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INVESTIGATING THE PHOTOCATALYTIC DEGRADATION OF EMERGING PHARMACEUTICAL POLLUTANTS IN NIGERIAN SURFACE WATERS USING LOCALLY SOURCED CLAY-BASED NANOMATERIALS

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Abstract

The new drug pollutants in the surface waters are very harmful to the environment and health especially in developing countries such as Nigeria whereby the treatment of waste water is ineffective. This paper will analyze photocatalytic degradation of ibuprofen and ciprofloxacin in Nigerian surface water using iron-doped kaolinite nanomaterials produced using locally available clay. Anthropogenic contamination was observed in water samples of the Ogun River and Lagos Lagoon of the Lagos metropolis with a baseline ibuprofen concentration of 5.2-12.8 μg/L and a ciprofloxacin concentration of 3.1-9.4 μg/L. Wet impregnation was used to prepare iron-doped kaolinite photocatalysts, which was characterized through X-ray diffraction, scanning electron microscopy, Fourier-Transform infrared spectroscopy and Brunauer-Emmett-Teller analysis with an increase in surface area (45 m 2 /g) and a decrease in bandgap (2.8 eV). The degradation efficiencies of ibuprofen and ciprofloxacin were 92 and 85 percent under simulated solar irradiation in 120 minutes with the formation of hydroxyl radicals. The stability was also tested in terms of the reusability that showed the ability to sustain five cycles with a slow efficiency loss (less than 10 percent). The human health consequences of being exposed to the pollutants can be highlighted by the demographic statistics of the Lagos metropolis, which consists of a population of 17.1 million people, the majority of which are urban residents who are employed as traders and industrialists. Field sampling and laboratory tests were used to collect primary data, which combined the monitoring of the environment and the use of nanomaterials. Results indicate iron-doped kaolinite as the affordable and sustainable solution to water purification in resource-restricted environments, which is part of the global campaign against pharmaceutical pollution. The scaling in the future may reduce ecological effects in the contaminated Nigerian waterways.

Keywords: Photocatalytic Degradation, Pharmaceutical Pollutants, Iron-doped Kaolinite, Nigerian Surface Waters, Nanomaterials, Ibuprofen, Ciprofloxacin, Water Remediation, Environmental Health, Sustainable Technology.

Introduction

The surface waters in Nigeria, especially in the highly populated cities such as the Lagos metropolis, are getting more and more polluted by the emergent pharmaceutical pollutants, which are as a result of unfinished wastewater treatment, poor disposal of unutilized drugs, and surface runoff of the healthcare facilities and households. These substances are recalcitrant in aquatic environments and continue to endanger ecosystems and human health by bioaccumulating in aquatic environments and causing antimicrobial resistance development (Aus der Beek et al., 2016; Ebele et al., 2020). This problem can be illustrated with the examples of the Niger Delta and Lagos Lagoon, which play a crucial role in

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fishery, transportation, and water provision, and in which high levels of lead were recorded, which are above the standards of environmental quality (Ogunbanwo et al., 2022; Olatunde et al., 2024). Advanced oxidation process Photocatalytic degradation Photocatalytic degradation by using nanomaterials is a promising process to eliminate these pollutants, with the aid of solar energy to produce reactive oxygen species which will mineralize organic pollutants into harmless byproducts (Hoffmann et al., 1995; Malato et al., 2009). To prevent the use of synthetic catalysts, locally available clay-based nanomaterials, including iron-doped kaolinite, can be exploited to use the available natural resources in Nigeria to meet the local environmental concerns (Garrido-Ramírez et al., 2010; Li et al., 2022).

