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Article

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Ethical Approval

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Efficiency of Photocatalytic Ozonation Process in the Presence of CoFe₂O₄/ZnTiO₃/Ag Nanocomposite for the Removal of Bisphenol A from Aqueous Solutions

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ABSTRACT

Bisphenol A (BPA), a ubiquitous endocrine-disrupting compound, persists in aquatic environments and resists conventional wastewater treatment, posing significant ecological and human health risks. This study introduces a novel magnetic CoFe₂O₄/ZnTiO₃/Ag ternary nanocomposite synthesized via a facile co-precipitation and calcination method, and evaluates its performance as a heterogeneous catalyst in the synergistic photocatalytic ozonation process for BPA degradation under UV-C irradiation. The nanocomposite was comprehensively characterized using XRD, FTIR, FESEM, EDX, and VSM, confirming successful integration of crystalline phases, strong magnetic separability, and enhanced surface properties. Batch experiments systematically investigated key operational parameters, including initial BPA concentration (10–50 mg/L), catalyst dosage (25–75 mg/L), pH (4–10), and reaction time (up to 240 min), under continuous ozone input (0.2 g/h). Under optimized conditions (pH 7, 50 mg catalyst, 10 mg/L BPA), the process achieved 91.30% BPA removal and 82.5% COD reduction within 240 min. Comparative experiments revealed pronounced synergy among UV-C, ozone, and the nanocomposite, significantly outperforming the corresponding individual and binary

processes (e.g., UV-C/O₃: 76.09%; O₃ alone: 70.65%). Statistical analyses (ANOVA, $p < 0.05$) confirmed significant interactions. The catalyst exhibited excellent reusability, retaining 83.47% efficiency after seven cycles, attributable to its magnetic recoverability and structural stability. These findings demonstrate that CoFe₂O₄/ZnTiO₃/Ag -assisted photocatalytic ozonation offers a promising, efficient, and sustainable advanced oxidation strategy for removing recalcitrant endocrine disruptors from aqueous media, which demonstrates promising potential for wastewater treatment applications.

Keywords: Bisphenol A; CoFe₂O₄/ZnTiO₃/Ag; Magnetic catalyst; Photocatalytic ozonation; Synergistic degradation; Ternary nanocomposite.

1. Introduction

Emerging pollutants (EPs) comprise synthetic or naturally occurring chemical substances that are not routinely monitored in the environment but pose significant risks to ecological systems and human health. Their increasing detection in aquatic environments has raised global concern due to their persistence, bioaccumulation potential, and incomplete removal by conventional treatment systems [1,2]. Among EPs, endocrine-disrupting compounds (EDCs) have attracted considerable attention because of their ability to interfere with hormonal regulation even at trace concentrations [3].

Bisphenol A (BPA) is one of the most widely produced and extensively studied EDCs. It is primarily used as a monomer in the manufacture of polycarbonate plastics and epoxy resins, resulting in its continuous release into water bodies through industrial effluents and leaching from consumer products [4,5]. Although epidemiological evidence in humans remains limited, numerous in vivo and in vitro studies have demonstrated that BPA exposure is associated with reproductive disorders, endocrine dysfunction, metabolic disturbances, and hormone-related cancers, including breast, ovarian, and prostate cancers [5,6]. In addition to human health risks, BPA adversely affects aquatic organisms, disrupts ecological balance, and threatens biodiversity [7-9]. Due to its chemical stability and phenolic structure, BPA is resistant to biodegradation, making its complete removal in wastewater treatment plants challenging [5,10].

Various treatment technologies have been applied for the removal of BPA and similar contaminants, including adsorption, membrane filtration, coagulation, biological treatment, and

advanced oxidation processes (AOPs) [4,11,12]. However, these methods often suffer from significant limitations such as high operational costs, secondary pollution, sludge generation, membrane fouling, and limited mineralization efficiency. In this context, AOPs have emerged as promising alternatives due to their ability to generate strongly reactive species, particularly hydroxyl radicals ($\cdot\text{OH}$). These radicals are capable of non-selectively oxidizing recalcitrant organic pollutants [13]. Ozonation is an effective AOP that degrades contaminants either through direct electrophilic attack by ozone molecules or indirectly via hydroxyl radicals formed during the ozone decomposition, especially under alkaline conditions [14].

Recently, photocatalytic ozonation has gained increasing attention as a hybrid advanced oxidation technology that integrates the advantages of heterogeneous photocatalysis and ozonation. Under ultraviolet (UV) irradiation, semiconductor photocatalysts generate electron–hole pairs that enhance reactive oxygen species (ROS) formation and improve the ozone decomposition efficiency. This synergistic interaction leads to accelerated pollutant degradation, reduced ozone consumption, and improved mineralization compared to single processes [15,16].

Titanium dioxide (TiO_2) remains one of the most extensively investigated photocatalysts due to its chemical stability and non-toxicity [17,18]. However, its practical application is limited by rapid electron–hole recombination and its limited visible-light absorption. To overcome these limitations, TiO_2 -based materials have been modified with noble metals such as silver (Ag), which enhance charge separation and facilitate interfacial electron transfer [19,20]. Moreover, zinc-based semiconductors, including ZnO and ZnTiO_3 , exhibit favorable electronic structures and strong photocatalytic performance under UV irradiation [21–23]. Incorporating magnetic materials such as cobalt ferrite (CoFe_2O_4) into photocatalytic systems not only improves catalytic stability but also enables facile magnetic recovery, reducing secondary contamination and operational costs [24,25].

Despite numerous studies on individual photocatalytic or ozonation systems, limited research has focused on the synergistic integration of magnetic ternary nanocomposites with photocatalytic ozonation for BPA degradation. Recent advances have explored Ce-doped Bi-MOF-derived $\text{Bi}_2\text{O}_3/\text{CeO}_2$ with oxygen vacancies (achieving 97.6% mineralization) [26], nano CeO_2 in catalytic/photocatalytic ozonation (99.97% removal efficiency) [27], metal cationic defect-enriched ZnMnAl -LDHs under visible light (>90% mineralization) [28], and Fe-Mn co-doped TiO_2 for enhanced ozonation (98.08% degradation) [29]. However, the combined effects

of CoFe_2O_4 , ZnTiO_3 , and Ag within a single magnetic ternary nanocomposite structure and their interaction with ozone under UV irradiation remain insufficiently explored.

Therefore, the present study aimed to synthesize and characterize a novel $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ magnetic nanocomposite and evaluate its performance in a photocatalytic ozonation system for the removal of Bisphenol A from aqueous environments. The effects of operational parameters, process synergy, and catalyst reusability were systematically investigated to assess the feasibility of this integrated advanced oxidation approach for water treatment applications.

2. Materials and Methods

2.1. Materials

All chemical reagents used in this study were of analytical grade and used as received without further purification. Bisphenol A (BPA, $\text{C}_{15}\text{H}_{16}\text{O}_2$, CAS No. 80-05-7, purity >99%) was purchased from Sigma-Aldrich. For the synthesis of the magnetic ternary nanocomposite, cobalt nitrate hexahydrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), iron nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), tetra-n-butyl titanate ($\text{Ti}(\text{OC}_4\text{H}_9)_4$), silver nitrate (AgNO_3), ammonia solution (25% NH_3), and absolute ethanol (99.5%) were acquired from Merck or Sigma-Aldrich. The pH of the solutions was adjusted using 1 M sulfuric acid (H_2SO_4) and 1 M sodium hydroxide (NaOH). Double-distilled water was utilized for the preparation of all aqueous solutions and stock preparations.

2.2. Synthesis of $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ Ternary Nanocomposite

The $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ ternary nanocomposite was synthesized via a systematic two-step coprecipitation and calcination route. In the first step, the magnetic CoFe_2O_4 cores were prepared by dissolving stoichiometric amounts of cobalt and iron nitrates in deionized water, followed by the dropwise addition of ammonia solution under vigorous stirring to precipitate the metal hydroxides. The resulting precipitate was filtered, washed several times with deionized water and absolute ethanol, dried, and subsequently calcined at 700 °C for 2 h to yield crystalline, magnetic CoFe_2O_4 nanoparticles. In the second step, coating and Ag deposition were achieved by dispersing 1 mmol of the as-synthesized magnetic CoFe_2O_4 core nanoparticles in absolute ethanol under ultrasonication to ensure a stable, homogeneous suspension. Separately, precursor solutions were prepared by dissolving 1 mmol of zinc nitrate hexahydrate, 1 mmol of tetra-n-

butyl titanate (as the titanium source), and 1 mmol of silver nitrate each in 30 mL of deionized water. These precursor solutions were then added dropwise to the agitated cobalt ferrite suspension under continuous ultrasonication to promote uniform mixing, homogeneous nucleation, and optimal interfacial contact. The resulting mixture was dried in an oven at 90 °C until complete solvent evaporation, and the obtained dry powder was transferred to a muffle furnace and calcined at 700 °C for 2 h with a heating rate of 5 °C/min (followed by natural cooling to room temperature) to induce crystallization, phase formation, and strong interfacial bonding among the synthesized $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3$, and Ag phases.

2.3. Photocatalytic Ozonation Experimental Setup

The batch photocatalytic ozonation experiments were conducted in a custom-designed cylindrical quartz reactor with a 250 mL working volume. Constant agitation of the reaction mixture (at 300 rpm) was maintained using a magnetic stirrer to ensure uniform catalyst dispersion and prevent particle sedimentation. Illumination was provided by a 400 W metal halide lamp (Pars Company, Iran) positioned vertically above the reactor for uniform exposure. The light source emitted across both ultraviolet (including UV-C, ~200–280 nm) and visible wavelengths to effectively photo-excite the semiconductor components. Ozone gas was continuously generated from dry oxygen using an ARDA ozone generator (France) at a constant output rate of 0.2 g/h and introduced into the reactor through a porous diffuser situated at the bottom of the vessel to optimize gas-liquid mass transfer. The reactor temperature was strictly maintained at 25 ± 2 °C throughout the experiments using an external water-jacket cooling system to eliminate thermal fluctuations caused by the lamp.

2.4. Experimental Procedure and Analytical Methods

A stock solution of BPA (1000 mg/L) was prepared in double-distilled water, from which working solutions (10, 30, and 50 mg/L) were prepared daily by dilution. In a typical run, 250 mL of the BPA solution was transferred to the reactor, and the pH was adjusted to the desired level (4, 6, 7, 9, or 10) using 1 M H_2SO_4 or NaOH. A specific dosage of the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ catalyst (25, 50, or 75 mg) was then dispersed in the solution. The suspension was stirred in the dark for 20 min to establish adsorption-desorption equilibrium before initiating the reaction. The light source and ozone input were turned on simultaneously to mark the beginning of the photocatalytic ozonation process. At pre-selected time intervals (up to 240 min), samples were withdrawn and the magnetic catalyst was rapidly separated from the

suspension using an external magnet (or further clarified via a 0.22 μm syringe filter). The residual concentration of BPA was quantified spectrophotometrically using a UV-Vis spectrophotometer. To measure BPA at a maximum absorbance wavelength of 460 nm, a standard colorimetric derivatization method was performed using 4-aminoantipyrine (4-AAP) and potassium ferricyanide under alkaline conditions. The BPA removal efficiency (%) was calculated as follows:

$$\text{Removal Efficiency (\%)} = (C_0 - C_t) / C_0 \times 100$$

where C_0 and C_t are the initial and residual concentrations of BPA (mg/L), respectively. The chemical oxygen demand (COD) was determined using the standard closed reflux dichromate method to evaluate the mineralization degree of the system. For the reusability study, the recovered catalyst was thoroughly washed with deionized water, dried at 60 $^{\circ}\text{C}$ under vacuum, and reused in consecutive runs under optimal reaction conditions.

