, and endocrine-disrupting chemicals. These findings demonstrate the technical viability of localized advanced oxidation technologies and provide empirical baseline data to inform regulatory discharge standards in low- and middle-income settings.

## 1. INTRODUCTION

### 1.1 Background of Study

Pollution is defined as the presence of substances and energy in environmental media (air, water, and land) whose nature, location, or quantity produces undesirable environmental effects and impairs the natural quality and usefulness of resources (UN Statistics/UNDRR, 2025). As of 2025, pollution remains the largest environmental cause of disease and premature death globally, undermining sustainable economic growth and exacerbating poverty, particularly in low- and middle-income regions (World Bank, 2025).

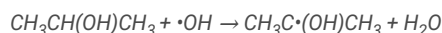
In environmental chemistry and water resources management, it is essential to distinguish between natural water and wastewater. Water refers to fresh water from natural sources like rivers and aquifers intended for consumption or ecosystem support. Wastewater is water that has been adversely affected in quality by anthropogenic activities, containing liquid waste from domestic, industrial, and agricultural sources. Water pollution occurs when toxic substances are released directly into receiving water bodies without adequate treatment (Yang *et al.*, 2022). Over the past 35 years, global climate dynamics have worsened droughts, increasing water shortages worldwide (European Commission, 2019). Developing countries face severe environmental and economic challenges due to the overuse and contamination of freshwater resources (Fernández-García *et al.*, 2015).

Recently, global scientific concern has intensified regarding micropollutants in wastewater, specifically chemicals from medicines, personal care products, and hormone-disrupting substances (Alessandra & Vincenzo, 2016). Many of these synthetic compounds are unregulated and categorized as "emerging contaminants" (European Commission, 2019). Conventional municipal and industrial wastewater treatment plants are not engineered to remove these complex micro-organics (José Antonio *et al.*, 2019). Endocrine-disrupting compounds (EDCs) mimic or block natural hormones, impairing wildlife and human health (Adhena *et al.*, 2022; Guo *et al.*, 2019). Priority EDCs include steroid estrogens (estrone, 17 $\beta$ -estradiol) and industrial additives such as bisphenol A (BPA) and phthalate plasticizers (Combarrous, 2017; Liu *et al.*, 2019). Even at trace concentrations (ng/L to  $\mu\text{g/L}$ ), EDCs can induce birth defects, neurological disorders, immune suppression, and hormonal cancers (B-Moon *et al.*, 2019; Jung *et al.*, 2015).

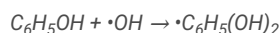
In addition to EDCs, pharmaceuticals and personal care products (PPCPs) comprising antibiotics, antihistamines, analgesics, synthetic steroids, preservatives, disinfectants, and UV filters regularly enter aquatic systems through manufacturing discharges and municipal runoffs (Jiao *et al.*, 2022; Wang *et al.*, 2016). Advanced Oxidation Processes (AOPs) represent specialized chemical treatment methods designed to degrade recalcitrant organic pollutants by generating highly reactive oxygen species (ROS), predominantly hydroxyl radicals ( $\cdot\text{OH}$ ) (Jiao *et al.*, 2022).

First introduced in 1980 for drinking water purification (José Antonio *et al.*, 2019), AOPs generate hydroxyl radicals with a very high standard oxidation potential ( $E^\circ = 2.80 \text{ V}$ ), enabling non-selective degradation of organic molecules through three main pathways (Deng *et al.*, 2015):

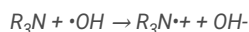
1. Hydrogen Abstraction: The hydroxyl radical abstracts a hydrogen atom from a saturated carbon-hydrogen bond, producing an organic radical ( $\text{R}\cdot$ ) and water.



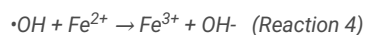
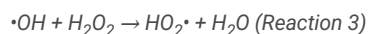
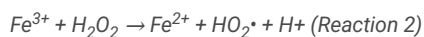
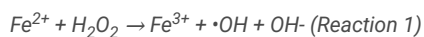
2. Radical Pairing (Electrophilic Addition): The  $\cdot\text{OH}$  radical adds to unsaturated carbon-carbon double bonds or aromatic rings, forming cyclohexadienyl-type organic radicals that destabilize ring systems.



3. Electron Transfer: The  $\cdot\text{OH}$  radical accepts an electron from an electron-rich organic donor (such as amines or sulfur compounds), forming radical cations that undergo rapid hydrolysis.



Iron ( $\text{Fe}^{2+}$ ) is the primary transition metal catalyst used to decompose hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) into hydroxyl radicals in classic Fenton chemistry (Yang and Renzun, 2015):



Because Reaction 2 is substantially slower than Reaction 1, optimizing the molar ratio of  $\text{Fe}^{2+}$  to  $\text{H}_2\text{O}_2$  is essential to prevent radical scavenging by excess reagents (Ebrahiem *et al.*, 2015). Microwave irradiation synergizes Fenton chemistry by providing rapid dielectric heating and accelerating the thermal reduction of  $\text{Fe}^{3+}$  back to  $\text{Fe}^{2+}$ , thereby maintaining high radical generation rates (Cheng *et al.*, 2023; Homem *et al.*, 2023).

## 1.2 Statement of Problem

Industrialized regions in developing nations, face severe freshwater contamination from unregulated industrial effluent discharge. Pharmaceutical and cosmetic manufacturing wastewater represents a highly complex, hazardous matrix containing pharmaceutically active compounds (PhACs), endocrine-disrupting chemicals (EDCs), and phthalate plasticizers (such as DEHP and DBP). These compounds are highly stable, bioaccumulative, and resistant to natural degradation. Even at trace levels, they impair neurological and reproductive functions in aquatic organisms and pose chronic health risks to downstream human populations. Conventional wastewater treatment systems are inherently ineffective at removing these emerging organic micropollutants. In Nigeria, this failure is compounded by a critical regulatory gap: neither local regulatory agencies (NESREA nor NAFDAC) enforces national effluent standards specifically targeting PhACs or EDCs from manufacturing facilities. Consequently, manufacturers routinely discharge untreated, biologically active waste into regional aquatic systems. While Advanced Oxidation Processes (AOPs) like the conventional Fenton reaction can mineralize recalcitrant organics, they suffer from prolonged reaction times (30-120 minutes) and generate excessive volumes of toxic iron sludge. Microwave-enhanced Fenton processes offer a rapid alternative, but their efficacy has not been systematically evaluated on real, multi-component Nigerian industrial wastewater. Therefore, the core research problem addressed in this study is that in the absence of effective, locally validated treatment technologies and targeted regulatory standards, hazardous pharmaceutical and cosmetic effluents continue to threaten Nigerian water bodies.

