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Kinetic Study of Advanced Oxidation of Ibuprofen: Influence Of H_2O_2, Fe (II), And Ph on TOC Removal

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Abstract-This study investigates the kinetics of total organic carbon (TOC) removal from ibuprofen (IP)-contaminated water through a Fenton-based advanced oxidation process (AOP). A series of controlled experiments were conducted to examine the individual and combined effects of hydrogen peroxide (H_2O_2) concentration, ferrous ion (Fe^{2+}) dosage, and pH on the efficiency of organic degradation. The reaction kinetics were monitored by measuring TOC at regular intervals using varying volumes of H_2O_2 (1–8 mL), Fe(II) (1–7.5 mL), and pH values (2.1–3.1), while maintaining other conditions constant. Results demonstrated that TOC removal increased with higher H_2O_2 concentrations, attributed to enhanced generation of hydroxyl radicals. The optimal pH for TOC removal was found to be 2.5, as lower or higher pH levels led to either radical quenching or catalyst precipitation. A complex factorial design revealed that the most effective removal (≈30% TOC reduction within 24 hours) occurred at an oxidant-to-catalyst (ox/cat) ratio of 15 and an oxidant-to-contaminant (ox/IP) ratio of 76. These findings provide insights into the optimization of Fenton-based AOPs for the treatment of emerging pharmaceutical contaminants in water systems.

Index Terms- Fenton Oxidation, Ibuprofen Degradation, TOC Removal, Water Treatment

I. INTRODUCTION

Pharmaceuticals and personal care products are becoming more prevalent in aquatic habitats as a result of their widespread use and, occasionally, misuse. Trace levels have been regularly found in surface waters, usually in the range of nanograms to micrograms per decimeter cubed [1]. In parallel, efforts are being made to develop efficient treatment techniques to remove these low-level contaminants from water and wastewater sources, while continuing research continues to examine the possible ecological and human health implications of these contaminants [2]. Research has indicated that traditional biological and physicochemical methods frequently fail to completely eradicate medications [3], underscoring the necessity for more sophisticated and effective methods, including advanced oxidation processes (AOPs), to break them down.

The foundation of AOP is the in situ creation of hydroxyl radicals (•OH), which produce CO_2, water, and inorganic ions as byproducts of the non-selective oxidation of organic contaminants to their ultimate oxidation degree, or mineralization. The breakdown of pharmaceutical pollutants has been effectively accomplished using a variety of AOPs, such as Fenton oxidation [4], [5].

The Fenton process is one of the most researched and used AOPs for the breakdown of different organic contaminants because of its high efficiency, affordability, and ease of use. Ferrous iron (Fe^{2+}) catalyzes the breakdown of hydrogen peroxide (H_2O_2) in the Fenton reaction, producing very reactive hydroxyl radicals (•OH) [6]. Pharmaceuticals like ibuprofen can be efficiently broken down by these strong oxidizing agents, which can also break down a variety of chemical molecules.

A common non-steroidal anti-inflammatory drug (NSAID) that has been regularly found in aquatic habitats is ibuprofen (IBP), which is not completely eliminated by traditional wastewater treatment facilities [7]. Since its presence in water bodies can endanger aquatic life as well as human health, its effective removal is a crucial environmental issue. As a result, there is a lot of interest in improving the Fenton method for ibuprofen removal.

This introduction will focus on the optimization of the Fenton process for ibuprofen removal, specifically examining the impacts of pH and oxidant-to-catalyst ratio on Total Organic Carbon (TOC) reduction. These parameters are crucial for the efficiency and economic viability of the Fenton process. Understanding their influence is essential for developing effective and sustainable treatment strategies for pharmaceutical contaminants in water. In the heterogeneous Fenton process, the aqueous solution's pH is crucial because it greatly influences the production of hydroxyl radicals (•OH) from the oxidant [8]. Studies on the breakdown of ibuprofen using bimetallic catalysts reveal that substantial mineralization rates (70–80%) happen at pH 3–4. But at pH 5, efficiency drops to 50–55% [9]. As the oxidant breaks down into oxygen and water, decreasing its reactivity, mineralization drastically decreases to 10–20% at pH 6 and 8 [10]. Consequently, total organic carbon (TOC) removal is improved and chemical consumption and metal leaching are decreased when pH is optimized, ideally at or around 4 [11]. This makes the process more efficient and financially viable.