3. Results and Discussion

3.1. Characterization of the Photocatalyst and Process Performance

The physicochemical properties of the synthesized $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ ternary nanocomposite were first characterized using various analytical techniques. Subsequently, the effects of key operational parameters, including pH, catalyst dosage, initial Bisphenol A (BPA) concentration, and reaction time, were systematically investigated. In addition, the performance of individual processes, namely ozonation alone and UV-C irradiation alone, was evaluated and compared with the integrated photocatalytic ozonation process to assess the overall BPA removal efficiency.

3.1.1. X-ray Diffraction (XRD) Analysis

Figure 1 presents the X-ray diffraction (XRD) pattern of the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ ternary nanocomposite calcined at 700 $^{\circ}\text{C}$. XRD was employed as a primary technique to determine the crystalline phases present, evaluate phase purity, analyze structural characteristics, and confirm the successful formation of the desired ternary composite.

The diffraction pattern exhibits several sharp and well-defined peaks, reflecting the high crystallinity of the synthesized material. These observed diffraction peaks can be successfully

indexed to three distinct crystalline phases. Specifically, the characteristic reflections observed at $2\theta \approx 18.3^\circ, 30.1^\circ, 35.5^\circ, 43.1^\circ, 57.0^\circ$, and 62.6° correspond to the (111), (220), (311), (400), (511), and (440) planes, respectively, of the cubic spinel structure of CoFe_2O_4 (JCPDS card no. 00-001-1121 or 22-1086). Additionally, the prominent peaks at $2\theta \approx 38.1^\circ, 44.3^\circ, 64.4^\circ$, and 77.5° are assigned to the (111), (200), (220), and (311) planes of cubic metallic silver (Ag) (JCPDS card no. 01-087-0717 or 04-0783). Finally, the remaining key diffraction peaks at $2\theta \approx 32.8^\circ\text{--}33.0^\circ, 35.3^\circ\text{--}35.5^\circ, 40.5^\circ, 48.9^\circ$, and 53.2° are indexed to the (104), (110), (113), (024), and (116) planes of rhombohedral (or ilmenite-type) ZnTiO_3 (JCPDS card no. 00-025-0671).

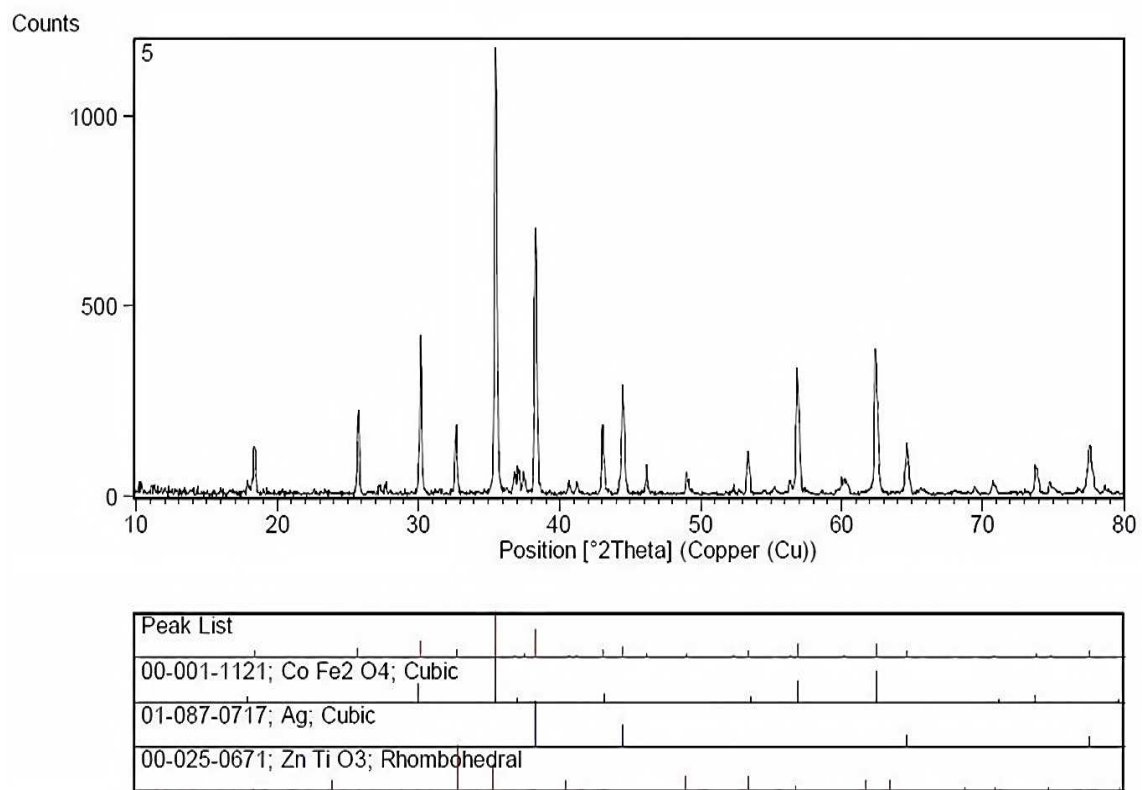


Figure 1. XRD pattern of the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ nanocomposite calcined at 700°C , showing characteristic peaks of cubic CoFe_2O_4 (JCPDS 00-001-1121), cubic Ag (JCPDS 01-087-0717), and rhombohedral ZnTiO_3 (JCPDS 00-025-0671).

The simultaneous presence of these three distinct sets of diffraction peaks, without any significant peak overlap or additional unindexed reflections from impurity phases, confirms the successful integration of the magnetic CoFe_2O_4 core, the perovskite-like ZnTiO_3 component,

and the metallic Ag nanoparticles within the unified ternary nanocomposite. The high intensity and sharpness of the reflections directly reflect the excellent crystallinity achieved through the calcination process at 700 °C, while the complete absence of broad amorphous humps or unindexed reflections further indicates the high phase purity of the synthesized product. The XRD spectrum also includes a detailed peak list annotation that highlights the matching JCPDS reference cards for each distinct crystalline phase, plotted along a 2θ scale spanning from 10° to 80° (obtained using Cu K α radiation with $\lambda=1.5406$ Å). Minor variations in peak intensity or the slight broadening of certain reflections in the composite samples compared to their pure phase counterparts are commonly and reasonably attributed to the nanoscale crystallite size of the components, interfacial lattice strain at the phase boundaries, or minor amorphization occurring at the grain interfaces during high-temperature thermal treatment.

3.1.2. Fourier-Transform Infrared (FTIR) Spectroscopy Analysis

FTIR spectroscopy was employed to identify the surface functional groups and confirm the chemical bonding states within the synthesized CoFe₂O₄/ZnTiO₃/Ag ternary nanocomposite. The FTIR spectrum (plotted as Figure 2) was recorded in the wavenumber range of 4000–400 cm⁻¹ and exhibits several characteristic absorption bands that verify the successful chemical integration of the ternary structure. The broad and intense absorption band centered at approximately 3477 cm⁻¹, along with the medium-intensity peak at 1631 cm⁻¹, are assigned to the O–H stretching and H–O–H bending vibrations, respectively. These bands originate from adsorbed water molecules and surface-bound hydroxyl groups, indicating the strongly hydrophilic nature of the nanocomposite surface. This hydrophilicity is particularly advantageous for aqueous-phase photocatalytic applications, as surface hydroxyl groups act as primary capture sites for photogenerated holes, thereby promoting the generation of highly reactive hydroxyl radicals ($\cdot$ OH). The weak and broad absorption bands observed in the 1508–1419 cm⁻¹ region is typically attributed to the C–O stretching vibrations of residual carbonate species or organic precursor fragments adsorbed on the surface, which remain in minor quantities even after high-temperature calcination at 700 °C. In the lower wavenumber fingerprint region, the distinct peaks appearing at 1269 cm⁻¹, 1133 cm⁻¹, and 907 cm⁻¹ are associated with the characteristic Ti–O–Ti or Zn–O–Ti stretching modes, confirming the successful formation of the perovskite-like ZnTiO₃ phase. Furthermore, the sharp absorption bands observed at 687 cm⁻¹, 584 cm⁻¹, 532 cm⁻¹, 504 cm⁻¹, and 439 cm⁻¹ correspond to the metal–oxygen (M–

OM–O) lattice vibrations of the octahedral and tetrahedral interstitial sites, which are strongly characteristic of the cubic spinel structure of the magnetic CoFe_2O_4 core. The incorporation of metallic silver (Ag) nanoparticles did not result in the emergence of new distinct oxide bands, which is consistent with the plasmonic, non-oxidized, and metallic nature of silver in the composite rather than an oxidized state. Conclusively, the FTIR results corroborate the successful integration of the magnetic ferrite core, the titanate shell, and the metallic silver nanoparticles without introducing any crystalline or amorphous organic impurities, which supports the structural stability of the composite for synergistic photocatalytic ozonation applications.

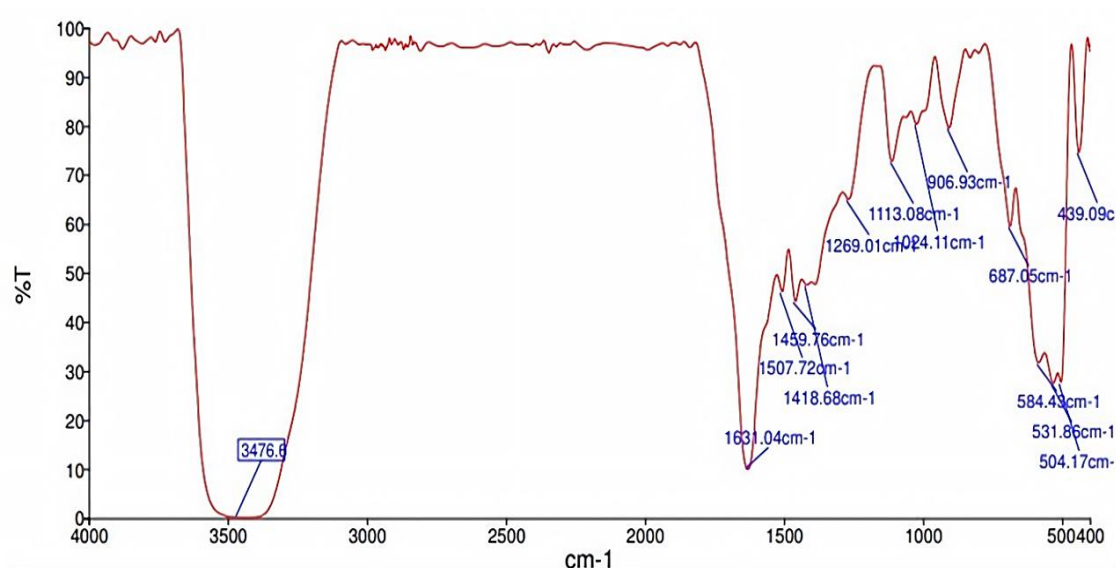


Figure 2. FTIR spectrum of the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ nanocomposite, highlighting O–H vibrations from adsorbed water and characteristic metal–oxygen bonds in the composite lattice.

