

Enhanced Degradation of Metronidazole (MNZ) in Aqueous Solutions Using Combined Ultrasonic, Hydrogen Peroxide, and Peroxymonosulfate Processes

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Abstract

Background: Metronidazole (MNZ) is a widely used pharmaceutical compound that has recently emerged as a significant environmental contaminant. Due to its potential mutagenic properties, MNZ poses risks of DNA damage and carcinogenesis.

Methods: This experimental study was conducted at the laboratory scale to investigate the efficiency of MNZ removal in aqueous solutions using a novel combination of ultrasonic waves (US), hydrogen peroxide (H₂O₂), and peroxymonosulfate (PMS).

Results: In the US/PMS/H₂O₂ process, the removal efficiency ranged from 4.3% to 99.5% under the best conditions (PMS dose=6 mmol/L, H₂O₂ dose=20 mmol/L, time=30 min, US power=120 W, MNZ concentration=2 mg/L). A comparative analysis of the maximum removal efficiency between the two processes showed that incorporating US significantly increased MNZ removal, resulting in a 3-fold increase in efficiency under different conditions. Furthermore, in the PMS/H₂O₂ process, the H₂O₂ dose exerted the most pronounced impact, followed sequentially by reaction time, PMS dosage, MNZ concentration, and pH. In the US/PMS/H₂O₂ process, reaction time was identified as the most influential factor, followed by MNZ concentration, PMS dosage, US power, and H₂O₂ dosage. The results of the Taguchi design model further underscored the substantial contribution of ultrasonic irradiation to MNZ removal.

Conclusion: Overall, the findings demonstrate that US treatment is highly effective at significantly reducing MNZ concentrations in water and wastewater, highlighting the critical importance of parameter optimization in developing efficient US-based treatment technologies.

Keywords: Ultrasonics, Peroxymonosulfate, Hydrogen peroxide, Wastewater, Water

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Introduction

The growth of the global population, coupled with rapid urbanization and industrialization, has intensified water pollution challenges, leading to the release of an increasing array of emerging micropollutants that pose significant threats to the availability of clean, safe water resources (1,2). Owing to their chemical stability and lipophilic nature, these compounds can persist within biological systems for extended periods (3).

The overuse of antibiotics has accelerated the emergence of antibiotic-resistant pathogenic microorganisms, necessitating higher doses to achieve effective bacterial control (4). Furthermore, antibiotics are not completely metabolized within the body, with approximately 30–90% of the administered compounds remaining biologically

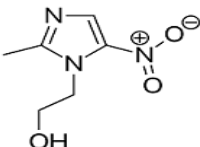
active upon excretion (5). Conventional wastewater treatment processes are insufficient for removing emerging pollutants, as they are predominantly designed to target biodegradable, soluble organic contaminants (6).

MNZ is a widely used pharmaceutical compound that has become a prominent environmental concern in recent years (1). MNZ (C₆H₉N₃O₃) belongs to the nitroimidazole class of antibiotics, known for its potent antimicrobial properties (7). It is widely used to treat and prevent both systemic and local infections (8). A summary of the physicochemical characteristics of MNZ is provided in Table 1(9)

MNZ is potentially mutagenic, capable of inducing DNA damage and promoting carcinogenesis (4). It readily accumulates in aquatic environments due to its low soil



Table 1. Physicochemical characteristics of MNZ (9)

Parameters	Characteristics
Molecular structure	
Molecular formula	$C_6H_9N_3O_3$
Molecular weight	171.15
pK_a	2.55
Water solubility (g.L ⁻¹)	9.5
Melting point (°C)	159-163
Biological half-life in the body	(10)

absorption, non-biodegradability, high water solubility, mobility, and high toxicity (10). MNZ is the only nitroimidazole designated as an essential medicine by the World Health Organization (WHO) (11). Consequently, both the WHO and the US National Institutes of Health classify MNZ as a highly toxic and potentially carcinogenic substance (12).

Conventional wastewater treatment methods are often insufficient for removing MNZ, prompting the development of alternative technologies (13). However, these methods are often hindered by challenges, including high operational costs, inefficient pollutant removal, sludge generation, and concerns about disposal (5,14).

Recent studies have demonstrated that advanced oxidation processes (AOPs) are highly effective in removing pollutants from wastewater (15). AOPs are recognized for their capacity to degrade hazardous organic pollutants by generating potent oxidants, such as hydroxyl radicals, which non-selectively react with stable organic contaminants in aqueous solutions, ultimately achieving complete mineralization (16,17). The application of US in AOPs has garnered significant attention due to its ability to enhance pollutant removal efficiency substantially. The US is particularly valued for its operational simplicity, energy efficiency, and the absence of secondary pollutants and sludge production (18). In US-assisted processes, hydroxyl radicals are generated through the pyrolysis of water molecules, while ultrasonic waves simultaneously enhance reaction rates by improving mass transfer (19).

In a 2021 study by Sheikhmohammadi et al (18), it was demonstrated that US combined with TiO_2/Fe_3O_4 achieved higher MNZ removal efficiency than pure TiO_2 or Fe_3O_4 nanoparticles, with 97.5% MNZ removal under optimal conditions.

Lee et al (20) investigated the activation of peroxydisulfate (PDS) and PMS by US and found that US alone was effective for degrading ibuprofen (IBP). The removal efficiency of IBP improved with the addition of PDS, PMS, and H_2O_2 .

Another study by Seid-Mohammadi et al (21) reported the removal of the antibiotic cephalexin from aqueous solutions using a hybrid process involving US, H_2O_2 , and nickel oxide nanoparticles. The removal efficiencies for COD, cephalexin (CEX), and TOC were 86.9%, 46.7%, and 55.5%, respectively.

