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Optimizing UV-based advanced oxidation processes for effective TOC removal in wastewater

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Abstract. The growing water demand requires advanced treatment technologies to efficiently remove recalcitrant organic compounds from wastewater for its reuse. This study evaluated and compared the performance of various advanced oxidation processes (AOPs) for total organic carbon (TOC) removal, focusing on UV-based systems. Among the tested methods, UV/PS and UV/TiO_2/Persulfate (PS) achieved the highest TOC removal efficiencies of 89.1% and 92.6%, respectively, under optimized conditions. The UV/TiO_2/PS process, which combines persulfate activation with TiO_2 photocatalysis, demonstrated superior performance under conditions 1 g/L persulfate, 1 g/L TiO_2, and 39 W UV output, making it a promising option for wastewater reuse. Non-UV-based AOPs, including US/H_2O_2 and US/PS, also exhibited high TOC removal efficiencies (87.8% and 81.9%, respectively) due to ultrasonic cavitation. However, their high energy intensity requirements challenges for process scalability. O_3/H_2O_2 achieved moderate efficiency (70.5%), while Fenton-based processes (Fenton and Ultrasound (US)/Fenton) showed lower efficiencies (59.5% and 71.6%) due to their sensitivity to pH conditions. Overall, UV-based systems outperformed others in efficiency and adaptability, with UV/TiO_2/PS identified as the most effective for treating recalcitrant pollutants. In the UV/PS and UV/TiO_2/PS systems, sulfate ion generation confirmed effective persulfate activation, resulting in sulfate radical formation. Compared to Fenton-based processes, which generate substantial sludge, these systems represent an environmentally favorable alternative.

Keywords: advanced oxidation processes; sustainable waster reuse; total organic carbon removal; persulfate activation; UV-based system; wastewater treatment

1. Introduction

The increasing frequency of extreme weather events, such as heavy rainfall and severe droughts driven by climate change—along with rising industrial water demand, the need to secure raw water resources, and ensuring a stable supply of water and wastewater services have emerged as critical policy challenges in South Korea. The annual volume of reclaimed wastewater from public treatment facilities with capacities exceeding 500 m^3/day has steadily grown, from 167 million tons (reuse rate: 2.9%) in 2000 to 712 million tons (10.8%) in 2008, 943 million tons (13.5%) in 2014, and 1,113 million tons (15.5%) in 2018, reaching 1,137 million tons (15.4%) in 2022 (Ministry of Climate, Energy and Environment, 2024).

Current processes for producing reclaimed water primarily include coagulation and filtration, membrane filtration, activated carbon adsorption, and ozonation. However, the growing emphasis on reclaimed water utilization has intensified research into AOPs. The AOPs offer stable treatment efficiency and effective removal of recalcitrant compounds, enabling effluent TOC treatment to meet stringent levels required for reuse as raw water in critical industries such as semiconductors and displays (Kwon *et al*. 2020, Zhao *et al*. 2020, Ligaray *et al*. 2019, Lee *et al*. 2018). Refractory pollutants, such as carbamazepine, dimethyl phthalate, pharmaceuticals, and endocrine disruptors are difficult to treat effectively using conventional physical or biological processes, necessitating tertiary treatment through AOPs (Jaimes-López *et al*. 2024, Rizwan and Bilal 2022). AOPs have proven highly effective in degrading recalcitrant compounds due to their ability to generate strong oxidants, such as hydroxyl radicals (•OH) and sulfate radicals ($SO_4^{\bullet -}$), which facilitate rapid and efficient oxidation reactions (Oh *et al*. 2023, Heydari *et al*. 2021, Khulbe *et al*. 2012).

Various AOP technologies include Fenton reactions, ozone-based AOPs, and physical AOPs, often combining different oxidants and catalysts to enhance radical generation. The Fenton reaction, a traditional AOP, is effective without the need for external energy input, requiring only a catalyst and oxidant. However, its limitations include the generation of $Fe(OH)_3$ sludge and limited decolorization efficiency. Consequently, research has shifted from conventional homogeneous Fenton reactions to heterogeneous Fenton systems (Thomas *et al*. 2021, Sun *et al*. 2023). Furthermore, Fenton processes have been integrated with other techniques, such as photo-Fenton (Romero *et al*. 2016), ultrasonic-Fenton (Serna-Galvis *et al*. 2023), and electro-Fenton (Su *et al*. 2012) systems, to improve performance. In addition, pseudo-Fenton reactions, which use various oxidants and catalysts, are actively being studied as extensions of traditional Fenton processes (Ribeiro *et al*. 2024, Babuponnusami and Muthukumar

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2014).

Ozone-based AOPs, such as Peroxone (O_3+H_2O_2), have been widely used in domestic water treatment plants and industrial wastewater facilities. However, these processes require additional equipment for ozone generation and containment, and residual ozone or its by-products may remain in the treated water. Ozone-based AOPs can be enhanced by combining ozone with UV, (photo)catalysts, ultrasound (US), or Fenton reactions to improve performance (Lu *et al.* 2024, Epelle *et al.* 2023, Rekhate and Srivastava 2020).

AOPs utilizing US, such as US/H_2O_2, offer advantages, including the absence of sludge and residual by-products. However, these processes can generate high-temperature effluent, requiring cooling systems when needed (de Carvalho Costa *et al.* 2021, Camargo-Perea *et al.* 2020, Madhavan *et al.* 2019). US-based systems can further improve efficiency by incorporating oxidants (e.g. H_2O_2, PS, O_3) and catalysts.

UV-based AOPs, such as UV/H_2O_2, produce no sludge or residual by-products and generally result in lower effluent temperatures compared to US-based processes. Like US-based systems, UV systems can be enhanced by combining with oxidants (e.g. H_2O_2, PS, O_3, Cl_2) or catalysts (e.g. TiO_2, Fe^{2+}) (Tanos *et al.* 2023, Lei *et al.* 2020, Miklos *et al.* 2018).

Son *et al.* (2024) evaluated the treatment of actual textile wastewater using UV/H_2O_2 and found that hydrogen peroxide concentration was the most influential factor affecting both color and TOC removal. Their multilayer perceptron (MLP) analysis, which evaluated the impact of operating variables, showed that under optimal conditions (high hydrogen peroxide concentration and prolonged reaction time), over 99% color removal could be achieved. The MLP model itself demonstrated high predictive accuracy (R^2 = 0.99) for color removal based on experimental data (Son *et al.* 2024).

Among these UV-based AOPs, TiO_2 has been extensively studied for its applications as a photocatalyst, with ongoing research focused on improving its performance through modifications (Shafeei *et al.* 2019) and doping (Zhang *et al.* 2019).

Lee *et al.* (2016) evaluated the removal efficiency of methyl orange (MO) using the microwave/UV/TiO_2/PS process. Under ambient conditions, with an initial MO concentration of 8.72×10^{-2} M, a microwave power of 0.5 kW, synthesized anatase-phase TiO_2, a PS concentration of 236.25 mg/L, and a reaction time of 60 min, the process achieved approximately 80% MO removal (Lee *et al.* 2016).

