



Original Research Article

Combined Removal of Pemetrexed Drug Using Adsorption and Photocatalytic Degradation from Polluted Water

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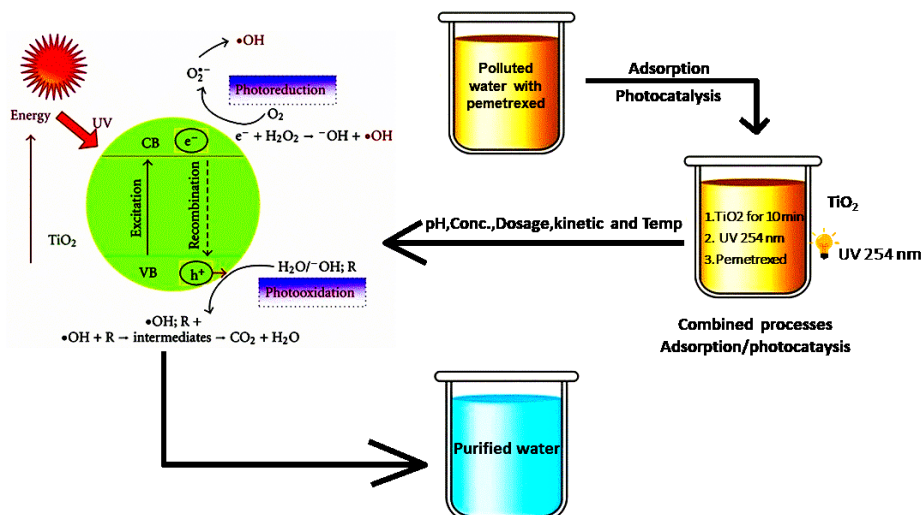
Advanced oxidation processes (AOP's)

Water treatment

ABSTRACT

Phenyl-substituted imidazoles, such as pemetrexed (PEME), pose substantial environmental risks due to their capacity for prolonged persistence within aquatic ecosystems. This study evaluates a combined adsorption-photocatalysis approach using TiO₂/UV-C for PEME removal, optimizing key parameters including pH (4.0–8.0), adsorbent dosage (0.125–1.0 g TiO₂), and initial PEME concentration (25–200 ppm). The findings reveal optimal adsorption efficacy (92.6%) at a pH of 4.0, propelled by electrostatic attraction between protonated TiO₂ (Ti-OH₂⁺) and anionic PEME. Photocatalytic degradation further enhanced removal (98.7% at pH 4.0) through hydroxyl radical (•OH) generation under UV-C irradiation. The equilibrium data demonstrated a strong correlation with the Freundlich isotherm ($R^2 > 0.98$), indicating multi-layer adsorption on heterogeneous TiO₂ surfaces. Temperature variations ranging from 15 to 40 °C exhibited an insignificant impact, while elevated doses of TiO₂ demonstrated a positive correlation with enhanced degradation. The TiO₂/UV-C system has been demonstrated to be a scalable and efficient solution for PEME-contaminated water, particularly under acidic conditions, thereby advancing the application of advanced oxidation processes (AOPs) for the treatment of pharmaceutical wastewater.

GRAPHICAL ABSTRACT



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Introduction

The United Nations highlights water scarcity as a critical factor affecting most Millennium Development Goals [1], reflecting its growing global priority. A growing body of research underscores that a myriad of organic contaminants originating from municipal, agricultural, and industrial wastewater sources are being increasingly identified in freshwater bodies. Termed as Contaminants of Emerging Concern (CECs), these substances have been associated with substantial health effects and environmental consequences, even at exceptionally low concentrations [2-6]. Computational screening of chalcogenide photocatalysts underscores the need for materials with broad pH tolerance, a challenge addressed in this study through TiO₂ surface charge modulation [7]. Unlike conventional methods (*e.g.*, precipitation-coagulation and biological treatment), advanced oxidation processes (AOPs) more effectively remove organic contaminants from water. Therefore, the development and adoption of advanced technologies are imperative to mitigate the impacts stemming from the uncontrolled disposal of these contaminants [8].