This study aimed to investigate the efficiency of MNZ removal using an advanced ultrasound (US) oxidation process combined with peroxymonosulfate (PMS) and hydrogen peroxide (H_2O_2). The integration of the US with PMS and H_2O_2 is expected to enhance the degradation rate of pollutants through key mechanisms such as the formation of localized hot spots and sonoluminescence. Although several studies have examined MNZ removal using various advanced oxidation processes, there is a notable lack of research specifically addressing the removal efficiency of MNZ through advanced US oxidation processes combined with PMS and H_2O_2 .

Materials and Methods

This experimental study was conducted at the laboratory scale to investigate the removal of MNZ from aqueous environments using US in combination with PMS and hydrogen peroxide H_2O_2 . Optimal operating conditions were determined using Design of Experiments (DOE) software.

To prepare the stock solution, 1 g of pure MNZ powder (purity 99.9%) was dissolved in 1000 mL of distilled water. The mixture was stirred using a magnetic stirrer for 10 minutes to ensure complete dissolution. The prepared solution was subsequently stored at refrigeration temperature until use.

Chemicals

The chemicals used in this study included metronidazole (MNZ, purity 99.9%) and acetonitrile (HPLC grade), both purchased from Merck, Germany. Additional chemicals, such as peroxymonosulfate, hydrogen peroxide (35%), sodium thiosulfate, hydrochloric acid, sodium hydroxide, sodium carbonate, calcium chloride, sodium bromide, potassium chloride, magnesium chloride, monosodium phosphate, manganese chloride, sodium sulfate, sodium chloride, sodium nitrate, tributylamine, ethanol, potassium iodide, humic acid, parabenzoquinone, and sodium bicarbonate, were also obtained from Merck, Germany.

Equipment

An ultrasonic homogenizer (Sonopuls Bandelin HD3200, Germany) equipped with a 25 kHz probe and a power range of 20–140 W was used to generate ultrasound. The probe was positioned at the center of the reactor at laboratory temperature.

The primary and secondary concentrations of MNZ were determined using a high-performance liquid

chromatography (HPLC–UV) system (Jasco UV-2.75 Plus-FP-2020, Japan) equipped with an autosampler, a UV–Vis detector, and a C18 column. Analyses were performed at 320 nm with a run time of 12 minutes. The mobile phase consisted of acetonitrile and distilled water at an 80:20 (v/v) ratio, with a sample injection volume of 20 µL. The mobile phase flow rate was 0.75 mL/min. A C18 column with a diameter of 5 cm and a length of 20 cm was used.

The limit of detection (LOD) and limit of quantification (LOQ) were calculated and reported based on signal-to-noise ratios of 3 and 10, respectively, and were found to be 0.012 and 0.032 mg/L, respectively.

Experiment design

To determine the optimal conditions for MNZ removal, a DOE–Taguchi test design was employed. Table 2 presents the factors and their levels used to eliminate MNZ. The experiments were conducted in two stages, each with five 4-level factors, yielding a total of 20 samples per stage. Each experiment was performed in triplicate, and the results were analyzed using the Taguchi model. The mean

Table 2. Factors and levels of MNZ removal in PMS/H₂O₂ and PMS/H₂O₂/US processes

Factor	Level 1	Level 2	Level 3	Level 4
dose PMS (mmol/L)	1	2	4	6
dose H ₂ O ₂ (mmol/L)	10	15	20	30
Time (min)	15	30	60	75
pH	5	7	9	11
MNZ concentration (mg/L)	2	3	5	10
US intensity (W)	30	60	90	120

Table 3. MNZ removal values using the PMS/H₂O₂ process

Run	PMS dose (mmol/L)	H ₂ O ₂ dose (mmol/L)	Time (min)	pH	MNZ (mg/L)	Removal percentage of MNZ (%)
1	2	15	15	11	5	4.5±0.4
2	6	15	60	5	10	5.31±0.3
3	1	20	60	9	5	4.96±0.4
4	1	30	75	11	10	18.10±0.8
5	1	15	30	7	3	12.49±0.7
6	4	20	15	7	10	3.74±1.0
7	1	10	15	5	2	1.44±0.5
8	6	20	30	11	2	4.64±1.1
9	2	20	75	5	3	3.42±0.7
10	6	10	75	7	5	6.09±1.5
11	4	15	75	9	2	4.95±0.2
12	2	30	60	7	2	1.98±1.2
13	6	30	15	9	3	13.86±0.9
14	2	10	30	9	10	0.95±0.9
15	4	30	30	5	5	33.04±0.6
16	4	10	60	11	3	3.44±0.7

and standard deviation of the results were reported.

Since no significant effect of pH on removal efficiency was observed in the first step and no significant difference in removal efficiency was observed across the tested pH levels, pH 7 was selected as the optimal condition.

Consequently, all subsequent experiments involving the PMS/H₂O₂/US process were conducted at pH 7, with US intensity as a test parameter.

MNZ removal efficiency was calculated using the following equation:

$$R = (C_0 - C_t) / C_0 \times 100$$

In this equation, (C_0) and (C_t) represent the initial and final concentrations of MNZ in mg/L, respectively. The effects of scavengers and inorganic anions were also investigated. For this experiment, the conditions were as follows: PMS dose = 2 mmol/L, H₂O₂ dose = 10 mmol/L, reaction time = 30 min, US power = 60 W, and pH = 7.

In a 500 ml beaker containing 250 ml of stock solution, H₂O₂, PMS, a scavenger, and the desired anion were added. After adjusting the pH, the sample was treated with US for 30 minutes. The treated sample was analyzed by HPLC to determine the secondary concentration and the removal percentage.

Results

As shown in Table 3, the MNZ removal efficiency using the PMS/H₂O₂ process ranged from 1% to 33%, with the highest removal observed in experiment No. 15, achieving approximately 33%.