3.1.3. Vibrating Sample Magnetometer (VSM) Analysis

The magnetic properties, room-temperature separation feasibility, and magnetic field response of the bare CoFe_2O_4 nanoparticles and the ternary $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ nanocomposite were quantitatively investigated using vibrating sample magnetometer (VSM) measurements at room temperature. The obtained magnetic hysteresis (M–H) loops are displayed in Figure 3. Both the bare ferrite core and the ternary nanocomposite exhibit characteristic ferromagnetic hysteresis loops. The bare CoFe_2O_4 nanoparticles demonstrate a high saturation magnetization ($M_s \approx 24$ – 25 emu/g) along with a relatively wide loop indicative of higher coercivity (H_c). Conversely, the ternary $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ nanocomposite exhibits a markedly reduced M_s value (≈ 14 –

15 emu/g) and a much narrower hysteresis loop, showing softer magnetic behavior. The notable decline in M_s is primarily attributed to the "magnetic dilution effect" caused by the incorporation of the non-magnetic ZnTiO_3 and metallic Ag phases, which encapsulate or disperse around the magnetic spinel core and effectively weaken the exchange interactions between the magnetic ions (Fe^{3+} and Co^{2+}) in the CoFe_2O_4 lattice. Furthermore, the markedly increased interfacial boundary areas in the ternary composite can lead to spin canting, lattice distortion, or structural disorder at the grain boundaries, which collectively lower the net magnetic moment. The observed decrease in H_c and the narrower loop configuration of the composite suggest a reduction in the effective magneto crystalline anisotropy and a transition from a relatively hard magnetic phase to a softer magnetic phase. This soft magnetic transition is especially beneficial for heterogeneous catalysis; it minimizes magnetic remanence (M_r) and coercivity, thereby preventing permanent particle aggregation or irreversible agglomeration via dipole-dipole attractions when the external magnetic field is removed, which preserves the catalyst's dispersibility in aqueous media.

Despite the reduction in the overall saturation magnetization, the ternary nanocomposite still retains an entirely sufficient magnetization (>10 emu/g), which is fully adequate to ensure rapid and remarkably efficient magnetic separation from treated water using an external neodymium magnet. This robust magnetic response makes the synthesized nanocomposite a remarkably suitable, easily recoverable, and reusable heterogeneous catalyst for advanced photocatalytic ozonation processes.

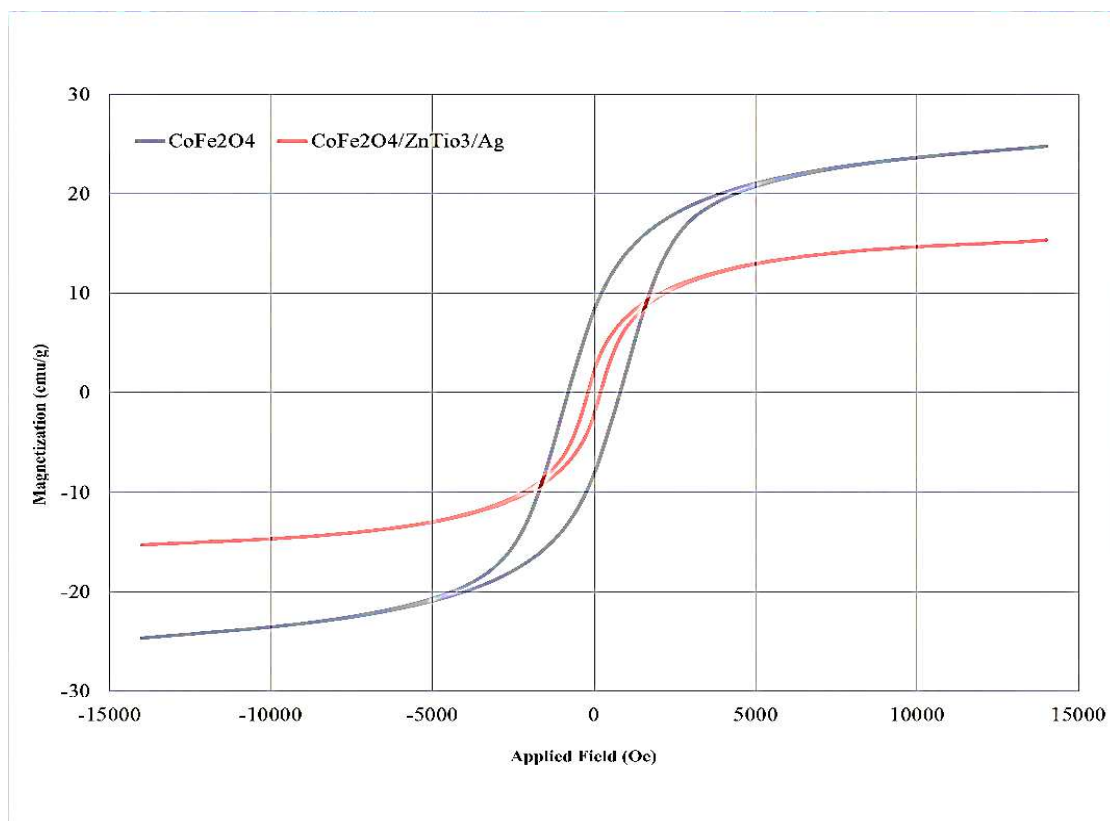


Figure 3. Room-temperature magnetization hysteresis loops of CoFe_2O_4 and $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ nanocomposite, demonstrating the impact of non-magnetic components on magnetic properties.

3.1.4. Field Emission Scanning Electron Microscopy (FESEM) Analysis

Field emission scanning electron microscopy (FESEM) was utilized to comprehensively evaluate the surface morphology, grain size, shape, and spatial distribution of the synthesized $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ ternary nanocomposite. FESEM is especially useful for analyzing magnetic nanomaterials, as its high-brightness electron source and optimized electron optics minimize charging artifacts and beam deflection induced by the local magnetic fields of the ferrite core, yielding sharp, high-resolution micrographs without the need for thick conductive coatings. The representative FESEM micrographs obtained at various magnifications (ranging from $330\times$ to $100\text{ k}\times$) are displayed in Figure 4. The images reveal a heterogeneous, crystalline morphology predominantly consisting of aggregated, quasi-spherical to polyhedral nanoparticles. The individual grains exhibit sizes ranging from 50 to 200 nm, with an average

particle size of approximately 80–150 nm based on scale-bar estimations. The observed agglomeration of these nanoparticles into larger clusters is a typical characteristic of ferrite-based composites prepared via high-temperature calcination (700 °C). This agglomeration is primarily driven by strong magnetic dipole-dipole interactions among the magnetic CoFe_2O_4 cores, attractive van der Waals forces, and localized thermal sintering effects during the calcination process.

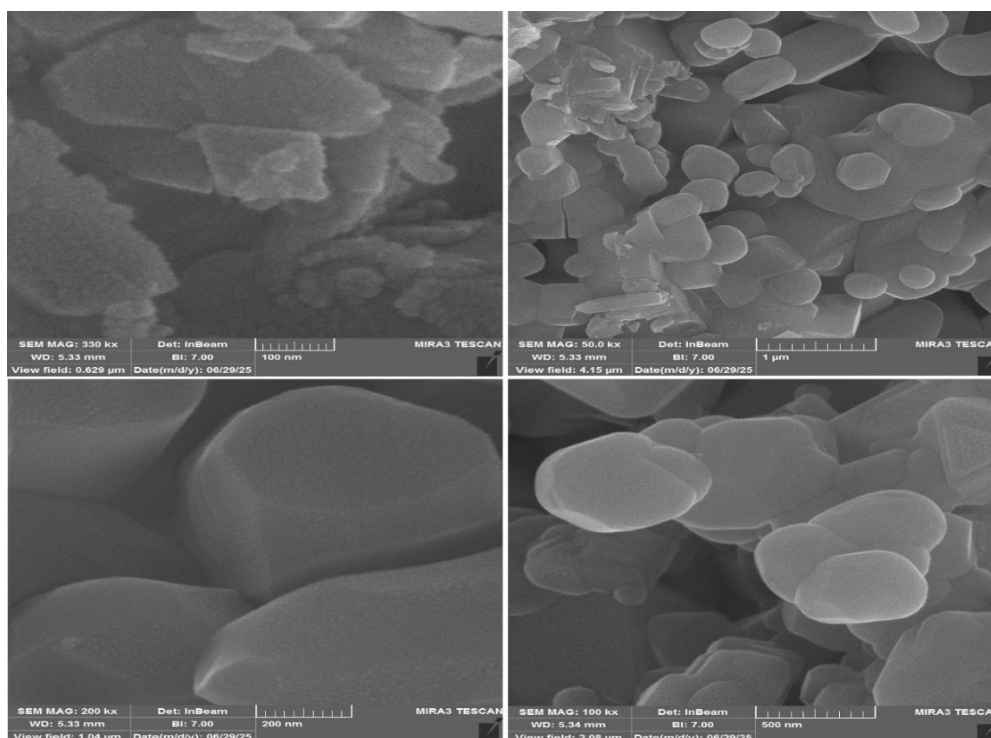


Figure 4. Representative FESEM micrographs of the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ nanocomposite at different magnifications, illustrating aggregated quasi-spherical nanoparticles with sizes in the 50–200 nm range and evidence of good phase integration.

At higher magnifications (Figure 4), the surface features of the larger grains show smooth facets decorated with well dispersed, smaller bright nanoparticles and distinct nanoscale protrusions. These bright, contrasting spots are attributed to the metallic Ag nanoparticles and localized ZnTiO_3 domains distributed uniformly along the grain boundaries of the CoFe_2O_4 core. Due to the higher atomic number and superior electrical conductivity of metallic silver, these domains exhibit higher secondary electron emission, appearing as bright spots under the electron beam. The close interfacial contact and the absence of phase segregation or microcracking confirm strong physical adhesion and structural integrity post-synthesis. From a catalytic perspective, the nanoscale dimensions and the resulting surface roughness enhance the active

surface area and promote pollutant adsorption. Although the localized aggregation slightly restricts the free dispersibility of the catalyst in the aqueous suspension, this effect is heavily mitigated by the material's excellent magnetic recoverability, preventing any significant loss of active catalyst during practical wastewater treatment.

3.1.5. Energy-Dispersive X-ray Spectroscopy (EDX) Analysis

Energy-dispersive X-ray spectroscopy (EDX) coupled with FESEM was employed to determine the elemental composition, evaluate chemical purity, and confirm the presence of the constituent elements within the synthesized $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ ternary nanocomposite. The obtained EDX spectrum is presented in Figure 5, and the corresponding quantitative elemental results are summarized in Table 1.

The EDX spectrum (Figure 5) clearly identifies the characteristic X-ray emission lines corresponding to all the targeted elements: titanium (Ti $K\alpha$, Ti $K\beta$), iron (Fe $K\alpha$, Fe $K\beta$), cobalt (Co $K\alpha$, Co $K\beta$), zinc (Zn $K\alpha$, Zn $K\beta$), and silver (Ag $L\alpha$, Ag $L\beta$). Crucially, no additional peaks corresponding to foreign impurities or precursors were detected in the spectrum. This complete absence of impurity signals indicates the high chemical purity and successful calcination of the ternary nanocomposite at 700 °C. The prominent cobalt and iron signals are closely consistent with the dominant CoFe_2O_4 spinel phase, while the simultaneous presence of zinc, titanium, and silver signals confirms the successful integration of the ZnTiO_3 and metallic Ag components within the composite matrix.