## 1.3 Justification of the Study

This study is scientifically and methodologically justified as the first integrated investigation to apply a microwave-enhanced Fenton advanced oxidation process to real, multi-component industrial wastewater collected directly from active Nigerian manufacturing lines. While Fenton-based technologies are well-documented in high-income countries, their application within the operational constraints of low- and middle-income regions (Like, Sub-Saharan Africa) remains severely under-researched. This project bridges this gap by establishing empirical baseline metrics for highly complex matrices characterized by high lipid fractions and Chemical Oxygen Demand (COD) values ranging from 1,000 to 5,300 mg/L and testing a rapid tertiary treatment strategy (700 W, 10 min) across diverse operational streams, including methylated spirits, boric acid lines, and dense softener plant brines.

These findings will directly benefit multiple stakeholders:

- i. **Regulatory Advocacy & Compliance:** Provides concrete data to inform the development of stricter national effluent discharge standards under NESREA and NAFDAC guidelines.
- ii. **Technology Transfer:** Establishes the technical viability, rapid reaction kinetics, and cost-effectiveness of localized AOPs, serving as an empirical engineering foundation for technology transfer to domestic manufacturing facilities.
- iii. **Public Health & Environmental Protection:** Addresses the urgent socio-economic need for rapid, scalable tertiary treatment solutions that protect vulnerable aquatic ecosystems and downstream public health without imposing prohibitive operational costs on domestic manufacturers in Sub-Saharan Africa.

## 1.4 Aim and Objectives

### 1.4.1 Aim:

To evaluate the efficiency of a microwave-enhanced Fenton advanced oxidation process ( $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ ) in removing emerging organic contaminants (PhACs, EDCs, and PCPs) from industrial wastewater and to determine the resulting physicochemical and biological transformations.

### 1.4.2 Objectives of the Study:

1. Evaluate the operational process performance and track shifts in bulk physicochemical characteristics (specifically pH, electrical conductivity [EC], and oxidation-reduction potential [ORP]) of twenty raw and microwave-Fenton treated pharmaceutical and cosmetic wastewater matrices.
2. Quantify macro-organic degradation and biotransformation dynamics by assessing Chemical Oxygen Demand (COD) removal efficiencies, Dissolved Oxygen (DO) responses, and Biological Oxygen Demand ( $\text{BOD}_{5j}$ ) variations across the treated effluent stream.

3. Determine the secondary remediation efficiency of the process regarding specific biological, nutrient, and mineral parameters, explicitly evaluating coliform counts, heavy metal elimination, phenolic compound degradation, water hardness ( $\text{Ca}^{2+}/\text{Mg}^{2+}$ ) co-precipitation, total salt, and total nitrogen levels.
4. Profile organic micropollutants using Gas Chromatography-Mass Spectrometry (GC-MS) across five representative sample pairs to determine target pollutant removal efficiencies, identify newly formed oxidation transformation by-products, and evaluate their associated health hazards to humans and animals.

### 1.5 Scope and Limitations

#### Scope:

The study evaluates microwave-assisted Fenton degradation ( $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ , 700 W, 70°C, 10 min) across 20 industrial wastewater matrices collected from Kano and Kaduna States, Nigeria. Water quality metrics, macro-pollutant removals, and GC-MS micro-organic profiling across 5 representative sample pairs were systematically evaluated.

#### Limitations:

Analytical evaluations were constrained by instrument measurement limits and resource availability for pilot-scale continuous flow systems. Operational parameters were evaluated under batch laboratory conditions.

## 2. LITERATURE REVIEW

Advanced oxidation technologies have been extensively evaluated for high-strength industrial effluent remediation. Agabo-García et al. (2021) demonstrated that the Photo-Fenton process utilizing an iron oxyhydroxide catalyst achieved a 62.8% COD reduction and 88.9% total phenolic compound (TPC) removal, maintaining over 90.5% catalytic efficiency across three cycles. Agabo García and Hodaifa (2017) confirmed > 90% organic carbon and phenol removal in olive oil mill wastewater at pH 3.0. Berberidou et al. (2022) achieved 90% mineralization of clopyralid pesticide within 120 minutes under solar/UV photo-Fenton systems. Jiao et al. (2022) reviewed free-radical mechanisms in PPCP removal, underscoring the dominance of  $\cdot\text{OH}$  and sulphate radical pathways.

Odabasi and Buyukgungor (2024) benchmarked Fenton,  $\text{UV}/\text{H}_2\text{O}_2$  and  $\text{UV}/\text{TiO}_2$  systems, proving classical Fenton achieved > 99.9% PPCP removal at superior cost-efficiency. Starling et al. (2020) demonstrated persulfate-enhanced Photo-Fenton solar disinfection at neutral pH. Specialized sorbents have also been investigated: Kamarehie et al. (2018) achieved 22.28 mg/g Bisphenol A adsorption on  $\text{MgO}$ -coated activated carbon, while Guo et al. (2019) used corn straw biochar to immobilize PFOS and EE2 in aquatic sediments. B-Moon et al. (2019) demonstrated metal-organic frameworks (MOFs) with high EDC adsorption capacities (138.4–200.4 mg/g).

Monitoring studies in developing and emerging economies highlight widespread aquatic contamination. Caldas et al. (2019) detected 30 target pesticides and pharmaceuticals over a 4-year survey in Brazilian surface waters. Sun et al. (2019) identified 40 PPCPs along the Jinsha River. Microwave-assisted Fenton systems offer accelerated kinetics through localized dielectric superheating; Cheng et al. (2023) demonstrated rapid COD removal using microwave activation. Deng et al. (2015) and José Antonio et al. (2019) emphasized that incomplete AOP oxidation can yield polar transformation products, necessitating comprehensive GC-MS tracking as conducted in this study. Ecotoxicological studies by Adhena et al. (2022), Ahmed et al. (2018), Combarnous (2017), Jung et al. (2015), Liu et al. (2019), and Wang et al. (2016) confirm that residual EDCs like DEHP and DBP induce endocrine disruption, bioaccumulation, and reproductive toxicity in aquatic organisms.

Microwave heating (Milestone ETHOS X) operates via dipole rotation and ionic conduction within an oscillating electromagnetic field (300 MHz to 300 GHz) (Lui et al., 2023). Homem et al. (2023) demonstrated that microwave radiation accelerates  $\text{Fe}^{3+}$  reduction to  $\text{Fe}^{2+}$ , sustaining high  $\cdot\text{OH}$  production. Gas Chromatography-Mass Spectrometry (Agilent 7890B GC/5977A MSD) provides gold-standard compound resolution based on capillary partitioning and electron impact (EI) mass fragment identification (Kazemzadeh et al., 2020; Koster et al., 2023).

