

## Microwave-assisted synthesis of M/TiO<sub>2</sub>/C (M=Ni, Cu, Ni–Cu) photocatalysts for CO<sub>2</sub> reduction: structural evolution and photocatalytic properties

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**ABSTRACT** This study presents the synthesis of a TiO<sub>2</sub>-based composite material with transition metal (Ni, Cu) nanoparticles using microwave radiation. The obtained materials were characterised using X-ray powder diffraction, and the size of the nanoparticles was determined using the Scherrer equation. The photocatalytic activity of the synthesised composites was studied in reaction of CO<sub>2</sub> reduction to CO and CH<sub>4</sub> under the visible light with a wavelength of 400 nm. Microwave treatment of a mixture of TiO<sub>2</sub> with transition metal salts (Ni, Cu) and graphite was founded to decrease a photocatalytic activity in CO<sub>2</sub> reduction reaction, while a mechanical mixture of TiO<sub>2</sub> and graphite, not subjected to microwave treatment, demonstrated increased catalytic activity compared to unmodified TiO<sub>2</sub> Evonik P25. The decrease in catalytic activity of the case of microwave-treated samples is associated with an irreversible phase transition of the photoactive anatase phase into the catalytically inert rutile phase and formation of TiO<sub>2-x</sub> phases. This process is induced by overheating during microwave synthesis, where graphite (C<sub>g</sub>) acts as an effective microwave absorber and a reducing agent for Ti<sup>4+</sup> cations in TiO<sub>2</sub>. The obtained results are interesting for the development of efficient TiO<sub>2</sub>-based photocatalysts for CO<sub>2</sub> reduction.

**KEYWORDS** titanium dioxide, photocatalysis, transition metal nanoparticles, carbon dioxide reduction, X-ray diffraction, green chemistry

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### 1. Introduction

Global climate change caused by greenhouse gas emissions is one of the most serious environmental problems of our time. One of the main greenhouse gases contributing to climate change is carbon dioxide [1]. In view of this, developing of effective methods to convert CO<sub>2</sub> into useful chemical compounds or molecules that are more reactive than carbon dioxide is a pressing issue [2].

The photocatalytic reduction of carbon dioxide using TiO<sub>2</sub> is a promising method for converting CO<sub>2</sub> into valuable chemical compounds and is fully consistent with the fundamental principles of “green” chemistry [3]. The main advantage of this approach is the ability to perform the reaction under the influence of sunlight, which minimizes energy costs and reduces the carbon footprint of the process [4]. During the photocatalytic process on TiO<sub>2</sub>, CO<sub>2</sub> can be reduced to various products, including carbon monoxide (CO), methane (CH<sub>4</sub>), methanol (CH<sub>3</sub>OH), and formic acid (HCOOH), etc. [5]. However, the disadvantage of using unmodified titanium dioxide is its low photoactivity and the predominant formation of CO as a product, which limits its practical application as a photocatalyst [6]. A solution to this problem can be

the modification of  $\text{TiO}_2$  with transition metal nanoparticles (TMNPs), such as nickel and copper, including modification with bimetallic particles, that allows not only to increase the efficiency of  $\text{CO}_2$  reduction, but also to control the selectivity of the process towards target products, such as methane and  $\text{C}_{2+}$  products, which are of great practical importance [7–9]. In addition, the modification of titanium dioxide with carbon materials, such as graphene, is promising, allowing the absorption edge of  $\text{TiO}_2$  to be shifted to a longer-wavelength region [10].

The aim of this work is to obtain nanocomposite materials based on  $\text{TiO}_2$ , modified with TMNPs (Ni, Cu) and graphite ( $\text{C}_g$ ), and to investigate their structural and catalytic properties in the process of  $\text{CO}_2$  reduction under visible light irradiation. These materials were synthesized via microwave (MW) assisted thermal decomposition of metal acetylacetone precursors in the presence of graphite under reduced pressure, enabling rapid formation of the nanostructured system.

## 2. Experimental

### 2.1. Materials and measurements

Graphite spectral electrodes (graphite purity is 99.999 %) were used as a source of graphite. The rods were crushed using a metal file, and then the resulting powder was additionally ground in a mortar. The commercially available reagents used in the experiments were copper (II) acetylacetone (Chemkraft, > 99 %), nickel (II) acetylacetone (Chemkraft, > 99.5 %), titanium dioxide (Evonik P25), methanol (purified by distillation). Deionized distilled water was used for photocatalyst synthesis, ultrapure water was used for kinetic experiments. Ultrapure water was produced by “NuZar Q” water system set.