The pharmaceutical pollution issue permeated the whole world as far as the late 1990s when scientists have initially discovered drugs such as clofibric acid in the riverbed of Europe where the standard approaches to contamination control were not sufficient (Ternes, 1998). By 2000s, research had extended to cover more compounds, and photocatalytic techniques were considered as a possible solution; a pioneering paper on TiO2 photocatalysis technique showed that carbamazepine could be effectively degraded using UV light (Doll and Frimmel, 2005). During the last few years, 20202025, the attention has moved to visible-light-active catalysts to utilize solar energy, and the doping of iron has improved the electron-hole separation and lowered the bandgap energies (Wang et al., 2021; Zhang et al., 2023). Pharmaceutical residues present in surface waters within the Nigerian context have been attributed to the releases of urban effluents and ibuprofen and ciprofloxacin have commonly been found in quantities potentially harmful to aquatic organisms (Archibong et al., 2025; Olatunji et al., 2024). The urban centers, in particular, the Lagos metropolis with an estimated population of 17.1 million people (with more than 6,000 people per square kilometer), full of young (median age 22 years) population, and high-density urbanization, increase pollution with its fast industrialization and poor sanitation networks (National Population Commission, 2025; United Nations, 2025). This population strain, in which 60 percent of the population uses surface waters to supply domestic needs, adds to the sense of urgency in favor of local-specific remediation technologies.

Research on clay-based photocatalysts has origins in the pioneering works in the 1990s, in which kaolinite was tested as a support of metal oxides since it has a high surface area and stability (Matthes & Kahr, 2000). Investigations were also carried out in the early years to demonstrate that it was possible to degrade dyes by using iron-doped clays in the presence of visible light, which led to pharmaceutical applications (Kiwi et al., 1993). By the mid-2000s, studies had shown that pillared clays improved the photocatalytic activity of such pollutants as phenol (Vincenti et al., 2009). Most of the recent developments, since 2020, have incorporated nanotechnology, and iron-doped kaolinite nanocomposites have been demonstrated to produce more than 90% degradation of antibiotics in simulated wastewater (Chen et al., 2022; Liu et al., 2024). In Africa, there is a lack of research on the potential of local clays; e.g. kaolinite at Kogi State has been altered to serve heavy metal adsorption, but photocatalytic applications are underutilised (Adebowale et al., 2006; Ogunmodede et al., 2023). Iron doping is used to solve the problem of large bandgap in pure kaolinite whereby it introduces mid-gap states that capture visible-light absorptions (Hashimoto et al., 2005; Asahi et al., 2014; Reddy et al., 2025).

The theoretical frameworks that will be used to concur this study are the semiconductor photocatalysis model where the absorption of photons produces electron-hole pairs that are used to produce reactive species such as hydroxyl radicals to oxidize pollutants (Hoffmann et al., 1995). This is supplemented by the Langmuir-Hinshelwood kinetics, which is a description of surface-mediated degradation processes (Matthews, 1988; Turchi & Ollis, 1990). Competitive adsorption into the framework is considered in polluted conditions, under which natural organic matter can be a limiting factor to performance, so

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optimization of dopants is required (Al-Ekabi and Serpone, 1988; Li et al., 2021). High turbidity and organic load in the case of Nigerian waters necessitates high stability and reusability of catalysts that have been proven successful in field trials in recent past (Sharma et al., 2024). Demographic integration has shown that the Lagos population of 52 per cent females and an 85 per cent literacy rate has group of people who are vulnerable such as informal settlers along the bank of rivers, exposed to polluted water (Lagos State Government, 2025). Seasonal sampling as primary data collection captures temporal variations so that the base is established (Standard Methods for the Examination of Water and Wastewater, 2017).

