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Original Article

Photocatalytic Degradation of Co-amoxiclav Antibiotic Using $Ag_2O/GO/TiO_2$ Nanocomposite From Aqueous Solutions

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Abstract

Background: In this study, the removal of Co-amoxiclav antibiotic from aqueous solutions was investigated using $Ag_2O/GO/TiO_2$ nanocomposite during a photocatalytic process.

Method: The effect of pH parameters (11-3), time (120 min), initial concentration of antibiotics (5-50 mg/L) and dose of nanocomposite (0.04-0.01 g) in removing the studied antibiotic was investigated. During the experiments, a UV lamp was used to study the photocatalytic degradation, and the remaining concentration of co-amoxiclav and cephalexin antibiotics was determined by UV-VIS spectrophotometer.

Results: The results demonstrated that the optimal parameters for the degradation of co-amoxiclav utilizing the nanocomposite are as follows: pH 9, nanocomposite dosage of 0.02 g/L, a reaction time of 60 min, resulting in a removal efficiency of 66.92%.

Conclusion: This study showed a moderate efficiency of nanocomposite in removing antibiotics, which can be of interest in the research on development of nanotechnology in removing pharmaceutical compounds from water. However, more attention should be paid to the effects of their residual nanomaterials on the environment.

Keywords: Toxic compounds, Pollution, Environment, Nanocomposite

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Introduction

The health and environmental effects of excessive use of antibiotics in the world is one of the serious human challenges. Antibiotics are widely used to treat bacterial infections, which can be detected in different types of water due to their continuous use and incomplete absorption in the body during metabolism.[1-3] The presence of antibiotics in the environment in low concentrations, leads to the development of antibiotic-resistant pathogens that potentially threaten the functioning of the ecosystem and human health. In recent years, the incidence of antibiotic-resistant bacteria has increased, and many researchers believe that this increase is due to the indiscriminate use of antibiotics.[4-6] So far, different physical, chemical and biological purification methods have been used to remove antibiotics. The aforementioned methods, despite their advantages, are encumbered by inherent limitations that render their widespread applicability challenging in many instances. In general, the normal water and wastewater treatment process is not able to remove these compounds.[7,8] Nowadays, owing to its affordability, simplicity, rapid separation, and high effectiveness, the absorption method has emerged as a favorable technique for pollutant removal. Concurrently, magnetic nano adsorbents have garnered considerable attention and employment in this context. These adsorbents exhibit notable capacity for both absorption and decomposition of organic compounds and various pollutants. Furthermore, characterized by high chemical stability, low toxicity, and exceptional recyclability, these adsorbents yield low-risk byproducts, thereby fostering the advancement of this method in water and wastewater treatment for pollutant mitigation.[9-13] Biological methods are different anaerobic and aerobic methods that use microorganisms to remove drugs and toxic compounds. Conventional methods of purifying polluted water, including biological processes, are generally not efficient in removing antibiotics.[14] Consequently, advanced oxidation processes coupled with nanotechnology, particularly the photocatalytic process, have gained prominence. This method relies on the generation of hydroxyl radicals or other highly reactive mediators, which exhibit strong reactivity towards toxic and non-biodegradable pollutants.[15,16] Graphene oxide stands as a pivotal derivative of graphite, garnering significant attention in recent years. Its utilization stems from its substantial surface area, distinctive two-dimensional structure, and noteworthy chemical properties. Researchers have extensively explored its application in various configurations, either independently or in conjunction with other semiconductors such as titanium dioxide, particularly in the realm of removing diverse organic compounds resistant to biological degradation.[17,18] Among the most widely used antibiotics in Iran and the world, we can mention Cefixime, Tetracycline, Azithromycin, *Penicillin's*, Amoxicillin and co-amoxiclav.[1,2,8,19] Given the extensive body of research focusing on the removal of various antibiotics and the relatively limited studies addressing co-amoxiclav, this study explores the photocatalytic removal of co-amoxiclav under novel conditions employing a new nanocomposite. Co-amoxiclav, a combination antibiotic comprising amoxicillin and clavulanic acid, stands out as one of the most potent antibiotics for infection treatment. Its efficacy extends to the management of urinary tract infections, middle ear infections, respiratory tract infections, chronic bronchitis, and sinusitis.[20-22] In this research, the concurrent utilization of UV irradiation and a novel nanocomposite, $Ag_2O/GO/TiO_2$, will be explored for the removal of co-amoxiclav antibiotics. Despite previous studies demonstrating the efficacy of graphene or TiO_2 in removing various antibiotics, the present nanocomposite is novel, with no prior investigation into its capacity for co-amoxiclav removal. Therefore, this study aims to establish the optimal conditions for the photocatalytic removal of co-amoxiclav from aqueous solutions using the $Ag_2O/GO/TiO_2$ nanocomposite under UV irradiation.

