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Investigation of the Photocatalytic Activity of TiO₂ after Neutron Irradiation

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Abstract

This work focused on the investigation of titanium dioxide (TiO₂) modified with magnesium (Mg) and aluminum (Al) ions (0.5–2%) before and after neutron irradiation. The influence of irradiation on the morphological and optical characteristics of the samples was evaluated using scanning and transmission electron microscopy, as well as UV-Vis diffuse reflectance spectroscopy. The resulting photocatalysts were tested for the degradation of methylene blue under UV irradiation. The photocatalytic tests demonstrated a maximum methylene blue removal efficiency of 98.3% for the TiO₂:Al–0.5% sample after 1 h of neutron irradiation. These results demonstrate that the Mg/Al modification and neutron irradiation can influence the photocatalytic properties of modified TiO₂, highlighting the potential of this approach for future environmental applications.

1. Introduction

Environmental pollution remains one of the major global challenges, highlighting the need for the development of effective and sustainable approaches to environmental remediation [1, 2]. Titanium dioxide (TiO₂) is a widely used semiconductor material due to its chemical stability, non-toxicity, cost-effectiveness, and high photocatalytic activity. Despite these advantages, the practical application of TiO₂ as an effective photocatalyst is limited by several factors, including its spectral response being confined to the ultraviolet (UV) region and the high recombination rate of photogenerated electron-hole pairs [3–6].

Currently, various methods of structural and surface modification of TiO₂ are used to overcome these limitations, including the formation of heterojunctions [7–10], thermal treatment [11–13], and metal and non-metal doping [14–16]. Among these approaches, metal doping of TiO₂ is widely used [17].

The introduction of Mg²⁺ and Al³⁺ ions contributes to the formation of additional active sites, improves the separation efficiency of photogenerated electron-hole pairs, and inhibits their recombination, thereby enhancing the photocatalytic performance of the material [18–20].

At the same time, the functional properties, phase composition, and efficiency of the modified materials are largely determined by the nature and concentration of the introduced dopants. Another approach to enhancing the properties of semiconductor materials involves the application of ionizing radiation, particularly neutron irradiation. Neutron irradiation can induce structural and defect-related modifications in both the bulk and surface of semiconductor materials, including the formation of oxygen vacancies, and affect photocatalytic behavior [21–26].

The aim of the present study was to investigate the effects of Mg and/or Al modification at concentrations of 0.5–2% on the morphological, optical, and photocatalytic properties of TiO₂, as well as to evaluate the effects of subsequent neutron irradiation.

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2. Materials and methods

2.1 Materials

Titanium dioxide (TiO₂, ≈25 nm, 99.9%, MERYER, China), aluminium nitrate nonahydrate (Al(NO₃)₃·9H₂O, >99.99%, MERYER, China), magnesium nitrate hexahydrate (Mg(NO₃)₂·6H₂O, ≥99%, MERYER, China), methylene blue (C₁₆H₁₈ClN₃S·xH₂O, dye content ≥82%, Sigma–Aldrich, USA), ultrapure water (18.2 MΩ·cm at 25 °C, ELGA PURE Quest, UK).

2.2 Preparation of samples

Commercial TiO₂ (T) was used as received as the reference sample and was not subjected to impregnation, evaporation, or calcination. For the preparation of each modified sample, 1.0 g of commercial TiO₂ was used. TiO₂:Mg (TM), TiO₂:Al (TA), and TiO₂:Mg:Al (TMA) photocatalysts were prepared by an impregnation method followed by heat treatment [27, 28]. The numerical values in the sample notation indicate the nominal modifier content for TM and TA (TM-0.5, TM-1, TA-0.5, and TA-1) and the total nominal modifier content for TMA (TMA-1 and TMA-2). For TM and TA, molar ratios of 0.995:0.005 and 0.99:0.01 were used, corresponding to nominal modifier contents of 0.5% and 1%, respectively. For TMA, Ti:Mg:Al molar ratios of 0.99:0.005:0.005 and 0.98:0.01:0.01 were used, corresponding to total nominal modifier contents of 1% and 2%, respectively (Table 1). The required amounts of Mg(NO₃)₂·6H₂O and/or Al(NO₃)₃·9H₂O were dissolved in 10 mL of ultrapure water. TiO₂ powder was added to the precursor solution, and the resulting suspension was ultrasonicated for 30 min. The suspension was then heated in a water bath at approximately 90 °C until the solvent evaporated, followed by drying at 60 °C for 12 h. The dried samples were heated from room temperature to 800 °C over 120 min, corresponding to an average heating rate of approximately 6.5 °C min^{−1}, and subsequently calcined at 800 °C for 1 h in air.