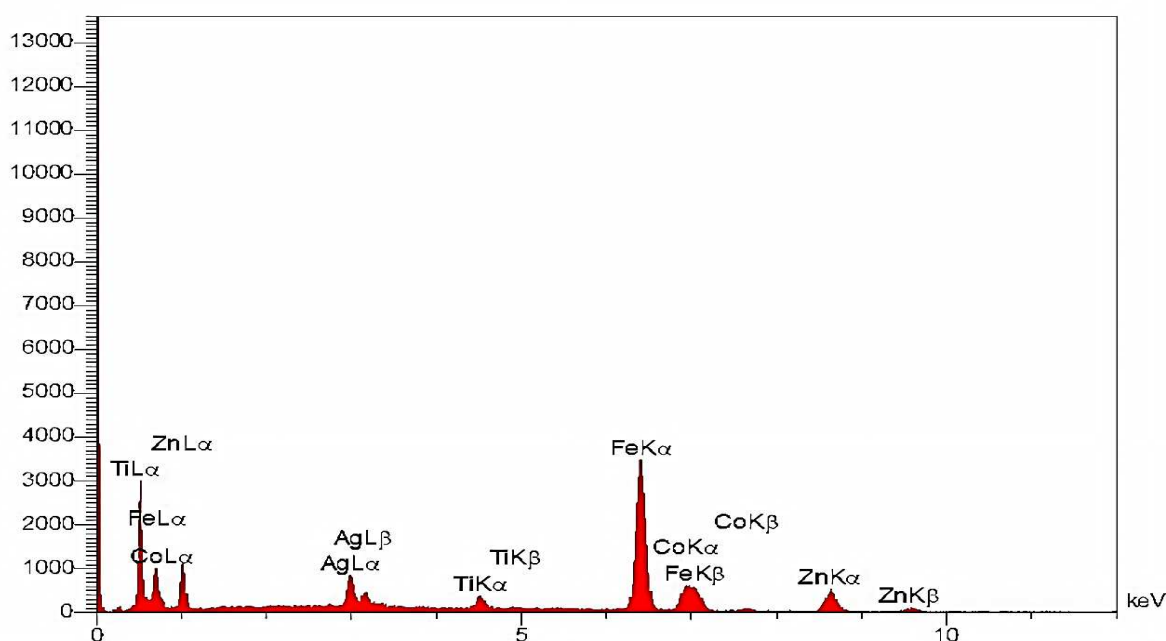


Figure 5. EDX spectrum of the CoFe₂O₄/ZnTiO₃/Ag nanocomposite, showing characteristic X-ray peaks of Co, Fe, Zn, Ti, and Ag.

The quantitative results presented in Table 1 confirm that iron is the dominant metallic element, contributing 57.46 wt% (62.00 at%), which reflects the iron-rich nature of the magnetic CoFe₂O₄ core. Cobalt contributed 11.33 wt% (11.59 at%), which is consistent with the expected stoichiometric Co:Fe atomic ratio of approximately 1:2 in the pure spinel cobalt ferrite core phase. Zinc and titanium accounted for 12.12 wt% (11.18 at%) and 6.56 wt% (8.25 at%), respectively. The lower titanium content compared to zinc is due to the dilution of the 1:1 Zn:Ti stoichiometry of ZnTiO₃ by the much heavier surrounding cobalt ferrite matrix. Silver was detected at 12.53 wt% (7.00 at%), confirming the successful deposition and incorporation of metallic silver; its atomic percentage is notably lower than its weight percentage due to the high atomic mass of silver.

Table 1. Quantitative EDX elemental composition of the CoFe₂O₄/ZnTiO₃/Ag nanocomposite (ZAF-corrected).

Elt	Lin	Int	W%	A%	ZAF	Pk/Bg	Clas	LCon	HConf
	e						s	f	
Ti	Ka	50.8	6.56	8.25	0.9908	29.69	A	6.32	6.79
Fe	Ka	409.9	57.46	62.00	1.0023	215.91	A	56.73	58.19
Co	Ka	78.2	11.33	11.59	0.9932	41.82	A	11.00	11.66
Zn	Ka	71.9	12.12	11.18	0.9210	33.35	A	11.76	12.49
Ag	La	93.2	12.53	7.00	0.8773	78.57	A	12.19	12.86
			100.00	100.00					

The slight deviations from the ideal bulk stoichiometric ratios within the ternary composite are fully anticipated in such multi-component nanostructures, where surface enrichment of specific phases, minor volatilization during high-temperature calcination, or incomplete precursor distribution during step-by-step precipitation can occur. The ZAF-corrected quantification yields particularly reliable values, with ZAF factors close to 1.0 for most elements, indicating negligible matrix effects. Overall, the EDX data provide robust evidence for the successful synthesis of the target CoFe₂O₄/ZnTiO₃/Ag ternary nanocomposite, supporting the structural, physical, and functional characteristics observed in the XRD, FTIR, and FESEM analyses.

3.2. Influence of Operational Parameters on BPA Degradation

3.2.1. Effect of Catalyst Dosage

The dosage of the heterogeneous catalyst plays a pivotal role in advanced oxidation processes by determining the total available surface area and the density of active catalytic sites. The effect of different CoFe₂O₄/ZnTiO₃/Ag catalyst dosages (25, 50, and 75 mg) on BPA removal efficiency was systematically evaluated over a 240 min reaction period, with the corresponding response surface illustration presented in Figure 6.

To establish statistical validity, a one-way analysis of variance (ANOVA) was performed, and the descriptive statistics are summarized in Table 2. The ANOVA results (F-statistic=14.503, p-value=0.002<0.05) confirm highly significant differences in the mean BPA removal efficiencies across the evaluated dosage levels. Specifically, the 50 mg dosage yielded the highest mean removal efficiency of 65.22% (with a 95% confidence interval ranging from 58.45% to 71.98% and a standard deviation of 2.93%), achieving a maximum final removal efficiency of 91.30% at 240 min. In contrast, the lowest mean removal was recorded at the minimum dosage of 25 mg (43.91%; 95% CI:37.15%–50.68%). Increasing the dosage beyond the optimum to 75 mg resulted in a decline in the mean removal efficiency to 60.44% (95% CI:53.67%–67.20%), indicating a characteristic non-linear, peak-shaped response.

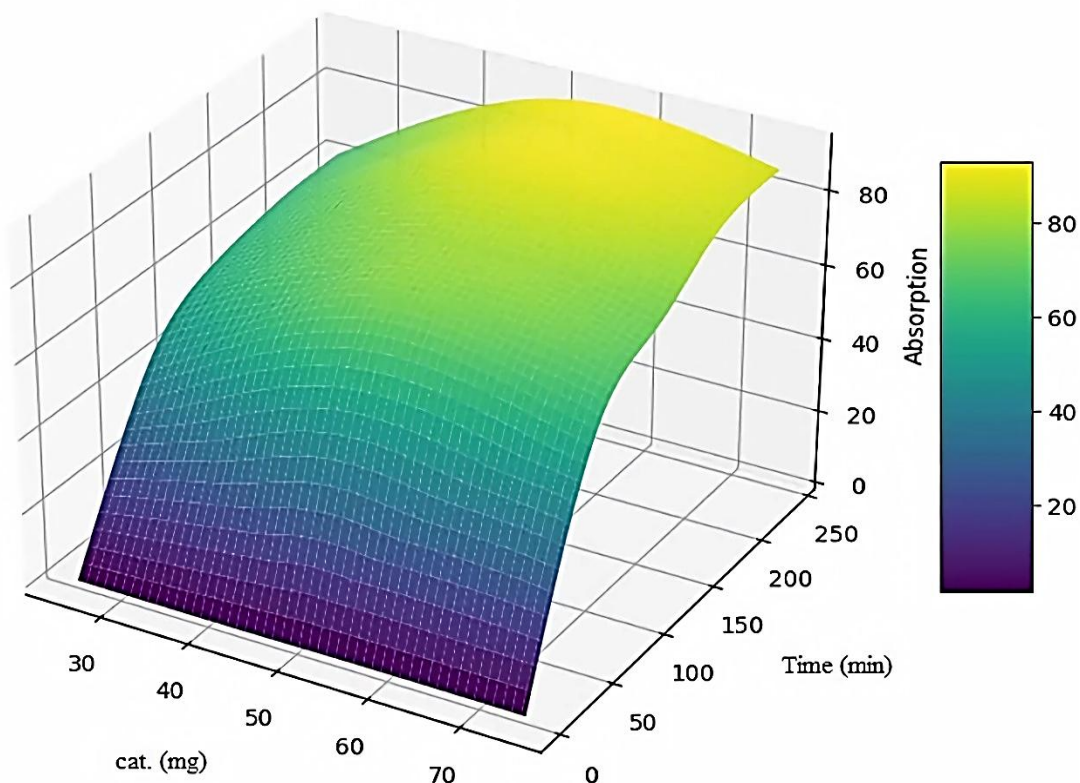


Figure 6. Effect of different catalyst dosages on Bisphenol A removal in the photocatalytic ozonation process

Table 2. Descriptive statistics of Bisphenol A removal percentage at different dosage levels of the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ catalyst.

Dosage (mg)	Mean (%)	Standard Error (SD)	95% Confidence Interval (Lower Bound)	95% Confidence Interval (Upper Bound)	F-statistic	p-value
25	43.914	2.935	37.146	50.682	14.503	0.002
50	65.216	2.935	58.448	71.984	—	—
75	60.436	2.935	53.668	67.204	—	—

The observed increase in BPA degradation when escalating the catalyst dosage from 25 to 50 mg is attributed to the enhanced availability of active surface sites and the corresponding increase in photogenerated charge carriers (e^-h^+ pairs) under UV irradiation. These active sites promote robust interactions between the catalyst surface groups, ozone molecules, and adsorbed BPA molecules, thereby accelerating the generation of highly reactive oxygen species (ROS). However, the slight decline in efficiency observed at 75 mg is a well-documented phenomenon in heterogeneous photocatalysis, primarily driven by two antagonistic effects. First, an excessive concentration of suspended particles increases the turbidity of the solution, leading to a "light-shielding effect" that scatters and blocks light penetration into the reactor core, thereby limiting the photo-excitation of the catalyst. Second, higher particle densities can accelerate the collision and recombination of photogenerated charge carriers and scavenge active radicals, ultimately reducing the net concentration of reactive $\cdot\text{OH}$ and superoxide ($\text{O}_2^{\bullet-}$) radicals. These findings align with previous investigations by Liu et al. [30] and Kanwal et al. [31], which established that optimal catalyst loading is critical to balancing active site availability against light-scattering limitations.

3.2.2. Effect of Solution pH

The pH of the aqueous medium is a critical parameter influencing the surface charge of the heterogeneous catalyst, the ionization state of the target pollutant molecules, and the pathways of ozone decomposition. The impact of the initial solution pH on BPA removal was systematically evaluated across five levels (4, 6, 7, 9, and 10) under optimized catalyst loading, as illustrated in the 3D response surface plot in Figure 7.

The corresponding statistical parameters and one-way ANOVA results are summarized in Table 3. The highly significant F-value of 12.839 and $p\text{-value} = 0.001 < 0.05$ confirm that the solution pH exerts a statistically decisive influence on the mean BPA removal percentages. The data reveal a distinct optimal performance at neutral pH (pH 7.0), achieving the highest mean removal of 65.22% (95% CI: 59.38%–71.06%) and a peak degradation efficiency of 91.30% after 240 min. In contrast, strongly acidic conditions (pH 4.0) yielded the poorest performance, with a mean removal of only 40.65% (95% CI: 34.81%–46.49%). Intermediate but statistically lower removal efficiencies were recorded in both acidic-to-neutral transition (pH 6.0; 43.91%) and alkaline regimes (pH 9.0, 55.00%; and pH 10.0, 46.96%).

