

# Microwave-Assisted Synthesis of TiO<sub>2</sub>/GO Composite and Its Adsorption-Photocatalysis Property under Visible Light

*by Sigit Priatmoko*

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**Submission date:** 06-Jul-2023 11:19AM (UTC+0700)

**Submission ID:** 2127103305

**File name:** 67.\_Microwave-Assisted\_Synthesis\_of\_TiO.pdf (1.02M)

**Word count:** 3990

**Character count:** 22037

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To cite this article: S Setiawan *et al* 2021 *IOP Conf. Ser.: Mater. Sci. Eng.* **1143** 012055

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## 1 Microwave-Assisted Synthesis of TiO<sub>2</sub>/GO Composite and Its Adsorption-Photocatalysis Property under Visible Light

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**Abstract.** The photocatalytic performance of low-grade Titanium Dioxide (TiO<sub>2</sub>) anatase was successfully improved by compositing with graphene oxide (GO) via microwave-assisted, activated by UV and Xe light. The various experimental conditions were conducted. X-ray diffraction (XRD), Particle size analyzer (PSA), Prepared TiO<sub>2</sub>/GO photocatalyst was characterized by several instruments such as Specific surface area (BET) and Scanning electron microscopy (SEM). Effect of various annealing times (30 to 120 minutes) and temperatures (200-1000°C) were studied to reach the optimum pure TiO<sub>2</sub> as a further based material in composite with GO. The result showed that both annealing time and temperature influenced crystallinity and particle size only, not the phase structure of TiO<sub>2</sub>. A similar phenomenon was founded by adding GO in a small concentration. A comparison of photocatalytic activity under UV and Xe irradiation has been proposed to show the GO effect independently. Under UV irradiation, the presence of GO enhancing the active sites of the catalyst. 1.0 wt% of GO succeeds in decomposing 8ppm of RhB more than 90%. Meanwhile, under Xe irradiation, the efficiency of the degradation of RhB was only 30%, resulted in using 1.2wt% of GO. Interestingly, the photodecomposition rate (*k*) of TiO<sub>2</sub>/GO composite under Xe irradiation was 3 times over pure TiO<sub>2</sub> higher than that of UV irradiation (1 time). The study present that the small concentration of GO affected not only the active site of the TiO<sub>2</sub>/GO composite but also the electronic properties, which give different effects under different energy activated.

**Keyword:** Photoadsorption, Rhodamine B, Organic dye, Water treatment

### 1. Introduction

Rhodamine B is a commercial azo dye for textile industries with high sunlight energy to decompose [1]. Since their toxicity, the high effort regarding the degradation of this compound from the environment is proposed. Unfortunately, the conventional method to remove Rhodamine B from water, such as precipitation, separation, and adsorption, is inefficient [2]. Almost the technology only converts the non-biodegradable dye into sludge, resulting in further post treatment [3]. Furthermore, green technology to solve this problem is needed.



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For decades, photocatalytic technology attracts environmental remediation applications research. Semiconductor material as a photocatalyst material has been known effectively to decompose organic pollutants [4]. One of the common photocatalyst material is Titanium dioxide ( $\text{TiO}_2$ ). As well as their advantages, their disadvantages, such as fast recombination of the electron ( $e^-$ ) and hole ( $h^+$ ) during the process and broader bandgap energy making their only active under UV irradiation, are the problems[5]. Accordingly, many research works have been proposed. Composite with another semiconductor, metal-doped, and inorganic doped are the several techniques to answer the disadvantageous[5,6].

Among the co-catalyst material, Graphene Oxide (GO) is a promising material that can be considered. GO known as an oxide of grapheme which has excellent adsorption properties[7]. Their reactive oxygen functional groups make this material suitable for forming Van der Waals force with  $\text{TiO}_2$ [8,9]. Moreover, GO could enhance the adsorption process due to their high specific surface area, while,  $\text{TiO}_2$ -GO bonding resulted in decreasing bandgap energy and electron-hole recombination[9]. Composite of  $\text{TiO}_2$  and GO for decompose organic pollutants has been proposed by several researchers [10][11][12][13]. Kumaran et al. proposed the synthesis of nanocomposite GO- $\text{TiO}_2$  for dye wastewater treatment [14]. The studies showed that GO succeeded in the improvement of photoadsorption organic dyes. Unfortunately, almost the reports used  $\text{TiO}_2$  P25, which has been known as a highly photocatalytic activity. Some of them synthesize  $\text{TiO}_2$  from the  $\text{TiO}_2$  salt. Importantly, they only used UV or visible light. Based on those of literature studies, GO's effect on the photocatalytic activity of low-grade  $\text{TiO}_2$  and their comparison study under different energy sources to deeply understanding is hard to find.