Table 1. Sample codes and nominal modifier contents of the prepared TiO₂ photocatalysts

Sample	Composition	Nominal modifier content
T	TiO ₂	0
TM-0.5	TiO ₂ :Mg	0.5% Mg
TM-1	TiO ₂ :Mg	1% Mg
TA-0.5	TiO ₂ :Al	0.5% Al
TA-1	TiO ₂ :Al	1% Al
TMA-1	TiO ₂ :Mg:Al	0.5% Mg + 0.5% Al
TMA-2	TiO ₂ :Mg:Al	1% Mg + 1% Al

2.3 Neutron irradiation

Neutron irradiation was performed at two nuclear facilities of the Institute of Nuclear Physics (Almaty, Kazakhstan). The samples were irradiated for 1, 2, and 5 h in the core of a critical facility, a zero-power research reactor operating at a thermal power of 100 W. Demineralized light water serves as both the moderator and reflector in this facility, where the maximum thermal neutron flux density at the central core positions is on the order of 10⁹ n/cm²·s [29]. In addition, the samples were irradiated for 17 min at the WWR-K research reactor in a peripheral irradiation channel located in the reactor tank beyond the beryllium reflector. Owing to the higher neutron flux density at this position, the accumulated neutron fluence was equivalent to that obtained after approximately 10 h of irradiation at the critical facility. The WWR-K reactor was used to achieve the required irradiation conditions, as longer irradiation periods at the critical facility were restricted by radiation safety constraints. A peripheral irradiation position with a neutron energy spectrum similar to that of the critical facility was therefore selected. This approach enabled the investigation of neutron fluence effects over approximately one order of magnitude. The WWR-K is a 6 MW, light-water-moderated, tank-type multipurpose research reactor, with thermal neutron flux densities ranging from 10¹² to 10¹⁴ n/cm²·s at irradiation positions within the reactor core [30, 31].

Before irradiation, the samples were sealed in quartz ampoules and placed in a standard irradiation capsule made of an aluminum alloy and cooled by the reactor coolant. The quartz ampoules contained air at atmospheric pressure. During irradiation, the sample temperature did not exceed 50 °C and was assumed to be approximately equal to the coolant temperature, which was monitored using standard temperature sensors installed in both facilities. At the critical facility, the accumulated thermal neutron fluences ($E < 0.55$ eV) after 1, 2, and 5 h were 6.3×10¹², 1.3×10¹³, and 3.2×10¹³ n/cm², respectively. The corresponding fast neutron fluences ($E > 1.15$ MeV) were 8.7×10¹¹, 1.7×10¹², and 4.3×10¹² n/cm². For the 17 min irradiation at the WWR-K reactor, the thermal and fast neutron fluences were 6.3×10¹³ and 7.9×10¹² n/cm², respectively.

Radiation damage was evaluated using a Monte Carlo simulation performed with the MCNP 6.3 code and the ENDF/B-VII.1 nuclear data library for the irradiation position at the WWR-K reactor. Atomic displacement damage was estimated using the NRT

model and the corresponding threshold displacement energies for Ti and O. For the 17 min irradiation at the WWR-K reactor, corresponding to the highest neutron fluence investigated in this study, the calculated total displacement damage was approximately 6.4×10^{-9} dpa. The calculated contributions from thermal neutrons ($E < 0.5$ eV), epithermal neutrons (0.5 eV–0.1 MeV), and fast neutrons ($E > 0.1$ MeV) were approximately 6.4×10^{-9} , 3.4×10^{-11} , and 3.1×10^{-13} dpa, respectively.

The calculated absorbed gamma-ray doses were approximately 88, 181, 446, and 877 Gy for the 1, 2, and 5 h irradiations at the critical facility and the 17 min irradiation at the WWR-K reactor, respectively.

Post-irradiation gamma spectrometry revealed induced radioactivity primarily associated with ^{47}Sc (595 Bq) and ^{65}Zn (215 Bq). The samples were stored in a lead-shielded area for approximately 24 h until the measured radiation levels decreased to background levels. They were subsequently stored at controlled room temperature before characterization and photocatalytic testing.