Advanced oxidation processes (AOPs), particularly Fenton-based technologies, rely on the generation of highly reactive hydroxyl radicals  $\cdot\text{OH}$  to unselectively degrade complex organic pollutants into non-toxic end products (Neyens and Baeyens 2003; Pera-Titus et al. 2004). While classical Fenton systems are governed by the catalytic decomposition of hydrogen peroxide by ferrous ions under acidic conditions (Neyens and Baeyens 2003), their operational efficiency in treating recalcitrant pharmaceutical and industrial effluents can be significantly enhanced through thermal or microwave assistance (Pera-Titus et al. 2004).

## 3. MATERIALS AND METHODS

### 3.1 Reagents and Materials

All reagents were of analytical grade: Hydrogen peroxide ( $\text{H}_2\text{O}_2$  35% w/w), Ferrous sulfate heptahydrate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ), Sulfuric acid ( $\text{H}_2\text{SO}_4$  98%), Sodium hydroxide (NaOH pellets, > 99%), Dichloromethane (DCM, HPLC grade), Potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ), Silver sulfate ( $\text{Ag}_2\text{SO}_4$ ), Mercury sulfate ( $\text{HgSO}_4$ ), Ferrous ammonium sulfate ( $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ ) and Ferroin indicator solution. High-purity distilled water was used throughout.

Analytical equipment included an Agilent 7890B GC/5977A MSD system, Milestone ETHOS X Microwave Synthesizer, Radwag Partner XA 82/220 balance ( $\pm 0.1$  mg), Jenway 3510 pH meter, and BLE-C600 multi-parameter meter.

### 3.2 Sampling and Preservation

Twenty composite wastewater samples (n = 20) were collected across industrial point sources in Kano and Kaduna States, Nigeria. Sampling comprised four (4) points from pharmaceutical facility [Facility A] (Cough Syrup, MMT Tank 1, MMT Tank 2, Metro Suspension), 3 points from an industrial plant [Facility B] (Boiler Drain, Softener Plant, RO + EDI+Softener), 10 points from regional healthcare center [Facility C] (5 Methylated Spirit lines, 5 Boric Acid lines), and 3 matrix-matched simulated cosmetic wastewater controls (CSW, PWS, PW). Samples were collected in pre-cleaned amber glass containers to prevent photodegradation and stored at 4°C in accordance with standard protocols (APHA, 2017).

### 3.3 Experimental Procedure

The microwave-assisted Fenton oxidation protocol followed Odabasi and Buyukgungor (2024) and Ebrahiem et al. (2015) with minor optimization. Wastewater aliquots (50 mL) were acidified to pH 2.5–3.5 using 0.1 M H<sub>2</sub>SO<sub>4</sub>. Then, 50 mL of 1.70 × 10<sup>-4</sup> M FeSO<sub>4</sub> solution was added and stirred for 20 min. Next, 50 mL of 5% v/v H<sub>2</sub>O<sub>2</sub> was added slowly under continuous stirring for 10 min. The mixture was placed in a Milestone ETHOS X microwave workstation and irradiated at 700 W (70°C) for 10 min. Post-treatment, the sample was neutralized to pH 8.0–8.5 using 0.1 M NaOH to precipitate iron as Fe(OH)<sub>3</sub> flocs. After 2 hours of gravity settling, the clarified supernatant was filtered through 110 mm Whatman No. 1 paper (dry weight recorded). Organic compounds were extracted from the clarified supernatant using liquid-liquid extraction with dichloromethane (DCM) and analyzed via GC-MS.

### 3.4 Analytical Methods and Calculations

Chemical Oxygen Demand (COD) was determined via open-reflux dichromate digestion followed by ferrous ammonium sulfate titration using Ferroin indicator

**COD (mg/L) =** 
$$\frac{(\text{mL blank} - \text{mL sample}) \times \text{Normality} \times 800}{\text{mL sample}}$$

Where “a” is titrant volume for blank (mL), “b” is titrant volume for sample (mL), and “N” is normality of FAS.

Removal efficiency was calculated as:

**Removal Efficiency (%) =** 
$$\frac{(C_0 - C_t)}{C_0} \times 100$$

Biological Oxygen Demand (BOD5) was measured via 5-day incubation at 20°C using an Extech DO600 DO meter:

**BOD5 (mg/L) = DO1 - DO5**

GC-MS analysis was conducted on an Agilent 7890B GC linked to a 5977A MSD using an HP-5ms Ultra Inert capillary column (30 m × 250 μm × 0.25 μm). Chromatographic peaks were integrated using Agilent MassHunter and matched against the NIST14.L spectral library with a minimum 80% similarity threshold. Relative peak area percentage was calculated as:

**Relative Area (%) =** 
$$\left( \frac{\text{Area}_i}{\sum \text{Area}_i} \right) \times 100$$

## 4. RESULTS AND DISCUSSION

### 4.1 Treatment Process Performance

Table 1 outlines the operational parameters recorded during the microwave-Fenton treatment across all 20 wastewater matrices investigated.

**Table 1: Operational Parameters of the Fenton Treatment Process for All Wastewater Samples**

| Sample Matrix            | Initial pH | Adjusted pH | Stirring Time (min) | Heating Time (min) | pH After Heating | Final pH (Fe(OH) <sub>3</sub> ) | Filter Paper Wt (g) |
|--------------------------|------------|-------------|---------------------|--------------------|------------------|---------------------------------|---------------------|
| Cough Syrup              | 5.60       | 3.12        | 30                  | 10                 | 3.79             | 8.60                            | 0.159               |
| MMT Suspension (Tank 1)  | 10.60      | 3.14        | 30                  | 10                 | 4.60             | 8.16                            | 0.151               |
| MMT Suspension (Tank 2)  | 9.90       | 3.15        | 30                  | 10                 | 4.51             | 8.68                            | 0.153               |
| Metro Suspension         | 6.90       | 3.00        | 30                  | 10                 | 4.56             | 8.27                            | 0.149               |
| Boiler Drain Point       | 11.20      | 2.82        | 30                  | 10                 | 6.56             | 8.05                            | 0.150               |
| RO + EDI + Softener      | 7.20       | 3.01        | 30                  | 10                 | 6.80             | 8.02                            | 0.147               |
| Softener Plant           | 5.90       | 2.73        | 30                  | 10                 | 6.05             | 8.00                            | 0.149               |
| Methylated Spirit 1      | 7.10       | 2.58        | 30                  | 10                 | 4.79             | 8.25                            | 0.149               |
| Methylated Spirit 2      | 6.70       | 2.64        | 30                  | 10                 | 7.09             | 8.07                            | 0.150               |
| Methylated Spirit 3      | 7.06       | 3.16        | 30                  | 10                 | 7.50             | 8.12                            | 0.148               |
| Methylated Spirit 4      | 6.60       | 2.58        | 30                  | 10                 | 6.94             | 8.18                            | 0.147               |
| Methylated Spirit 5      | 6.62       | 2.52        | 30                  | 10                 | 6.92             | 8.45                            | 0.152               |
| Boric Acid 1             | 6.80       | 3.04        | 30                  | 10                 | 6.90             | 8.04                            | 0.155               |
| Boric Acid 2             | 6.66       | 3.00        | 30                  | 10                 | 5.88             | 8.41                            | 0.156               |
| Boric Acid 3             | 6.90       | 2.51        | 30                  | 10                 | 6.00             | 8.10                            | 0.150               |
| Boric Acid 4             | 6.55       | 3.15        | 30                  | 10                 | 6.72             | 8.20                            | 0.149               |
| Boric Acid 5             | 7.07       | 2.79        | 30                  | 10                 | 5.97             | 8.01                            | 0.151               |
| Simulated Sample 1 (CSW) | 6.37       | 3.24        | 30                  | 10                 | 6.83             | 8.30                            | 0.149               |
| Simulated Sample 2 (PWS) | 6.20       | 3.19        | 30                  | 10                 | 4.75             | 8.47                            | 0.159               |
| Simulated Sample 3 (PW)  | 6.38       | 3.03        | 30                  | 10                 | 6.87             | 8.09                            | 0.148               |