Materials and Methods

Synthesis and characterization of $Ag_2O/GO/TiO_2$ nanocomposite

In this study, we utilized the $Ag_2O/GO/TiO_2$ nanocomposite synthesized which was characterized in our previous publication.[23]

Chemicals and devices

The chemicals used for the synthesis were procured from Merck (Darmstadt, Germany) and were available in the laboratory of Ahvaz Unit. The antibiotic studied was obtained from Soban Pharmaceutical Company (Iran). Additionally, the research utilized a shaker device (Vibramax 100 Heidolph) for stirring the solution, a spectrophotometer (T90 + UV/VIS) to determine the remaining concentration, and a pH meter (Inolab-Sentix 41) to adjust the pH.

Optimization tests

Co-amoxiclav antibiotic removal tests were conducted using the $Ag_2O/GO/TiO_2$ nanocomposite on a laboratory scale in a discontinuous manner. The experiments were performed in 500 mL Erlenmeyer flasks containing 100 mL of the sample at a laboratory temperature of 25 ± 1 °C. The study investigated the effects of various parameters, including pH (ranging from 3 to 11), contact time (up to 120 min), initial antibiotic concentration (5-50 mg/L), and the dose of $Ag_2O/GO/TiO_2$ nanocomposite (0.01-0.04 g). The pH of the solutions was also adjusted by adding hydrochloric acid. To prepare the solution, 100 mg of the antibiotic was dissolved in 40 mL of methanol using ultrasonication. Distilled water was then added to bring the volume of the solution to 100 mL. Using this solution, the final volume was adjusted to 250 mL in subsequent steps. The resulting suspension was stirred with a magnetic stirrer in the dark for 30 min. After this period, a UV lamp (UV-C lamp, 254 nm, 6 W, PHILIPS) was placed in the central area of the sample. The residual concentration of co-amoxiclav was determined using a UV-VIS spectrophotometer. The antibiotic removal efficiency was calculated using the equation 1.

$$R\left(\%\right)=\frac{C_0-C_e}{C_0}\times 100 \qquad (1)$$

Where, R is the antibiotic removal efficiency (percent), C_0 and C_e are the primary and secondary concentration of the antibiotic in mg/L, respectively.

Results and Discussion

Table 1 presents the results regarding the amount of nanomaterials and the efficiency of antibiotic removal. It was observed that the removal efficiency of co-amoxiclav increased with the increase in the adsorbent dose. Although the highest removal efficiency of 67.61% was achieved with a nanocomposite dose of 0.04 g/L, a dose of 0.02 g/L, with an efficiency of 66.92%, was deemed more economically optimal and was selected as the optimal dose for further experiments.

The removal tests for co-amoxiclav antibiotic using the $Ag_2O/GO/TiO_2$ nanocomposite under UV irradiation were conducted for 60 min at a dose of 0.02 g/L, pH 9, and ambient temperature, varying the initial concentrations of co-amoxiclav. The results indicated that as the initial concentration of co-amoxiclav increased, the removal efficiency decreased (Table 2). The highest removal efficiency was 98.62% at an initial concentration of 5 mg/L, while the lowest removal efficiency was 65.57% at an initial concentration of 30 mg/L. Additionally, the reaction rate decreased with increasing antibiotic

Table 1. The effect of Nanocomposite on the Photocatalytic Degradation Efficiency at C (mg/L) = 20, pH = 9 and Contact Time (min) = 60

Dose (g)	Removal (%)
0.01	46.39
0.02	66.92
0.03	67.18
0.04	67.61

concentration. Consequently, an initial concentration of 20 mg/L was selected as the optimal concentration for subsequent experiments.