Table 4 presents the effects of key variables and removal efficiencies in the PMS/H₂O₂/US process. The highest

Table 4. MNZ removal efficiency by PMS/H₂O₂/US process

Run	PMS dose (mmol/L)	H ₂ O ₂ dose (mmol/L)	Time (min)	US (W)	MNZ (mg/L)	Removal percentage of MNZ (%)
1	4	15	75	90	2	99.52±0.01
2	4	10	60	120	3	97.51±3.5
3	2	30	60	60	2	37.53±0.5
4	6	10	75	60	5	98.59±2.1
5	1	30	75	120	10	45.31±0.2
6	2	20	75	30	3	78.19±0.5
7	6	15	60	30	10	12.68±0.4
8	6	20	30	120	2	99.56±0.01
9	2	10	30	90	10	14.78±0.6
10	2	15	15	120	5	4.33±2.2
11	1	20	60	90	5	26.49±0.3
12	4	20	15	60	10	4.68±4.8
13	6	30	15	90	3	17.86±0.9
14	4	30	30	30	5	40.64±0.4
15	1	10	15	30	2	10.43±0.1
16	1	15	30	60	3	54.83±8.9

MNZ removal was recorded in experiment No. 8, with an efficiency of 99.5%. Notably, experiment No. 1 also achieved a similarly high removal rate of 99.5%, with only a negligible difference between the two tests. If we increase the ultrasonic intensity by 30%, we can halve the reaction time while maintaining the same efficiency.

This study investigated the effect of pH on the removal efficiency of MNZ using the PMS/H₂O₂ process, which is shown in Figure 1(A). The effect of the initial concentration of MNZ on the removal efficiency in both the PMS/H₂O₂ and PMS/H₂O₂/US processes was investigated, as shown in Figure 1(B). To assess the decomposition efficiency over time, Figure 1(C) illustrates the PMS/H₂O₂ process and the PMS/H₂O₂/US process. Figure 1(D) illustrates the effect of H₂O₂ concentration on MNZ removal efficiency in the PMS/H₂O₂ process and US/PMS/H₂O₂ process. Figure 1(E) illustrates the effect of varying PMS concentrations on MNZ removal efficiency in the PMS/H₂O₂ process and the US/PMS/H₂O₂ process. Figure 1(F) shows the effect of ultrasonic power in the US/PMS/H₂O₂ process.

In Table 4, experiments 1 and 8 achieved nearly identical efficiencies despite different conditions. The results of these two rows show that by increasing the ultrasonic intensity from 90 to 120 W and the PMS dose concentration from 4 to 6 mmol/L, the time can be reduced by about 45 minutes (60%) while achieving the same desired result. Figure 2 shows the contribution of factors on MNZ degradation using PMS, H₂O₂, US, H₂O₂/US, PMS/US, PMS/H₂O₂, and PMS/H₂O₂/US processes in MNZ removal. Table 5 shows the effects of factors on MNZ degradation using the PMS/H₂O₂ and PMS/H₂O₂/US processes. Table 6 shows the effect of scavengers on the removal of MNZ from aqueous solution during the US/PMS/H₂O₂ process.

Table 7 shows the effect of organic substances on

Table 5. The effect of the contribution of factors on MNZ degradation using the PMS/H₂O₂ and PMS/H₂O₂/US processes

PMS/H ₂ O ₂ process		PMS/H ₂ O ₂ /US process	
Parameters	Contribution%	Parameters	Contribution%
H ₂ O ₂ Dose	49.89	Time	50.47
Time	16.23	MNZ	24.07
PMS Dose	15.40	PMS Dose	12.26
MNZ	12.77	US	7.92
pH	5.11	H ₂ O ₂ Dose	4.48
R ²	0.99	R ²	0.99
Residuals	0.71	Residuals	7.76

removing MNZ from an aqueous solution in the US/PMS/H₂O₂ process. Table 8 shows the effect of inorganic anions in removing MNZ from an aqueous solution in the US/PMS/H₂O₂ process.

Discussion

Effect of pH on MNZ removal

This study examined the influence of pH on MNZ removal efficiency using the PMS/H₂O₂ process. As illustrated in Figure 1(A), the highest removal efficiency was observed at pH 5, indicating that acidic conditions favored removal. A decline in efficiency was noted at neutral (pH 7) and mildly alkaline (pH 9) conditions; however, a subsequent increase in removal efficiency was recorded at pH 11.

pH is a crucial parameter that influences the solubility and activity of oxidants, reaction kinetics, radical production, catalyst surface properties, and pollutants (22). Organic pollutants can exist in different molecular or ionic forms depending on the pH, due to their distinct pK_a values. These variations significantly influence their interactions with reactive species in the oxidation