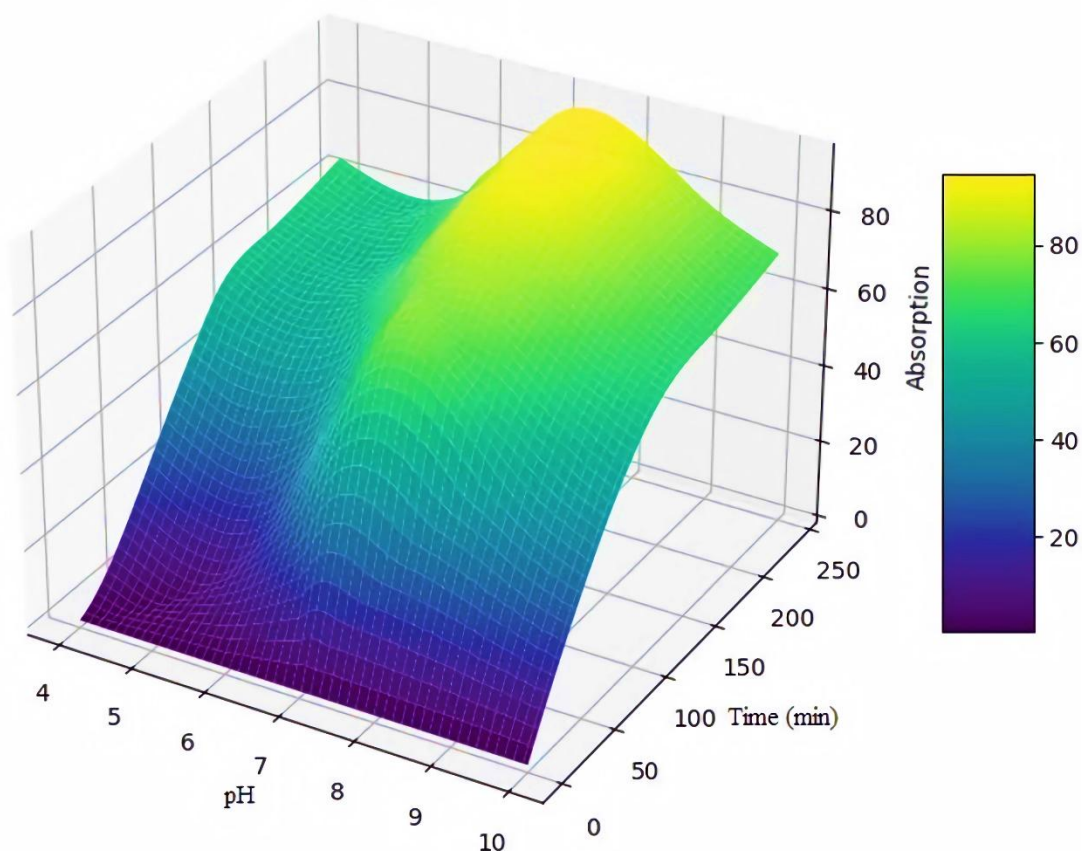


Figure 7. Effect of different pH levels on Bisphenol A removal in the photocatalytic ozonation process.

Table 3 presents the descriptive statistics and one-way ANOVA results ($F = 12.839$, $p = 0.001$) for Bisphenol A (BPA) removal percentages across five pH levels (4, 6, 7, 9, and 10) in the photocatalytic ozonation process with the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ nanocomposite. The highly significant p-value confirms clear differences in mean removal efficiencies among the pH conditions, with a consistent standard error of 2.754% and reported 95% confidence intervals.

Table 3. Descriptive statistics of Bisphenol A removal percentage at different pH levels using the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ catalyst.

pH	Mean (%)	Standard Error (SE)	95% Confidence Interval (Lower Bound)	95% Confidence Interval (Upper Bound)	F-statistic	p-value
4	40.652	2.754	34.813	46.491	12.839	0.001
6	43.914	2.754	38.075	49.753	—	—

7	65.216	2.754	59.377	71.055	—	—
9	55.002	2.754	49.163	60.841	—	—
10	46.956	2.754	41.117	52.795	—	—

The influence of solution pH on the degradation process can be mechanistically interpreted through its effects on the catalyst's surface charge, the aggregation of the nanoparticles, and the mechanism of ozonation. At highly acidic conditions (pH 4.0), the catalyst surface becomes highly protonated and positively charged, which can alter the electrostatic adsorption of BPA. Additionally, under acidic conditions, ozone molecules directly attack target organic compounds via slow and highly selective electrophilic pathways, resulting in lower overall degradation rates. Conversely, in alkaline media (pH 9.0 and 10.0), ozone rapidly decomposes into highly reactive, non-selective hydroxyl radicals ($\cdot\text{OH}$) via an indirect radical-based mechanism. While this indirect pathway generates more $\cdot\text{OH}$ radicals, excessively alkaline conditions can negatively affect the catalyst surface charge (deprotonation) and cause electrostatic repulsion of the ionized BPA molecules ($\text{pK}_a \approx 9.6\text{--}10.2$), thereby restricting interfacial reaction steps. Furthermore, higher pH levels can accelerate the auto decomposition of ozone before it can interact effectively with the catalyst surface. Therefore, near-neutral conditions (pH 7.0) optimize the synergistic interplay between direct ozonation, indirect radical oxidation, and surface-catalyzed reactions, facilitating maximum pollutant-catalyst contact without electrostatic repulsion or premature ozone loss. This neutral pH optimum is closely consistent with findings reported by Lawan et al.[32] and Wang et al. [33], who documented similar peak degradation efficiencies for phenolic compounds under neutral pH conditions due to optimized radical generation and balanced electrostatic interactions.

3.2.3. Effect of Initial BPA Concentration

The concentration of the target contaminant in the influent is a crucial factor that dictates the treatment capacity and efficiency of heterogeneous advanced oxidation systems. The influence of the initial BPA concentration on degradation efficiency was systematically investigated at three levels: 10, 30, and 50 mg/L, with the corresponding 3D response surface plot displayed in Figure 8.

The one-way ANOVA and descriptive statistics for this parameter are presented in Table 4. The statistical analysis confirms highly significant differences among the concentration groups (F-

statistic=13.770, p-value=0.003<0.05), with a uniform standard error of 3.57%. The data demonstrate a clear, statistically significant inverse relationship between the initial BPA concentration and its removal efficiency. The highest mean removal efficiency of 65.22% (with a 95% confidence interval of 56.99% to 73.44%) was achieved at the lowest tested concentration of 10 mg/L, resulting in a maximum final degradation efficiency of 91.30% after 240 min. As the initial concentration was increased to 30 mg/L, the mean removal dropped to 51.14% (95% CI:42.91%–59.37%), and further decreased to 38.76% (95% CI:30.53%–46.99%) at the highest concentration of 50 mg/L (representing a final degradation of 52.60% at 240 min).

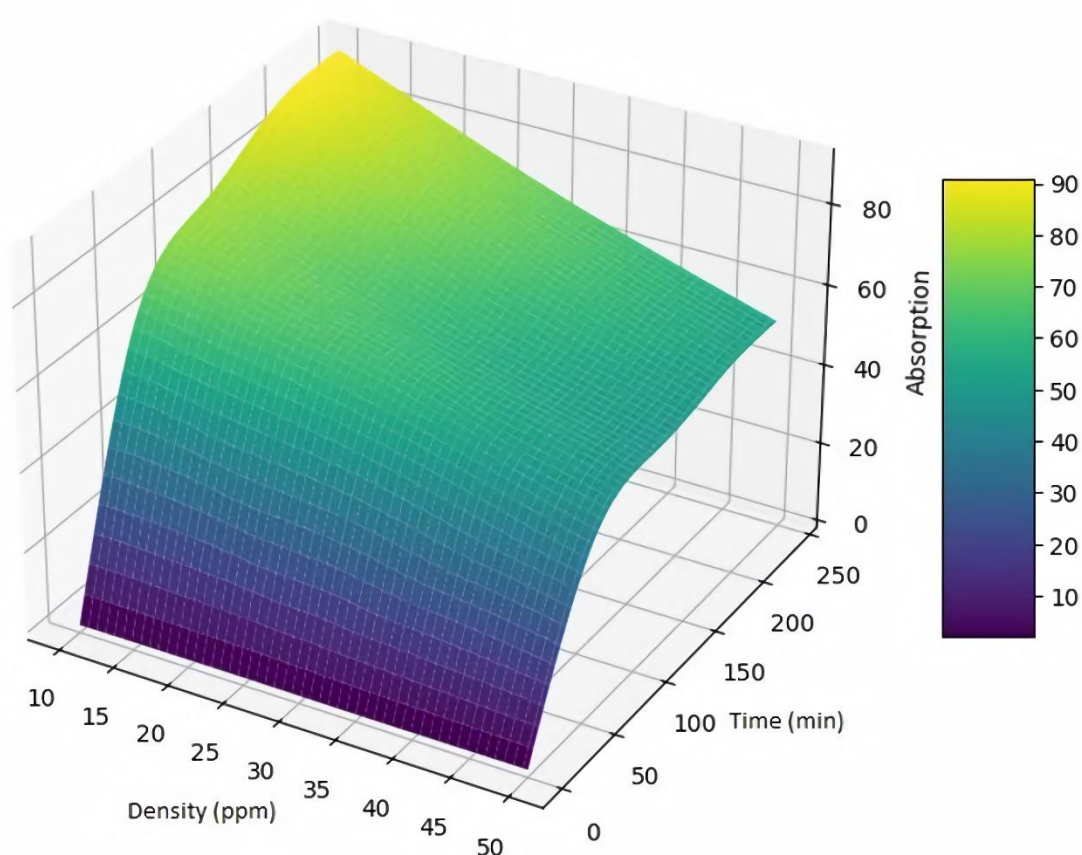


Figure 8. Effect of different initial Bisphenol A concentrations in the photocatalytic ozonation process.

Table 4-10 summarizes the descriptive statistics and one-way ANOVA results for Bisphenol A (BPA) removal percentages at three initial concentrations (10, 30, and 50 mg/L) in the photocatalytic ozonation process using the CoFe₂O₄/ZnTiO₃/Ag nanocomposite. The ANOVA confirms highly significant differences among the groups ($F = 13.770$, $p = 0.003 < 0.05$), with a uniform standard error of 3.567% across all levels.

Table 4. Descriptive statistics of Bisphenol A removal percentage at different levels of initial pollutant concentration.

Concentration (ppm)	Mean (%)	Standard Error (SE)	95% Confidence Interval (Lower Bound)	95% Confidence Interval (Upper Bound)	F-statistic	p-value
10	65.216	3.567	56.990	73.442	13.770	0.003
30	51.140	3.567	42.914	59.366	—	—
50	38.760	3.567	30.534	46.986	—	—

This statistically meaningful inverse trend, characterized by minimal overlap in the 95% confidence intervals between 10 and 50 mg/L, is mechanistically anticipated in heterogeneous photocatalytic ozonation processes. At lower initial concentrations (10 mg/L), the number of BPA molecules is relatively small compared to the abundance of active catalytic sites available on the CoFe₂O₄/ZnTiO₃/Ag surface. This favorable ratio facilitates rapid and unhindered adsorption of BPA molecules and their subsequent immediate oxidation by continuously generated hydroxyl radicals ($\cdot\text{OH}$) and ozone. However, as the initial concentration escalates to 50 mg/L, the target pollutant molecules and their intermediate degradation products (such as low-molecular-weight organic acids and phenolic intermediates) increasingly saturate the available active catalytic sites. This leads to a severe competitive adsorption effect on the catalyst's surface, where intermediates block active sites and scavenge photogenerated charge carriers, thereby lowering the overall degradation rate of parent BPA molecules. These findings indicate that the integrated system performs exceptionally well for low-to-trace level concentrations of BPA (≤ 10 mg/L), which is highly relevant for practical treatment scenarios since BPA is typically detected at trace-to-low levels in actual municipal and industrial wastewater effluents.

3.2.4. Effect of Reaction Time

To optimize the operational and energy efficiency of wastewater treatment processes, evaluating the influence of reaction time is of paramount importance. The progress of BPA degradation as a function of reaction time (0, 60, 120, 180, and 240 min) under optimized conditions (pH 7.0, 50 mg catalyst, and 10 mg/L initial BPA concentration) is plotted in Figure 9.

The ANOVA results validating the statistical significance of reaction time are presented in Table 5. The highly significant F-value of =8.645 and p-value= 0.001<0.05 confirm that the duration of the advanced oxidation treatment has a statistically decisive impact on the achieved removal yield. The results show a continuous, monotonic increase in BPA removal as the reaction progressed from 0 to 240 min. The maximum removal efficiency of 91.30% was achieved at the terminal time of 240 min.