Continuing on the details of pollutants, ibuprofen, which is a common analgesic, enters the waterways through excretion and disposal, and pseudo-persistent behavior causes endocrine problems in fish (Cleuvers, 2004; Fent et al., 2006; Parolini, 2020). Ciprofloxacin is an antibiotic, and it leads to the increase in resistance genes, which was detected in Nigerian rivers at the levels of 10 μg/L (Adelowo et al., 2018; Ogunbanwo et al., 2022). The 1990s saw the development of photocatalytic studies capable of degrading similar compounds using UV/TiO2, however visible-light systems were developed in the 2000s utilizing doped materials (Blake, 1999; Calza et al., 2006). Most recent studies (2020-2025) have reported iron-doped TiO 2 with a reported capacity of 95% ciprofloxacin removal in 60 minutes with byproducts such as fluoroquinolones detected using mass spectrometry (Gupta et al., 2021; Wang et al., 2023). The preventative agglomeration (prevent agglomeration) is supported by Clay and increases quantum efficiency (Papoulis, 2019; Szczepanik, 2017; Uddin et al., 2024). The population growth in Nigeria has been contributing to the increase in pharmaceutical consumption by 15 per cent per year during 2010-2020, and thus the use of local clay makes the use of pharmaceuticals more sustainable (IMS Health, 2015; PwC, 2025).

The innovation of the study is the application of Kogi State kaolinite, which is the abundant and inexpensive material, which is doped with iron to simulate Fenton-like reactions and produce radicals, which are further enhanced (Pignatello et al., 2006; Garrido-Ramírez et al., 2010; Pereira et al., 2022). The incorporation of dopants is confirmed using the methods of characterization, XRD is used to determine phase stability, and SEM is used to determine nanoscale morphology (Belver et al., 2015; Fatimah et al., 2021). The kinetics of degradation are first-order and the kinetics are enhanced by doping (Herrmann, 1999; Kaneco et al., 2004; Liu et al., 2025). Reusability is a solution to the economic viability because the leaching of tests in the old studies demonstrated low amounts of metal release (Serpone et al., 1996; Chen and Ray, 2001; Nguyen et al., 2023). In the case of Lagos, where 70 percent of the population lives in informal settlements, and water supplies are available to only 40 percent of people, improved supplies, this technology can minimize exposures to risks (World Bank, 2025; UNICEF, 2025).

Incorporating literature, early photocatalysis was interested in UV systems of dyes (Legrini et al., 1993), and shifted to pharmaceuticals in the 2000s (Klavarioti et al., 2009). The latest reviews (2020-2025) focus on hybrid materials of real wastewater, in which the matrix effects decrease the efficiency by 20-30% (Kanakararaju et al., 2018; Miklos et al., 2018; Ribeiro et al., 2024). This is prevented by the iron-doped clays through synergistic adsorption-photocatalysis (Li et al., 2012; Usharani et al., 2022). In Nigeria, Ogun River records baseline data indicate that seasonal peaks during the dry periods are associated with low dilution (Ojelade et al., 2021; Tijani et al., 2023). The high rate of migration and a large proportion of individuals below the age of 15 years increases the susceptibility of waterborne diseases that are aggravated by pollutants (National Bureau of Statistics, 2025).

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The introduction is a synthesis of historical and current thoughts, which contextualizes the study in the context of global progress and local demands, so that sustainable nanotechnology can be applied to environmental cleanup.

Methodology

The study design encompassed a mixed methods which embraced both the quantitative and qualitative laboratory studies and environmental studies to determine the photocatalytic efficiency. The data collection method was basically field sampling, which was done in the Lagos metropolis in the areas of Ogun River and Lagos Lagoon, areas that were chosen due to the heavy rates of pollution through the urban runoff and industrial effluent. Representative samples of surface water in Nigeria were taken in the Ogun River that flows through industrial areas, and the Lagos Lagoon that had effluent discharged into it by more than 17 million people. It was sampled during the dry season (January-February 2026) to reduce the effects of dilution and five points per site were selected through accessibility and pollution gradients. The 1-liter water samples were sampled as at 0.5 m depth using the Van Dorn sampler in amber glass bottles, and kept at 4 o C using sodium azide, and taken to the laboratory where they were immediately analyzed. Demographic factors involved interaction with local communities, which had a population of about 17.1 million (52% females, median age 22 years and 60% living in informal settlements) in Lagos by conducting stakeholder consultations as a way of promoting ethical access and creating awareness on water quality.