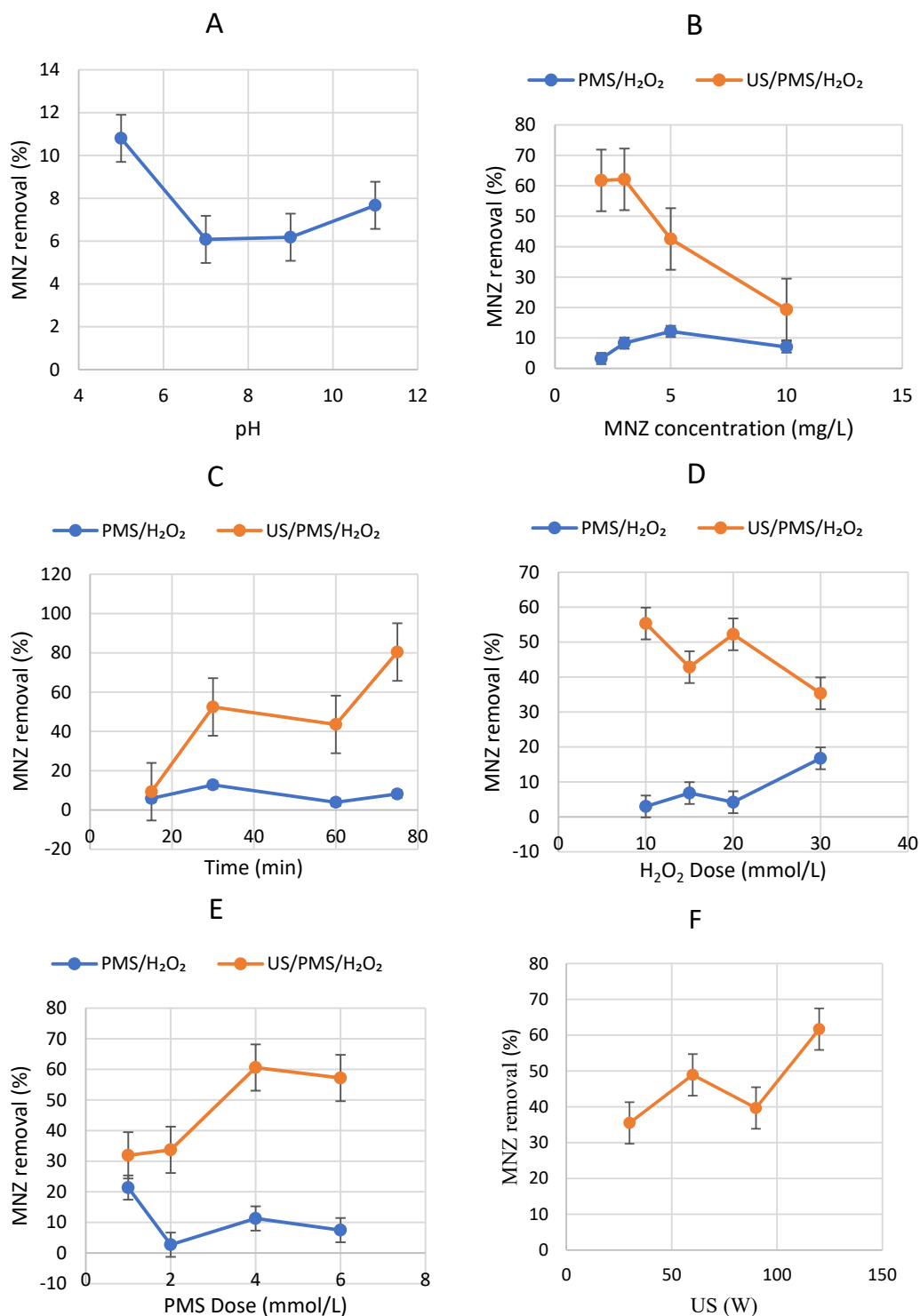


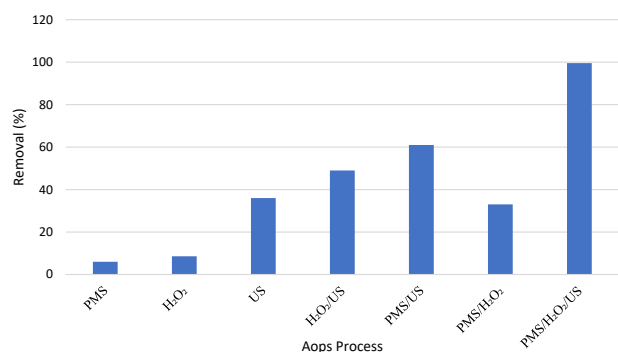
Figure 1. (A) Effect of pH on MNZ removal in PMS/H₂O₂ process (B) Effect of MNZ concentration on MNZ removal in PMS/H₂O₂ process and US/PMS/H₂O₂ process (C) Effect of contact time on MNZ removal in PMS/H₂O₂ and US/PMS/H₂O₂ process (D) Effect of H₂O₂ dose on MNZ removal in PMS/H₂O₂ process and US/PMS/H₂O₂ process (E) Effect of PMS dose on MNZ removal in PMS/H₂O₂ and US/PMS/H₂O₂ process (F) Effect of ultrasonic power in the US/PMS/H₂O₂ process

Table 6. The effect of scavengers in removing MNZ from an aqueous solution in the US/PMS/H₂O₂ process

Run	PMS Dose (mmol/L)	H ₂ O ₂ Dose (mmol/L)	Time (min)	US (W)	MNZ (mmol/L)	pH	Scavengers	scavengers concentration (mmol/L)	Removal percentage of MNZ (%)
1	2	10	30	60	5	7	TBA	15	1.3
2	2	10	30	60	5	7	Etanol	15	27.1
3	2	10	30	60	5	7	PBQ	15	10.2

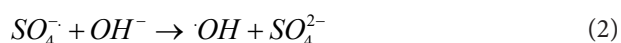
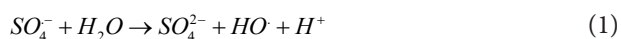
Table 7. The effect of organic substances on removing MNZ from an aqueous solution in the US/PMS/H₂O₂ process

Run	MNZ (mmol/L)	Organic materials	Humic Acid concentration (mmol/L)	Removal percentage of MNZ (%)
1	5	Humic acid	5	14.30
2	5	Humic acid	10	0.1
3	5	Humic acid	20	0.1

**Figure 2.** Contribution of factors on MNZ degradation using the PMS, H₂O₂, US, H₂O₂/US, PMS/US, PMS/H₂O₂, and PMS/H₂O₂/US processes

system. Moreover, the nature and oxidative potential of the generated free radicals are also pH-dependent, further affecting the overall efficiency of the oxidation process (19).