The effectiveness of the Fenton process, particularly for the removal of total organic carbon (TOC), depends on the ratio of oxidant to catalyst. For ibuprofen degradation to be efficient, the oxidant (such as H_2O_2) and catalyst (such as Fe^{2+} or ZVI) must be properly balanced [12]. Excess of either can decrease treatment effectiveness and decrease the availability of •OH [13]. Good degradation and resource conservation can be achieved in electro-Fenton systems with minimal ZVI dosage and excellent H_2O_2 generation [14]. While adding more iron might speed up processes, doing so too much can limit performance by causing iron precipitation and depleting •OH radicals [15]. Therefore, maximizing TOC reduction and preserving the Fenton process's cost-effectiveness in the removal of ibuprofen depend on optimizing this ratio.

The Fenton process stands as a powerful advanced oxidation technology for the removal of recalcitrant organic pollutants like ibuprofen from aqueous environments. Its effectiveness hinges significantly on critical operational parameters, particularly pH and the oxidant-to-catalyst ratio. As demonstrated, maintaining an optimal pH range, typically acidic, is crucial for maximizing the generation of highly reactive hydroxyl radicals and preventing their unproductive decomposition. Similarly, a precise balance in the oxidant-to-catalyst ratio is essential to ensure efficient radical utilization and avoid scavenging effects that can hinder degradation efficiency and increase operational costs. Understanding the intricate degradation mechanisms and the formation of intermediate products further aids in fine-tuning the process for complete mineralization and TOC reduction. Continued research and optimization of these parameters are vital for developing sustainable and economically viable solutions for pharmaceutical wastewater treatment, ultimately contributing to cleaner water resources and mitigating environmental risks posed by emerging contaminants.

II. REVIEW OF RELATED WORK

The environmental persistence and toxicity of ibuprofen as an emerging contaminant has garnered significant attention in recent literature. Ibuprofen's widespread occurrence in environmental matrices has been well-documented across multiple studies. Research indicates that ibuprofen enters aquatic environments primarily through domestic sewage and industrial discharge pathways, where it demonstrates considerable resistance to conventional treatment methodologies and poses substantial risks to ecosystem integrity [16]. The compound's persistence in environmental systems stems from its physicochemical properties, which limit natural biodegradation processes and contribute to its classification as a priority pharmaceutical pollutant.

Recent advances in treatment technology have identified the electro-peroxone (E-peroxone) process as a highly effective approach for ibuprofen remediation. This advanced technique, which integrates ozonation with electrolysis, has demonstrated exceptional performance in pharmaceutical wastewater treatment applications. Research findings indicate that E-peroxone achieved complete ibuprofen degradation within 5–15 minutes while simultaneously reducing toxicity levels to 5% inhibition of Vibrio fischeri bioluminescence, compared to 22% for ozonation alone and 88% for electrolysis alone [17]. These results position E-peroxone as a promising solution for addressing pharmaceutical contamination in wastewater treatment facilities.

Ozonation has emerged as a prominent treatment approach for ibuprofen removal across various operational scales. Laboratory-scale investigations comparing non-catalytic and catalytic ozonation demonstrated that non-catalytic ozonation achieved 93% degradation of 10 mg/L ibuprofen solutions within 4 hours under optimal conditions [18]. The study further revealed that incorporation of H-Beta and Fe-H-Beta zeolite catalysts in rotating bed reactors enhanced degradation efficiency, highlighting the potential of zeolite-catalyzed ozonation for addressing ibuprofen's environmental persistence.

A comprehensive environmental assessment by researchers highlighted ibuprofen's ubiquitous presence in both aquatic and terrestrial ecosystems, where it induces cytotoxic and genotoxic effects, promotes oxidative stress responses, and causes developmental abnormalities in aquatic organisms [19]. The study emphasized that ibuprofen's extensive therapeutic use, combined with its poor environmental degradability, establishes it as a significant ecological threat requiring immediate attention. While biodegradation approaches have shown limited efficacy due to the compound's recalcitrant physicochemical characteristics, bacterial degradation pathways present potential solutions that warrant further investigation.