Based on that rationale, the purpose of this study was to prepare the  $\text{TiO}_2$ /GO composite by microwave-assisted. Here, low-grade  $\text{TiO}_2$  particles have been used. The low price of the material making this research feasible to be developed on a large scale. Photocatalytic activity of the prepared composite  $\text{TiO}_2$ /GO was studied under UV and Xe light irradiation independently. An interesting result was founded when the GO present affected the catalyst's active site dominantly under UV light and affected the electronic properties dominantly under Xe light irradiation. It is shown by different efficiency and photodecomposition rate results between UV and Xe irradiation.

## 2. Experimental

### 2.1. Material

Low-grade Titanium Dioxide anatase ( $\text{TiO}_2$ , Bratachem) powder has been used as a  $\text{TiO}_2$  source. The materials used to prepare Graphene Oxide (GO) in this study were as follows: Graphite powder (<20 nm, Aldrich), potassium permanganate ( $\text{KMnO}_4$ , Baker), fuming nitric acid ( $\text{HNO}_3 \geq 99.5\%$ , sigma Aldrich), hydrogen peroxide ( $\text{H}_2\text{O}_2$  (aq) 35%, Acros), Nitric acid ( $\text{HNO}_3$  (aq), 69%, Panreac), and hydrochloric acid ( $\text{HCl}$  (aq) 37%), scharlau). Rhodamine B (RhB; Wako pure chemical industries Ltd, Japan) was used as a waste model organic dye.

### 2.2. Preparation of $\text{TiO}_2$ /GO composite

Graphene oxide has been prepared by a modified Hummers method described in detail in our previous work [15]. The annealing process has optimized  $\text{TiO}_2$ . The annealing condition, such as various temperatures (from 200 to 900°C) and various times (from 30 to 120 minutes), was conducted. The optimum generated annealed  $\text{TiO}_2$  was further composited with prepared GO by microwave-assisted synthesis at 300 watts for 10 minutes. A small concentration of GO from 0.6 to 1.2 wt% was directed.

### 2.3. Characterizations

Phase analysis of the prepared particles has been done by X-ray diffraction (XRD, PANALITICAL, with  $\text{Cu K}\alpha$  radiation; angular domain 20-80° ( $2\theta$ )), where the crystallite size was measured by Scherrer's equation[16]. The morphology and surface area were analyzed by Scanning Electron Microscopy (SEM, JEOL JIB 4610F), Tunneling Electron Microscopy (TEM, Tecnai G2 20 S-TWIN), Particle Size Analyser (PSA), and Brunauer-Emmett-Teller (BET), respectively.

To investigate the photocatalytic performance, 8 ppm Rhodamine B dissolved in 250 ml for one minute. Further, the wastewater model was mixed with the prepared photocatalyst (1.6 g/L). The solution was left in the dark for 30 minutes under continuous agitation to established equilibrium (adsorption/desorption) between RhB and the prepared photocatalysts and continuously irradiated by UV-A (365 nm) for 90 minutes. Xenon lamp was used as a visible light source with the wavelength peak at 450 and 540 nm as depicted in Figure 1. Further, photodegradation of RhB was monitored for 90 minutes. The degradation of RhB was measured by UV-Vis spectrophotometer (UV-3150, Shimadzu, Japan).

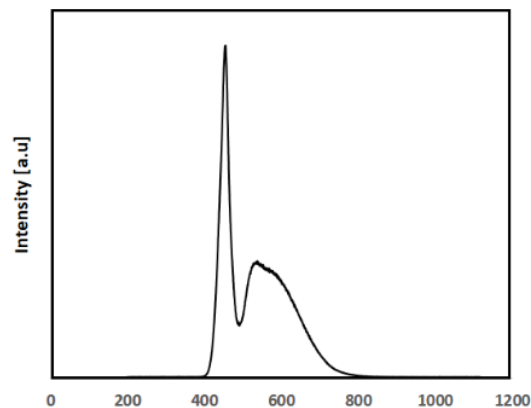


Figure 1. Wavelength characteristic of Xenon lamp.