As shown in Table 3, there is no significant difference between pH 5 and pH 11, as both can be considered optimal. This may be because acidic conditions at pH 5 are favorable for the production of active hydrogen. In contrast, different pH levels alter the types of radicals present and their effectiveness in degrading antibiotics (19). Under acidic conditions, the system predominantly generates sulfate radicals (SO₄^{•−}). In neutral to slightly alkaline environments, both sulfate (SO₄^{•−}) and hydroxyl (OH[•]) radicals are produced, whereas in strongly alkaline conditions, hydroxyl radicals (OH[•]) become the dominant species (23). The decrease in efficiency observed at pH 7 and 9 may be attributed to increased production of hydroxyl radicals, which can scavenge sulfate radicals, thereby reducing the overall effectiveness of the process (22,24).



According to Table 5, pH had no significant effect on removal efficiency, accounting for only 5.1% of MNZ removal. Thus, no substantial differences in removal efficiency were observed across different pH levels.

The study by Hou et al (25) demonstrated that the initial pH did not significantly impact the removal efficiency

Table 8. The effect of the presence of inorganic anions on removing MNZ from an aqueous solution in the US/PMS/H₂O₂ process

Run	MNZ (mmol/L)	anions	anions concentration (mmol/L)	Removal percentage of MNZ (%)
1	5	Na ₂ C ₂ O ₃	15	61.9
2	5	CaCl ₂	15	19.8
3	5	NaBr	15	22.9
4	5	KCl	15	20.8
5	5	MgCl ₂	15	0.5
6	5	NaH ₂ PO ₄	15	11.2
7	5	MnCl ₂ ·4H ₂ O	15	9.4
8	5	NaSO ₄	15	82.0
9	5	NaCl	15	10.5
10	5	Na ₂ NO ₃	15	27.9
11	5	NaN ₃	15	18.3
12	5	NaHCO ₃	15	49.8

of tetracycline in the Fe₃O₄/Na₂S₂O₈/US system within the pH range of 3.7 to 9. Similarly, Chen et al (7) found that the removal efficiency of MNZ increased as the pH decreased.

In the research by Tavasol et al (19), which investigated the effect of solution pH on cephalexin degradation, the sonophotocatalytic process with H₂O₂ showed higher efficiency at a pH close to neutral. The removal of cephalexin initially increased but then decreased at pH 3. The highest removal rate (87%) was observed at pH 8, while at pH 9, the removal rate decreased slightly to 78.8%.

Another study by Sun et al (23), focusing on the effect of initial pH on amoxicillin (AMX) removal, revealed that AMX was effectively removed at pH 3 and 5 after about 30 minutes of reaction. The removal rate decreased at pH 7 and pH 9 but increased again at pH 11.

Effect of MNZ concentration on MNZ removal

Based on Tables 3 and 4, the effect of the initial MNZ concentration on decomposition efficiency was investigated in both the PMS/H₂O₂ and PMS/H₂O₂/US processes. As shown in Figure 1(B), in the PMS/H₂O₂ process, MNZ removal efficiency increases with the concentration up to 5 mg/L. However, beyond this concentration, the removal efficiency declines as MNZ concentration increases, indicating that the optimal initial concentration is 5 mg/L. Conversely, in the PMS/H₂O₂/US process, as illustrated in Figure 1(B), the removal efficiency decreases with increasing MNZ concentration.

At higher concentrations of MNZ, pollutants and intermediate molecules produced during the oxidation process compete for reaction with hydroxyl radicals, leading to reduced removal efficiency. Additionally, increased MNZ concentration leads to higher consumption of oxidants and prolongs the decomposition process (3).

The study by Yazdan Panah et al (3) indicates that

removal efficiency decreases with increasing MNZ concentration. Similarly, Su et al found that as the amoxicillin concentration increased from 0.072 to 0.119 mmol/L, COD removal efficiency improved from 76% to 93.4%. However, when the initial concentration exceeded 0.119 mmol/L, the COD removal efficiency decreased slightly (16).

As illustrated in Figure 1(B), the two processes differ in MNZ removal: the PMS/H₂O₂ system shows the lowest removal at 2 mg/L, whereas the US/PMS/H₂O₂ system achieves the highest removal efficiency at the same concentration. At 3 mg/L, the concentration in the PMS/H₂O₂ system improves slightly but remains below the peak value observed in the US/PMS/H₂O₂ process. The PMS/H₂O₂ system reaches its maximum removal efficiency at 5 mg/L, whereas the US/PMS/H₂O₂ system begins to decline at this point. In both systems, a further increase to 10 mg/L results in a continued decrease in removal efficiency. Overall, although the US/PMS/H₂O₂ system showed reduced performance at higher concentrations, it consistently achieved higher MNZ removal than the PMS/H₂O₂ process.

Effect of time on MNZ removal

To assess the removal efficiency over time, Figure 1(C) illustrates that in the PMS/H₂O₂ process, the removal efficiency increases steadily up to 30 minutes. Beyond this point, a decline in efficiency is observed until 60 minutes, followed by a subsequent increase up to 75 minutes. The highest removal rate occurs at 30 minutes, likely due to the optimal H₂O₂ concentration at this stage, which enhances hydroxyl radical generation and thereby improves removal efficiency.

As shown in Figure 1(C), when US is incorporated, the removal efficiency also increases with reaction time, indicating a direct correlation between exposure duration and MNZ degradation. Prolonged reaction times facilitate the continuous generation of hydroxyl radicals, thereby enhancing the overall removal efficiency of MNZ (21). This suggests that the combined US/PMS/H₂O₂ system provides a more effective and sustained generation of oxidative species, resulting in enhanced degradation performance over time.