A systematic evaluation of multiple advanced oxidation processes (AOPs) provided valuable insights into their relative effectiveness for ibuprofen removal. The comparative analysis examined Fenton, UV/H_2O_2, and UV/TiO_2 systems, revealing that all methods except UV treatment alone achieved over 99.9% ibuprofen removal efficiency, while UV alone reached 85% effectiveness [20]. The Fenton process emerged as the most efficient and economically viable option for integration into treatment facilities targeting pharmaceutical micropollutants.

Toxicological research has provided crucial insights into ibuprofen's biological effects. Studies in Rattus norvegicus demonstrated that oral ibuprofen administration (10–30 mg/kg over 21 days) elevated lipid peroxidation levels and enhanced antioxidant enzyme activity (SOD and CAT) [21]. Although some parameters returned to baseline after a 7-day withdrawal period, the results confirmed ibuprofen's potential for oxidative toxicity in mammalian systems, raising concerns about chronic exposure effects in non-target organisms.

Pilot-scale validation studies treating municipal effluent confirmed ozonation's practical applicability, demonstrating that ibuprofen and similar acidic pharmaceuticals underwent 90–99% oxidation using ozone concentrations ≥2 mg/L [22]. Notably, the presence of suspended solids showed minimal interference with oxidation efficiency, supporting ozonation's implementation across diverse effluent compositions and treatment scenarios.

Advances in analytical methodology have facilitated improved ibuprofen detection and monitoring capabilities. Researchers developed an innovative electrochemical detection system using screen-printed carbon electrodes modified with manganese oxide nanoparticles (MnO_2NPs) for simultaneous ibuprofen and metoprolol detection in wastewater [23]. The sensor demonstrated excellent catalytic performance, achieving a detection limit of 3.81 pM with linear response across 0.97–5.82 pM concentrations. Successful validation with actual wastewater samples indicates strong potential for real-time pharmaceutical pollutant monitoring applications.

Integrated treatment approaches combining multiple technologies have shown promise for enhanced ibuprofen removal. A hybrid system combining ozone-based advanced oxidation (peroxone) with activated carbon adsorption effectively reduced pharmaceutical industrial effluent chemical oxygen demand (COD) levels [24]. The peroxone process alone delivered 75–88.5% COD reduction within 3 hours, while subsequent adsorption enhanced overall removal to 85.4–92.7%. Optimization studies revealed pseudo-first-order degradation kinetics, validating this integrated approach for industrial applications.

Recent comparative research extending to photocatalysis, ozonation, and gas-phase pulsed corona discharge (PCD) demonstrated >90% removal efficiency across all methods, with conventional treatment already achieving >90% ibuprofen reduction [25]. Among the advanced oxidation processes evaluated, PCD exhibited the lowest energy consumption (0.28 kWh/m³), followed by ozonation (0.55 kWh/m³), making both technologies suitable for meeting anticipated regulatory requirements under the European Union's Urban Wastewater Treatment Directive.

Photocatalytic degradation represents an emerging area of research for ibuprofen treatment. Recent investigations utilizing BiOCl nanosheets under 254 nm UV irradiation achieved ≥97% ibuprofen degradation (50 μM) within 60 minutes [26]. Mechanistic studies revealed that hydroxyl radical scavengers had minimal impact on degradation efficiency, while hole scavengers significantly reduced performance. These findings indicate that BiOCl's photocatalytic mechanism operates primarily through hole oxidation rather than hydroxyl radical pathways, establishing it as a sustainable alternative to conventional photocatalysts such as TiO_2.

III. RESEARCH METHODOLOGY AND MATERIALS

Dilutions of H_2O_2 and IP were made using DI water and volumetric flasks. Fe(II) was accurately weighed and dissolved using analytical methods to maintain precision. All components were mixed in a specific order: IP and Fe(II) were added first, and H_2O_2 was added last to initiate the oxidation reaction. The moment of H_2O_2 addition was marked as time zero for kinetic monitoring. This approach ensured reproducibility and accuracy across experiments.