The initial pH ranged from 5.60 to 11.20, reflecting extreme variation in industrial effluent chemistry. Target acidification (pH 2.50–3.24) maximized Fe<sup>2+</sup> solubility for radical generation (Odabasi & Buyukgungor, 2024; Yang & Renzun, 2015). Post-heating pH rose to 3.79–7.50 due to proton consumption during radical chain reactions. Final neutralization (pH 8.00–8.68) precipitated iron hydroxide flocs, increasing filter paper dry weights above the 0.146 g blank, with highest sludge production in Cough Syrup (0.159 g) and Simulated Sample 2 (0.159 g) due to higher organic mass loads (Agabo-García *et al.*, 2021).

#### 4.2 Physicochemical Characteristics

Tables 2 and 3 summarize the physicochemical characteristics of untreated and microwave-Fenton treated wastewater samples, respectively.

**Table 2: Physicochemical Characteristics of Untreated Wastewater Samples**

| S/N | Sample Matrix       | pH   | EC (µS/cm) | ORP (mV) | DO1 (mg/L) | DO5 (mg/L) | BOD (mg/L) | COD (mg/L) | Antioxidant |
|-----|---------------------|------|------------|----------|------------|------------|------------|------------|-------------|
| 1   | Cough Syrup         | 4.82 | 1470       | 294.5    | 8.10       | 8.10       | 0.00       | 3000       | -ve         |
| 2   | MMT (Tank 1)        | 8.49 | 3210       | 132.5    | 8.24       | 6.00       | 2.24       | 3000       | +ve         |
| 3   | MMT (Tank 2)        | 8.22 | 2165       | 158.5    | 7.99       | 5.98       | 2.01       | 3100       | +ve         |
| 4   | Metro Suspension    | 6.13 | 1359       | 212.0    | 8.00       | 8.00       | 0.00       | 3000       | -ve         |
| 5   | Boiler Drain Point  | 9.48 | 1010       | 130.5    | 8.20       | 7.90       | 0.30       | 4000       | +ve         |
| 6   | RO+EDI + Softener   | 7.43 | 606        | 208.5    | 8.30       | 5.70       | 2.60       | 3000       | +ve         |
| 7   | Softener Plant      | 5.78 | 110000     | 223.0    | 7.90       | 7.90       | 0.00       | 1000       | -ve         |
| 8   | Methylated Spirit 1 | 6.26 | 156        | 197.0    | 7.90       | 7.90       | 0.00       | 4000       | -ve         |
| 9   | Methylated Spirit 2 | 6.19 | 158.5      | 213.0    | 7.90       | 7.90       | 0.00       | 4300       | -ve         |
| 10  | Methylated Spirit 3 | 6.57 | 159.2      | 209.0    | 7.90       | 7.90       | 0.00       | 4100       | -ve         |
| 11  | Methylated Spirit 4 | 6.22 | 156.9      | 199.7    | 7.90       | 7.90       | 0.00       | 4000       | -ve         |
| 12  | Methylated Spirit 5 | 7.11 | 155        | 192.9    | 7.90       | 7.90       | 0.00       | 3900       | -ve         |
| 13  | Boric Acid 1        | 6.81 | 202        | 191.0    | 8.00       | 8.00       | 0.00       | 5000       | -ve         |
| 14  | Boric Acid 2        | 6.77 | 218        | 202.0    | 7.90       | 7.90       | 0.00       | 5200       | -ve         |
| 15  | Boric Acid 3        | 6.49 | 220        | 187.0    | 8.00       | 8.00       | 0.00       | 5300       | -ve         |
| 16  | Boric Acid 4        | 6.64 | 211        | 198.0    | 8.00       | 8.00       | 0.00       | 5000       | -ve         |
| 17  | Boric Acid 5        | 6.79 | 222        | 186.0    | 8.00       | 8.00       | 0.00       | 5100       | -ve         |
| 18  | Simulated 1 (CSW)   | 6.37 | 507        | -61.0    | 8.10       | 7.80       | 0.30       | 5000       | -ve         |
| 19  | Simulated 2 (PWS)   | 6.20 | 828        | -92.0    | 6.40       | 4.44       | 1.96       | 3000       | -ve         |
| 20  | Simulated 3 (PW)    | 6.38 | 608        | -73.0    | 6.65       | 4.07       | 2.58       | 3000       | -ve         |