### 2.2. Synthesis of modified titanium dioxide

Commercial  $\text{TiO}_2$  (Evonik P25) was used as a base for the photocatalyst synthesis. To perform MW treatment, a mixture was prepared from titanium dioxide (0.4 g) and metal salts ( $\text{Ni}(\text{acac})_2$  and/or  $\text{Cu}(\text{acac})_2$ ) in an amount corresponding to 10 wt.% of metal. In the case of synthesis of a bimetallic (Ni–Cu) catalyst, the mass fraction of the metals was maintained in a ratio of 1:1, while their total content was also 10 wt.%. The weighed portions of the salt and  $\text{TiO}_2$  were mixed, then 15 – 20 ml of methanol was added until the complete dissolution of the salt. The resulting solution was dispersed in an ultrasonic bath for 1 hour, and after that the solvent was removed by heating in an oil bath. The dried powder was mixed with graphite (0.2 g) and thoroughly ground in an agate mortar until the mixture attained a uniform colour. The resulting mixture was placed in a quartz test tube, which was connected to a vacuum line to create a reduced pressure in the range of 80 – 100 Pa. The tube was placed horizontally, ensuring uniform distribution of the mixture in a thin layer approximately 2 cm long. MW treatment was performed using a Hyundai HYM-M2045 household microwave oven with an output power of 700 W. Irradiation was performed at the power 700 W for 5 minutes. Control samples containing only  $\text{C}_g$  and  $\text{TiO}_2$  were prepared, with ( $\text{MW-TiO}_2/\text{C}_g$ ) and without ( $\text{TiO}_2/\text{C}_g$ ) MW treatment.

### 2.3. Photocatalyst characterization

The prepared photocatalysts were analysed by X-ray diffraction (XRD). XRD patterns of the photocatalysts was obtained using a D8 ADVANCE diffractometer equipped with a LYNXEYE linear detector (Bruker AXS GmbH, Karlsruhe, Germany) at room temperature in the  $2\theta$  of 15 – 85° with a step of 0.05° with Ni-filtered  $\text{Cu K}\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ). The phase composition of the photocatalysts was quantitatively analysed by the Rietveld refinement method using GSAS-II software packages [11]. The coherent scattering region (CSR) of crystallites were estimated from the full width at half maximum of corresponding peaks using the Scherrer formula.

### 2.4. Photocatalytic experiments

The photocatalytic reduction of  $\text{CO}_2$  was carried out in a static glass reactor (170 ml) with a quartz window ( $16 \text{ cm}^2$ ). A 30 mg suspension in 500  $\mu\text{L}$  of ultrapure water was pre-sonicated and deposited on a glass substrate ( $8 \text{ cm}^2$ ), followed by drying at 50 °C in air. Prior to the reaction, the catalyst was illuminated with a 397 nm LED for 15 min to remove organic contaminants from the catalyst surface, then transferred to the reactor containing 1 mL of ultrapure water. The system was first purged with argon for 20 min to remove oxygen, followed by ultrapure  $\text{CO}_2$  (99.995 %) purging for 20 minutes. Then it was illuminated with LED with an intensity maximum at a wavelength of 397 nm. The gas phase composition was determined using a Khromos GC-1000 gas chromatograph equipped with both a flame ionization detector and a thermal conductivity detector. The sample was collected once every 1 hour, the duration of the photocatalytic experiment was 5 hours.

## 3. Results and discussion

In this research, we propose an original method for modification of  $\text{TiO}_2$  by TMNPs (Ni, Cu) based on the reduction of metal salts with graphite under the action of MW radiation. The key advantage of this method is that there is one-stage synthesis of graphene-encapsulated nickel nanoparticles by high-temperature decomposition of nickel acetylacetone on graphite [12], which can contribute to increasing their catalytic activity in the carbon dioxide reduction reaction. The use

of other metal salts containing chlorides or other anions is not desirable as halogens and some other anions can strongly decrease the catalytic activity when present in the catalytic system. Other advantages include the high synthesis rate, environmental friendliness and simplicity of this approach. The study investigated several sample types:  $TiO_2/C_g$  without MW treatment, MW- $TiO_2/C_g$  subjected to MW treatment, 10 %  $Ni/TiO_2/C_g$ , 10 %  $Cu/TiO_2/C_g$ , and a bimetallic sample of 5 %  $Cu$  5 %  $Ni/TiO_2/C_g$  – after MW irradiation.