A. Instruments

1) Analytical Balance: Mettler Toledo ML 204
2) Magnetic Stirrer: VELP Scientifica MICROSTIRRER (Max speed: 1100 rpm)
3) pH Meter: WTW Multi 3320

B. Total Organic Carbon Analyzer

TOC analysis was conducted using the Shimadzu TOC-V CPH series analyzer. The technique quantifies organic contamination by oxidizing organic matter to CO_2, which is detected by a non-dispersive infrared (NDIR) sensor.

The Non-Purgeable Organic Carbon (NPOC) method was employed. Water samples were acidified with HCl to remove inorganic carbon (TIC), then purged with a carrier gas. The remaining organic carbon was oxidized at 720°C using a platinum catalyst, and the released CO_2 was measured

IV. RESULTS AND DISCUSSION

This section presents the kinetic results of the oxidation process. Reaction kinetics, a branch of physical chemistry, focuses on studying the rates of chemical reactions. A standard approach in kinetic studies involves maintaining all initial conditions (such as pH, temperature, and concentrations) constant, except for the specific variable under investigation. In this research, three series of kinetic experiments were conducted: one in which the oxidant concentration was varied, and another in which the initial Fe (II) concentration was systematically changed and the third in which the pH was varied.

The experiments were carried out by preparing 0.5, 1.0 and 2.0 litre of reaction mixtures which was transferred into multiple TOC vials. The organic carbon content of these vials was measured at different times, therefore, creating the kinetic curve.

A. H_2O_2 dependence of the removal of TOC

A result can be shown below in determining the H_2O_2 dependence on the removal of TOC in IP, 190ml of IP was used and kept constant throughout the 4 different mixtures, 35 ml of Fe(II) was also used constantly in all four mixtures. The first mixture M1 contains 1 ml, second mixture contain 2.5 ml, third mixture contain 5 ml and fourth mixture contain 8 ml volumes of H_2O_2 in order.

The mixtures have oxidant/ibuprofen (ox/IP) ratios of 8.3, 20.7, 41.4, 66.3 for mixture 1, 2, 3, and 4. The ox/cat raios of 2.4, 5.9, 11.8, 19.0 for 1, 2, 3, and 4 respectively.

The result of the oxidations can be represented on the spreadsheet graph below.

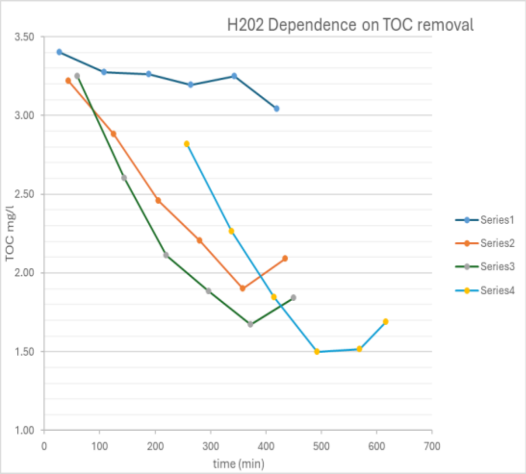

Figure 1: H_2O_2 dependence on TOC removal with respect to time.

On the above figure series 1, 2, 3, and 4 represents the four mixtures M1, M2, M3 and M4 orderly.

It was observed that the removal of the TOC was more effective with a higher H_2O_2 concentrations because H_2O_2 is a strong oxidizing agent that helps break down organic compounds into carbon dioxide and water. In advanced oxidation processes, H_2O_2 can generate hydroxyl radicals, when combine with catalysts (like Fe^{2+} in Fenton reactions). With more oxidants present, the reaction rate increases, organic molecules are degraded more quickly and efficiently.

B. pH dependence of the removal of TOC

A result can be shown below in determining the pH dependence on the removal of TOC in IP. Initial IP was kept constant throughout the three different mixtures, similarly to the initial concentrations of of Fe(II) and H_2O_2. The pH of first mixture was set using H_2SO_4 to 2.1, second mixture 2.5, and third mixture 3.1.