**Table 3: Physicochemical Characteristics of Treated Wastewater Samples**

| S/N | Sample Matrix       | pH   | EC (µS/cm) | ORP (mV) | DO1 (mg/L) | DO5 (mg/L) | BOD (mg/L) | COD (mg/L) | Antioxidant |
|-----|---------------------|------|------------|----------|------------|------------|------------|------------|-------------|
| 1   | Cough Syrup         | 6.14 | 1088       | 386.0    | 11.50      | 8.40       | 3.10       | 1800       | +ve         |
| 2   | MMT (Tank 1)        | 6.07 | 2180       | 321.0    | 8.24       | 6.00       | 2.24       | 1800       | +ve         |
| 3   | MMT (Tank 2)        | 6.48 | 1526       | 362.0    | 8.14       | 5.99       | 2.15       | 1700       | +ve         |
| 4   | Metro Suspension    | 6.16 | 1006       | 382.0    | 8.30       | 7.90       | 0.40       | 1200       | +ve         |
| 5   | Boiler Drain Point  | 6.53 | 872        | 350.0    | 8.30       | 7.90       | 0.40       | 1800       | +ve         |
| 6   | RO+EDI + Softener   | 6.48 | 720        | 352.0    | 8.40       | 8.00       | 0.40       | 1100       | +ve         |
| 7   | Softener Plant      | 6.04 | 45500      | 368.0    | 8.80       | 7.60       | 1.20       | 900        | +ve         |
| 8   | Methylated Spirit 1 | 6.55 | 581        | 293.0    | 8.30       | 7.80       | 0.50       | 900        | -ve         |
| 9   | Methylated Spirit 2 | 6.55 | 601        | 290.0    | 8.40       | 7.70       | 0.70       | 700        | -ve         |
| 10  | Methylated Spirit 3 | 6.46 | 605        | 291.0    | 8.35       | 7.80       | 0.55       | 800        | -ve         |
| 11  | Methylated Spirit 4 | 6.76 | 599        | 295.0    | 8.33       | 7.75       | 0.58       | 600        | -ve         |
| 12  | Methylated Spirit 5 | 6.56 | 610        | 298.0    | 8.30       | 7.80       | 0.50       | 800        | -ve         |
| 13  | Boric Acid 1        | 6.38 | 720        | 352.0    | 8.30       | 7.90       | 0.40       | 1200       | +ve         |
| 14  | Boric Acid 2        | 6.47 | 626        | 290.0    | 8.40       | 7.95       | 0.45       | 1200       | +ve         |
| 15  | Boric Acid 3        | 6.35 | 722        | 292.0    | 8.44       | 7.89       | 0.55       | 1100       | +ve         |
| 16  | Boric Acid 4        | 6.88 | 743        | 298.0    | 8.30       | 7.70       | 0.60       | 1000       | +ve         |
| 17  | Boric Acid 5        | 6.45 | 689        | 325.0    | 8.35       | 7.90       | 0.45       | 900        | +ve         |
| 18  | Simulated 1 (CSW)   | 6.55 | 789        | 275.0    | 8.50       | 8.20       | 0.30       | 1300       | +ve         |
| 19  | Simulated 2 (PWS)   | 6.59 | 797        | 268.0    | 8.60       | 8.20       | 0.40       | 900        | +ve         |
| 20  | Simulated 3 (PW)    | 6.46 | 790        | 294.0    | 8.46       | 8.15       | 0.31       | 900        | +ve         |

Post-treatment pH normalized to 6.04–6.88 across all matrices, fully complying with NESREA effluent guidelines (pH 6.0–9.0) (NESREA, 2011). Electrical conductivity decreased in mineral-rich effluents (e.g., Softener Plant brine dropped by 58.6% from 110,000 to 45,500  $\mu\text{S}/\text{cm}$ ) due to floc co-precipitation. Conversely, EC increased in dilute Methylated Spirit matrices (155  $\rightarrow$  610  $\mu\text{S}/\text{cm}$ ) due to residual reagent sulfate and iron ions (Cheng et al., 2023; Starling et al., 2020). ORP values universally increased to +268...+386 mV, confirming a complete transition from reducing to strongly oxidizing conditions (Berberidou et al., 2022; Li et al., 2024).

4.3 Macro-Organic Degradation and Biotransformation Dynamics

Table 4 compares the BOD<sub>5</sub> and DO dynamics, while Table 5 details the Chemical Oxygen Demand (COD) removal yields across the sample portfolio.

Table 4: BOD and Dissolved Oxygen Profile: Untreated vs. Treated Wastewater Samples

| Sample Matrix       | Untreated BOD (mg/L) | Treated BOD (mg/L) | BOD Shift/Removal                    | Untreated DO1 (mg/L) | Treated DO1 (mg/L) |
|---------------------|----------------------|--------------------|--------------------------------------|----------------------|--------------------|
| Cough Syrup         | 0.00                 | 3.10               | BOD Emergence (0 $\rightarrow$ 3.10) | 8.10                 | 11.50              |
| MMT (Tank 1)        | 2.24                 | 2.24               | 0.0%                                 | 8.24                 | 8.24               |
| MMT (Tank 2)        | 2.01                 | 2.15               | -7.0% (slight increase)              | 7.99                 | 8.14               |
| Metro Suspension    | 0.00                 | 0.40               | BOD Emergence (0 $\rightarrow$ 0.40) | 8.00                 | 8.30               |
| Boiler Drain Point  | 0.30                 | 0.40               | -33.0% (slight increase)             | 8.20                 | 8.30               |
| RO + EDI + Softener | 2.60                 | 0.40               | 84.6% removal                        | 8.30                 | 8.40               |
| Softener Plant      | 0.00                 | 1.20               | BOD Emergence (0 $\rightarrow$ 1.20) | 7.90                 | 8.80               |
| Methylated Spirit 1 | 0.00                 | 0.50               | BOD Emergence (0 $\rightarrow$ 0.50) | 7.90                 | 8.30               |
| Methylated Spirit 2 | 0.00                 | 0.70               | BOD Emergence (0 $\rightarrow$ 0.70) | 7.90                 | 8.40               |
| Methylated Spirit 3 | 0.00                 | 0.55               | BOD Emergence (0 $\rightarrow$ 0.55) | 7.90                 | 8.35               |
| Methylated Spirit 4 | 0.00                 | 0.58               | BOD Emergence (0 $\rightarrow$ 0.58) | 7.90                 | 8.33               |
| Methylated Spirit 5 | 0.00                 | 0.50               | BOD Emergence (0 $\rightarrow$ 0.50) | 7.90                 | 8.30               |
| Boric Acid 1        | 0.00                 | 0.40               | BOD Emergence (0 $\rightarrow$ 0.40) | 8.00                 | 8.30               |
| Boric Acid 2        | 0.00                 | 0.45               | BOD Emergence (0 $\rightarrow$ 0.45) | 7.90                 | 8.40               |
| Boric Acid 3        | 0.00                 | 0.55               | BOD Emergence (0 $\rightarrow$ 0.55) | 8.00                 | 8.44               |
| Boric Acid 4        | 0.00                 | 0.60               | BOD Emergence (0 $\rightarrow$ 0.60) | 8.00                 | 8.30               |
| Boric Acid 5        | 0.00                 | 0.45               | BOD Emergence (0 $\rightarrow$ 0.45) | 8.00                 | 8.35               |
| Simulated 1 (CSW)   | 0.30                 | 0.30               | 0.0%                                 | 8.10                 | 8.50               |
| Simulated 2 (PWS)   | 1.96                 | 0.40               | 79.6% removal                        | 6.40                 | 8.60               |
| Simulated 3 (PW)    | 2.58                 | 0.31               | 88.0% removal                        | 6.65                 | 8.46               |

Zero baseline BOD in high-COD raw effluents (Cough Syrup, Methylated Spirits, and Boric Acid) indicates that recalcitrant micro-organics and bactericidal solvents suppressed microbial activity (Jiao et al., 2022; Wang et al., 2016). Post-treatment BOD emergence (0  $\rightarrow$  0.40–3.10 mg/L) proves radical cleavage converted non-biodegradable parent molecules into assimilable organic fragments (Agabo García & Hodaifa, 2017; Agabo-García et al., 2021). In effluents with initial biodegradable carbon (RO+EDI+Softener, Simulated 2 & 3), BOD reductions reached 79.6%–88.0%. Treated BOD:COD ratios improved to 0.23–0.34, crossing the 0.2 threshold required for biological polishing (Li et al., 2024; Starling et al., 2020).