The results of the Co-amoxiclav antibiotic removal tests employing the $Ag_2O/GO/TiO_2$ nanocomposite under UV irradiation are detailed in Table 3. These experiments were conducted across diverse time intervals, employing a Co-amoxiclav concentration of 20 mg/L, an adsorbent dosage of 0.02 g/L, at a pH of 9, and ambient temperature conditions. Following a 120 min exposure period, the removal efficiency of the antibiotic reached 63.23%. Subsequently, beyond the 120 min mark, a decline in removal efficiency was observed. Consequently, based on the efficiencies observed during the experimentation, a duration of 60 min was determined as the optimal timeframe for subsequent tests. It is noteworthy that the negative time value of -20 corresponds to the initial mixing period preceding the commencement of the experiment.

The results of the co-amoxiclav antibiotic removal test using the $Ag_2O/GO/TiO_2$ nanocomposite under UV exposure, at a co-amoxiclav concentration of 20 mg/L, a dosage of 0.02 g/L, with a contact time of 60 min, and maintained at ambient temperature, are presented in Table 4. The highest removal efficiency was achieved at pH 11, reaching 67.01%, while the lowest absorption efficiency was observed at pH 3, yielding 47.33%. Based on the efficiencies obtained, pH 9 was identified as the optimal pH condition and was subsequently utilized for further investigations in the process.

As illustrated in Figure 1, panel c, the dose of the nanocomposite was investigated as a parameter affecting the absorption and removal capacity of antibiotics by the $Ag_2O/GO/TiO_2$ nanocomposite. An increase in the nanocomposite dose corresponded to an enhanced degradation efficiency of antibiotics. This improvement in efficiency can be attributed to the specific internal pores of the nanocomposite. However, despite the increased removal efficiency with higher dosages, the amount of co-amoxiclav absorbed per gram of nanocomposite decreased. The increase in removal efficiency in this case is attributed to the increased surface area of the adsorbent, which facilitates greater access of the antibiotic molecules to the adsorption sites on the $Ag_2O/GO/TiO_2$ nanocomposite, despite the fixed quantity of antibiotic molecules. In the study by Yaghmaiyan et al, which investigated the removal of amoxicillin from aqueous media using standard activated carbon, the absorption rate similarly increased with higher amounts of adsorbent.[24] Additionally, Mohammed and Kareem demonstrated that the removal of tetracycline improved with an increased dose of ZnO adsorbent.[25]

The initial concentration of the antibiotic was investigated as a variable influencing the absorption capacity of the antibiotic by the $Ag_2O/GO/TiO_2$ nanocomposite (Figure 1d). The results indicated that an increase in the initial concentration of the antibiotic negatively affects its removal efficiency, meaning that the removal efficiency decreases as the initial concentration of the antibiotic increases. This observation aligns with the study by Kakavandi et al[26] which found that the removal of amoxicillin from aqueous environments using magnetic activated carbon decreased with higher initial concentrations of the antibiotic. Additionally, while increasing the initial concentration of antibiotics negatively impacts removal efficiency, it has a positive effect on the total absorption and removal capacity. Similarly, Samarghandi et al[27] demonstrated that increasing the amount of adsorbent led to a corresponding increase in the absorption of cephalexin.

The increase in the absorption and removal of antibiotics with extended contact time is attributed to the higher likelihood of antibiotic molecules interacting with the surface of the nanomaterial (Figure 1a). Prolonging the contact time enhances the amount of absorption and removal (photocatalytic degradation) by allowing more interactions between the functional groups of the antibiotic and the adsorption sites within the nanocomposite structure.[1,2,9,28,29] In a study conducted by Balarak et al[30] to remove the antibiotic penicillin from aqueous solutions using a modified cannula, the effect of contact time was

Table 2. The Effect of Nanocomposite Concentration on Photocatalytic Degradation Efficiency at Dose (gr) = 20, pH = 9 and Contact Time (min) = 60

Concentration (mg/L)	Removal (%)
5	98.62
10	79.55
15	67.46
20	66.92
30	65.57

Table 3. Effect of Time on Photocatalytic Degradation Efficiency at Dose (g) = 0.02, pH = 9 and C (mg/L) = 20

Contact Time (min)	Removal (%)
-20	0
0	11.09
10	26.34
20	43.47
40	62.11
60	66.92
80	66.49
100	64.43
120	63.23
140	62.71

Table 4. Effect of pH on Photocatalytic Degradation Efficiency at Dose (g) = 0.02, Contact Time (min) = 60 and C(mg/L) = 20

pH	Removal (%)
3	47.33
5	53.09
7	62.02
9	66.92
11	67.01

investigated and it was determined that by increasing the contact time up to 75 min, the removal rate of penicillin increases and after 75 min, the amount of absorption remains constant and absorption or elimination does not take place. The contact time of 75 min was recorded as the optimal contact time with 80% removal of penicillin.