The result can be shown on the following charts.

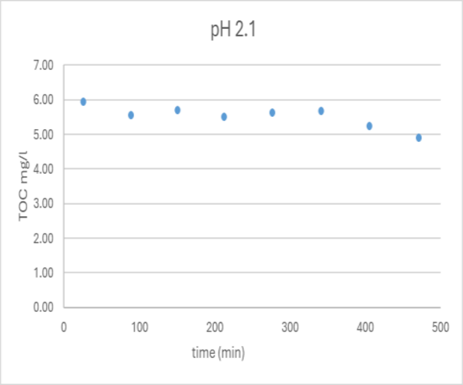

Figure 2: pH 2.1 on TOC removal with respect to time.

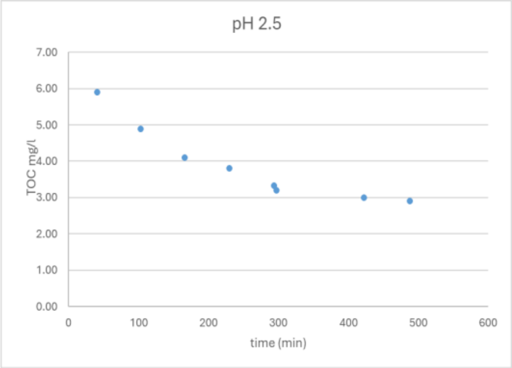

Figure 3: pH 2.5 on TOC removal with respect to time.

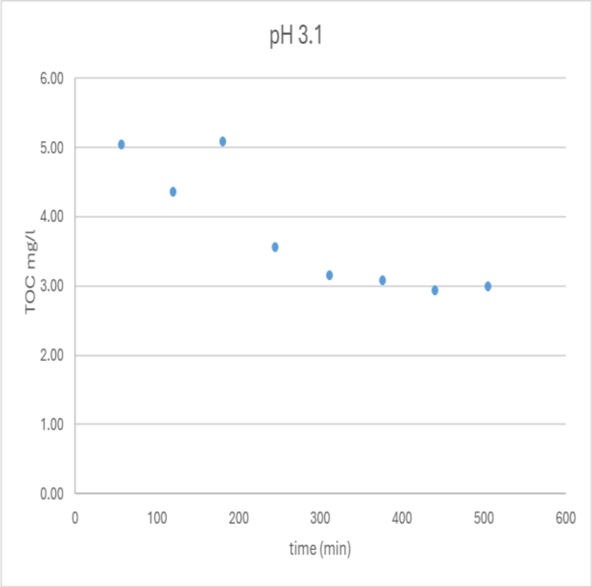

Figure 4: pH 3.1 on TOC removal with respect to time.

TOC removal is more effective at pH 2.5 because acidic conditions enhance the generation and stability of reactive oxidizing species. At pH 2.5, iron stays soluble, which is essential for the reaction and at pH 2.5, the balance between H_2O_2 stability and reactivity favors maximum hydroxyl radicals' production, boosting organic matter breakdown. If pH is too low H_2O_2 becomes more stable and less reactive, if pH is too high (>3), iron may precipitates as $Fe(OH)_3$, reducing radical production. At higher pH, side reactions may increase, including the formation of carbonate and bicarbonate ions which can scavenge hydroxyl radicals, reducing effectiveness.

C. Complex dependence

This complex experiment aimed to study the effect of the concentrations of H_2O_2 and Fe(II) on the removal of TOC. Altogether 8 different mixtures were prepared by systematically changing the aforementioned parameters (Table 1).