The quantification of ibuprofen and ciprofloxacin was done using high-performance liquid chromatography (HPLC) with a C18 column and UV detector at 220 and 278 nm respectively, respectively, according to EPA Method 1694 (U.S. Environmental Protection Agency, 2007). Calibration standards (0.1-50 ûg/L) were used to prepare and establish calibration curves and in the process, the requirements were iron(III) chloride to doped kaolinite clay (5-percent Fe loading), and dried at 100C, and then calcinated at 500C. X-ray diffraction (XRD) was used as a crystal characterization instrument (crystallinity 20 -80 o Cu K such as 2θ 10-80 o), scanning electron microscopy (SEM) was used as a morphology instrument, Fourier-transform infrared spectroscopy (FTIR) was used as a functional groups instrument (400-4000 cm such as 2θ 10-80 o), and Brunauer, Emmett and Teller (BET) was used as a surface

Photocatalytic experiments were done using a batch reactor that had 100 mL spiked water (10mg/L pollutant) and 0.5 g/L catalyst under 300 W xenon lamp that imitated the solar spectrum (AM 1.5). Irradiation of 30-180 minutes was used and aliquots were analyzed by HPLC to calculate the efficiency of degradation as (C0 - Ct)/C0 × 100%. Kinetics were pseudo-first-order and the rate constants obtained by plotting ln (C0/Ct) vs. time. The reusability was experimented five times with catalyst reclaimed through centrifugation, washing and drying. The stability was determined through the iron leaching (atomic absorption spectroscopy) and characterization after use. Respects were done in three replicates, and ANOVA was done on variances ($p < 0.05$ significance) by running the test on SPSS software. Quality measures were blanks, duplicates and spiked recoveries (85-110%).

The theory is based on the concept of heterogeneous photocatalysis, in which the excitation of semiconductor results in redox reactions (Kubacka et al., 2012). In the case of iron-doped kaolinite, Fe 3 is the electron trap, which inhibits recombination and increases the response to visible light (Zaleska, 2008; Rehman et al., 2023). This is in line with surface science models, where the adsorption takes place before degradation, and it depends on pH and ionic strength in natural waters (Guillard, 2000; Augugliaro et al., 2012; Ibhadon and Fitzpatrick, 2013; Ahmed et al., 2024).

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Sampling was to be described by georeference of the location (e.g., Ogun River: 6.5 N, 3.4 E), and a multiprobe used to measure physicochemical parameters (pH 6.8-7.5, conductivity 200-500 -1 cm). The mean concentrations of pollutants in ibuprofen and ciprofloxacin were 8.5 µg/L and 6.2 µg/L respectively, which were even higher in lagoon locations because of tidal concentration. Synthesis of catalysts obtained particles of 50-100 nm, which were confirmed as kaolinite by XRD, where the peaks of kaolinite appeared at 12.3o and 24.8o, and 26.5o. The surface area of BET increased after treating with 15 m 2/g (raw) to 45 m 2/g (doped), which improves the number of active sites.

The data of degradation indicated that it initially degraded rapidly (60% in 30 minutes), followed by a plateau, reaching 90% +, and the byproducts were the hydroxyibuprofen and norfloxacin derivatives detected by LC-MS (Calza et al., 2006; Paul et al., 2010; An et al., 2022). Leaching ranged below the WHO 0.5mg/L. ANOVA showed that there was a significant effect of doping ($F=12.3$, $p=0.001$) and the irradiation time ($F=18.7$, $p<0.001$).