Yahiaoui *et al.* (2024) investigated the photocatalytic degradation of acetaminophen (ACE) under visible light conditions using TiO_2 (P25) coated on glass plates via a thermal attachment method. The glass plates were coated 2-7 times and subjected to visible light irradiation at an intensity of 450 W/m², at 35°C, pH 6.5, with varying ACE concentrations (5-65 mg/L) and flow rates (6-17.5 mL/s). The highest ACE removal efficiency was observed at a flow rate of 14 mL/s, achieving 100% removal within 3 h under optimal conditions. Even after six reuses of the TiO_2-coated glass plates, which were washed with distilled water at 60°C and dried, the ACE removal efficiency remained at 97% after 3 h (Yahiaoui *et al.* 2024).

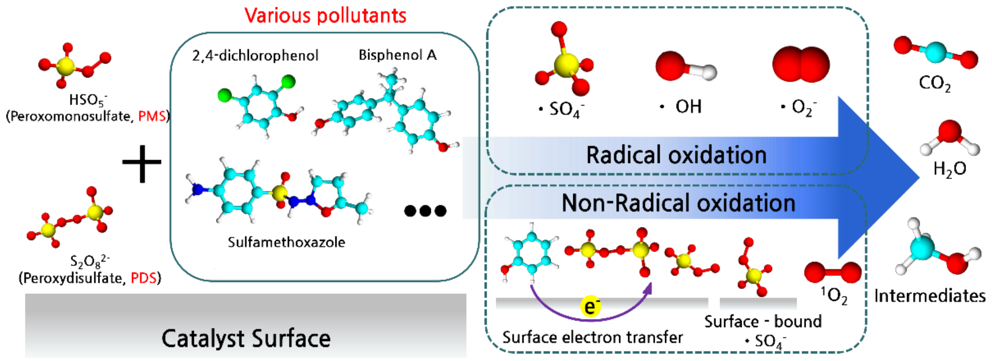
Laghrib *et al.* (2024) explored the degradation of methylene blue (MB) and total organic carbon (TOC) using TiO_2 nanoparticles either singly doped or co-doped with Mn and Sr via the sol-gel method. Under conditions of 0.12 g TiO_2/L, 30 mg MB/L, and UV irradiation at 365 nm (15 W), pure-TiO_2 achieved 43% MB degradation efficiency, while 1% Mn-TiO_2 and 1% Sr-TiO_2 exhibited efficiencies of 86% and 92%, respectively. Co-doped Mn-Sr-TiO_2 demonstrated the highest MB degradation efficiency at 95%, substantially exceeding that of singly doped TiO_2, which exhibited efficiencies ranging from 54% to 62% (Laghrib *et al.* 2024).

While extensive research has been conducted on individual AOPs such as UV/TiO_2, O_3/H_2O_2, Fenton, and persulfate (PS) treatments, comprehensive investigations into multi-component AOPs (involving three or more oxidation mechanisms) for wastewater effluent reuse remain limited. Although UV/PS and UV/TiO_2 systems have been widely studied individually, their integrated application under direct UV irradiation remains largely unexplored, particularly in the context of optimizing effluent reuse. This study aimed to optimize a UV-based multi-AOP by simultaneously generating sulfate radicals ($SO_4^{\bullet-}$) from persulfate and hydroxyl radicals (•OH) from titanium dioxide. This combined oxidative approach was intended to enhance the TOC removal efficiency from wastewater effluents by increasing the overall oxidative potential of the system. The research sought to evaluate the efficiency and applicability of UV/PS and UV/TiO_2/PS technologies for wastewater effluent treatment, assess organic matter removal capabilities across alternative oxidation processes (including Fenton, ozonation, hydrogen peroxide, and ultrasonic treatments), analyze their oxidative potential, and identify the most optimal AOP configuration for sustainable wastewater effluent reuse.

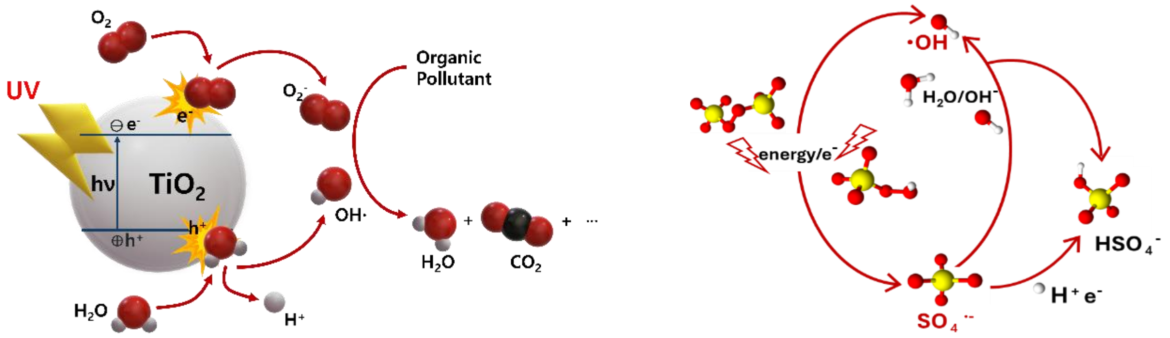
2. Theory of UV-based AOP and recent applications

TiO_2 exhibits exceptional properties, including chemical and biological inertness, high stability against photo-corrosion and chemical degradation, and significant economic advantages compared to other commonly used photocatalysts, such as SiO_2, ZnO, WO_3, CdS, and ZnS. TiO_2 crystals occur in several crystalline phases, with anatase and rutile being the most commercially relevant (Khan and Shah 2023, Jiang *et al.* 2021, Gatto *et al.* 2015, Shon *et al.* 2010). When a photocatalyst absorbs light (hν) with energy exceeding its band gap, it generates an electron-hole (e^--h^+) pair, which subsequently separates into photo-generated free electrons and holes. The band gap energy is a critical parameter in the photocatalytic process; excessively narrow or wide band gaps can hinder photocatalytic efficiency, making an optimal band gap essential for effective performance (Xu *et al.* 2017).

To overcome the limitation of TiO_2 being activated only in the ultraviolet spectrum, research has focused on



(a) Catalyst-activated persulfate (PS) oxidation



(b) UV/TiO_2 (c) UV/PS

Fig. 1 Schematic diagrams of UV-based AOPs reaction mechanisms

modifying its band gap energy to extend photocatalytic activity into the visible light range, thus broadening its potential applications (Oh *et al.* 2021, Seo *et al.* 2019, Khan *et al.* 2014).

UV-based AOPs are characterized by the use of specific ultraviolet wavelengths, primarily UV-C, in combination with various radical-generating agents. These processes facilitate efficient oxidative decomposition of aqueous organic pollutants while minimizing the generation of byproducts, such as sludge. Recent studies have indicated that PS provides superior cost-effectiveness and pollutant removal efficiency compared to hydrogen peroxide (H_2O_2) in UV-based AOPs. Additionally, ongoing research has explored the modification of TiO_2 through doping to enhance the economic feasibility of these technologies.