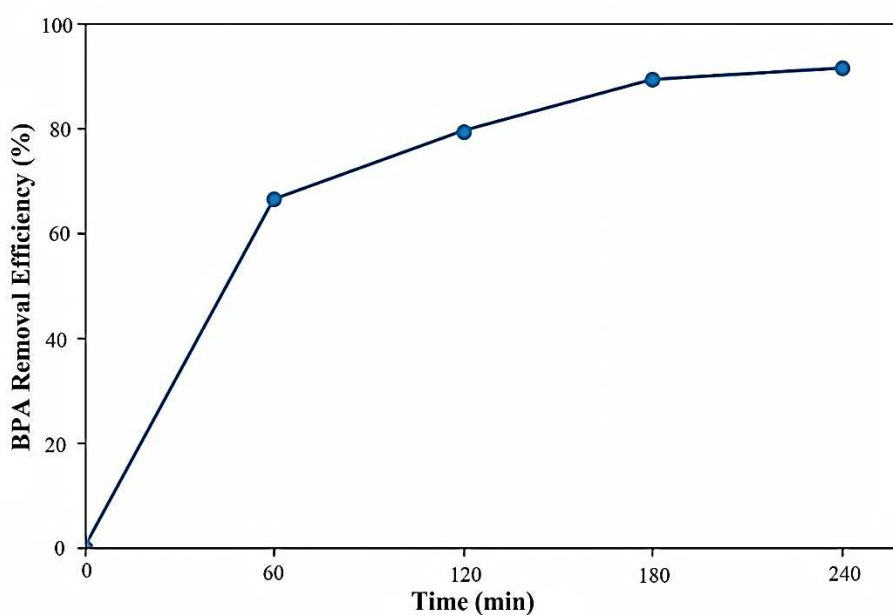


Figure 9. Effect of reaction time on Bisphenol A removal in the photocatalytic ozonation process.

Table 5. ANOVA test results for the effect of reaction time on Bisphenol A removal.

Source of Variation	Sum of Squares	Degrees of Freedom (df)	Mean Square	F-statistic	p-value
Time (Between Groups)	16797.742	4	4199.435	8.645	0.001
Error (Within Groups)	13115.102	27	485.745	—	—

Total	29912.844	31	—	—	—
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The consistent increase in degradation with longer exposure times is directly related to the continuous, time-dependent accumulation of active oxidants in the reactor. Under constant UV illumination and continuous ozone flow (0.2 g/h), longer contact times allow for a higher total cumulative yield of photogenerated electrons/holes and a greater rate of catalytic ozone decomposition into $\cdot\text{OH}$ radicals. This continuous radical generation ensures sustained oxidative attack on both the remaining parent BPA molecules and their primary degradation intermediates, leading to progressive and thorough pollutant degradation over the 240 min treatment period.

3.3.2. Effect of Solar Light Irradiation on Photocatalytic Ozonation

To evaluate the applicability of the integrated system under natural light conditions and assess its dependence on external light sources, the photocatalytic ozonation efficiency of the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ nanocomposite was comparatively investigated in the presence and absence of natural solar light irradiation. The degradation curves of BPA over a 240 min reaction period under both conditions are presented in Figure 10. To statistically evaluate the significance of solar light on the degradation efficiency and catalyst adsorption capacity, an independent-samples t-test was performed, with the statistical results compiled in Table 6. The Levene's test for equality of variances showed no significant difference between the variances ($F=0.001$, $p=0.970>0.05$). Crucially, the t-test yielded a highly non-significant p-value ($p=0.995>0.05$, with a negligible mean difference of -0.132%), statistically confirming that the presence of natural sunlight does not introduce any significant difference in either the BPA removal efficiency or the catalyst's adsorption capacity.

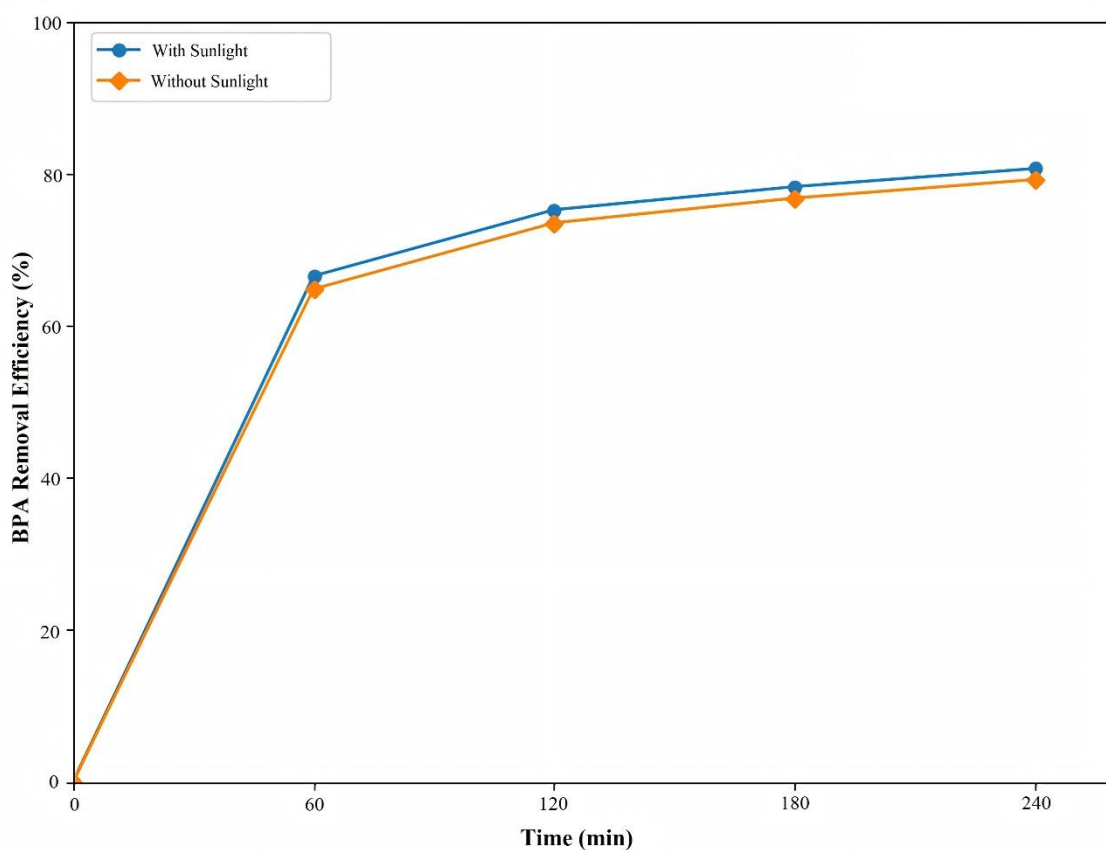


Figure 10. Effect of photocatalytic ozonation in the presence of $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ nanocomposite on Bisphenol A removal under sunlight.

Table 6. Independent t-test results for the comparison of BPA removal percentage and adsorption capacity in the presence and absence of natural sunlight.

Variable	Levene's Test (F)	Leven e's Test (Sig.)	t-value	df	Sig. (2-tailed)	Mean Difference	95% Confidence Interval (Lower Bound)	95% Confidence Interval (Upper Bound)
Removal Percentage (%)	0.001	0.970	-0.006	8	0.995	-0.132	-49.723	49.459
Adsorption Capacity (qe, mg/g)	0.001	0.970	0.006	8	0.995	0.0012	-0.455	0.457

As shown in Figure 10, the degradation profile of BPA in the presence of natural sunlight (achieving ~81.3% removal) closely mirrors that obtained in the absence of sunlight

(~80.1% removal). This lack of statistically significant difference can be interpreted through two distinct scientific perspectives. First, from an operational standpoint, the high-power 400 W metal halide lamp utilized in the experimental setup provides a highly intense and saturated flux of UV-C and visible light. This intense artificial irradiation already maximizes the photo-excitation of the semiconductor components (ZnTiO_3 and the CoFe_2O_4 magnetic core), saturating the generation rate of photogenerated charge carriers (e^-h^+ pairs) and active $\cdot\text{OH}$ radicals. Consequently, the addition of natural solar light does not further enhance the degradation rate, as the catalytic active sites are already operating at their maximum capacity. Second, from an environmental engineering perspective, this result highlights the exceptional stability and reliability of the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ photocatalytic ozonation system. It demonstrates that the process performance remains particularly robust and unaffected by diurnal cycles, weather fluctuations, or cloud cover, making it a highly reliable and consistent technology for continuous, year-round municipal or industrial wastewater treatment.

3.3.3. Comparative Process Efficiency and Synergy Analysis

To quantitatively evaluate the necessity and synergistic interaction of the individual process components, BPA degradation was comparatively investigated under five distinct treatment configurations: (i) nanocomposite adsorption alone, (ii) UV-C photolysis alone, (iii) ozonation (O_3) alone, (iv) combined UV-C/ O_3 photolytic ozonation, and (v) the integrated UV-C/ O_3 /Nanocomposite photocatalytic ozonation process. The comparative degradation curves over the 240 min reaction period are presented in Figure 11.

The multi-factor ANOVA test results evaluating the significance and contribution of each individual component are summarized in Table 7. The statistical model is highly significant (F-statistic=20.570, p-value=0.001<0.05). Among the individual parameters, ozone was identified as the most dominant single factor influencing the system (F-statistic=126.874, p-value=0.001), followed closely by the introduction of the nanocomposite catalyst (F=47.105, p=0.001) and UV-C irradiation (F=46.135, p=0.001).

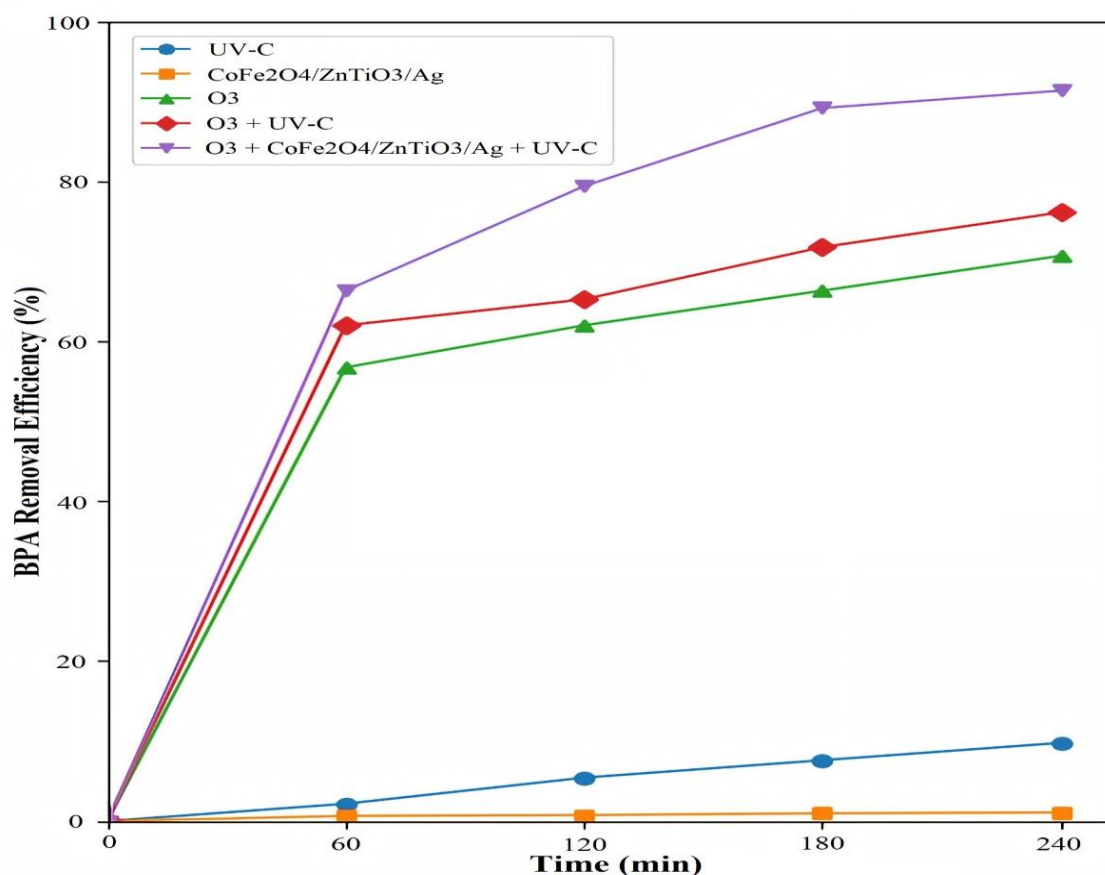


Figure 11. Comparison of the efficiency of ozonation, UV-C radiation, nanocomposite, and UV-C/O₃ in Bisphenol A removal.