Table 1: Complex dependence reaction mixture table

Number of samples	IP (ml)	H_2O_2 (ml)	Fe^{2+} (ml)	V total	IP (mM)	H_2O_2 (mM)	Fe^{2+} (mM)	ox/IP ratio	ox/cat ratio
M1	40	0	0	50	0.025	0	0	0.0	0.0
M2	40	0.4	5.0	50	0.025	0.393	0.052	15.7	7.5
M3	40	0.8	5.0	50	0.025	0.787	0.052	31.5	15.0
M4	40	1.2	5.0	50	0.025	0.180	0.052	47.2	22.5
M5	40	2.5	5.0	50	0.025	2.46	0.052	98.3	46.8
M6	40	1.2	1.0	50	0.025	1.180	0.010	47.2	112
M7	40	1.2	2.5	50	0.025	1.180	0.026	47.2	45.0
M8	40	1.2	7.5	50	0.025	1.180	0.078	47.2	15.0

For each mixture, the solutions described in Table 1 were prepared and tested. In all cases, IP and Fe(II) were first mixed, and the desired pH was adjusted using sulfuric acid (H_2SO_4). Oxidation was then initiated by the addition of the oxidant, and the time of initiation was recorded. After allowing the mixtures to react for 24 hours – based on prior experiments, which indicated that the reaction completes within this time – TOC measurements were performed. The removal efficiency (RE) of the organic carbon from IP was calculated and expressed as a percentage using the following formula:

$$\mathrm{RE}\ (\%) = \left(\frac{\mathrm{TOC_{ref}} - \mathrm{TOC_{sample}}}{\mathrm{TOC_{ref}}} \right) \times 100$$

where $\mathrm{TOC_{ref}}$ is the TOC of a solution containing the IP in the absence of oxidant or Fe (II), while $\mathrm{TOC_{sample}}$ is the TOC of the solution containing all the reactants.

Among all tested conditions, the highest removal efficiency (RE%) was achieved with an Fe(II) concentration of 0.079 mM and an H_2O_2 concentration of 1.18 mM. This corresponds to an oxidant-to-catalyst ratio of 15 and a 76-fold excess of H_2O_2 relative to IP. Under these conditions, about 30% of the initial organic carbon content was removed within 24 hours.

It can be concluded that the optimal conditions for the oxidative treatment were observed at approximately pH 2.5, with an oxidant-to-catalyst (ox/cat) ratio between 10 and 15. This finding aligns reasonably well with previous studies on the Fenton process. For instance, in a study focused on the removal of 3-aminopyridine, an emerging contaminant, the optimal ox/cat ratio was reported to be between 10 and 40 [27]. Another investigation targeting the removal of organic content from produced water identified the optimal ratio in the range of 10 to 25 [28]. The variation among studies is expected, given the differences in the nature and reactivity of the contaminants investigated.

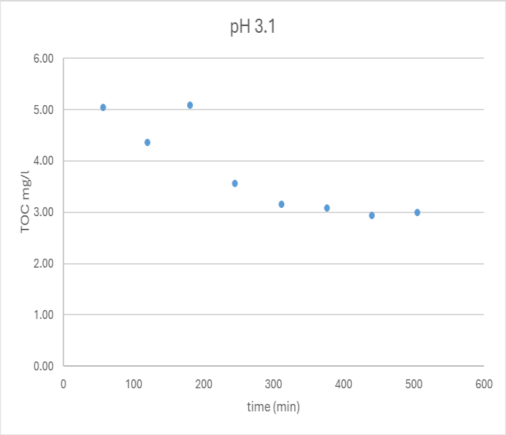

Figure 5: The dependence of the removal efficiency on the oxidant/catalyst ratio.
c(IP) = 0.025 mM, pH = 2.5, solvent: DI water

CONCLUSION

This study demonstrated the effectiveness of the Fenton oxidation process for the removal of total organic carbon (TOC) from ibuprofen-contaminated water. Kinetic experiments revealed that increasing the concentration of hydrogen peroxide (H_2O_2) and optimizing the pH significantly enhanced TOC removal efficiency. The optimal conditions were observed at pH 2.5 with an oxidant-to-catalyst (ox/cat) ratio between 10 and 15. Under these conditions, approximately 30% of the organic carbon was removed within 24 hours. These findings align with previously reported optimal ranges for Fenton-based treatments and highlight the importance of parameter optimization in advanced oxidation processes. The results contribute to the broader understanding of pharmaceutical degradation and offer a foundation for the development of efficient water treatment strategies targeting emerging contaminants.

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