Xiao *et al.* (2016) conducted a comparative evaluation of UV/PS and UV/H_2O_2 processes based on the degradation of iodoacetic acid (IAA). Under experimental conditions of 5 W UV irradiation, 1.5 μM IAA, and 60 μM oxidant concentrations, the UV/PS process achieved nearly 90% IAA removal, approximately twice the efficiency of the UV/H_2O_2 process. The pseudo-first-order rate constants for the UV/PS process were 2.54, 3.43, and 4.80 times higher than those of UV/H_2O_2 at oxidant/IAA molar ratios of 40, 120, and 300, respectively (Xiao *et al.* 2016).

Kang *et al.* (2024) investigated the UV/PS process for bisphenol A (BPA) degradation and TOC removal in coagulated industrial wastewater. Using 11 mg/L BPA, 6 W UV, and 400 mg/L PS, they achieved a BPA removal efficiency of 98.9%. In a subsequent experiment with 100 mg/L industrial wastewater, 6 W UV, 8,000 mg/L PS, and a 180-minute reaction time, the process demonstrated TOC removal rates of 92.2%, 85.7%, and 89.9% for metal processing, food processing, and adhesive production wastewaters, respectively (Kang *et al.* 2024).

Dhaka *et al.* (2017) employed a UV (low-pressure mecury lamp)/PS process for methyl paraben (MP) degradation. Their findings showed a 98.9% MP removal under conditions of 32.8 μM MP, 1 mM PS, pH 6.5, and a 90-minute reaction period. Radical scavenging experiments using ethanol (EtOH) and *tert*-butyl alcohol at 100 mM concentrations highlighted the critical role of radical species: MP removal efficiency decreased to 52.7% with EtOH and 77.1% with *tert*-butyl alcohol. Additionally, the presence of 20 mg/L humic acid further reduced the removal efficiency to 53%. The study confirmed that sulfate radicals were the primary active species responsible for MP degradation under acidic and neutral pH conditions (Dhaka *et al.* 2017).

Eqs. (1)-(9) illustrate the chemical reactions involved in the UV/TiO_2 mechanism, while Fig. 1(b) provides a schematic representation of these processes (Al-Mamun *et al.* 2019, Al-Anbari *et al.* 2017).

$$TiO_2 + h\nu \rightarrow TiO_2\,(e^- + h^+) \tag{1}$$

$$TiO_2\,(h^+) + H_2O \rightarrow H^+ + \bullet OH \tag{2}$$

$$TiO_2\,(h^+) + OH^- \rightarrow TiO_2 + \bullet OH \tag{3}$$

$$TiO_2\,(e^-) + O_2 \rightarrow TiO_2 + O_2^{\bullet -} \tag{4}$$

$$TiO_2\,(2e^-) + O_2 + 2H^+ \rightarrow H_2O_2 \tag{5}$$

$$TiO_2(e^-) + H_2O_2 \rightarrow \bullet OH + OH^- \quad (6)$$

$$\text{Organic compound} + \bullet OH \rightarrow \text{Degradation products} \quad (7)$$

$$\text{Organic compound} + TiO_2(h^+) \rightarrow \text{Oxidation products} \quad (8)$$

$$\text{Organic compound} + TiO_2(e^-) \rightarrow \text{Reduction products} \quad (9)$$

PS has been extensively studied as an oxidant due to its high solubility in water, low cost, and ease of storage. Upon activation, PS generates sulfate radicals ($SO_4^{\bullet-}$) with an oxidation potential comparable to or even higher than hydroxyl radicals (•OH) ($E^0(SO_4^{\bullet-})$ = 2.5-3.1 V vs. NHE). These radicals exhibit a longer lifetime ($3\text{-}4 \times 10^{-5}$ s), high radical conversion efficiency, and low self-decomposition rates (Tian *et al.* 2022, Li *et al.* 2022, Lee *et al.* 2018). These properties make PS a promising oxidant for degrading recalcitrant pollutants.

PS compounds consist of a sulfate group bonded to a peroxo group and are classified into peroxymonosulfate (PMS; HSO_5^-) and peroxydisulfate (PDS; $S_2O_8^{2-}$). Among these, PDS offers advantages over peroxymonosulfate in terms of radical generation efficiency, dissociation energy, and cost-effectiveness (Bakhshaei *et al.* 2024, Wu *et al.* 2024, Park *et al.* 2024, Yang *et al.* 2020, Luo *et al.* 2016).

Fig. 1(a) schematically depicts the oxidation reaction mechanism of PS when activated by catalysts. PS exhibits relatively weak UV absorption at wavelengths above 300 nm, thereby making UV-C light (wavelength: 254 nm) the most suitable for PS activation via UV irradiation. The quantum yield, defined as the ratio of reaction occurrences to absorbed photons, is a crucial parameter in PS activation. The UV wavelength significantly influences the quantum yield, with UV-C wavelengths, particularly at 254 nm, being predominantly used due to its higher efficiency (Choi and Park 2020).

Fig. 1(c) illustrates the reaction mechanism of the UV/PS process. PDS, with a high oxidation-reduction potential of 2.01 V, acts as a strong oxidant, as shown in Eq. (10). Upon UV irradiation, cleavage of the O-O bond in the PDS results in the formation of sulfate radicals ($SO_4^{\bullet-}$), as described in Eq. (11). Additionally, as shown in Eq. (12), hydrogen radicals (H·) generated during the process can react with PDS to form sulfate radicals via the pathway outlined in Eq. (13).

$$PDS(S_2O_8^{2-}) + 2e^- \rightarrow 2SO_4^{2-} \qquad E^0 = 2.01\ V \quad (10)$$

$$PDS + UV \rightarrow 2SO_4^{\bullet-} \qquad E^0 = 2.50\ V \quad (11)$$

$$H_2O \rightarrow H\bullet + \bullet OH \quad (12)$$

$$PDS + H\bullet \rightarrow SO_4^{\bullet-} + SO_4^{2-} + H^+ \quad (13)$$

3. Experimental section

The samples analyzed in this study were effluents from a local municipal wastewater treatment plant, treated through biological processes. The effluent from the wastewater treatment plant, which was used as the influent for the AOP experiment, had an average TOC concentration of 6.8 mg/L, ranging from 5.5 to 8.0 mg/L. Additionally, the influent contained an average TN concentration of 8.5 mg/L and an average TP concentration of 0.3 mg/L, along with varying concentrations of other water quality parameters, including TCOD, SCOD, pH, TSS, VSS, and FSS.

For the AOP experiments, TiO_2 (TMC-BA100, anatase phase) supplied by Dongwoo TMC Co., Ltd. was used as the photocatalyst. Potassium persulfate ($K_2S_2O_8$, 98-102%, SAMCHUN) was employed as the oxidant. A waterproof UV-C lamp (ZY-UV1, ZhiYang Co., 13 W, 254 nm), featuring a cylindrical design measuring 2 cm in diameter and 13 cm in length, was employed as the UV source. Additionally, to analyze the effects of organic matter excited by UV irradiation, fluorescence intensity was measured using a fluorescence analyzer (Lumilux 5100, Scinco). During the analysis, the excitation wavelength (EX) range was set to 190-650.