Antibiotics, analgesics, anti-inflammatories, anticancer, and antiseptics have a high fluctuation in quantity, a low carbon-to-nitrogen (C/N) ratio, a high suspended solid (SS) concentration, a high sulfate concentration, a complicated composition, and a high biological toxicity [9]. The presence of these contaminants in aquatic environments poses a substantial environmental concern, effecting on both fauna and flora, as well as terrestrial organisms. The potential for severe disruption of various biological activities highlights the gravity of this issue. Even humans have not been immune to the adverse effects of these toxic biological compounds [10]. Pemetrexed (PEME) functions as an anti-folate employed in the treatment of

various cancers, including lung cancer and mesothelioma. Classified as a chemotherapeutic medication, it operates by inhibiting the effect of folic acid, thereby impeding or preventing the proliferation of cancer cells, as elucidated by Baldwin and Perry [11]. Structurally, pemetrexed is an *N*-acyl glutamic acid with the *N*-acyl group 4-[2-(2-amino-4-oxo-4,7-dihydro-1H-pyrrolo[2,3-*d*] pyrimidin-5-yl) ethyl] (Figure 1), as outlined by Secrétan *et al.* [12]. Due to its chemical attributes, including water solubility, nonvolatility, and limited metabolism, the compound has been identified in surface water, as a consequence of its increasing utilization over time. Despite the significance of its function, it is harmful to living organisms at very low concentrations [13].

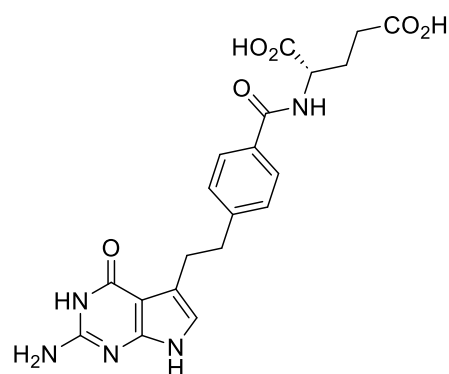
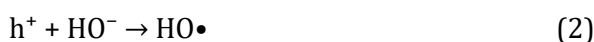


Figure 1. Structure of pemetrexed drug.

Photocatalytic oxidation is an optimal method for degrading contaminants found in our water. This approach entails expediting a photoreaction through the utilization of a solid semiconductor photocatalyst, examples of which include TiO₂, ZnO, and ZnS [14]. The photocatalytic sequence initiates with the photocatalyst activation through an external irradiation source, such as a UV-A lamp or sunlight. This activation leads to the generation of various highly reactive radical species, such as hydroxyl radicals (OH•), initiating redox reactions that actively participate in the degradation of organic contaminants. Amidst the various photocatalysts employed for

photocatalysis, the application of titanium dioxide combined with ultraviolet radiation (UV-C/ TiO₂) stands out as a promising technology within the realm of advanced oxidation processes. Recent work by Arjomandi Rad *et al.* on Mg/Cu-doped TiO₂ demonstrates enhanced visible-light absorption and charge separation, suggesting that tailored dopants could further optimize photocatalytic efficiency for pharmaceutical degradation [15]. This methodology presents potential advantages over numerous existing AOPs, encompassing the ability to efficiently degrade a diverse spectrum of organic pollutants in water. Noteworthy benefits include the limited formation of hazardous by-products, ready availability, and cost-effectiveness [16-18]. Further enhancing TiO₂ photocatalysis is the exceptionally high oxidation potential of the valence band holes, measuring 3.1 eV at strong acidic conditions. This elevated oxidation potential enables photo-excited TiO₂ to effectively oxidize a broad spectrum of organic molecules. Upon absorption of UV-C light, the electron (e⁻) and hole (h⁺) pair generated can migrate to the surface of the anatase particle, where they engage in electron transfer reactions with adsorbed oxygen, water, hydroxide ions, or organic species. Both water and hydroxide ions serve as electron donors to the holes (h⁺) of the catalyst, leading to the generation of hydroxyl radicals. This process is elucidated by the following equations [19]:



Generally speaking, comprehensive kinetics analyses/designs may be troublesome due to the extreme difficulty of scaling up complicated photocatalytic reactors to full size. To assess different reaction systems under consistent operational parameters and provide estimated values for scaling-up considerations, simplified models prove to be a valuable alternative, as

emphasized by Hermann *et al.* [20]. The primary aim of this investigation is to ascertain the feasibility of purifying and eliminating Pemetrexed from aqueous solutions through the application of advanced oxidation processes (AOP).