In a study conducted by Dehghani et al (26), the removal efficiency of amoxicillin was reported as 84.9%, 81.1%, 81.9%, and 87.9% for reaction times of 2, 5, 10, and 15 minutes, respectively. Similar findings were reported by Seyed Mohammadi et al (21), who demonstrated that cephalexin removal efficiency increased with longer reaction times.

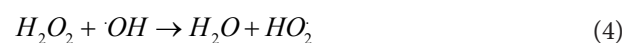
As illustrated in Figure 1(C), the maximum removal efficiency in the PMS/H₂O₂ process was observed at 30 minutes, reaching approximately 8%. In contrast, the highest MNZ removal in the US/PMS/H₂O₂ process occurred at 75 minutes, achieving around 80%. At 15

minutes, the removal efficiency in the US/PMS/H₂O₂ system was already higher than that in the PMS/H₂O₂ system. Both processes exhibited an overall increase in removal efficiency over time; however, at 30 minutes, the efficiency was approximately 10% in the PMS/H₂O₂ system and over 50% in the US/PMS/H₂O₂ system. Following 60 minutes, both systems showed a temporary decline in removal efficiency. Nonetheless, by 75 minutes, removal efficiency increased again, reaching about 10% in the PMS/H₂O₂ process and approximately 80% in the US/PMS/H₂O₂ process.

Effect of H₂O₂ dose on MNZ removal

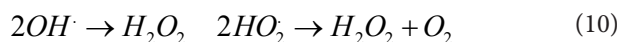
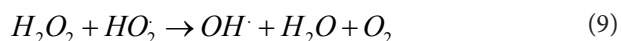
The type and concentration of the oxidizing agent play a critical role in determining the performance and efficiency of AOPs (21). H₂O₂ is a primary source of hydroxyl radicals (•OH) and plays a pivotal role in advanced oxidation processes. However, excessive H₂O₂ dosages can reduce removal efficiency through radical scavenging and increase operational costs (3). As shown in Figure 1(D), which depicts the effect of varying H₂O₂ concentrations on MNZ removal in the PMS/H₂O₂ process, the removal efficiency of MNZ increases with higher doses of H₂O₂. This enhancement is primarily due to increased production of hydroxyl radicals (•OH), which play a key role in oxidative degradation. The addition of H₂O₂ not only accelerates electron transfer reactions but may also react with •OH radicals to generate perhydroxyl radicals (HO₂•), further contributing to the oxidation process (27). As shown in the figure, increased radical formation enhances overall removal efficiency.

Figure 1(D) also illustrates the effect of H₂O₂ concentration on MNZ removal efficiency in the US/PMS/H₂O₂ process. Initially, an increase in H₂O₂ concentration enhances removal efficiency by increasing the •OH radical concentration. However, beyond an optimal concentration, further increase in H₂O₂ leads to a decline in efficiency. This reduction is attributed to the scavenging effect of excess H₂O₂, which can act as a hydroxyl radical quencher, thereby decreasing the availability of active •OH radicals for effective oxidation. At higher concentrations, H₂O₂ reacts with •OH to produce HO₂•, which possesses a lower oxidation potential than •OH. The following reactions illustrate these inhibitory pathways:(3,21).



For effective oxidation, •OH must be efficiently transferred from the liquid phase to the surface of MNZ molecules. A considerable portion of H₂O₂ is typically consumed in generating these reactive radicals. The

application of US enhances this process through acoustic cavitation, which produces numerous microscopic air bubbles. Upon collapse, these bubbles generate localized high temperatures and pressures, significantly improving the decomposition of H_2O_2 and the formation of $\cdot OH$ radicals. Moreover, cavitation promotes enhanced mass transfer by increasing turbulence and mixing in the solution, thereby facilitating the transport of reactive species to pollutant surfaces. The combined effects of intensified mass transfer, radical generation, and extreme microenvironments created by cavitation contribute to the improved degradation of MNZ and other organic contaminants (27).



In a study by Pourzmani et al it was observed that humic acid decomposition increased with higher H_2O_2 concentrations (27). Similar findings were reported by Seid-Mohammadi (21), who investigated the effect of H_2O_2 on cephalixin removal in the US/ H_2O_2 /NiO process. Their study showed that the removal efficiency initially increased with rising H_2O_2 concentrations but then decreased after reaching an optimal level. Additionally, Yazdanpanah et al demonstrated that increasing the H_2O_2 concentration from 0.2 mmol to 1 mmol increased MNZ removal efficiency from 79% to 98%. However, further increasing the H_2O_2 concentration from 1 mmol to 2 mmol decreased the removal efficiency from 98% to 81% (3). This underscores the critical importance of optimizing oxidant dosage to maximize efficiency and minimize potential scavenging effects in advanced oxidation processes.

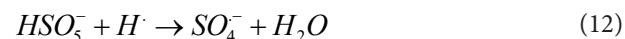
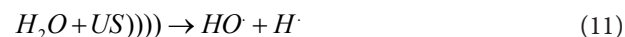
Comparing the two processes, Figure 1(D) shows that, in the absence of US, the highest MNZ removal efficiency was achieved at a H_2O_2 concentration of 30 mmol/L. However, in the presence of the US, the highest removal efficiency was observed at H_2O_2 concentrations of 10 and 20 mmol/L.

Effect of PMS dose on MNZ removal

Figure 1(E) illustrates the effect of varying PMS concentrations on MNZ removal efficiency in the presence of H_2O_2 . The results indicate that removal efficiency is higher at PMS concentrations of 1 and 4 mmol, while a decline in efficiency is observed at 2 and 6 mmol. This reduction may be attributed to radical–radical interactions that occur prior to their reaction with organic pollutants. Specifically, excess sulfate ions (SO_4^{2-}) can

inhibit the formation of hydroxyl radicals by scavenging reactive species or participating in side reactions, thereby diminishing the system's overall oxidative capacity (28).