Effluent samples from the wastewater treatment plant were collected in 2 L sterile sampling bottles. To ensure sample homogeneity prior to the experiments, the effluents were vacuum-filtered using Whatman GF/C filters (1.2 μm). TOC concentrations were determined using the high-temperature combustion oxidation method (TOC-L CPH, Shimadzu), while chemical oxygen demand (COD_{cr}) concentrations were measured with a UV/Vis spectrophotometer (DR 5000, HACH).

The AOP experiments were conducted in a 3 L acrylic reactor with an effective working volume of 2 L, using magnetic stirring to maintain homogeneous oxidation reactions. UV experiments employed direct contact with LED UV-C lamps. To minimize external light interference, all experiments were performed in a dark box.

The oxidation efficiency (%) of the wastewater effluent treated with UV-based AOPs was calculated as the percentage reduction in TOC concentration. This was determined by the difference between the initial TOC concentration before AOP treatment and the TOC concentration after oxidation, as shown in Eq. (14).

$$AOP\ efficiency\ (\%) = [1 - \frac{C_t}{C_0}] \times 100 \quad (14)$$

where, C_t: TOC concentration (mg/L) before AOPs C_o: TOC concentration (mg/L) after AOPs

3.1 Evaluation of parameters for multi-AOPs

The oxidation efficiency of AOP processes under varying oxidant concentrations, TiO_2 catalyst dosages, and UV outputs was evaluated by measuring changes in TOC concentration to identify optimal operating conditions for wastewater effluent reuse. The experimental conditions included an initial water temperature of approximately 20°C, which increased to ~37°C after 3 h, a UV irradiation time of 180 min, and a UV output ranging from 26 to 52 W. Additionally, In the dual-AOP experiments, the UV/PS process was conducted by varying the PS dosage (1.85-7.40 mM) to determine the optimal dosage. For the UV/TiO_2

process, experiments were performed within a TiO_2 dosage range of 1-3 g/L, and subsequent experiments used the optimal TiO_2 dosage.

3.2 Kinetic analysis of the multi-AOPs

The reaction rate constants (k and k') for the AOP experiments, including UV/TiO_2, UV/PS, and UV/TiO_2/PS processes, were calculated using pseudo-first-order and pseudo-second-order rate equations. These calculations were based on the temporal changes in TOC concentrations (C_t) during the oxidation reactions. For the UV/TiO_2 experiments, the rate constant (k) was calculated using the pseudo-first-order rate Eq. (15):

$$k = -\frac{\Delta(ln[C_t])}{\Delta t} \tag{15}$$

where C_t is the TOC concentration (mg/L) at time t (min), and $\ln|C_t|$ is the natural logarithm of the TOC concentration.

The rate constant (k) was derived from the slope of the linear regression between $\ln|C_t|$ and reaction time (t).

For UV/PS and UV/TiO_2/PS experiments, where higher-order reactions may have occurred due to the interaction of PS and radicals, the pseudo-second-order rate constant (k') was determined using the following Eq. (16):

$$k' = \frac{\Delta(\frac{1}{[C_{t'}]})}{\Delta t'} \tag{16}$$

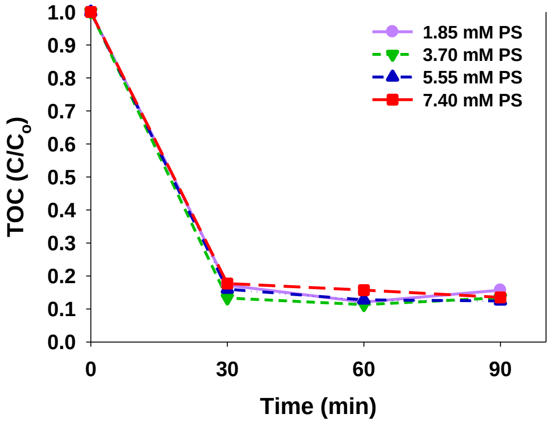
where $1/C_{t'}$ is the inverse of TOC concentration at time t' and $\Delta t'$ is the change in reaction time.

The rate constant (k') was obtained by analyzing the slope of the linear relationship between $[C_{t'}]^{-1}$ and reaction time (t'). Time-concentration data obtained from each AOP run were imported into *SigmaPlot 10* and subjected to linear regression. The slope of each regression line was adopted as the pseudo–first-order rate constant**,** while the coefficient of determination (R^2) served as an indicator of model fit. By comparing these parameters across experiments, the kinetic behavior of the various multi-AOP configurations was quantitatively evaluated.

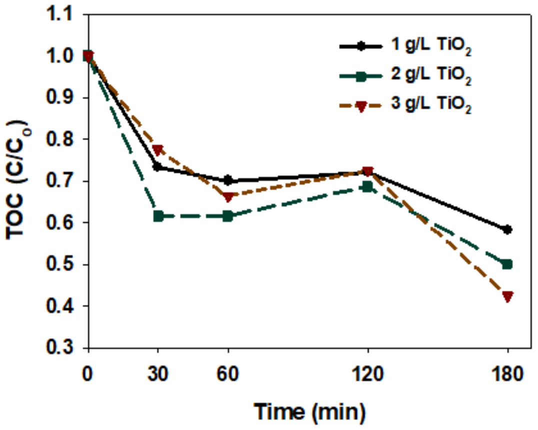
4. Results and discussion

4.1 Performance assessment of combined AOPs: UV/peroxydisulfate and UV/TiO₂

Fig. 2 compares the TOC removal performance of the UV/TiO_2 process and the combined UV/PS system. Fig. 2(a) shows the UV/PS process with varying PS concentrations, and Fig. 2(b) presents the UV/TiO_2 process with varying TiO_2 dosage. The UV/PS system was established with reference to previous studies (Gao *et al*. 2019, Ghauch *et al*. 2017, Liu *et al*. 2016). The UV/TiO_2 system was established with reference to previous studies (Munien *et al*. 2023, Akter *et al*. 2022, Zweigle *et al*. 2022, Park *et al*. 2022). In this study, the TiO_2 dosage was set in the range of 1–3 g/L. Zweigle *et al*. (2022) reported that excessively high concentrations of TiO_2 could decrease photocatalytic efficiency, emphasizing the importance of optimizing TiO_2 dosage to ensure both effective pollutant removal and economic feasibility. In this study, the PS dosage was varied in the range of 1.85-7.40 mM (500-2,000 mg/L).