X-ray diffraction (XRD) analysis confirmed that the sample ( $TiO_2/C_g$ ) contains anatase and rutile, that is typical for the commercial  $TiO_2$  Evonik P25 sample, and heat treatment leads to the phase transition from anatase to rutile (Fig. 1, Table 1). This is consistent with literature data, that this transition occurs in the range of 400 – 1200 °C [13]. At the same time, with MW treatment, the CSR of both anatase and rutile increases, reaching 200 nm in some cases. Carbon in the photocatalysts is represented by nanosized graphite with a mass fraction of 20 – 40 %. In addition, the presence of a metallic phase corresponding to  $M^0$  ( $M=Ni, Cu$ ) can be noted in the diffraction patterns.

It has been found that rutile is reduced to mixed-valence oxides (general formula  $TiO_{2-x}$  where  $0.125 < x < 0.5$ ) during MW processing (Fig. 1). In the case of MW- $TiO_2/C_g$  sample,  $Ti_8O_{15}$ ,  $Ti_6O_{11}$  phases are formed only. The composition of these phases slightly differs from the stoichiometric one. The presence of metals, especially Cu, promotes a deeper reduction of  $Ti^{4+}$  cations in the oxide structure, in particular, with the formation of  $Ti_2O_3$  (Fig. 2). This may be due to the fact that metal ions in oxidation state 2+ are able to reduce the energy required for deformation of the structure and accelerate the transition of anatase to rutile, [14, 15]. The CSR of the resulting TMNPs have been found as 42 nm for 10 %  $Ni/TiO_2/C_g$  sample; 90 nm for 10 %  $Cu/TiO_2/C_g$  sample; for the mixed sample 5 %  $Cu$  5 %  $Ni/TiO_2/C_g$   $d_{NiNPs}$  was equal to 13 nm and  $d_{CuNPs}$  was equal 24 nm. The mass fraction of the metals was close to that laid down during the synthesis, taking into account the measurement error.

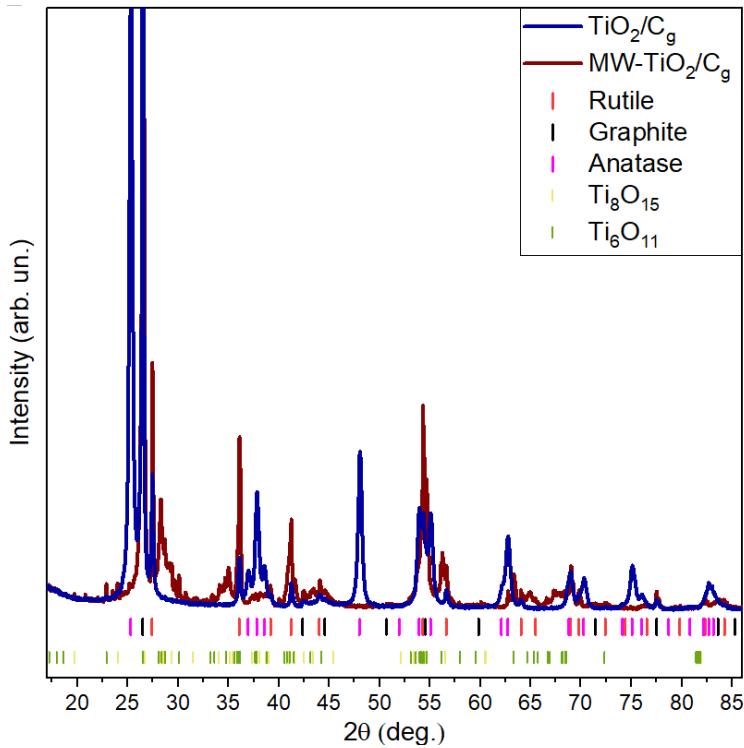


FIG. 1. XRD patterns of  $TiO_2$  before and after MW processing

The obtained samples were investigated in the process of photocatalytic  $CO_2$  reduction. The experiments were conducted in a photocatalytic reactor, where the LED with a maximum of intensity at the wavelength of 397 nm was used as a radiation source (Fig. 3). Based on the data obtained, the rates of the formation of carbon dioxide reduction products were calculated, as well as the total reduction rate ( $\omega(CO_2RR)$ ). The rate of products ( $CH_4, CO$ ) formation was determined by linear approximation of kinetic curves. The total reduction rate was calculated according to the following formula (1):