The activation of PMS is strongly influenced by US frequency, as different frequencies generate distinct cavitation effects. Figure 1(E) demonstrates the influence of varying PMS concentrations (2–8 mmol/L) on the efficiency of MNZ removal under ultrasonic treatment. The results show a clear trend: removal efficiency increases with rising PMS concentration. This improvement can be attributed to the enhanced generation of reactive species, which accelerates the degradation process. Specifically, higher PMS levels promote the formation of more sulfate and hydroxyl radicals, intensifying the oxidative attack on organic molecules, boosting removal efficiency.



Similar findings were reported by Xu et al (29), who observed that increasing PMS concentration enhanced chloramphenicol (CAP) degradation. Specifically, as PMS concentration increased from 2 mmol to 4 mmol, the removal percentage improved from 51% to 79.2%. Further increasing the PMS concentration to 6 mmol continued to enhance degradation. However, raising the concentration to 8 mmol did not yield a significant increase in removal efficiency.

Similar results were observed in a study by Malkutian et al (28), who found that increasing persulfate concentration enhanced both reaction rate and tetracycline removal efficiency.

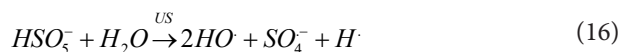
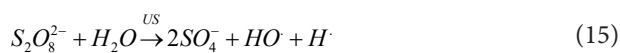
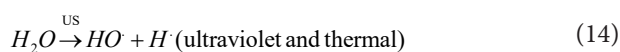
In both processes, the removal efficiency was maximized at a PMS concentration of 4 mmol/L, whereas the lowest efficiency was observed at 2 mmol/L.

Effect of ultrasonic intensity on MNZ removal

US power is a critical parameter that significantly influences the cavitation phenomenon (16). As shown in Figure 1(F), removal efficiency increased with higher US power, reaching nearly 50% at 120 W. Higher US power leads to greater energy transfer into the system, resulting in more intense cavitation bubble collapse and enhanced formation of reactive radicals (18). Consequently, highly reactive free radicals such as $\cdot HO$, $\cdot OH$, and $\cdot H$ are generated during the propagation of US due to cavitation (30).

In this process, cavitation occurs in three stages: the formation, rapid growth, and explosion of microbubbles within the water. This leads to a momentary release of substantial energy. Additionally, cavitation generates hot spots (up to 5000 K) and high pressures (around 1800 atm) for a brief period (18). These conditions facilitate

the activation of PMS, leading to the production of free radicals. The removal of organic substances is further enhanced by the activation of H_2O_2 and PMS, as described by the following equations (20).



Sulfate radicals generated by AOPs have recently attracted attention for their ability to remove antibiotics. These radicals can be produced by activating PS (persulfate) or PMS using various methods, including heat, transition metals, and US (31). Additionally, H_2O_2 has been shown to enhance sonication rate, resulting in the production of more radicals (19).

These findings align with results from other studies, which show that increasing ultrasonic power enhances the removal efficiency of MNZ. For example, Nasser et al (32) observed that increasing ultrasonic power increased the tetracycline degradation rate from 57.7% to 88.5%. Similarly, Pourzmani et al (33) found that raising the ultrasonic power from 50 to 120 W improved the decomposition of diclofenac.

Comparison of two processes in MNZ removal

In this study, the effectiveness of the PMS/ H_2O_2 and US/PMS/ H_2O_2 processes for MNZ removal under optimal conditions was evaluated. As illustrated in Figure 2, the incorporation of US resulted in nearly complete elimination of MNZ from aqueous solutions. This indicates that the US significantly enhances the degradation rate of MNZ, likely by intensifying cavitation effects, increasing the generation of reactive radicals, and improving mass transfer within the system.

In the combined PMS/ H_2O_2 /US process, the simultaneous addition of H_2O_2 and PMS during ultrasonic treatment resulted in a synergistic enhancement in $^{\cdot}OH$ production. This increase in reactive species contributed to improved oxidant transfer and distribution within the solution. The observed performance enhancement was not solely attributable to the mechanical effects of ultrasound, indicating that the chemical interaction between PMS and H_2O_2 under ultrasonic irradiation played a critical role. To quantitatively assess these effects, statistical analysis of the experimental model was performed using analysis of variance (ANOVA), with the results summarized in Table 5.

As shown in Table 5, all factors significantly influenced MNZ removal (P -value < 0.01), but their interactions did

not. Additionally, Table 5 shows that, while the considered variables each affected MNZ removal to varying degrees, the H_2O_2 dose had the greatest impact. The results of the statistical analysis for MNZ removal using the PMS/ H_2O_2 /US process are presented in Table 5, highlighting the contribution of each factor to MNZ degradation.

According to Table 5, all factors significantly influenced MNZ removal (P -value < 0.01), but their interactions were not significant. Unlike the PMS/ H_2O_2 process, where H_2O_2 dosage was the most critical factor, in the PMS/ H_2O_2 /US process, the duration of US exposure had the greatest impact on MNZ removal.