(a) The effect of PS dosages in UV/PS



(b) The effect of TiO_2 dosages in UV/TiO_2

Fig. 2 Comparison of ultraviolet (UV)/ persulfate (PS) and UV/TiO_2 (UV: 39 W, PS: 1.85-7.40 mM, TiO_2: 1-3 g/L)

In the UV/PS process, all PS concentrations demonstrated rapid initial TOC removal, with C/C_0 ratios decreasing to approximately 0.2 within 30 minutes**.** The optimal PS concentration was observed at 3.70 Mm (1,000 mg/L), achieving the highest TOC removal efficiency with a final C/C_0 ratio of approximately 0.1**.** The UV/PS AOP clearly displays faster kinetics for TOC reduction than UV/TiO_2. This reflects fundamental differences between a homogeneous radical reaction in bulk solution versus a heterogeneous surface-catalyzed process. In published comparisons, homogeneous UV/oxidant processes (like UV/KPS or UV/H_2O_2) often outperform photocatalysis in initial rates (Saien, 2010). The reaction approximately follows pseudo-first-order kinetics with respect to TOC. Although more persulfate yields more radicals and better TOC removal, excessively high PS concentrations can lead to radical self-scavenging – sulfate radicals recombine with each other or even react with surplus persulfate instead of attacking organic molecules.

This enhanced performance is attributed to the generation of sulfate radicals and hydroxyl radicals through UV photolysis of PS, which rapidly oxidize and decompose organic compounds in the solution. However, at higher PS concentrations (5.55 and 7.40 mM), the TOC removal efficiency slightly decreased compared to the optimal concentration, likely due to radical scavenging reactions where excess PS molecules compete for radicals, as described in Eq. (17).

The UV/TiO_2 system in Fig. 2(b) demonstrated a fundamentally different degradation pattern compared to the UV/PS system. The TOC removal efficiency showed a gradual decline throughout the entire treatment period, with initial TOC ratios starting at 1.0 and decreasing to approximately 0.4-0.6 by 180 minutes. Notably, the system exhibited minimal sensitivity to TiO_2

$$SO_4^{\bullet -} + SO_4^{\bullet -} \rightarrow S_2O_8^{2-} \ (k = 4 \times 10^8\ M^{-1}s^{-1}) \qquad (17)$$

Early in the reaction, abundant active sites and organics yield a relatively faster rate; as intermediates form and occupy sites, or as the easier-to-oxidize fraction is depleted, the rate decreases. The net result is a slow, asymptotic approach to an incomplete TOC removal over hours rather than the quick near-total removal seen with UV/PS.

The UV/TiO_2 AOP also demonstrated a non-monotonic trend, which may be attributed to either the formation of intermediate oxidation products temporarily registering as organic carbon. Overall, UV/TiO_2 leads to substantially slower TOC decay than UV/PS AOP highlighting that photocatalysis mineralizes the organics only partially in the given timeframe, with no abrupt elimination phase as seen in UV/PS. The differences in profiles emphasize that the UV/PS system exhibits rapid and extensive TOC removal (≈80–90%) within minutes, in contrast to the gradual, moderate removal (≈50%) achieved by UV/TiO_2 over hours.

This suggests that the heterogeneous photocatalytic mechanism in UV/TiO_2 generates different reactive species that follow distinct reaction pathways compared to the sulfate radical-mediated degradation in UV/PS systems. This difference shows the superior oxidative capacity of sulfate radicals and their ability to achieve complete mineralization of organic contaminants under optimized conditions.

4.2 Multi-AOPs test-UV/TiO_2/PS

4.2.1 Effect of TiO_2 dosage

To evaluate the TOC removal efficiency of the UV/TiO_2/PS process at varying TiO_2 dosages, experiments were conducted using TiO_2 concentrations ranging from 1.0 to 3.0 g/L. The UV/TiO_2/PS system was established with reference to preliminary tests and previous studies, in which the optimal dosage conditions were identified as TiO_2 0.05-0.25 g/L and PS 0.3-2.5 g/L (Bakhshaei *et al.* 2024, Zhang and Chu 2022, Ding and Hu 2020, Monteagudo *et al.* 2020). In this study, radical generation was found to saturate above 1.0 g/L TiO_2, and given the use of a direct-contact UV setup, higher PS concentrations were also evaluated to reflect differing UV exposure conditions. Previous studies, such as Bakhshaei *et al.* (2024), reported using up to 9.3 mM PS in similar systems. Fig. 3 illustrates the normalized TOC concentration (C/C_0) over 180 min for 2.0 L of wastewater effluent, with a fixed PS dosage of 1 g/L and varying TiO_2 dosages. The inset in Fig. 3 shows the reciprocal of TOC concentration (1/C) during the initial 60 min, assuming a second-order reaction, to calculate the reaction rate constant.

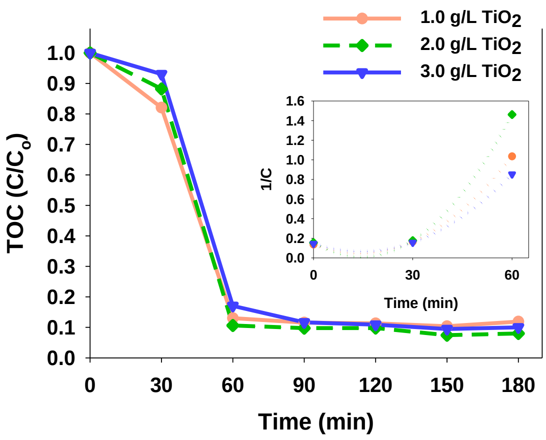


Fig. 3 The effect of TiO_2 dosages (UV: 39 W, persulfate (PS): 3.7 mM (= 1 g/L), TiO_2: 1-3 g/L)

The UV/TiO_2/PS multi-AOP experiments revealed that TOC removal approached maximum efficiency at approximately 60 min, contrasting with the UV/PS process. The addition of TiO_2 provided greater stability in TOC removal, reducing the fluctuations observed in the UV/PS process. Although the UV/PS system achieved TOC removal more rapidly, it exhibited higher variability over extended UV irradiation. This suggests that TiO_2, while acting as a photocatalyst, may partially inhibit the oxidation reaction of PS when used simultaneously.

The optimal TiO_2 dosage was determined to be 2.0 g/L, resulting in a maximum TOC removal efficiency of 92.6% after 150 min. Although the addition of TiO_2 improved TOC removal compared to the UV/PS process alone, the oxidation effect of PS contributed more significantly to TOC removal than the photocatalytic effect of TiO_2. Therefore, while TiO_2 served as a secondary enhancement, the UV/PS process alone was highly effective for TOC removal.