Table 7. ANOVA test results for the comparison of different treatment conditions and individual process components.

Source of Variation	Sum of Squares	Degrees of Freedom (df)	Mean Square	F-statistic	p-value
Corrected Model	42556.581	13	3273.583	20.570	0.001
Time	14614.227	4	3653.557	22.958	0.001
Nanocomposite	7496.414	1	7496.414	47.105	0.001
UV-C	7342.071	1	7342.071	46.135	0.001
Ozone	20190.874	1	20190.874	126.874	0.001
Error	3341.971	21	159.141	—	—
Total	73504.651	35	—	—	—

As shown in Figure 11, the adsorptive removal of BPA by the CoFe₂O₄/ZnTiO₃/Ag nanocomposite alone (in the dark) was minimal and virtually

negligible, indicating that physical adsorption is not the primary pathway for BPA elimination. Similarly, UV-C photolysis alone yielded extremely low removal (9.78% at 240 min), suggesting that direct photolytic cleavage of the stable BPA ring structure is extremely inefficient. Ozonation alone (O₃) and the photolytic UV-C/O₃ process achieved intermediate removal efficiencies of 70.65% and 76.09%, respectively. Notably, the integrated photocatalytic ozonation system (UV-C/O₃/Nanocomposite) achieved the highest degradation efficiency of 91.30% within 240 min, demonstrating a considerable synergistic effect that substantially outpaces the sum of the individual processes.

To statistically confirm this synergy, the interaction effects between the different components were analyzed, and the results are compiled in Table 8. The interaction of the Nanocomposite with Ozone (F=24.289, p=0.001) and the interaction of UV with Ozone (F=34.573, p=0.001) were both highly significant, validating that the combination of these factors creates a powerful, cooperative chemical environment.

Table 8. Significant interaction effects of ozone, UV-C, nanocomposite, and reaction time on BPA removal.

Interaction Effect	Degrees of Freedom (df)	F-statistic	p-value
Nanocomposite × Ozone	1	24.289	0.001
UV-C × Ozone	1	34.573	0.001
Time × Ozone	4	4.231	0.011

Mechanistically, the exceptionally significant synergy observed in the integrated UV-C/O₃/Nanocomposite system is driven by the robust interfacial interactions and sequential electron-transfer pathways between the ternary catalyst and ozone molecules [34]. Upon UV-C excitation of the semiconductor components (ZnTiO₃ and the CoFe₂O₄ magnetic core), photogenerated electrons (e⁻) are promoted to the conduction band, leaving holes (h⁺) in the valence band. Within this heterostructure, the metallic Ag nanoparticles governed by their localized surface plasmon resonance and the formation of a Schottky barrier at the metal-semiconductor interface serve as an ideal electron sink. This configuration dramatically accelerates interfacial charge separation and suppresses the undesirable recombination of photogenerated e⁻-h⁺ pairs [35,36].

Concurrently, dissolved ozone (O_3) molecules, acting as highly electrophilic electron acceptors, rapidly capture the Ag-channeled surface electrons. By scavenging these photogenerated electrons, ozone rapidly decomposes into active ozonide radicals ($O_3^{\cdot-}$), which subsequently undergo protonation in the aqueous medium to yield an abundance of highly reactive, non-selective hydroxyl radicals ($\cdot OH$) [14,37]. This continuous electron-scavenging pathway not only ensures a high-yield generation of $\cdot OH$ radicals but also leaves a larger fraction of valence band holes free to directly oxidize water molecules or adsorbed organic species. Ultimately, this cascade of cooperative reactions, facilitated by the dual role of Ag (charge transfer mediator) and ozone (electron acceptor and radical propagator), yields a considerably higher concentration of active oxidants compared to single processes, driving the rapid and thorough mineralization of BPA.

3.4. Chemical Oxygen Demand (COD) Removal and Mineralization

While the degradation of parent pollutant molecules is important, the ultimate goal of advanced oxidation processes is the thorough mineralization of the contaminant into harmless inorganic end-products (CO_2 and H_2O), thereby preventing the accumulation of toxic intermediate degradation byproducts. Therefore, the mineralizing capability of the integrated UV-C/ O_3 /Nanocomposite process was evaluated by monitoring the reduction of Chemical Oxygen Demand (COD) over a 240 min treatment period under optimized conditions, as illustrated in Figure 12.

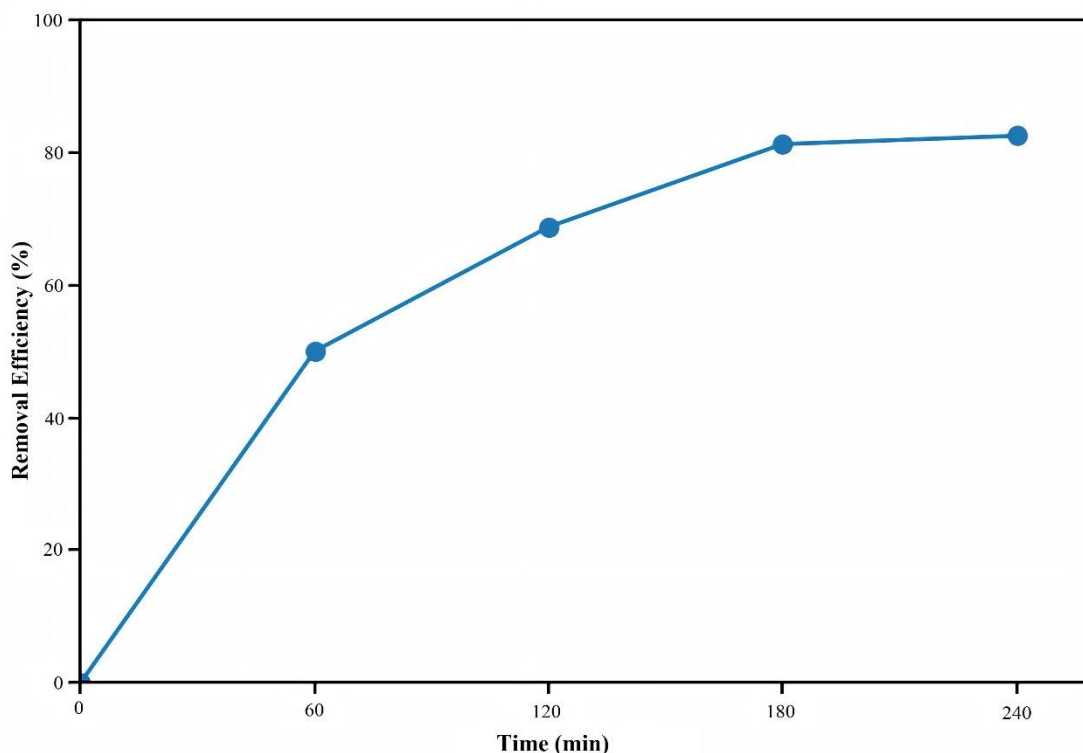


Figure 12. COD removal percentage in the photocatalytic ozonation process.

As depicted in Figure 12, the COD removal percentage increased progressively with longer reaction times, achieving a maximum mineralization efficiency of 82.5% within 240 min. The high COD removal efficiency (82.5%) closely mirrors the high parent BPA degradation efficiency (91.30%), indicating that the integrated system not only cleaves the initial phenolic structure of BPA but also successfully mineralizes the resulting intermediate compounds.

The slight gap between parent molecule degradation and COD reduction is a common phenomenon in advanced oxidation, representing the time-lag required to break down more stable aliphatic and aromatic intermediates (such as quinones, aldehydes, and low-molecular-weight dicarboxylic acids) into CO_2 and H_2O . As noted by Garoma et al. [38], the slight deceleration in COD removal in the advanced stages of treatment can be attributed to the temporary formation of viscous, gel-like reaction intermediates and organic byproducts that increase the mass transfer resistance of ozone in the liquid phase, thereby slightly limiting the rate of ozone diffusion and direct radical interaction at later stages of the process. Nevertheless, the achieved mineralization level of 82.5% demonstrates the exceptional potential of the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ -catalyzed photocatalytic ozonation process for thorough and environmentally safe water remediation.

3.5. Catalyst Reusability and Stability Analysis

From an environmental and industrial perspective, the stability, reusability, and recovery efficiency of a heterogeneous catalyst are critical parameters that determine its economic feasibility and long-term sustainability for continuous water treatment. In this study, the reusability of the synthesized $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ ternary nanocomposite was thoroughly evaluated under optimized reaction conditions (pH 7.0, 50 mg catalyst, and 10 mg/L initial BPA concentration) over seven consecutive treatment cycles. The residual BPA removal efficiency at the end of each 240 min cycle is presented in Figure 13.

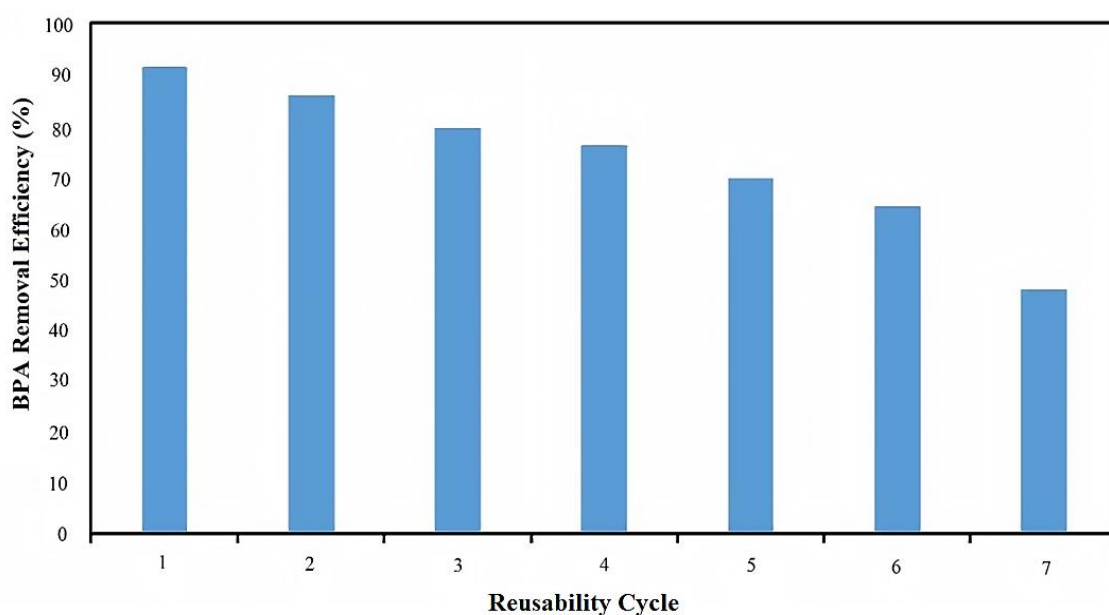


Figure 13. Reusability efficiency of the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ nanocomposite.