Materials and Methods

Pemetrexed disodium heptahydrate (batch No. -2197041) was procured from Teva company, while Potassium persulfate (K₂S₂O₈) and Titanium dioxide (TiO₂) P25 nanoparticles (comprising 80% Anatase and 20% Rutile) were obtained from Sigma Aldrich. High-performance liquid chromatography (HPLC) grade acetonitrile, buffer solutions, Sodium hydroxide, and Hydrochloric acid of analytical grades were sourced from Sigma Aldrich. The deionized water used in the experiments was prepared using a Millipore Milli-Q system with a resistance of 18 mΩ.

Instrumentations

A High-performance Liquid Chromatography (HPLC) which is a Shimadzu SPD-M20A, Model: CBM-20A, located at Abu-Dies/Al-Quds University was used in the study. The HANNA pH meter was used to conduct the pH measurements and the shaker used in the study was a Yamat, Model: MK2000, operating at 127 RPM. A system consisting of a double glass open Pyrex reactor equipped with a water-circulating system was constructed to maintain a constant temperature and to facilitate the photocatalysis. A Philips PL-L lamp (18 W, UV-C) provided irradiation at 253.7 nm. While we used pristine TiO₂ P25, recent studies on N-doped TiO₂ show that heteroatom incorporation can reduce charge recombination, suggesting a route for future catalyst optimization [21]. Furthermore, to guarantee the continuous homogenization of the solution during reactions, a magnetic stirrer was incorporated, while a lab jack was used to

optimize the stirring. The entire system was enclosed in a dark box to mitigate the light effect.

Adsorption experiments

All samples were first dissolved in a minimal amount of dioxane ($\leq 1\%$ of total volume) as a solubilizing agent, and then diluted in aqueous buffered solutions at varying pH levels to enhance PEME solubility. Centrifuge tubes of 50 mL were used for batch adsorption studies. Each tube contained 40 mL of PEME solution and varying adsorbent loads. During sorption tests, tubes were subjected to continuous shaking at a steady speed of 200 rpm using a mechanical shaker. Subsequently, the samples underwent filtration through 0.45 μm PVDF filters. HPLC was subsequently used to quantify the remaining PEME content in the filtrate, and no PEME was lost from the centrifuge tubes during the sorption procedure that was performed without ACs. 1000 ppm stock solution of PEME was prepared by dissolving 0.100 g of PEME in 100 mL MQ water then many diluted solutions were prepared. For adsorption experiments, 50 ppm PEME solution was mixed with 1.0 g TiO_2 and shaken in the dark at ambient temperature for 30 min. The pH was adjusted to 4.0-8.0, and samples were filtered through 0.45 μm membranes. the filtration was determined using HPLC.

We adjusted PEME solution pH (4.0–8.0) using HCl or NaOH to evaluate its impact on removal efficiency. Samples, comprising 50 ppm of PEME and 1.0 g of TiO_2 , were prepared with varying initial pH levels ranging from 4.0 to 8.0. Studying acidic conditions less than pH 4.0 was avoided, since carboxylic acid groups are completely undissociated and PEME precipitation under these conditions was detected. These samples were subjected to shaking at ambient temperature in the absence of light for 10 min, allowing them to reach equilibrium, as demonstrated in the subsequent results. Subsequently, the samples were exposed to a UV-

C lamp for 20 min, and subsequently analyzed using High-Performance Liquid Chromatography (HPLC). The effect of photocatalyst load variation: PEME solutions at a concentration of 50 ppm. PEME was mixed with different TiO_2 doses (1.0, 0.5, 0.25, and 0.125 g) in the Pyrex reactor, and then shaken at RT in the dark for 10 min to get the equilibrium (the pH of all solutions was adjusted to pH 4 and pH 7).

Analytical methods

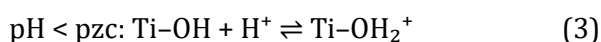
Using a high-performance Liquid Chromatography (HPLC), the in-water concentration of PEME was quantified coupled with a UV-Vis detector. Separation of the samples was attained by operating a C18 Column (3 mm x 100 mm, 3 μm). The mobile phase comprised 70% 0.005 M NaH_2PO_4 and 30% methanol (pH = 5.0), which was delivered at a 0.5 mL/min flow rate. An injection volume of 20 μL was employed, and the detection wavelength for PEME was set at 226 nm.