4.2.2 Effect of UV output

To investigate the TOC removal characteristics of the UV/TiO_2/PS process under varying UV outputs, experiments were conducted by adjusting the number of UV lamps to achieve outputs ranging from 26 W to 52 W. Fig. 4 shows the changes in TOC concentration over 180 min for 2.0 L of effluent under different UV outputs. The reaction kinetics during the initial 60 min were assumed to follow first-order behavior, and the y-axis was expressed as 1/C to calculate the rate constants. Contrary to expectations, UV outputs of 26 W and 39 W showed similar TOC removal performances. However, at the highest UV output (52 W), TOC removal efficiency decreased, which was attributed to excessive UV irradiation under the experimental conditions of 1 g/L TiO_2 and 3.7 mM PS, which appeared to cause

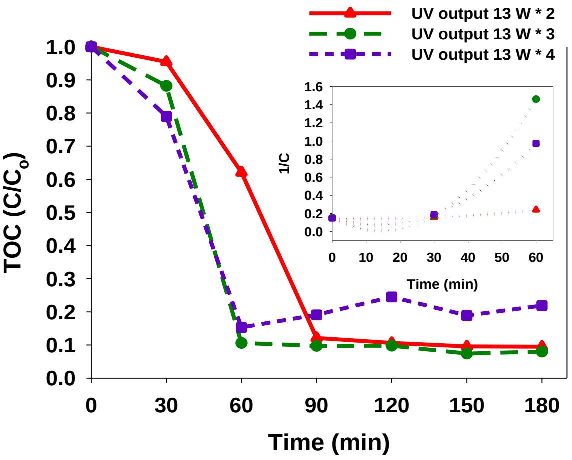


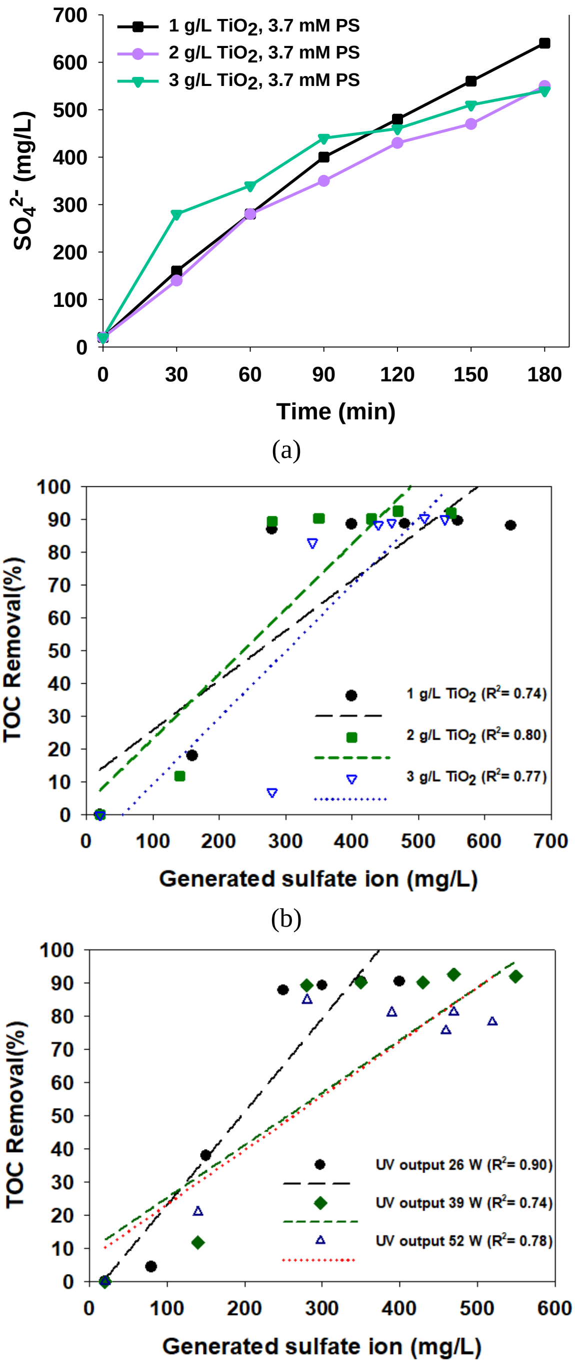
Fig. 4 The effect of ultraviolet (UV) output level (persulfate (PS): 3.7 mM, TiO_2: 2 g/L, UV: 26-52 W)

TOC increase instead of degradation. Under these conditions, the optimal UV output was determined to be 39 W (using three 13 W UV-C lamps), resulting in a maximum TOC removal efficiency of 92.6%. This finding is consistent with the results from Section 3.4.1, where the same UV output was identified as optimal in previous experiments.

4.2.3 Sulfate ion analysis and hazard assessment

To evaluate the hazard of treated effluent using the UV/TiO_2/PS process, the concentration of sulfate ions generated by PDS injection was analyzed. In the multi-AOP experiment utilizing UV/TiO_2/PS, sulfate ions were released from the applied PS, reaching a maximum concentration of 650 mg/L. While domestic discharge limits and river water quality standards do not explicitly define sulfate ion concentration thresholds, drinking water quality standards restrict sulfate concentrations to ≤ 200 mg/L. The primary objective of this multi-AOPs experiment was advanced treatment for wastewater reuse. Although this study does not directly address drinking water standards, the proposed AOP technology has broader applications for both wastewater treatment and water supply sources. Therefore, we systematically analyzed sulfate ion concentrations under varying experimental conditions and explored their correlation with TiO_2 dosage and UV irradiation intensity. PS, when activated by UV irradiation, transforms into sulfate radicals ($SO_4^{\bullet-}$). These generated radicals oxidize and decompose aqueous organic compounds, ultimately reducing to stable sulfate ions (SO_4^{2-}).

Fig. 5(a) illustrates the sulfate ion concentration over time under TiO_2 dosage conditions ranging from 1 to 3 g/L. Fig. 5(b) depicts the correlation between residual sulfate ion concentration and TOC removal efficiency following multi-AOP treatment at varying TiO_2 dosages. Fig. 5(c) shows sulfate ion concentrations over time under UV lamp output conditions ranging from 26 to 52 W. Fig. 5(d) presents the relationship between residual sulfate ion concentration and TOC removal efficiency at different UV irradiation outputs.



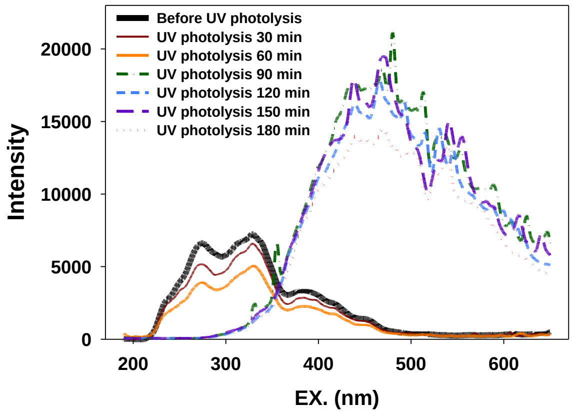
(a)

(b)

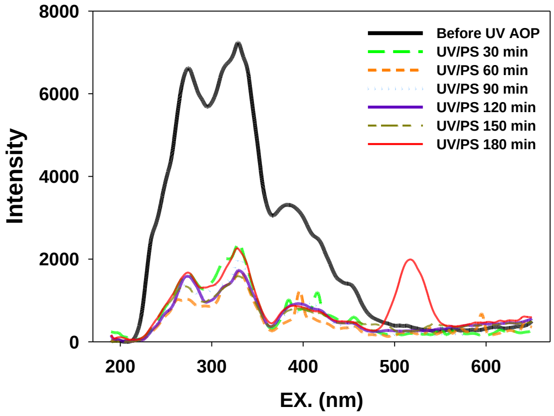
(c)