As illustrated in Figure 13, the BPA degradation efficiency decreased progressively from 91.30% in the first cycle to 47.83% in the seventh consecutive run. This gradual decline in photocatalytic ozonation efficiency is a common phenomenon in heterogeneous advanced oxidation, primarily driven by the progressive adsorption of recalcitrant organic intermediates and carboxylic acids on the active sites of the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}$ surface. This decline may be attributed to surface fouling by reaction intermediates, which could partially hinder light absorption and ozone transfer to active sites[39]. Additionally, minor physical loss of the catalyst nanoparticles during magnetic recovery, washing, and thermal drying at 60 °C reduces the active catalyst concentration in subsequent cycles, while minor pore clogging or slight leaching/oxidation of

the active Ag sites may further decrease the synergistic charge-separation capacity over time. Although the catalyst retained measurable activity after seven cycles, a substantial decline in degradation efficiency was observed, indicating the need for regeneration strategies. Crucially, its soft ferromagnetic properties enabled rapid magnetic separation within seconds using an external magnet, eliminating the need for energy-intensive centrifugation or membrane filtration. This confirms that the CoFe₂O₄/ZnTiO₃/Ag nanocomposite remains a particularly promising, stable, and magnetically recoverable catalyst for sustainable wastewater treatment.

3.6. Identification of BPA Degradation Intermediates and Environmental Implications

To elucidate the progressive oxidation pathways of BPA during the synergistic CoFe₂O₄/ZnTiO₃/Ag-catalyzed photocatalytic ozonation, intermediate compounds were identified using GC/MS (Figure 14).

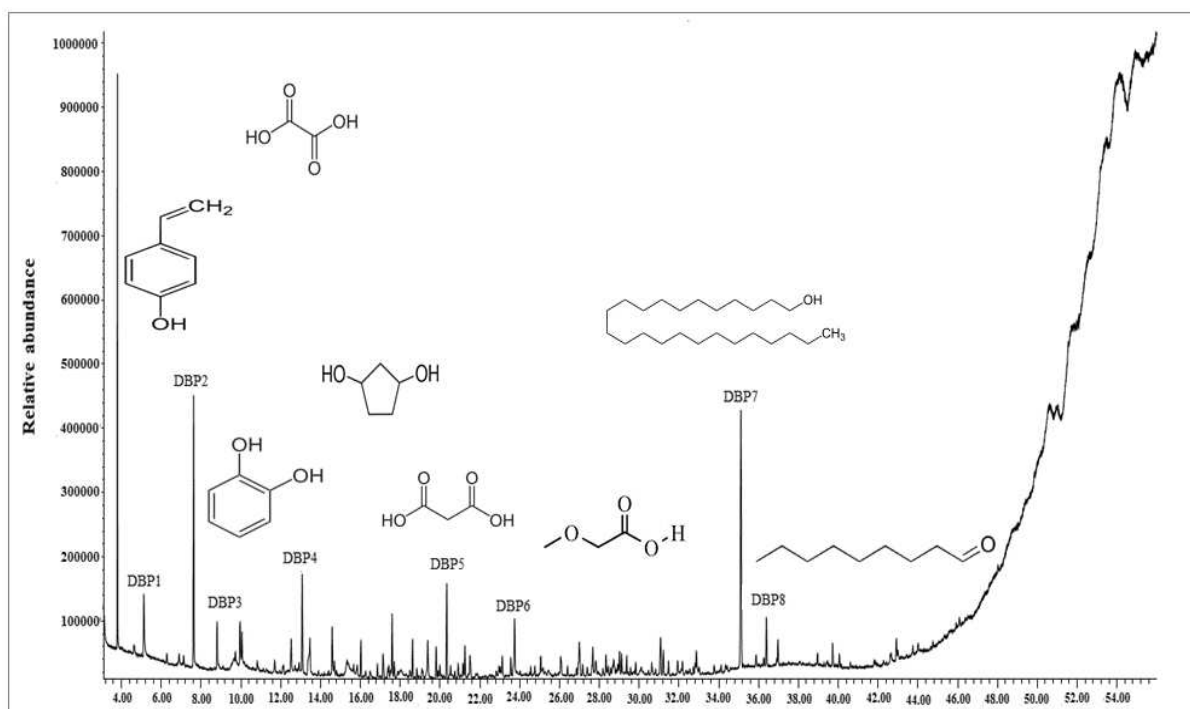


Figure 14. Identification of intermediate products from Bisphenol A degradation via photocatalytic ozonation analyzed by GC-MS.

Ten major degradation products were successfully characterized, and their physicochemical properties and toxicological profiles are summarized in Table 9.

Table 9. Chemical characteristics and toxicological profiles of the identified intermediates formed during the photocatalytic ozonation of BPA.

No.	Compound Name (Formula / MW)	Toxicological and Environmental Health Profiles	References
1	4-Vinyl-phenol (C ₈ H ₈ O/ 120 g/mol)	Moderate systemic toxicity, significant skin and eye irritation potential, and adverse effects on aquatic organisms at high environmental concentrations.	[40,41]
2	3-Methyl-2,3-dihydro-1-benzofuran (C ₉ H ₁₀ O/ 134 g/mol)	potential to induce mild respiratory and dermal irritation, while exhibiting relatively low persistence in the environment.	[42,43]
3	Ethanedioic acid (C ₂ H ₂ O ₄ / 90 g/mol)	Corrosive to tissues; harmful upon oral exposure at high doses; low ecotoxicity with limited environmental persistence.	[44,45]
4	1,2-Dihydroxy benzene (C ₆ H ₆ O ₂ / 110 g/mol)	Moderate acute toxicity; causes serious skin and eye irritation; highly toxic to aquatic organisms with potential long-term environmental effects.	[46,47]
5	Nonanal (C ₉ H ₁₈ O/ 142 g/mol)	May cause mild skin and eye irritation; readily biodegradable in aquatic environments; exhibits low acute toxicity toward aquatic organisms.	[42,48]
6	n-Tetracosanol-1 (C ₂₄ H ₅₀ O/ 354 g/mol)	Exhibits very low acute toxicity; readily biodegradable under environmental conditions; no significant adverse effects on human health or aquatic ecosystems have been reported.	[49,50]

7	Malonic acid (C ₃ H ₄ O ₄ / 104 g/mol)	May cause mild skin and eye irritation upon direct exposure; exhibits low aquatic toxicity and is readily biodegradable under environmental conditions.	[51]
8	4-Hydroxyacetophenone (C ₈ H ₈ O ₂ / 136 g/mol)	Causes mild to moderate skin and eye irritation; exhibits relatively low environmental persistence due to biodegradation; low to moderate aquatic toxicity.	[52]
9	1,3-Cyclopentanediol (C ₅ H ₁₀ O ₂ / 102 g/mol)	Limited toxicological data are available; however, based on its saturated diol structure, low acute toxicity and mild irritation potential are anticipated.	[53]
10	Methoxyacetic acid (C ₃ H ₆ O ₃ / 90 g/mol)	Associated with reproductive and developmental toxicity; causes severe eye damage and skin irritation; exhibits low to moderate environmental persistence due to biodegradation.	[53]

Based on the identified intermediates, a multi-step synergistic degradation pathway for BPA is proposed. The process initiates with the oxidative cleavage of the quaternary carbon at the central isopropylidene bridge, yielding monocyclic phenolic intermediates such as 4-vinylphenol, 4-hydroxyacetophenone, and 1,2-dihydroxy benzene (catechol). Concurrently, successive addition of $\cdot\text{OH}$ and O_3 onto the aromatic rings induces intramolecular cyclization to form heterocyclic furan-based derivatives (e.g., 3-methyl-2,3-dihydro-1-benzofuran). Continuous electrophilic attacks by $\cdot\text{OH}$ and O_3 subsequently trigger the opening of the benzene rings, generating saturated cyclic and aliphatic intermediates (e.g., 1,3-cyclopentanediol, nonanal, and n-tetracosanol-1). These compounds are progressively oxidized into short-chain carboxylic and ether-carboxylic acids (such as malonic, methoxyacetic, and ethanedioic acids), which are ultimately mineralized into CO_2 and H_2O , as supported by the 82.5% COD removal at 240 min.

While certain primary intermediates, such as methoxyacetic acid and catechol, possess moderate-to-high acute or reproductive toxicity (Table 9), they are transient species. Under the highly oxidizing conditions of the integrated $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}/\text{UV-C}/\text{O}_3$ system, these hazardous intermediates are rapidly decomposed into low-toxicity, biodegradable organic acids without byproduct accumulation, thereby ensuring an ecologically safe and purified final effluent.

4. Conclusion

In this study, a novel magnetic ternary $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}/\text{UV-C}/\text{O}_3$ nanocomposite was successfully synthesized via a co-precipitation and calcination route and evaluated as a heterogeneous catalyst in a photocatalytic ozonation system for the removal of Bisphenol A (BPA) from aqueous environments. Under optimized operational conditions (pH 7.0, 50 mg catalyst dosage, and 10 mg/L initial BPA concentration), the integrated system achieved an outstanding BPA removal efficiency of 91.30% and a Chemical Oxygen Demand (COD) reduction of 82.5% within 240 min, demonstrating particularly efficient degradation and mineralization of the target pollutant.

Furthermore, GC-MS analysis successfully identified ten major degradation intermediates, outlining a multi-step synergistic oxidation pathway. The degradation initiates with the cleavage of the central isopropylidene bridge, followed by intramolecular cyclization, benzene ring opening, and progressive oxidation of aromatic intermediates into short-chain carboxylic acids. Although certain transient intermediates (such as catechol and methoxyacetic acid) exhibit moderate-to-considerable initial toxicity, the highly oxidizing

$\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}/\text{UV-C}/\text{O}_3$ system ensured their rapid decomposition into low-toxicity, biodegradable organic compounds, resulting in an ecologically safe and purified final effluent.

Statistical analysis (ANOVA) confirmed remarkably significant synergistic interactions among UV-C, ozone, and the nanocomposite, proving that the integrated ternary system considerably outperforms individual or binary treatment processes. Furthermore, reusability and stability tests over seven consecutive cycles indicated that the catalyst maintains reasonable catalytic activity and high structural integrity. Despite a gradual decline in degradation efficiency due to surface poisoning by intermediates and minor physical mass loss, the soft ferromagnetic properties of the cobalt ferrite core allowed for rapid magnetic separation within seconds using an external magnet, bypassing the need for energy-intensive filtration or centrifugation. Overall, these findings suggest that the $\text{CoFe}_2\text{O}_4/\text{ZnTiO}_3/\text{Ag}/\text{UV-C}/\text{O}_3$ -catalyzed photocatalytic ozonation process is an exceptionally promising, reliable, and sustainable advanced oxidation technology

for the elimination of recalcitrant organic contaminants from wastewater. For future directions, further investigations focusing on intermediate degradation pathways, toxicity assessments of the treated effluent, and scale-up feasibility under continuous flow configurations are strongly recommended.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Ethical Approval

This study was officially approved by the Research Ethics Committee of Kashan University of Medical Sciences, Kashan, Iran (Ethics Approval Committee ID: IR.KAUMS.NUHEPM.REC.1402.051).

Disclosure of AI Usage for Language Editing

The authors acknowledge the use of **Perplexity AI** exclusively for copy-editing, language polishing, and improving the structural coherence of the text. The tool was not used for data generation, statistical analysis, or drawing scientific conclusions. The human authors carefully reviewed all AI-assisted language revisions and assume full and final responsibility for the content and integrity of this article.

Data Availability

All data generated or analysed during this study are included in this published article.

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