Results and Discussion

Adsorption phenomena optimization

The present study has explored the adsorption phenomena. The experimental setup involved conducting the experiments at ambient temperature (RT) with stirring of 50 ppm PEME for 30 min in the absence of light. During the experiments, the weight of TiO_2 was maintained at 1.0 g. However, the pH initially was systematically varied, ranging from pH 4.0 to pH 8.0 throughout the course of the experimental phase. [Figure 2](#) depicts PEME removal percentages at different pH values. The outcomes reveal an inverse relationship between pH and drug removal, indicating that as pH decreases, the percentage of drug removal increases. Specifically, at a pH of 8.0 (alkaline solution), the percentage of drug removal was approximately 25.90%. In contrast, at a pH of 4.0 (acidic

solution), the percentage of drug removal reached its maximum value of 92.60% within a 10-min timeframe. On different values of pH (pH = 5.0, 6.0, and 7.0), the efficiency of drug removal was 78.06%, 34.99%, and 29.98% respectively. Furthermore, the figure indicated that adsorption until 10 min has the highest removal efficiency whereas more 10 min is lost time without increasing the efficiency. Based on the results, it can be inferred that less than pH 5, the TiO_2 surface (the catalyst) will be positively charged according to Equation (3) [22]:



At pH 4.0, PEME carboxyl groups are partially ionized. Consequently, a negative charge is acquired by the PEME molecule from the carboxyl group, facilitating its robust adsorption

on the positively charged surface of the catalyst. These findings are in strong agreement with earlier reported results [23-25]. Noteworthy, these results revealed that the prevalent mechanism of PEME removal was by the physical adsorption and not due to photodegradation. pH 4.0 is the optimal for adsorbing PEME on TiO_2 . It keeps PEME carboxyl groups partially ionized and ensures totally positive charge at TiO_2 surface. In contrast, the kinetic analysis reveals a first-order reaction, as depicted in Figure 3. The observed linear relationship yields a straight line when plotting $\ln C$ at pH 5.0 (rate of adsorption is moderate compared to the other pH values) against time, and its slope corresponds to the rate constant. The following rates resulted by calculating the initial rate across the experiments at different pH values from Figure 2.

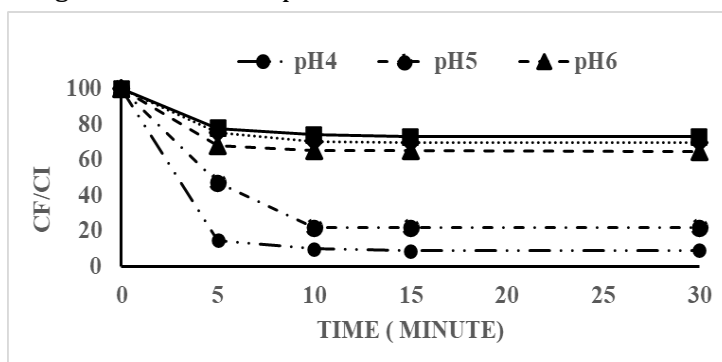


Figure 2. Adsorption optimization with TiO_2 at different pH values (R.T, 50 ppm PEME, $\text{TiO}_2 = 1.0$ g, in dark and retention time is 30 min).

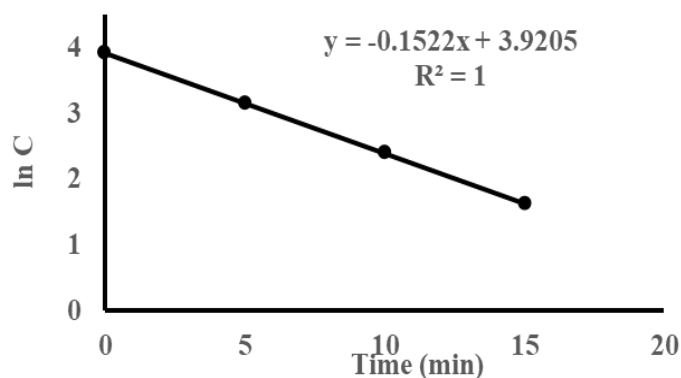


Figure 3. The kinetic study of the adsorption optimization at pH 5.0.