Table 5: Chemical Oxygen Demand (COD) Removal Efficiency Following Fenton Treatment

| Sample Matrix            | Untreated COD (mg/L) | Treated COD (mg/L) | Removal Efficiency (%) |
|--------------------------|----------------------|--------------------|------------------------|
| Cough Syrup              | 3000                 | 1800               | 40.00%                 |
| MMT Suspension (Tank 1)  | 3000                 | 1800               | 40.00%                 |
| MMT Suspension (Tank 2)  | 3100                 | 1700               | 45.20%                 |
| Metro Suspension         | 3000                 | 1200               | 60.00%                 |
| Boiler Drain Point       | 4000                 | 1800               | 55.00%                 |
| RO + EDI + Softener Tank | 3000                 | 1100               | 63.30%                 |
| Softener Plant           | 1000                 | 900                | 10.00%                 |
| Methylated Spirit 1      | 4000                 | 900                | 77.50%                 |
| Methylated Spirit 2      | 4300                 | 700                | 83.70%                 |
| Methylated Spirit 3      | 4100                 | 800                | 80.50%                 |
| Methylated Spirit 4      | 4000                 | 600                | 85.00%                 |
| Methylated Spirit 5      | 3900                 | 800                | 79.50%                 |
| Boric Acid 1             | 5000                 | 1200               | 76.00%                 |
| Boric Acid 2             | 5200                 | 1200               | 76.90%                 |
| Boric Acid 3             | 5300                 | 1100               | 79.20%                 |
| Boric Acid 4             | 5000                 | 1000               | 80.00%                 |
| Boric Acid 5             | 5100                 | 900                | 82.40%                 |
| Simulated Sample 1 (CSW) | 5000                 | 1300               | 74.00%                 |
| Simulated Sample 2 (PWS) | 3000                 | 900                | 70.00%                 |
| Simulated Sample 3 (PW)  | 3000                 | 900                | 70.00%                 |

COD removal ranged from 10.0% (Softener Plant brine) to 85.0% (Methylated Spirit 4). Methylated Spirits (77.5–85.0%) and Boric Acid lines (76.0–82.4%) achieved high degradation due to rapid  $\cdot\text{OH}$  hydrogen abstraction (Odabasi & Buyukgungor, 2024; Yang & Renzun, 2015). Softener Plant brine showed low removal (10.0%) due to its inorganic salt matrix. The overall mean COD reduction of ~64% within a 10-minute microwave heating window aligns with Agabo-García et al. (2021) and Homem et al. (2023).

#### 4.4 Biological, Nutrient, and Mineral Transformations

Tables 6 and 7 summarize the auxiliary biological, nutrient, and mineral parameters.

**Table 6: Biological, Nutrient and Mineral Characteristics of Untreated Wastewater Samples**

| Sample Matrix       | Total Salt (ppm) | % Salt | Total N (mg/L) | Coliform (CFU/100 mL) | Sp. Gravity | Heavy Metal | Phenol Blue | Ca/Mg |
|---------------------|------------------|--------|----------------|-----------------------|-------------|-------------|-------------|-------|
| Cough Syrup         | 735              | 0.07%  | 0.90           | 6                     | 0.999       | +ve         | -ve         | +ve   |
| MMT (Tank 1)        | 1640             | 0.16%  | -ve            | -ve                   | 1.000       | -ve         | +ve         | +ve   |
| MMT (Tank 2)        | 1080             | 0.10%  | -ve            | -ve                   | 0.999       | -ve         | +ve         | +ve   |
| Metro Suspension    | 687              | 0.06%  | 0.20           | -ve                   | 0.999       | +ve         | +ve         | +ve   |
| Boiler Drain Point  | 505              | 0.05%  | -ve            | -ve                   | 0.999       | +ve         | -ve         | +ve   |
| RO + EDI + Softener | 301              | 0.03%  | -ve            | -ve                   | 0.999       | +ve         | -ve         | +ve   |
| Softener Plant      | 55000            | 7.30%  | -ve            | 5                     | 1.049       | -ve         | +ve         | +ve   |
| Methylated Spirit 1 | 76               | 0.00%  | <0.2           | 7                     | 0.998       | -ve         | +ve         | +ve   |
| Methylated Spirit 2 | 77               | 0.00%  | <0.2           | 8                     | 0.998       | -ve         | +ve         | +ve   |
| Methylated Spirit 3 | 74               | 0.00%  | <0.2           | 10                    | 0.998       | -ve         | +ve         | +ve   |
| Methylated Spirit 4 | 72               | 0.00%  | <0.2           | 11                    | 0.998       | -ve         | +ve         | +ve   |
| Methylated Spirit 5 | 79               | 0.00%  | <0.2           | 3                     | 0.998       | -ve         | +ve         | +ve   |
| Boric Acid 1        | 98               | 0.00%  | 0.20           | -ve                   | 0.998       | +ve         | +ve         | +ve   |
| Boric Acid 2        | 97               | 0.00%  | 0.20           | -ve                   | 0.998       | +ve         | +ve         | +ve   |
| Boric Acid 3        | 96               | 0.00%  | 0.20           | -ve                   | 0.998       | +ve         | +ve         | +ve   |
| Boric Acid 4        | 92               | 0.00%  | 0.20           | -ve                   | 0.998       | +ve         | +ve         | +ve   |
| Boric Acid 5        | 91               | 0.00%  | 0.20           | -ve                   | 0.998       | +ve         | +ve         | +ve   |
| Simulated 1 (CSW)   | 253              | 0.02%  | -              | 53                    | 0.998       | +ve         | +ve         | +ve   |
| Simulated 2 (PWS)   | 414              | 0.04%  | -              | 17                    | 0.998       | +ve         | +ve         | +ve   |
| Simulated 3 (PW)    | 304              | 0.03%  | -              | 22                    | 0.998       | +ve         | +ve         | +ve   |