Fig. 5 Comparison of sulfate ion generation and TOC removal in ultraviolet (UV)/TiO_2/PDS process at UV output and TiO_2 dosages

In Fig. 5(a), sulfate ion concentrations increased linearly with reaction time across all TiO_2 dosages (1, 2, and 3 g/L). Notably, higher TiO_2 dosages resulted in greater sulfate ion production, with the 3 g/L TiO_2 condition achieving the highest concentration (~700 mg/L) at 180 min. This trend highlights the catalytic role of TiO_2 in enhancing the activation of PS and facilitating the production of SO_4^{2-} radicals. However, the diminishing difference between the 2 g/L and 3 g/L TiO_2 dosages suggests that excessive TiO_2



(a) UV photolysis



(b) UV/PS

Fig. 6 Comparison of UV photolysis and UV/PS AOP through fluorescence analysis

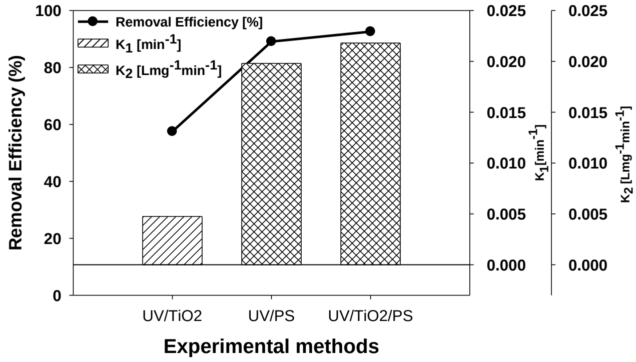


Fig. 7 Comparison of TOC removal efficiency and rate constants for treated wastewater effluent by various AOPs

may not proportionally increase PS activation, possibly due to aggregation or light scattering effects. Fig. 5(b) illustrates the correlation between sulfate ion concentration and TOC removal for different TiO_2 dosages. A positive linear correlation was observed, with the highest R^2 value (0.80) for the 2 g/L TiO_2 condition, indicating optimal performance. The lower correlation coefficients for 1 g/L (R^2= 0.74) and 3 g/L (R^2= 0.77) suggest suboptimal radical generation or utilization under these conditions. These results confirm that sulfate ion production is critical for TOC removal but must be balanced with efficient radical utilization.

Fig. 5(c) compares sulfate ion generation at different UV outputs (26 W, 39 W, and 52 W) with a fixed TiO_2 dosage (2 g/L) and PS concentration (1 g/L). Higher UV outputs resulted in faster and greater sulfate ion generation, with 52 W producing the highest concentration (~500 mg/L at 180 min). However, the rapid initial generation at higher outputs may have led to excessive radical recombination, reducing the availability of reactive species for TOC removal. This phenomenon underscores the importance of optimizing UV intensity to maximize radical efficiency while minimizing recombination. Fig. 5(d) shows the relationship between sulfate ion concentration and TOC removal under varying UV outputs. The strongest correlation (R^2= 0.90) was observed at 26 W, indicating efficient utilization of sulfate ions for TOC degradation. In contrast, higher UV outputs (39 W and 52 W) showed reduced correlation coefficients (R^2= 0.74 and R^2= 0.78, respectively), suggesting decreased efficiency due to radical recombination or secondary reactions. This finding reinforces the need to balance UV intensity and radical production to achieve optimal TOC removal. The optimal TiO_2 dosage was 2 g/L, as it provided the best balance between sulfate ion production and TOC removal efficiency without excessive aggregation or light scattering. A UV output of 39 W was found to be the most effective, balancing sulfate ion generation and radical utilization. Excessive UV intensity (52 W) led to radical recombination, reducing TOC removal efficiency. Sulfate ion generation was directly correlated with TOC removal, but only under conditions where radical production and utilization were balanced. Overproduction of radicals resulted in inefficiencies due to recombination.

4.3 Intermediate formation through UV photolysis and UV/PS by fluorescence analysis

As shown in Fig. 6(a), the control sample without UV photolysis exhibited a fluorescence intensity of approximately 6,000 in the 250-280 nm region (typically associated with protein-like substances), while the sample obtained after 90 minutes of UV exposure under a 52 W lamp demonstrated a newly formed, more intense peak at approximately 450 nm, reaching an intensity of 21,000. This observation suggests that high-molecular-weight organic matter underwent photodegradation, yielding intermediate byproducts with enhanced fluorescence characteristics.

In the UV alone treatment, it was observed that the primary spectral peak range differed significantly between the control group and the experimental conditions over time. Specifically, up to 60 minutes, the main peaks appeared in the 300-350 nm range, whereas from 90 to 180 minutes, the peaks shifted to the 400-450 nm range. This shift indicates that after 90 minutes, the initial organic compounds underwent transformation into intermediate products due to UV irradiation. Notably, as shown in Fig. 6(a), the intensity of the main peak after 90 minutes reached approximately 21,000, which is more than three times the initial peak intensity, highlighting an intriguing phenomenon.

Table 1 Comparison of tested advanced oxidation processes (AOPs) for the TOC removal using wastewater effluent

AOPs	TOC_0 (mg/L)	Reaction condition	Time (min)	TOC_t (mg/L) (Removal, %)
O_3/H_2O_2	9.5	O_3 flow rate: 10 L/min, O_3 conc.: 7.2 g/m^3	60	2.8 (70.5)
Fenton	7.4	Fe^{2+}: 15 mM, H_2O_2: 64 mM, N: 100 rpm	60	3.0 (59.5)
Electro-Fenton	7.8	Fe^{2+}: 15mM, H_2O_2: 64 mM, I: 1.5 A Anode & Cathode SUS	60	1.4 (82.1)
US*/Fenton	7.4	Fe^{2+}: 22 mM, H_2O_2: 174 mM, f: 80 kHz	60	2.1 (71.6)
US/H_2O_2	8.2	H_2O_2: 174 mM, f: 80 kHz	60	1.0 (87.8)
US/PS	7.7	PS: 43.5 mM, f: 80 kHz	60	1.3 (81.9)
US/PS/H_2O_2	10.4	PS: 0.52 mM, H_2O_2: 174 mM, f: 80 kHz	60	0.9 (91.1)
UV/PS	6.4	PS: 3.7 mM, P_{UV}: 26 W	60	0.7 (89.1)
UV/TiO_2/PS	6.5	TiO_2: 2 g/L, PS: 3.7 mM P_{UV}: 39 W	60	0.63 (90.3)

*US: Ultrasound

All fluorescence measurements were conducted using a Lumilux 5100 (Scinco) fluorescence spectrophotometer. These results suggest that UV photolysis alone demonstrates limited efficacy in completely mineralizing recalcitrant organic compounds in wastewater effluent. This phenomenon underscores the potential necessity of radical-based Advanced Oxidation Processes (AOPs) for achieving more complete mineralization of persistent organic matter.