The rate of reaction at different pH values was calculated to be: 8.6 ppm/min at pH 4.0, 5.8 ppm/min at pH 5.0, 3.2 ppm/min at pH 6.0, 2.38 ppm/min at pH 7.0 and, and 2.2 ppm/min at pH 8.0. It shows that decreasing pH increasing the rate of adsorption at TiO₂ surface.

Operating parameters of variable affecting photodegradation of peme by using TiO₂

Effect of solution pH

The investigation of adsorption concerning different pH values is perilously described in Figure 2.

Figure 4 summarizes the removal efficiency of PEME under variation pHs using combination of adsorption in the first 10 min, and then subjected to photodegradation using UV-C for 20 min.

It shows that the removal efficiency is enhanced using photodegradation comparing to the removal efficiency using adsorption alone. Under variation pHs, the removal efficiency decreases along with the increase of the pH value as the following: 98.7%, 90.7%, 84.6%, 56.7%, and 52.8% recorded at pH= 4.0, 5.0, 6.0, 7.0, and 8.0, respectively. The removal efficiency was enhanced with limited ratio using photodegradation comparing with only adsorption stage. Similar to observations with Mg/Cu-doped TiO₂ [15], our results highlight the

critical role of catalyst surface properties in governing adsorption-photocatalysis synergy. The percentage removal of adsorption in Figure 2 are 92.60%, 78.06%, 34.99%, 29.98%, and 25.90%.

Respectively at pH 4.0, pH 5.0, pH 6.0, pH 7.0, and pH 8.0. After the adsorption process any species present in the water matrix can be oxidized by the radicals generated from the photocatalysis reaction [26]. While our TiO₂/UV-C system achieved 98.7% removal, computational studies of Zn-chalcogenides identify materials with potential for wider pH stability, providing alternatives for complex wastewater matrices [7].

Effect of adsorbent dose

The effect on the PEME removal by TiO₂ dosage at pH values 4.0 and 7.0 is illustrated in Figure 5 and Figure 6. The findings indicate a proportional increase in drug degradation with the rise in adsorbent dose, ranging from 0.125 to 1 g of TiO₂. This trend is attributed to the aggregation of variable binding sites facilitated by the interaction between functional groups on the particles. A significant amount of adsorbed unoccupied surface area is resulted by the presence of excess TiO₂, contributing to enhanced drug removal [27,28].

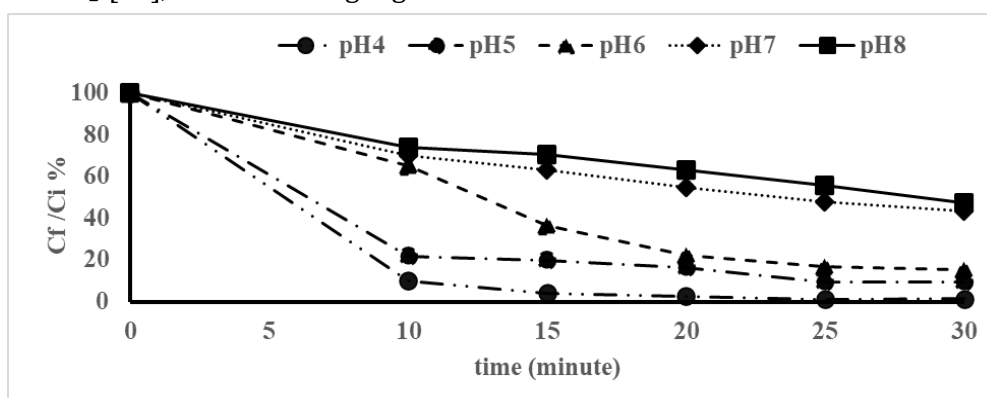


Figure 4. The PEME photodegradation with TiO₂ with different pH (R.T, 50 ppm PEME, TiO₂ = 1.0 g supplied with direct UV-C after keeping in the dark for 10 min with constant stirring 125 RPM).