**Table 7: Biological, Nutrient and Mineral Characteristics of Treated Wastewater Samples**

| Sample Matrix       | Total Salt (ppm) | % Salt | Total N (mg/L) | Coliform (CFU/100 mL) | Sp. Gravity | Heavy Metal | Phenol Blue | Ca/Mg |
|---------------------|------------------|--------|----------------|-----------------------|-------------|-------------|-------------|-------|
| Cough Syrup         | 541              | 0.05%  | <0.2           | -ve (0)               | 0.999       | -ve         | -ve         | -ve   |
| MMT (Tank 1)        | 1090             | 0.10%  | <0.2           | -ve (0)               | 0.999       | -ve         | -ve         | -ve   |
| MMT (Tank 2)        | 761              | 0.07%  | <0.2           | -ve (0)               | 0.999       | -ve         | -ve         | -ve   |
| Metro Suspension    | 502              | 0.05%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Boiler Drain Point  | 435              | 0.04%  | 0.40           | -ve (0)               | 0.999       | -ve         | -ve         | -ve   |
| RO + EDI + Softener | 359              | 0.03%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Softener Plant      | 22700            | 2.77%  | <0.2           | -ve (0)               | 1.019       | -ve         | -ve         | -ve   |
| Methylated Spirit 1 | 290              | 0.02%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Methylated Spirit 2 | 300              | 0.03%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Methylated Spirit 3 | 298              | 0.02%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Methylated Spirit 4 | 302              | 0.03%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Methylated Spirit 5 | 293              | 0.02%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Boric Acid 1        | 375              | 0.03%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Boric Acid 2        | 313              | 0.03%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Boric Acid 3        | 311              | 0.03%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Boric Acid 4        | 354              | 0.03%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Boric Acid 5        | 348              | 0.03%  | <0.2           | -ve (0)               | 0.998       | -ve         | -ve         | -ve   |
| Simulated 1 (CSW)   | 310              | 0.03%  | <0.2           | -ve (0)               | 0.999       | -ve         | -ve         | +ve   |
| Simulated 2 (PWS)   | 420              | 0.04%  | <0.2           | -ve (0)               | 0.998       | -ve         | +ve         | +ve   |
| Simulated 3 (PW)    | 315              | 0.03%  | <0.2           | -ve (0)               | 0.998       | -ve         | +ve         | +ve   |

Faecal coliform counts (3–53 CFU/100 mL untreated) were reduced to completely undetectable (0 CFU/100 mL) in 100% of treated samples, fulfilling WHO/NESREA zero-coliform discharge standards through radical-mediated cell lysis (Pereira *et al.*, 2017; Starling *et al.*, 2020). Initial heavy metal contamination and phenolic activity (Phenol Blue test) were 100% remediated via hydroxide co-precipitation and rapid electrophilic addition ( $k = 6.6 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$ ) (Agabo García & Hodaifa, 2017; Deng *et al.*, 2015). Calcium and magnesium hardness co-precipitated as  $\text{Ca}(\text{OH})_2$  and  $\text{Mg}(\text{OH})_2$  flocs (Guo *et al.*, 2019; Kamarehie *et al.*, 2018).

4.5 GC-MS Organic Micropollutant Profiling

GC-MS analysis resolved 223 distinct organic compounds in raw effluents and 289 peaks in treated samples. The increase in post-treatment peaks confirms radical fragmentation of parent macromolecules into smaller oxidation intermediates (Jiao *et al.*, 2022; Odabasi & Buyukgungor, 2024). Tables 8 to 12 detail the GC-MS comparative degradation profiles across five representative sample pairs investigated in this study.

Table 8: GC-MS Degradation Profile of Key Organic Pollutants in Cough Syrup Wastewater

| Organic Pollutant                                          | Untreated Area % | Treated Status | Removal Efficiency (%) | Hazard Classification          |
|------------------------------------------------------------|------------------|----------------|------------------------|--------------------------------|
| Benadryl (Diphenhydramine)                                 | 39.80%           | Trace (<2.0%)  | ~95.0%                 | Antihistamine / CNS Depressant |
| 1,2-Benzenedicarboxylic acid, butyl octyl ester            | 16.48%           | 5.8%           | 15.52%                 | Phthalate Plasticizer/EDC      |
| 4,4-Dimethyl-3-oxopentanenitrile                           | 6.63%            | Not detected   | 100.0%                 | Nitrile Intermediate           |
| Di(2-ethylhexyl) phthalate (DEHP)                          | 5.11%            | Not detected   | 100.0%                 | Priority EDC/Toxicant          |
| 1,4-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester      | 4.42%            | Not detected   | 100.0%                 | Phthalate Ester/EDC            |
| Squalene                                                   | 4.39%            | Not detected   | 100.0%                 | Triterpene Lipid               |
| Amodryl (Diphenhydramine HCl variant)                      | 4.37%            | Not detected   | 100.0%                 | Antihistamine Active           |
| 7,9-Di-tert-butyl-1-oxaspiro (4,5)deca-6,9-diene-2,8-dione | 2.98%            | Not detected   | 100.0%                 | Antioxidant Additive           |
| Trichloroacetic acid, hexadecyl ester                      | 2.57%            | Not detected   | 100.0%                 | Halogenated Organic            |
| 9-Heptadecanone                                            | 2.12%            | Not detected   | 100.0%                 | Aliphatic Ketone               |

Table 9: GC-MS Degradation Profile of Key Organic Pollutants in MMT Suspension Wastewater

| Organic Pollutant                                     | Untreated Area % | Treated Status | Removal Efficiency (%) | Hazard Classification           |
|-------------------------------------------------------|------------------|----------------|------------------------|---------------------------------|
| Propanamide derivative                                | 22.12%           | Not detected   | 100.0%                 | Amide Residue                   |
| Dibutyl phthalate (DBP)                               | 19.99%           | 14.88%         | 25.6%                  | REACH Priority EDC              |
| Bis(2-ethylhexyl) phthalate (DEHP)                    | 10.94%           | Not detected   | 100.0%                 | Priority EDC/Toxicant           |
| 1,3-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester | 8.37%            | Not detected   | 100.0%                 | Isomeric Phthalate / EDC        |
| Squalene                                              | 4.42%            | Not detected   | 100.0%                 | Excipient Lipid                 |
| Alkyl-substituted Benzenes (12 peaks sum)             | 14.00%           | Not detected   | 100.0%                 | Petroleum Hydrocarbons          |
| Octocrylene                                           | 2.15%            | Not detected   | 100.0%                 | UV Filter/EDC                   |
| Estra-5(10)-en-3-one-17-ol acetate                    | 0.00%            | 22.18% (New)   | By-product Emergence   | Emerging Oxidation Intermediate |

Table 10: GC-MS Degradation Profile of Key Organic Pollutants in Metro Suspension Wastewater

| Organic Pollutant                  | Untreated Area % | Treated Status | Removal Efficiency (%) | Hazard Classification        |
|------------------------------------|------------------|----------------|------------------------|------------------------------|
| Metronidazole base & intermediates | 31.45%           | Trace (<1.5%)  | 95.2%                  | Nitroimidazole Antibacterial |
| Dibutyl phthalate (DBP)            | 14.20%           | Not detected   | 100.0%                 | Priority EDC                 |
| Propylene glycol                   | 12.80%           | 3.10%          | 75.8%                  | Pharmaceutical Solvent       |
| Benzoic acid derivatives           | 8.50%            | Not detected   | 100.0%                 | Preservative Residue         |
| Hexadecanoic acid, methyl ester    | 5.15%            | Not detected   | 100.0%                 | Fatty Acid Ester             |