The pH of the solution is a crucial parameter affecting surface absorption and pollutant removal under photocatalytic conditions, significantly influencing the entire process. In this study, the effect of pH was examined within the range of 3 to 11. The results indicated that the highest percentage of co-amoxiclav removal occurred at higher pH levels. Specifically, as the pH increased from 3 to 9, the percentage of co-amoxiclav removal rose from 47 to 67% (Figure 1b). Similarly, in the study by Balarak et al,[31] it was demonstrated that key parameters, including initial pH, GO/TiO_2 dosage, UV intensity, and initial amoxicillin concentration, had a significant impact on amoxicillin degradation.

Kinetic studies are essential for investigating the factors influencing reaction rates. In this research, the data were collected, compared, and fitted to linear kinetic models. The results, based on the correlation coefficients of the kinetic models for the co-amoxiclav removal process using the nanocomposite, indicate that the process follows a pseudo-second-order kinetic model. This model better describes the data, as evidenced by the R^2 values of 0.982 and 0.986, respectively, shown in Table 5.

Kinetic studies are conducted to investigate the factors affecting reaction rates, and most environmental studies on pollutant removal typically follow second-order kinetics. Figure 2 presents the photocatalyst removal kinetic diagrams for Co-Amoxiclav. Similarly, in a study on the removal of vancomycin and cephalexin antibiotics using activated carbon, the equilibrium data followed pseudo-second-order kinetics. This finding suggests that chemical adsorption is the controlling process and the rate-limiting step.[1,32]

Conclusion

This study investigated the photocatalytic removal of the co-amoxiclav antibiotic from aqueous solutions using an $Ag_2O/GO/TiO_2$ nanocomposite. The effects of pH (range: 3-11), contact time (120 min), initial antibiotic concentration (5-50 ppm), and nanocomposite dosage (0.01-0.04 g/L) on antibiotic removal were examined. The results demonstrated that the optimal conditions for co-amoxiclav removal using the nanocomposite were achieved at pH 9, a nanocomposite dosage of 0.02 g/L, and a contact time of 60 min, resulting in a removal efficiency of 66.92%.