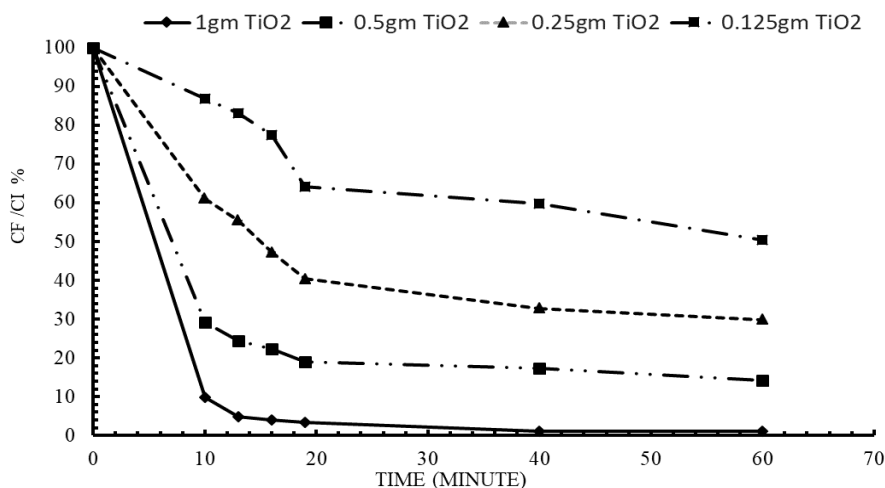


Figure 5. The PEME photodegradation with different catalyst loads of TiO₂ with a pH 4.0 (R.T, pH 4.0, and 50 ppm PEME supplied with direct UV-C after keeping in the dark for 10 min with constant stirring 125 RPM).

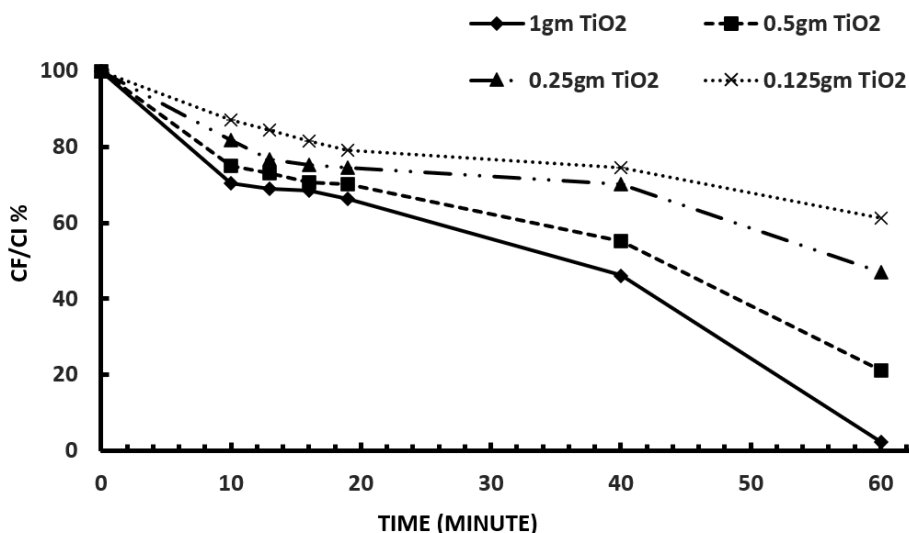


Figure 6. The PEME photodegradation with different catalyst loads of TiO₂ at a pH 7.0 (50 ppm PEME, R.T, and pH 7.0 supplied with direct UV-C after keeping in the dark for 10 min with constant stirring 125 RPM).

Effect of temperature

Figure 7 and Figure 8 respectively illustrate the adsorption capacity of PEME onto TiO₂ at fluctuating temperatures which spans from 15 to 40 °C at pH 4.0 and 7.0. Notably, the results demonstrate that the increase in temperature does not exert any discernible effect on the adsorption capacity. The values obtained at 15 °C and 40 °C are relatively close, suggesting a lack of significant temperature-dependent variation in

adsorption capacity. This can be attributed due to the increasing of PEME solubility as results of addition minimum amount of dioxin in the initial preparation solution.