2.4 Characterization

The surface morphology of the samples was examined by scanning electron microscopy (SEM) using a Thermo Scientific Quattro S microscope (Germany) at an accelerating voltage of 80 kV in secondary electron mode at various magnifications and by transmission electron microscopy (TEM) using a JEOL JEM-2100 microscope (Japan) operated at an accelerating voltage of 80 kV. The optical properties of the samples were investigated by UV-Vis diffuse reflectance spectroscopy (DRS) using an Agilent Cary 4000 spectrophotometer over the wavelength range of 200–800 nm. PTFE was used as the reference standard for the UV-Vis DRS measurements.

2.5 Photocatalytic test

The photocatalytic activity of the samples was evaluated based on the degradation efficiency of a 10 mg/L methylene blue (MB) solution. The experiments were conducted at room temperature in a parallel photochemical reactor (Xi'an Toption Instrument Co., Ltd.). A 300 W mercury lamp was used as the UV light source. All measurements were performed under isothermal conditions at an ambient temperature of 25 °C, and the temperature of the reaction mixture was regulated by tap circulation. For the photocatalytic tests, 20 mg of the

photocatalyst was dispersed in 50 mL of MB solution. The suspension was kept in the dark under continuous magnetic stirring for 30 min to establish adsorption-desorption equilibrium. Photocatalytic irradiation was then carried out for 60 min under continuous stirring. Aliquots of 1.5 mL were collected every 15 min and filtered through a PVDF syringe filter with a pore size of 0.22 μm . The MB concentration was determined by UV-Vis spectrophotometry at a wavelength of 664 nm. All photocatalytic experiments were performed in duplicate, and the reported values represent the mean of two independent measurements.

The photodegradation efficiency was calculated using the following Eq. (1):

$$\eta(\%) = \frac{(C_0 - C_t)}{C_0} \times 100, \quad (\text{Eq. 1})$$

where C_0 – dye concentration after the dark adsorption and C_t – dye concentration at UV irradiation time t , where t is counted from the beginning of UV irradiation after the dark stage.

3. Results and Discussion

The surface morphology of the samples was characterized by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) (Fig. 1). Analysis of the SEM images showed that all synthesized samples before irradiation (Fig. 1a–c) were composed of nanoparticle agglomerates with predominantly spherical morphology. The TA sample (Fig. 1a) exhibited a relatively loose structure with a moderate degree of particle aggregation. The TM sample (Fig. 1b) showed a more uniform particle distribution and the formation of compact agglomerates. In the case of combined Mg and Al modification (Fig. 1c), an agglomerated structure consisting of finely dispersed particles was observed. After neutron irradiation (Fig. 1e–g), no significant morphological changes were observed, indicating that the overall morphology of the samples was preserved. TEM images of the TMA sample before and after irradiation (Fig. 1d, h) revealed nanoscale particles with clearly defined boundaries. Comparing the samples before (Fig. 1d) and after irradiation (Fig. 1h), the general particulate morphology remained intact. The absence of pronounced morphological changes within the available resolution suggests morphological stability of the TMA photocatalyst under neutron irradiation.