Fig. 6(b) showed the synergistic effect of UV irradiation (52 W) and persulfate (3.7 mM) on the degradation of residual organic matter in wastewater effluent, as evidenced by fluorescence spectral analysis. In comparison to UV photolysis alone, the UV/PS (UV with persulfate) process demonstrated a marked reduction in fluorescence intensity across the ultraviolet spectrum (200-400 nm), indicating effective degradation of humic-like and protein-like substances by sulfate radicals generated from persulfate. The visible region (400-600 nm) remained relatively stable throughout most of the treatment duration, although samples irradiated for 180 minutes exhibited a modest intensity increase, potentially attributed to the formation of resistant byproducts or partial restructuring of chromophoric moieties under extended radical oxidation conditions. These results demonstrate that UV/PS treatment offers superior performance compared to UV photolysis alone in reducing fluorescence signatures associated with recalcitrant organic compounds, thereby enhancing mineralization efficiency. Thus, the implementation of UV/PS as an advanced oxidation process shows promise in meeting stringent water reuse criteria through effective reduction of persistent organic compounds in treated effluent.

4.4 Kinetics of the UV-based AOPs

Fig. 7 evaluates the TOC removal efficiency and reaction rate constants (k_1 and k_2) across three advanced oxidation processes: UV/TiO_2, UV/PS, and UV/TiO_2/PS hybrid system. The UV/TiO_2 process demonstrated good TOC removal efficiency, achieving approximately 60%. This performance can be attributed to the photocatalytic generation of hydroxyl radicals (•OH) through TiO_2 activation under UV irradiation. UV/TiO_2 exhibits superior oxidative efficiency compared to UV-only treatment, primarily because UV-only possesses limited oxidative capacity

While the removal efficiency of UV/TiO_2 alone was substantially lower than that of processes integrating persulfate (PS), such as UV/PS and especially UV/TiO_2/PS, the inclusion of TiO_2 in combined systems suggests its synergistic role in enhancing overall oxidation, despite its limitations when used without a strong co-oxidant like PS

For UV/PS and UV/TiO_2/PS, the results aligned with expectations, showing high removal efficiencies (>90%) and substantial reaction rate constants. These outcomes were attributed to the efficient activation of PS by UV light, which produces highly reactive sulfate radicals ($SO_4^{\bullet-}$) capable of degrading organic compounds. The addition of TiO_2 in the UV/TiO_2/PS system further enhanced catalytic activity, supporting more efficient TOC removal. In the

UV/PS and UV/TiO_2/PS experiments, TOC removal efficiencies were 88.7% at 60 min and 92.6% at 150 min, respectively. The corresponding kinetic constants (k) for the 0-60 min interval were 1.99×10^{-2} L/mg·min and 2.18×10^{-2} L/mg·min, respectively.

These findings highlight the critical role of incorporating additional oxidants, such as PS, to maximize the oxidative performance of UV-based processes. While UV/TiO_2 offered moderate removal efficiency, reliance on UV-only for TOC degradation was insufficient, necessitating hybrid approaches for effective wastewater effluent treatment.

Table 1 presents a comprehensive comparison of TOC removal efficiencies for various AOPs under specific reaction conditions. The UV/PS and UV/TiO_2/PS processes achieved the highest TOC removal efficiencies, reaching up to 89.1% and 92.6%, respectively. These results underline the advantages of combining UV irradiation with PS, which generates sulfate radicals ($SO_4^{\bullet-}$) with strong oxidation potential. Although both UV/PS and UV/TiO_2/PS methods performed remarkably well, UV/TiO_2/PS exhibited slightly higher removal efficiency, emphasizing the catalytic role of TiO_2 in enhancing the process.

US-assisted systems, such as US/Fenton, US/H_2O_2, and US/PS, exhibited moderate to high TOC removal efficiencies. The US/H_2O_2 process achieved a high TOC removal efficiency of 87.8%, indicating effective hydroxyl radical (•OH) generation through cavitation and the decomposition of hydrogen peroxide. The US/PS process followed with an efficiency of 81.9%, demonstrating the activation of PS through US-induced cavitation. Both the O_3/H_2O_2 and Fenton processes exhibited moderate TOC removal efficiencies of 70.5% and 59.5%, respectively. Although ozone is a strong oxidant, its efficiency depends on the decomposition of ozone into radicals, which can be relatively pH-dependent.

Processes such as Fenton and US/Fenton relied heavily on hydroxyl radicals for oxidation. These radicals were effective but less selective, which may explain the moderate TOC removal efficiencies observed. In contrast, UV/PS and UV/TiO_2/PS systems leveraged sulfate radicals, which are more effective than hydroxyl radicals, resulting in superior TOC removal efficiencies. The dual contribution of sulfate and hydroxyl radicals in UV/TiO_2/PS further enhanced its effectiveness.

The data in Table 1 emphasized the superior performance of the UV/PS and UV/TiO_2/PS processes, making them the most promising AOPs for wastewater treatment. While US-based and combined systems also showed high efficiencies, their practicality depended on energy consumption and operational complexity. Although the O_3/H_2O_2 and Fenton systems were less efficient, they remained valuable for specific applications where simplicity and cost were prioritized.

5. Conclusions

This study investigated and compared the performance of various AOPs for TOC removal from wastewater effluent, focusing on UV-based AOPs.

The results demonstrated that UV-based processes, particularly UV/PS and UV/TiO_2/PS, achieved the highest TOC removal efficiencies of 89.1% and 92.6%, respectively. The UV/TiO_2/PS system proved to be the most effective under optimized conditions, with 1 g/L PS, 2 g/L TiO_2, and a UV output of 39 W, making it a promising approach for treating recalcitrant organic pollutants in wastewater. The combination of PS activation and TiO_2 photocatalysis under UV light significantly enhanced oxidation efficiency, demonstrating the potential of these systems for wastewater reuse applications.

A critical insight revealed that while the UV/PS system achieved rapid initial TOC removal, the incorporation of TiO_2 into the UV/TiO_2/PS process provided enhanced stability in TOC removal kinetics, mitigating the TOC rebound effect observed in UV/PS alone at suboptimal concentrations. This suggests TiO_2 acts as a crucial secondary enhancement, ensuring more consistent and complete mineralization of organic compounds.

Fluorescence spectral analysis revealed that the UV/PS process demonstrated superior performance over UV photolysis alone in degrading recalcitrant organic compounds in wastewater effluent, particularly for protein-like and humic-like substances. This finding suggests that UV/PS treatment could be an effective advanced oxidation process for meeting water reuse requirements, despite the potential formation of resistant byproducts during extended treatment periods.

Among non-UV-based AOPs, the US/H_2O_2 and US/PS processes achieved relatively high TOC removal efficiencies of 87.8% and 81.9%, respectively, due to ultrasonic cavitation, which enhances the generation of reactive radicals. However, the high energy consumption of US-based systems may limit their applicability in large-scale operations. Similarly, the O_3/H_2O_2 process, widely used in water treatment, attained a moderate TOC removal efficiency of 70.5%.

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