$$\omega_{e-}(CO_2RR) = 2 \times \omega(CO) + 8 \times \omega(CH_4), \quad (1)$$

where 2 and 8 are the coefficients for accounting the electronic balance,  $\omega(CO)$  and  $\omega(CH_4)$  – the corresponding rates of the formation of  $CO$  and  $CH_4$ , respectively.

The photocatalytic activities of the samples in  $CO_2$  reduction are shown in Fig. 4(a,b) and Table 2. For reference, the experiments using commercial  $TiO_2$  Evonik P25 were performed.

TABLE 1. Structural properties of the photocatalysts based on XRD data

Sample	Phase	Mass fraction, %	CSR size, nm
TiO <sub>2</sub> /C <sub>g</sub>	TiO <sub>2</sub> (anatase)	66(4) <sup>*</sup>	32(2)
	TiO <sub>2</sub> (rutile)	11(1)	56(5)
	C <sub>g</sub> (graphite)	24(5)	68(3)
MW-TiO <sub>2</sub> /C <sub>g</sub>	TiO <sub>2</sub> (rutile)	22(5)	130(50)
	C <sub>g</sub> (graphite)	22(10)	58(5)
	Ti <sub>8</sub> O <sub>15</sub>	37(10)	50(20)
	Ti <sub>6</sub> O <sub>11</sub>	19(5)	50(20)
10 %Cu/TiO <sub>2</sub> /C <sub>g</sub> MW	TiO <sub>2</sub> (anatase)	17(5)	62(10)
	TiO <sub>2</sub> (rutile)	3(1)	70(20)
	C <sub>g</sub> (graphite)	35(10)	62(6)
	Ti <sub>4</sub> O <sub>7</sub>	20(5)	21(5)
	Ti <sub>3</sub> O <sub>5</sub>	5(2)	120(50)
	Ti <sub>2</sub> O <sub>3</sub>	8(2)	110(40)
	Cu	12(3)	90(10)
10 %Ni/TiO <sub>2</sub> /C <sub>g</sub> MW	TiO <sub>2</sub> (anatase)	16(3)	63(2)
	TiO <sub>2</sub> (rutile)	10(2)	45(10)
	C <sub>g</sub> (graphite)	33(10)	76(5)
	Ti <sub>4</sub> O <sub>7</sub>	23(4)	36(10)
	Ti <sub>3</sub> O <sub>5</sub>	8(2)	45(3)
	Ni	11(2)	42(5)
5 %Cu 5 %Ni/TiO <sub>2</sub> /C <sub>g</sub> MW	TiO <sub>2</sub> (anatase)	12(3)	125(50)
	TiO <sub>2</sub> (rutile)	1(0.5)	200(100)
	C <sub>g</sub> (graphite)	39(12)	51(5)
	Ti <sub>4</sub> O <sub>7</sub>	22(6)	18(6)
	Ti <sub>3</sub> O <sub>5</sub>	6(2)	92(50)
	Ti <sub>2</sub> O <sub>3</sub>	7(2)	78(30)
	Cu	5(2)	24(10)
	Ni	7(3)	13(5)