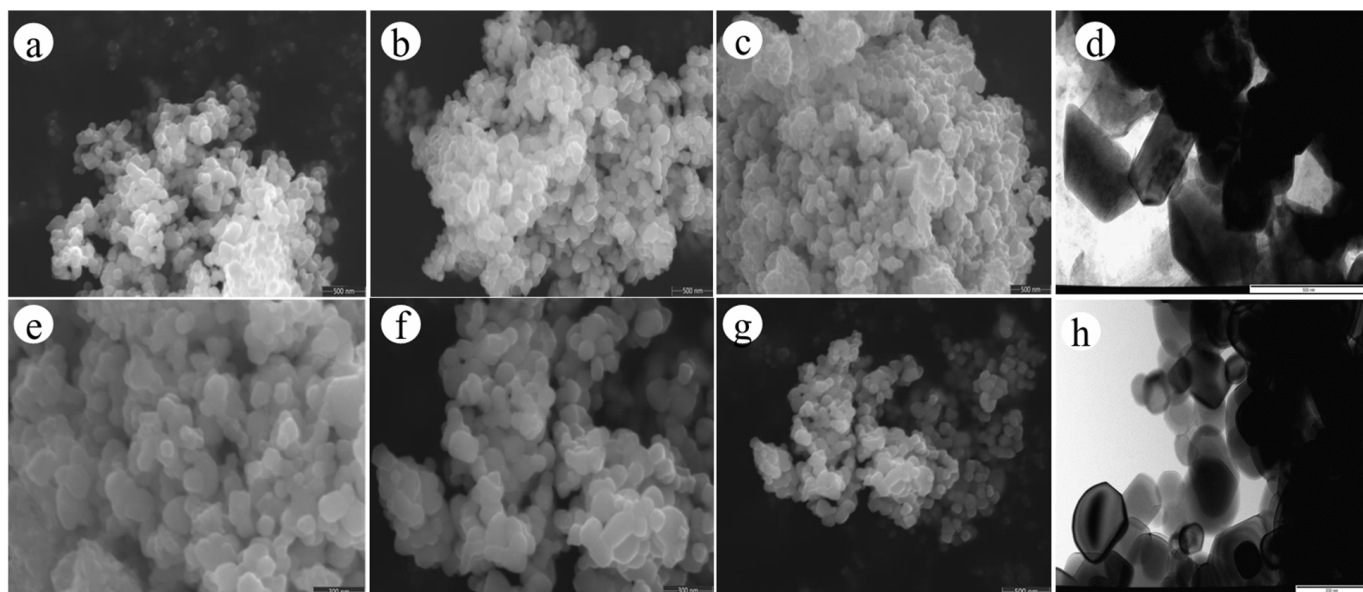


Fig. 1. (a–c, e–g) SEM, (d, h) TEM images of synthesized samples: (a) TA; (b) TM; (c) TMA – before irradiation; (e) TA; (f) TM; (g) TMA – after irradiation; (d) TMA – before irradiation; (h) TMA – after irradiation.

UV-Vis diffuse reflectance spectroscopy was employed to characterize the optical properties of the samples (Fig. 2). The obtained spectra showed that the studied materials exhibited light absorption at wavelengths up to 400 nm. The optical bandgap energies (E_g) of the samples were determined by extrapolating the linear regions on the Tauc plots. The obtained values were 3.23–3.31 eV for TiO₂, 3.13–3.16 eV for TM-0.5, 3.15–3.19 eV for TM-1, 3.15–3.20 eV for TA-0.5, and 3.14–3.19 eV for TA-1. These results indicate that Mg/Al modification of

TiO₂ followed by neutron irradiation is associated with a slight narrowing of the optical bandgap compared with the initial samples [32].

In our previous study, XRD analysis showed that Mg/Al modification and subsequent neutron irradiation did not result in detectable changes in the phase composition of TiO₂, and the anatase structure was preserved [28]. These findings suggest that the relatively low Mg and Al contents and neutron irradiation conditions employed in the present study are unlikely to induce major phase transformations.