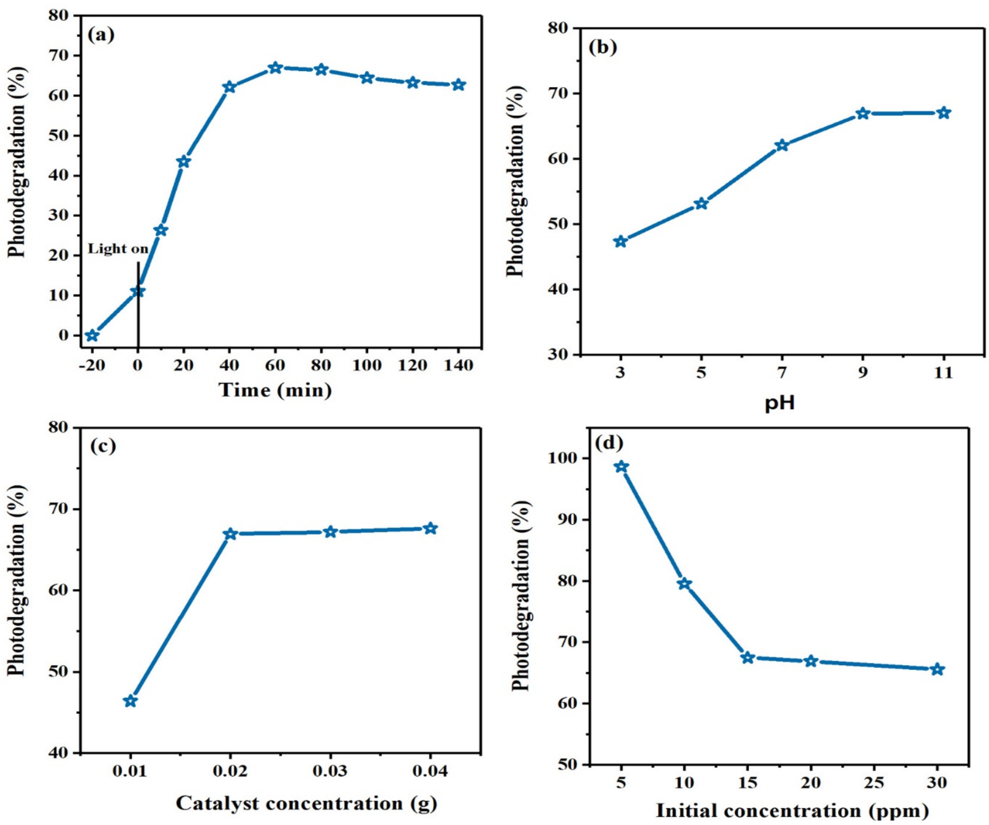

Figure 1. The Effect of Nanocomposite Dose (a), pH (b), Time (c), and Concentration (d) on the Photocatalytic Efficiency of Co-amoxiclav

Table 5. Kinetic Parameters of Co-Amoxiclav Removal

Antibiotic	Zero-Order		Pseudo-first Order		Pseudo-Second Order	
	K_0 (mgL· mim^{-1})	R_0^2	K_1 (min^{-1})	R_1^2	K_2 (Lmg^{-1}min^{-1})	R_2^2
Co-Amoxiclav	0.0074	0.97	0.0095	0.982	0.013	0.986

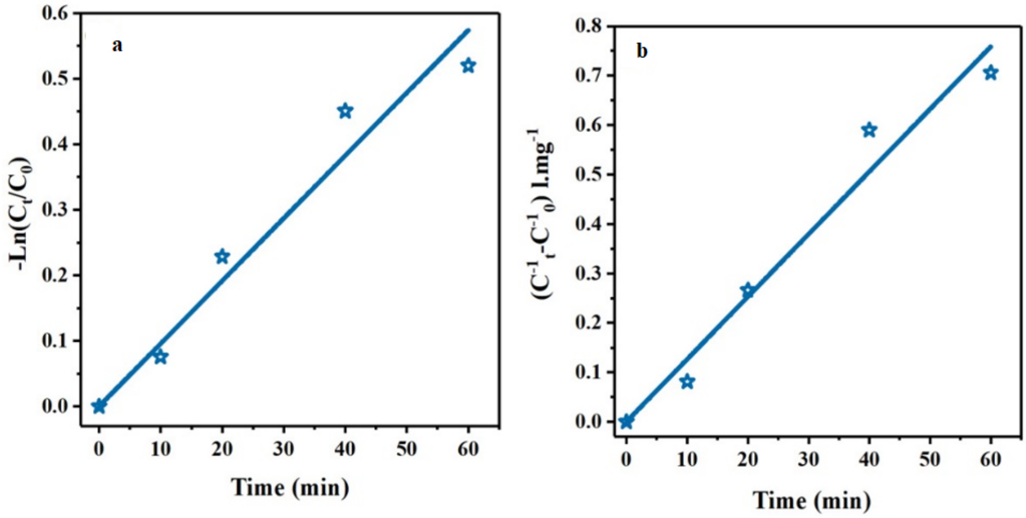

Figure 2. Kinetic Model of Co-Amoxiclav Photocatalytic Removal, (a) First-Order Kinetic (b) Second-Order Kinetic

It is noteworthy that in recent years, nanotechnology has emerged as a promising approach for the removal, absorption, and mitigation of resistant organic pollutants from the environment. However, despite their favorable efficiency, it is imperative to give adequate attention to cost estimation, nanomaterial performance evaluation, understanding removal mechanisms, and considering the potential impacts of nanomaterials on human health and their residues in the environment.

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Authors' Contribution
Conceptualization: Farzad Bayati, Mohammad Kazem Mohammadi, Reza Jalilzadeh Yengejeh, Ali Akbar Babaei.
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Supervision: Mohammad Kazem Mohammadi, Reza Jalilzadeh Yengejeh.
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Visualization: Mohammad Kazem Mohammadi.
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Competing Interests
The authors declared no conflict of interest.

Ethical Approval
There were no ethical considerations to be considered in this research.

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