<sup>\*</sup> the error in the last significant digit is indicated in brackets

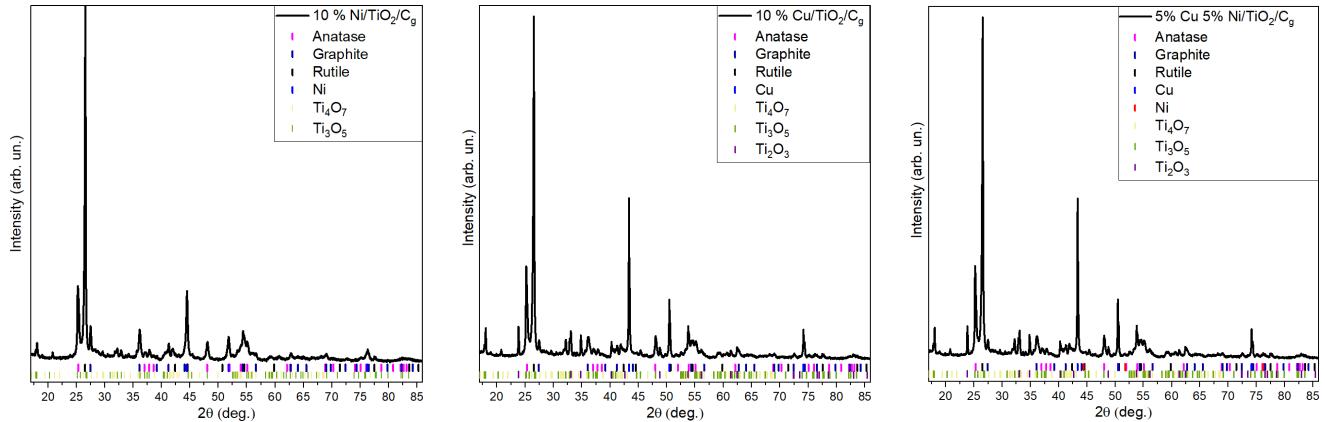


FIG. 2. XRD patterns of  $TiO_2$  modified by TMNPs: 10 % Ni/TiO<sub>2</sub>/C<sub>g</sub> (a), 10 % Cu/TiO<sub>2</sub>/C<sub>g</sub> (b), 5 % Cu 5 % Ni/TiO<sub>2</sub>/C<sub>g</sub> (c)

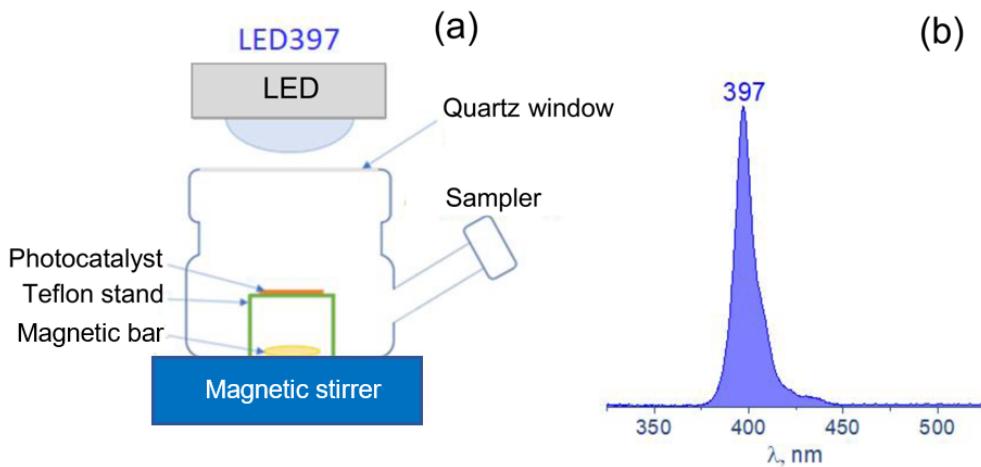


FIG. 3. Photocatalytic reactor used for  $CO_2$  reduction (a) and the spectrum of the LED used (b)