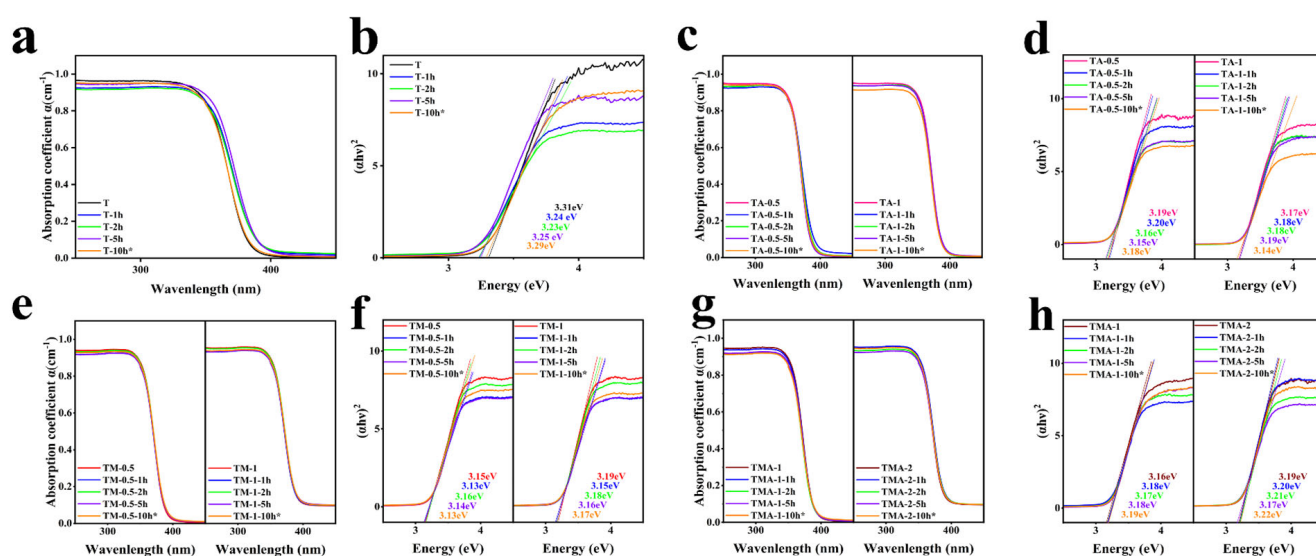


Fig. 2. UV-Vis diffuse reflectance spectra (a, c, e, g) and corresponding Tauc plots (b, d, f, h) of the photocatalysts. *Samples labeled “10 h” were irradiated at the WWR-K reactor (6 MW) for 17 min, equivalent in neutron fluence to 10 h at the critical facility (100 W).

Figure 3 presents the experimental data on the photocatalytic activity of the samples, evaluated based on their ability to degrade MB under 60 min of UV irradiation. The photocatalytic activity of the samples before neutron irradiation was investigated to evaluate the effect of irradiation on their photocatalytic performance. The experiments were conducted using a catalyst loading of 20 mg and an initial MB concentration of 10 mg·L⁻¹. In the blank experiment without a photocatalyst, MB concentration decreased by approximately 9% during the dark stage (Fig. 3), possibly due to adsorption on the reactor walls or losses during PVDF filtration. Figure 3a shows experimental data on MB degradation using commercial TiO₂ (T) before and after neutron irradiation. The highest removal efficiency of 88.2% was observed after 5 h of neutron irradiation. For the TM-0.5 sample (Fig. 3c), the removal efficiency was 86.2% before irradiation and 85.9% after 1 h of neutron irradiation, indicating that neutron irradiation did not improve the removal efficiency under these conditions. The TM-1 sample showed a lower removal efficiency, with a maximum value of 80.6% after 1 h of neutron irradiation. Among the TMA samples (Fig. 3g), the highest removal efficiency of 97.9% (including 35.50% adsorption) was observed for TMA-2 after 5 h of neutron irradiation. Among the Al-modified samples (Fig. 3e), the highest removal efficiency of 98.3% (including 25.38% adsorption) was observed for TA-0.5 after 1 h of neutron irradiation.

Thus, a comparative analysis of MB removal efficiency shows that neutron irradiation causes noticeable changes in the photocatalytic performance of the samples compared with the corresponding unirradiated samples [20, 33, 34].

A pseudo-first-order kinetic model was used to quantitatively describe the photocatalytic degradation of MB (Eq. 2):

$$\ln\left(\frac{C_0}{C_t}\right) = kt, \quad (\text{Eq. 2})$$

where k (min⁻¹) is the apparent rate constant, C_0 – initial dye concentration, C_t – MB concentration at irradiation time t . The experimental data were fitted using linear regression with a free intercept to account for deviations from the ideal kinetic model.

According to the kinetic plots (Fig. 3), the highest apparent pseudo-first-order rate constant was obtained for TMA-1 after 5 h of neutron irradiation (0.05780 ± 0.00750 min⁻¹; Fig. 3h), followed by TA-0.5 after 1 h of neutron irradiation ($0.05350 \pm$

0.00478 min⁻¹; Fig. 3f). For commercial TiO₂, the apparent rate constants were 0.01402 ± 0.00129 min⁻¹ before irradiation and 0.01927 ± 0.00219 min⁻¹ after irradiation at 10 h (WWR-K, 17 min) (Fig. 3b). As summarized in Table 2, the apparent rate constants varied depending on the modifier composition and neutron irradiation conditions.

The overall MB removal efficiencies, pseudo-first-order rate constants and coefficient of determination for all samples are summarized in Table 2. Overall, the effect of neutron irradiation on photocatalytic performance depended on the type and nominal content of the modifier as well as the irradiation time.