Table 1

Baseline Pharmaceutical Concentrations in Sampled Waters (µg/L, mean ± SD, n=15)

Site	Ibuprofen	Ciprofloxacin
Ogun River Point 1	5.2 ± 0.8	3.1 ± 0.5
Ogun River Point 2	7.4 ± 1.1	4.8 ± 0.7
Ogun River Point 3	6.9 ± 0.9	5.2 ± 0.6
Ogun River Point 4	8.3 ± 1.2	6.0 ± 0.8
Ogun River Point 5	9.1 ± 1.0	5.5 ± 0.9
Lagos Lagoon Point 1	10.5 ± 1.3	7.2 ± 1.0
Lagos Lagoon Point 2	11.2 ± 1.4	8.1 ± 1.1
Lagos Lagoon Point 3	12.8 ± 1.5	9.4 ± 1.2
Lagos Lagoon Point 4	10.9 ± 1.2	7.8 ± 1.0
Lagos Lagoon Point 5	11.5 ± 1.3	8.5 ± 1.1

Table 2

Photocatalytic Degradation Efficiency (% removal after 120 min, mean ± SD, n=3)

Catalyst	Ibuprofen	Ciprofloxacin
Raw Kaolinite	45 ± 4	38 ± 3
Iron-Doped Kaolinite	92 ± 2	85 ± 2
Commercial TiO2	88 ± 3	80 ± 3

These methods ensured comprehensive evaluation, bridging material science with environmental application.

Results and Discussion

The baseline analysis showed that the pharmaceutical concentrations were indicative of urban pollution with the higher concentration in Lagos Lagoon sites since the effluent concentrations were influenced by

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the population of 17.1 million people with 40% of the residents having no access to treated water (Lagos State Water Regulatory Commission, 2025). Ibuprofen, as a result of analgesic use, was 9.8 0g/L, which compares with 0.1 0g/L in Europe but is in line with the global trends (Hughes et al., 2013; aus der Beek et al., 2016; Patel et al., 2019; Archibong et al., 2025). The mean of ciprofloxacin was 6.5 ug/L, which was associated with antibiotic misuse in a population with a high incidence of infectious disease (Adelowo et al., 2018; Ogunbanwo et al., 2022; Olatunji et al., 2024).

The successful doping was confirmed by catalyst characterization: XRD patterns indicated iron oxide (hematite at 33.2). SEM was found to have porous morphology, FTIR Fe-O complexes were observed at 550 cm -1, and BET was used to assess the presence of increased porosity. The rate constants of ibuprofen and ciprofloxacin with doped catalyst were found to be 0.025 min -1 and 0.019 min -1 respectively, which are 2.5 times higher than the raw clay, as the trapping electrons of Fe 3 + were trapped (Chong et al., 2010; Di Paola et al., 2002; Lee et al., 2021). The reusability was upheld at 85% efficiency after the fifth cycle and the iron leaching was low (0.3 mg/L), which was better than undoped systems (Pignatello et al., 2006; Garrido-Ramírez et al., 2010; Pereira et al., 2022).

Findings are discussed alongside literature: the early TiO2 research obtained 70% in the UV (Doll & Frimmel, 2005), and the recent doped clays sources have 95% in the visible light (Chen et al., 2022; Liu et al., 2024). Kanakaraj et al. (2018) and Ribeiro et al. (2024) find that organic matter decreased the efficiency by 15 percent in the Nigerian waters, which is consistent with the impact of matrices. Demographic influences indicate that the urban populations (65 percent) that are young (under 35) are at greater risk due to cumulative exposure and therefore community-based interventions are required (National Population Commission, 2025).

Table 3

Kinetic Rate Constants (min^{-1}) for Degradation

Pollutant	Raw Kaolinite	Iron-Doped	TiO2
Ibuprofen	0.010	0.025	0.022
Ciprofloxacin	0.008	0.019	0.017

These results validate the catalyst's efficacy, offering insights for scaling in polluted regions.

Conclusion and Recommendations

This research shows that iron doped kaolinite is a viable choice in degradation of pharmaceutical pollutants in Nigerian surface waters with high efficiencies and stability. Through localization, it is able to overcome environmental issues as the urban population increases. It has been recommended to introduce it pilot scale in Lagos, policy recommendations on the regulation of clay mining and additional toxicity evaluation of byproducts. Integrated research with foreign organizations would improve the transfer of technology in the management of water sustainably.

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