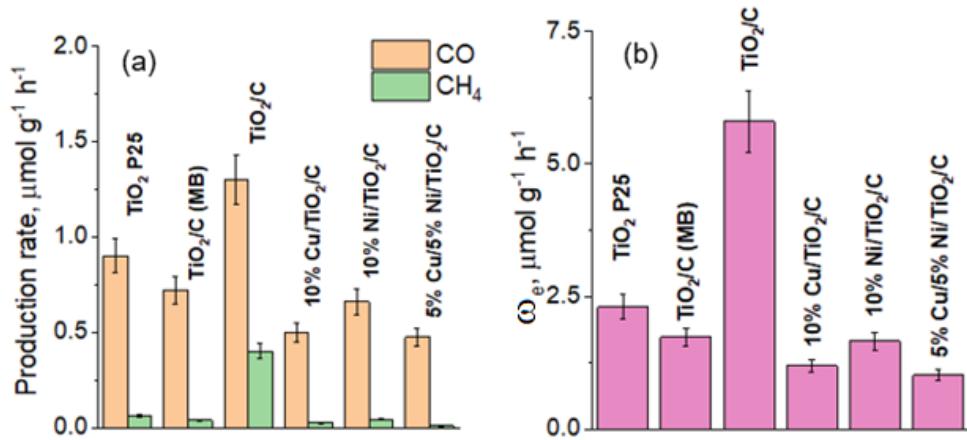


FIG. 4. (a) CO and CH<sub>4</sub> formation rates; (b) total  $CO_2$  reduction rates for  $TiO_2/C_g$  and  $X$  %  $M/TiO_2/C_g$  photocatalysts ( $M =$  Ni, Cu, or Ni–Cu). Conditions:  $M$  (cat.) = 30 mg,  $t$  (reaction) = 5 hours. Light source: LED with  $\lambda = 397$  nm

TABLE 2. Photocatalytic  $\text{CO}_2$  reduction activity of the synthesized catalysts

Catalyst	$\omega(\text{CO})$ , $\mu\text{mol g}_{\text{cat}}^{-1} \text{h}^{-1}$	$\omega(\text{CH}_4)$ , $\mu\text{mol g}_{\text{cat}}^{-1} \text{h}^{-1}$	$\omega_{e-}$ , $\mu\text{mol g}_{\text{cat}}^{-1} \text{h}^{-1}$
MW- $\text{TiO}_2/\text{C}_g$	0.72	0.04	1.74
$\text{TiO}_2/\text{C}_g$	1.30	0.40	5.80
10 % Cu/ $\text{TiO}_2/\text{C}_g$	0.50	0.02	1.19
10 % Ni/ $\text{TiO}_2/\text{C}_g$	0.66	0.04	1.66
5 % Cu/5 % Ni/ $\text{TiO}_2/\text{C}_g$	0.47	< 0.01	1.01
$\text{TiO}_2$ Evonik P25	0.90	0.06	2.28

The highest activity was possessed by the photocatalyst  $\text{TiO}_2/\text{C}_g$ , which was not exposed by MW-irradiation (Fig. 4(a,b)). The activity of this sample is more than two times higher than the activity of the commercial  $\text{TiO}_2$  Evonik P25, which may be related to the shift in the absorption edges of  $\text{TiO}_2$  due to the presence of graphite. It should be noted that the rate of methane formation over this catalyst was higher compared to other samples, suggesting that graphite acts like a metallic cocatalyst (Fig. 2(a)) [16].

For all photocatalysts subjected to MW treatment, a decrease in activity was observed compared to unmodified  $\text{TiO}_2$  Evonik P25. This can be attributed to a decrease in the content of catalytically active phase of anatase and the formation of reduced titanium oxide phases with overall formula  $\text{TiO}_{2-x}$  ( $0.125 < x < 0.5$ ), which do not show photocatalytic activity. In addition, a significant increase in the CSR of titanium oxides results in decreasing of the photocatalytic activity.

#### 4. Conclusion

As the result of this study, it was found that MW reduction of the investigated metals (Ni, Cu) on  $\text{TiO}_2$  surface leads to the formation of a composite material containing Ni and Cu nanoparticles. However, contrary to expectations, such a modification caused a decrease in photocatalytic activity compared to original  $\text{TiO}_2$ . XRD analysis showed that this decrease is due to two main factors: an irreversible phase transition of photoactive anatase to catalytically inert rutile and partial reduction of  $\text{TiO}_2$  during the synthetic process. These processes are initiated by a system local overheating caused by intensive absorption of MW radiation with graphite. The highest catalytic activity was demonstrated by the control sample  $\text{TiO}_2/\text{C}_g$ , which was not subjected to MW, which confirms the negative impact of the modification method on the functional properties of the material. The results obtained are important for the development of  $\text{TiO}_2$  modification strategies, indicating the need to use softer synthetic protocols that exclude structural and phase transformations of an oxide matrix. This work makes a significant contribution to the understanding of restrictions related to the use of microwave synthesis for the modification of photocatalysts, and offers new criteria for the development of effective catalytic systems based on  $\text{TiO}_2$ .

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