4. Conclusion

In this study, the photocatalytic activity of TiO₂ modified with Mg and Al at concentrations of 0.5–2% and subsequently subjected to neutron irradiation was investigated. SEM and TEM analyses revealed the formation of nanoparticle agglomerates and demonstrated that the overall morphology of the samples was preserved after neutron irradiation. The photocatalytic activity of the samples was evaluated using methylene blue (MB) degradation as a model reaction. The highest MB removal efficiency of 98.3% was achieved for TiO₂:Al-0.5% after 1 h of neutron irradiation, while the TiO₂:Mg, Al-2% sample exhibited a removal efficiency of 97.9% after 5 h of neutron irradiation. These results demonstrate that Mg/Al modification combined with neutron irradiation can influence the photocatalytic performance of TiO₂. Further studies will focus on explaining the underlying mechanisms of defect generation induced by neutron irradiation, evaluating the long-term stability, and testing their removal efficiency against a broader spectrum of organic pollutants in real wastewater matrices.

CRedit authorship contribution statement

Saule Dyussembekova: Conceptualization, Methodology, Investigation, Writing – original draft. **Aididar Sailaubek:** Investigation, Visualization, Writing – original draft. **Dina Utarova:** Visualization. **Slambek Kapan:** Software, Methodology. **Bulan Maikanov:** Software, Methodology. **Timur Khalila:** Investigation, Methodology. **Kirill Kisselyov:** Investigation, Methodology. **Magzhan Aitkulov:** Investigation, Methodology. **Diana Yessenkeldina:** Conceptualization, Methodology, Writing – original draft, Writing – review & editing.