Identification of active radical species in the decomposition of MNZ

To elucidate the reactive radical species responsible for MNZ degradation, a series of inhibition experiments and anion effect assessments were conducted under controlled conditions using the US/PMS/ H_2O_2 system. The experimental parameters were as follows: PMS concentration = 2 mmol/L, H_2O_2 concentration = 10 mmol/L, US power = 60 W, pH = 7.0, and initial MNZ concentration = 5 mg/L. Table 6 shows the effect of scavengers in removing MNZ from an aqueous solution in the US/PMS/ H_2O_2 process. The addition of parabenzoquinone (PBQ), tert-butyl alcohol (TBA), and ethanol resulted in marked decreases in MNZ removal efficiency, to 10.2%, 1.3%, and 27.1%, respectively. Among these, TBA, a well-known scavenger of hydroxyl radicals ($^{\cdot}OH$), exhibited the least inhibitory effect on MNZ degradation in the US system. These findings are consistent with previous studies demonstrating that the introduction of radical scavengers significantly impairs the removal efficiency of MNZ (22).

The effect of organic substances on the decomposition of MNZ

Humic acid, a representative form of natural organic matter (NOM) that contains functional groups such as quinones, alcohols, carboxylic acids, ketones, and phenols, can impede treatment by acting as a radical scavenger and competing with target pollutants for reactive species (34). Table 7 shows the effect of the presence of organic substances on removing MNZ from an aqueous solution in the US/PMS/ H_2O_2 process. The inhibitory effect of humic acid on PMS activity was assessed at concentrations of 5, 10, and 15 mg/L. The corresponding MNZ degradation efficiencies were 14.3%, 0.1%, and 0.1%, respectively, demonstrating a pronounced decline in treatment performance with increasing humic acid concentration. This substantial reduction is attributed to humic acid's ability to interfere with PMS activation, thereby reducing the generation of reactive radicals essential for MNZ degradation (35). Table 7 shows the effect of the presence of organic substances on removing MNZ from aqueous solution in the US/PMS/ H_2O_2 process.

Effect of anions in the decomposition of MNZ

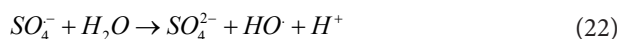
The oxidation processes can be significantly influenced by the presence of various anions, which may negatively affect treatment efficiency by scavenging reactive radicals or interfering with the generation of active species. Accordingly, the influence of various anions on the removal efficiency of MNZ in the PMS/H₂O₂/US process was systematically evaluated. As summarized in Table 8, all tested anions inhibited MNZ removal. The extent of inhibition followed the order: chloride > phosphate > bromide > nitrate > carbonate > sulfate. Among the chloride salts, magnesium chloride demonstrated the minimal impact on removal efficiency, resulting in only a 0.5% reduction.

One possible explanation for the observed inhibitory effect is the interaction of chloride ions with hydroxyl radicals ($\cdot\text{OH}$), leading to the formation of chlorine-based radicals such as $\text{Cl}\cdot$ and $\text{Cl}_2\cdot^-$, which possess lower oxidative potential. Chloride can also react with hydroxyl radicals to generate active chlorine species, including hypochlorous acid (HOCl). Under US irradiation, HOCl may further decompose into hydroxyl and chlorine radicals, both of which can contribute to the breakdown of metronidazole into smaller intermediate molecules. Additionally, chloride ions may react with H₂O₂ to form hypochlorite ions (ClO^-), further altering the oxidative environment and influencing MNZ degradation pathways (19).

The relatively lower inhibitory effect of carbonate is likely due to its ability to scavenge hydroxyl radicals, resulting in the formation of carbonate radicals ($\text{CO}_3^{\cdot-}$), as represented by the following reactions:



The minimal inhibitory effect of sulfate anions on MNZ degradation may be attributed to the generation of sulfate radicals ($\text{SO}_4^{\cdot-}$), which are potent oxidizing species. These radicals can participate in the deactivation of hydroxyl radicals; however, they also directly react with organic contaminants, facilitating their transformation into a range of intermediate compounds.



Conclusion

This study investigated the degradation of MNZ in aqueous solutions using the H₂O₂/PMS system, both

with and without US irradiation, and examined the influence of various operational parameters on process efficiency. The findings demonstrate the significant role of US waves in enhancing the process efficiency. Analysis of variance revealed that in the PMS/H₂O₂ process, the H₂O₂ dose had the most significant effect on MNZ degradation, followed by reaction time, PMS dose, MNZ concentration, and pH. However, for the US/PMS/H₂O₂ process, reaction time was the most influential parameter, followed by MNZ concentration, PMS dose, ultrasonic power, and H₂O₂ dose. Under optimal conditions, the highest removal efficiency in the PMS/H₂O₂ system was approximately 33.04%. However, when US waves were applied during the US/PMS/H₂O₂ process, the removal efficiency improved significantly, reaching 99.5% under optimal conditions. The removal efficiency of MNZ in the presence of various radical scavengers demonstrated that TBA, a known scavenger of hydroxyl radicals ($\cdot\text{OH}$), exhibited the most significant inhibitory effect on MNZ degradation. When assessing the impact of accompanying anions on MNZ decomposition, the order of effectiveness was chloride > phosphate > bromide > nitrate > carbonate > sulfate. Furthermore, humic acid was found to reduce the concentration of active radicals by interfering with PMS functionality, thereby leading to a notable decrease in MNZ removal efficiency.

In summary, this study demonstrated that the US can significantly reduce MNZ concentrations in both water and wastewater. The findings underscore the importance of various parameters in optimizing the US treatment process for effective MNZ degradation. However, given the potential high costs associated with large-scale application of US for water and wastewater treatment, a comprehensive economic feasibility evaluation is crucial before widespread implementation.

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Formal Analysis: Hamidreza Pourzamani, Ensiyeh Taheri, Zeinab Didari.

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Methodology: Hamidreza Pourzamani, Ensiyeh Taheri, Zeinab Didari.

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Competing interests

The author declares that he has no competing interests.

Ethical issues

The author hereby certifies that all data collected during the study are as stated in the manuscript, and no data from the study has been or will be published separately elsewhere (ethical code: IR.MUI.RESEARCH.REC.1401.320).

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