Adsorption isotherm

The effect of PEME dosage on adsorption capacity was assessed by altering the concentration of PEME within the range of 25-200 ppm at pH 4.0 (see Figure 9).

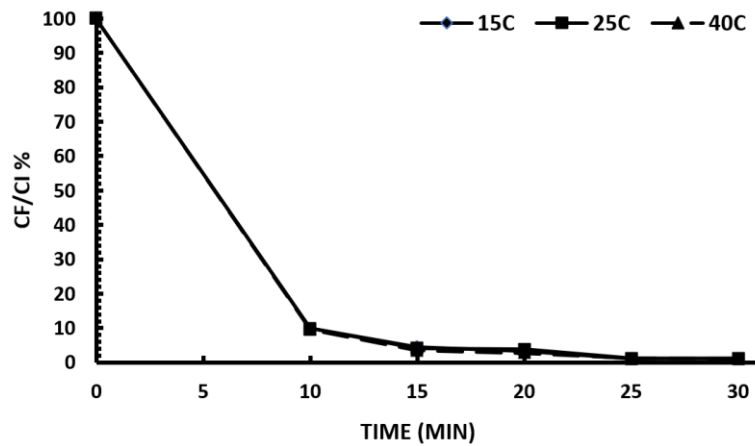


Figure 7. The photodegradation of PEME along with a pH of 4.0 with TiO_2 and changed temperature (50 ppm PEME, pH 4.0, $TiO_2 = 1.0$ g supplied with direct UV-C after keeping in the dark for 10 min with constant stirring 125 RPM).

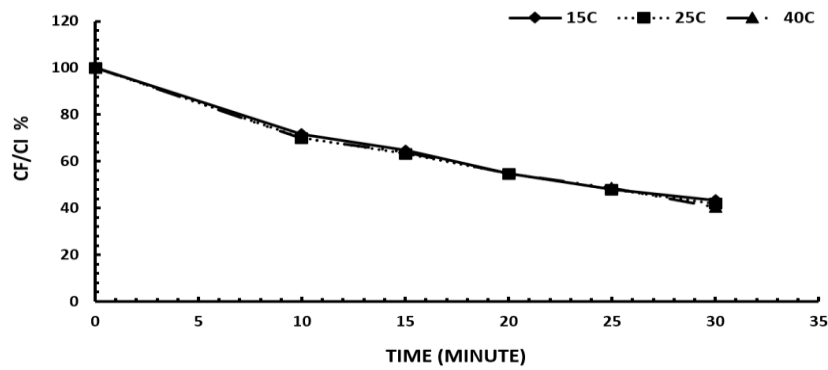


Figure 8. The PEME photodegradation along with a pH of 7.0 with TiO_2 and changed temperature (50 ppm PEME, pH 7.0, $TiO_2 = 1.0$ g supplied with direct UV-C after keeping in the dark for 10 min with constant stirring 125 RPM).

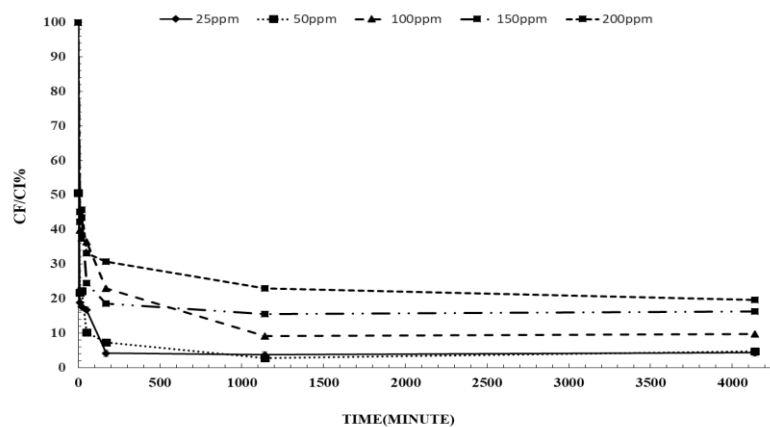


Figure 9. The adsorption isotherm for the different concentrations of PEME along with TiO_2 at the pH of 4.0 ($TiO_2 = 1.0$ g, pH 4.0, and R.T in the dark for three days with constant stirring 125 RPM).