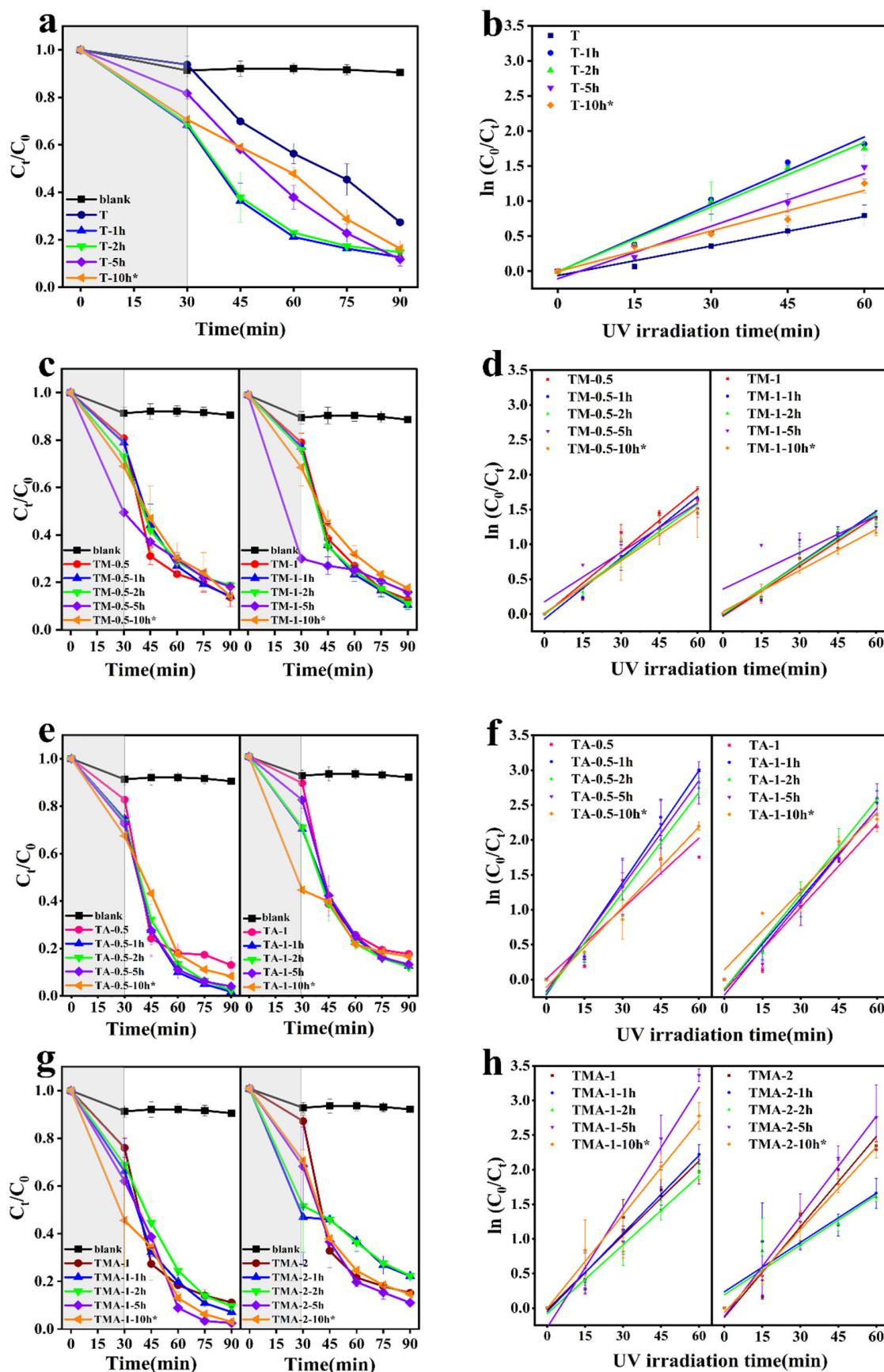


Fig. 3. Photocatalytic degradation of MB over T, TM, TA, and TMA photocatalysts under UV irradiation before and after neutron irradiation: (a, c, e, g) time dependence of C_t/C_0 ; (b, d, f, h) corresponding pseudo-first-order kinetic plots of $\ln(C_0/C_t)$. *Samples labeled "10 h" were irradiated at the WWR-K reactor (6 MW) for 17 min, equivalent in neutron fluence to 10 h at the critical facility (100 W).

Table 2. Comparison of the overall removal efficiency, pseudo-first-order, and R² values of the samples

Photocatalyst	Overall MB removal efficiency (%)					Pseudo-first-order constants (min ⁻¹)					R ²				
	Before	1 h	2 h	5 h	10 h*	Before	1 h	2 h	5 h	10 h*	Before	1 h	2 h	5 h	10 h*
T	72.70	87.40	85.20	88.20	83.80	0.01402 ± 0.00129	0.03200 ± 0.00234	0.03068 ± 0.00207	0.02490 ± 0.00231	0.01927 ± 0.00219	0.9753	0.9842	0.9864	0.9749	0.9626
TM-0.5	86.20	85.90	81.20	81.90	85.80	0.02993 ± 0.00500	0.02927 ± 0.00205	0.02633 ± 0.00187	0.02360 ± 0.00342	0.02498 ± 0.00117	0.9226	0.9855	0.9851	0.9406	0.9935
TM-1	78.40	80.60	79.90	75.60	74.00	0.02393 ± 0.00219	0.02481 ± 0.00271	0.02413 ± 0.00264	0.01740 ± 0.00691	0.01966 ± 0.00105	0.9755	0.9655	0.9653	0.6799	0.9915
TA-0.5	87.00	98.30	97.60	96.00	91.70	0.03359 ± 0.00757	0.05350 ± 0.00478	0.04791 ± 0.00383	0.05020 ± 0.00417	0.03825 ± 0.00308	0.8678	0.9766	0.9812	0.9798	0.9809
TA-1	90.70	96.30	96.70	95.40	91.80	0.03973 ± 0.00453	0.04306 ± 0.00263	0.04529 ± 0.00278	0.04460 ± 0.00433	0.03752 ± 0.00454	0.9625	0.9889	0.9888	0.9725	0.9579
TMA-1	88.80	92.85	90.35	97.50	97.00	0.03579 ± 0.00497	0.03761 ± 0.00149	0.03318 ± 0.00179	0.05780 ± 0.00750	0.04512 ± 0.00374	0.9454	0.9953	0.9914	0.9520	0.9798
TMA-2	93.43	85.70	85.60	97.90	94.00	0.04351 ± 0.00584	0.02371 ± 0.00550	0.02394 ± 0.00412	0.04810 ± 0.00286	0.03980 ± 0.00205	0.9488	0.8611	0.9184	0.9895	0.9921

*Samples labeled "10 h" were irradiated at the WWR-K reactor (6 MW) for 17 min, equivalent in neutron fluence to 10 h at the critical facility (100 W)

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.

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