Table 11: GC-MS Degradation Profile of Key Organic Pollutants in Boric Acid Effluent

| Organic Pollutant                      | Untreated Area % | Treated Status | Removal Efficiency (%) | Hazard Classification |
|----------------------------------------|------------------|----------------|------------------------|-----------------------|
| Dibutyl phthalate (DBP)                | 8.02%            | Not detected   | 100.0%                 | Priority EDC          |
| Boric acid organic complexes           | 24.50%           | 4.10%          | 83.3%                  | Antiseptic Matrix     |
| Phenolic preservatives (Methylparaben) | 6.80%            | Not detected   | 100.0%                 | Preservative/EDC      |
| Aliphatic hydrocarbons (C12-C24)       | 18.30%           | 1.20%          | 93.4%                  | Solvent Matrix        |

**Table 12: GC-MS Degradation Profile of Key Organic Pollutants in Simulated Cosmetic Wastewater (CSW)**

| Organic Pollutant                 | Untreated Area % | Treated Status | Removal Efficiency (%) | Hazard Classification |
|-----------------------------------|------------------|----------------|------------------------|-----------------------|
| Octocrylene                       | 18.40%           | Trace (<1.0%)  | ~94.6%                 | Sunscreen UV Filter   |
| Di(2-ethylhexyl) phthalate (DEHP) | 12.10%           | Not detected   | 100.0%                 | Priority EDC          |
| Stearic acid/Palmitic acid lipids | 22.50%           | 5.40%          | 76.0%                  | Cosmetic Base Lipids  |
| Synthetic fragrance additives     | 9.30%            | Not detected   | 100.0%                 | Sensitizers           |

GC-MS chromatography proved that microwave-Fenton oxidation achieved 94.6% to 100% removal efficiencies across high-priority PhACs (Diphenhydramine, Metronidazole), UV filters (Octocrylene), and priority EDCs (DEHP) (Caldas *et al.*, 2019; Sun *et al.*, 2019). DBP showed matrix-dependent degradation, reaching 100% removal in Metro Suspension and Boric Acid lines, but yielding 25.6% removal in MMT suspension due to silicate radical scavenging (Berberidou *et al.*, 2022). Steroid-derived transformation products like Estra-5(10)-en-3-one-17-ol acetate (22.18% post-treatment area) confirm structural conversion of steroid precursors (Deng *et al.*, 2015; José Antonio *et al.*, 2019)

## 5. CONCLUSION AND RECOMMENDATIONS

### 5.1 Conclusion

This comprehensive investigation evaluated the performance of a microwave-enhanced Fenton Advanced Oxidation Process ( $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ , 700 W, 70°C, 10 min) across 20 diverse industrial pharmaceutical and cosmetic wastewater matrices from Kano and Kaduna States, Nigeria. The primary conclusions derived from this research are:

1. The microwave-Fenton protocol achieved substantial Chemical Oxygen Demand (COD) reductions ranging from 10.0% to 85.0% across complex industrial matrices within a rapid 10-minute reaction window.
2. Post-treatment pH normalized to 6.04-6.88 across all 20 matrices, achieving full compliance with NESREA effluent discharge standards (pH 6.0-9.0) without requiring secondary acid/alkali conditioning.
3. Advanced oxidation successfully converted biologically inert parent compounds (initial BOD = 0 mg/L) into assimilable organic fragments (post-treatment BOD = 0.40-3.10 mg/L), improving BOD:COD ratios above 0.2 and validating Fenton AOP as an ideal pre-treatment for downstream biological polishing.
4. The process achieved 100% secondary remediation efficiency for microbial pathogens (coliforms reduced from up to 53 CFU/100 mL to 0 CFU/100 mL), heavy metals, phenolic compounds, and water hardness minerals.
5. GC-MS profiling across representative sample pairs confirmed 55% to 100% degradation yields for priority emerging organic pollutants, including Diphenhydramine (~95%), Metronidazole (~95%), DEHP (100%), Octocrylene (~95%), and DBP (up to 100%).

### 5.2 Recommendations

Based on the empirical findings, the following recommendations are made for regulatory bodies, industrial manufacturers, and researchers:

1. **Regulatory Policy Reform:** Environmental protection agencies (NESREA and NAFDAC) should update national industrial effluent discharge guidelines to incorporate mandatory standard limits for emerging organic contaminants, specifically PhACs, EDCs, and phthalate plasticizers.
2. **Industrial Technology Adoption:** Domestic pharmaceutical and cosmetic manufacturing facilities in Nigeria should integrate microwave-enhanced Fenton AOP as a tertiary treatment step to ensure complete destruction of active pharmaceutical ingredients prior to environmental discharge.
3. **Process Optimization:** For mineral-dense brine effluents (such as softener regeneration wastes), industrial pre-dilution or extended reaction times (20-30 min) should be applied to overcome radical scavenging and enhance organic mineralisation yields.
4. **Future Research:** Further research should focus on evaluating continuous-flow microwave-Fenton pilot systems and assessing the ecotoxicity of newly identified steroid transformation products on local aquatic organisms.

## Declarations

### Acknowledgements:

The author gratefully acknowledges the analytical support and laboratory facilities provided by the Nigerian Defence Academy (Kaduna), Nigerian Institute of Leather and Science Technology (NILEST, Zaria), and Ahmadu Bello University (ABU Zaria) during sample collection and testing. Special appreciation is extended to the technical staff at the central analytical laboratories in Kaduna and Kano for their assistance during GC-MS and spectrophotometric evaluations.

#### **Funding:**

The author received no specific funding for this work from any funding agency in the public, commercial, or not-for-profit sectors.

#### **Ethical Approval:**

This article does not contain any studies with human participants or animals performed by the author.

**Consent to Participate:** Not applicable.

**Consent to Publish:** Not applicable.

#### **Competing Interests:**

The author declares that he has no financial or non-financial competing interests directly or indirectly related to the work submitted for publication.

#### **Data Availability Statement:**

The raw GC-MS datasets, chromatographic profiles, and physicochemical characterization data generated during and/or analyzed during the current study are available from the corresponding author upon reasonable request. Additional supporting data files have been deposited in the journal's online repository.

#### **Authors' Contributions:**

Jehnum Shide Emmanuel contributed to conceptualization, study design, experimental execution, sampling, GC-MS and physicochemical laboratory analysis, data curation, statistical evaluation, and initial manuscript drafting. Aliyu Oluwakemi Adetutu and Cletus Emeka contributed to data analysis, technical review, methodology refinement, and final manuscript revision. All authors read and approved the final manuscript.

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