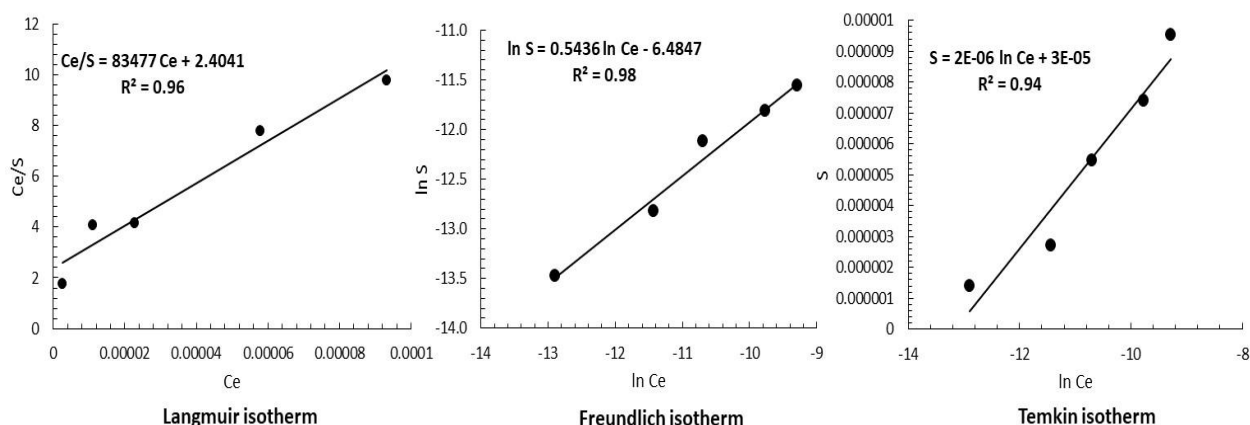


Figure 10. The isotherm study of Langmuir, Freundlich, and Temkin at the pH of 4.0.

The results distinctly illustrate that the adsorbed amount at equilibrium exhibits an increase with the rise in the initial concentration of PEME, ranging from 25 to 200 ppm. The Temkin and Freundlich isotherms assume a heterogeneous adsorbent surface with multilayer adsorption, non-uniform interactions between adsorbed species, and distributed active-site energies [29]. In contrast, the Langmuir isotherm model assumes a homogeneous surface, indicating the monolayer adsorption at particular active sites amid the adsorbent and no interactions even at the adjacent sites in between the adsorbates [30]. In Figure 10 demonstrates the calculated results of R^2 from the three isotherm models. Irrespective of the tested temperature, the equilibrium data obtained exhibited a better fit with the Freundlich isotherms ($R^2 > 0.98$), indicating a propensity for multilayer adsorption of PEME on the surface of this adsorbent.

Conclusion

This study demonstrates the efficacy of a combined adsorption-photocatalysis system ($\text{TiO}_2/\text{UV-C}$) for removing pemetrexed (PEME) from aqueous solutions. The study found that adsorption dominated PEME removal, achieving

92.6% efficiency at pH 4.0 due to electrostatic interactions between protonated TiO_2 surfaces (Ti-OH_2^+) and anionic PEME molecules. Photocatalysis further enhanced degradation, reaching 98.7% removal at pH 4.0 through hydroxyl radical ($\bullet\text{OH}$) generation. The Freundlich isotherm ($R^2 > 0.98$) provided the most adequate description of the adsorption process, thereby confirming multi-layer uptake on heterogeneous TiO_2 sites. While temperature variations (15-40 °C) had minimal effects, increasing TiO_2 dosage significantly improved removal efficiency. These findings highlight the $\text{TiO}_2/\text{UV-C}$ system as a promising, scalable solution for PEME-contaminated water, particularly under acidic conditions. Future research endeavors should prioritize the exploration of practical applications in authentic wastewater matrices and the assessment of long-term catalyst stability to facilitate the adoption of these catalysts in industrial settings. Additionally, exploring doped TiO_2 variants (*e.g.*, N-doped; [21]) may enhance UV-Visible light response and decrease the energy demands for scale-up. This work contributes to the expanding corpus of knowledge on AOP-based strategies for pharmaceutical pollutant remediation.

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Disclosure Statement

No potential conflict of interest was reported by the authors in this work.

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