### 3. Results and Discussion

#### 3.1 Effect of annealing time

Figure 2 depicted the physical properties of annealed-TiO<sub>2</sub> at 600°C for 30 to 120 minutes. The XRD patterns all prepared annealed TiO<sub>2</sub> has a similar peak with raw TiO<sub>2</sub>. The peaks generated at 25.27° [101] and 37.78° [004] represents the anatase phase, confirmed that annealing time did not influence the crystal structure. The annealing times were confirmed affected the crystallite and particle sizes, as shown in Figure 2b. Raw TiO<sub>2</sub>, as a reference, has the smallest at 34.39 nm for crystallite size and 800 nm in particle size. By increasing the annealing time from 30 to 120 minutes, the crystallinity increased from 45.34 up to 47.78 nm. Unlike crystallite size, the particle size of TiO<sub>2</sub> at 30 minutes shows the biggest size at 1054 nm. The further increase of annealing time resulted in a decrease of particle size down to 777 nm.

The photodegradation of RhB under UV irradiation over the times is shown in Figure 3. Annealing temperature from 30 to 90 minutes resulted in the remaining of RhB was at 30%. The highest photocatalytic activity was shown by using annealed TiO<sub>2</sub> at 120 minutes, with the remaining RhB was almost 20%. The photodegradation activity of RhB was significantly influenced by the size of crystallinity and particle of the prepared annealed TiO<sub>2</sub>. The smallest particle size and the biggest crystallite size enhanced the photocatalytic performance. A similar phenomenon was also proposed in detail by Arutanti et al. [17]. Based on this condition, 120 minutes was the optimum annealing time.

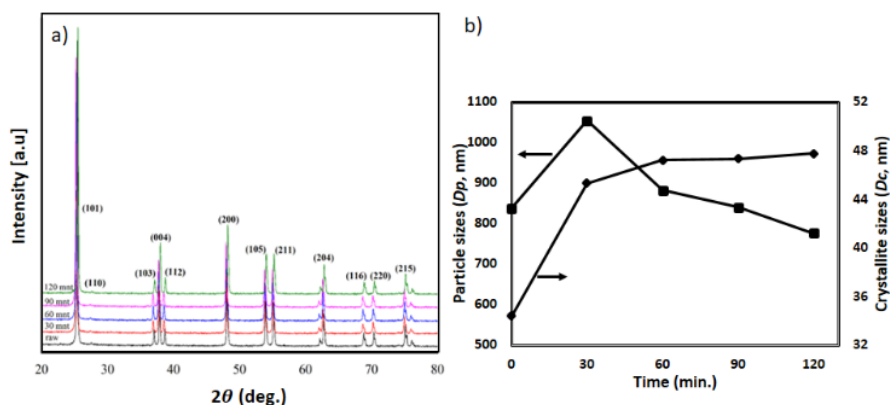


Figure 2. a) XRD patterns of the annealed low-grade TiO<sub>2</sub> for 600°C at different times, and b) correlation the particle and crystallite size of the prepared particles.

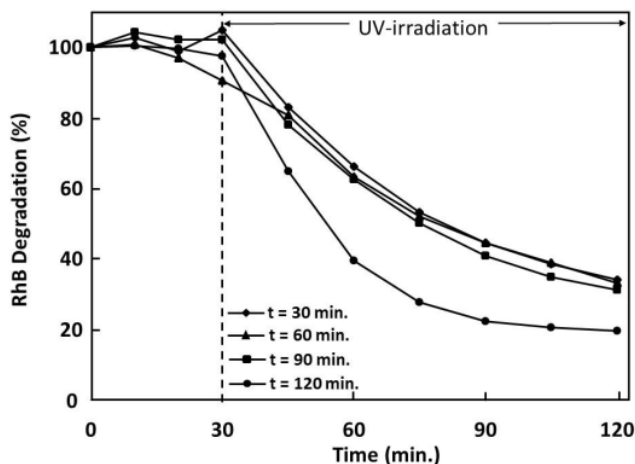


Fig. 3. Effect of annealing time on the photodegradation of 8 ppm RhB under UV-irradiation.

### 3.2 Effect of annealing temperature

The XRD peaks of the annealed TiO<sub>2</sub> from 200 to 1000°C are shown in Figure 4a. The results showed that various annealing temperatures did not affect the phase structure of TiO<sub>2</sub>, except the peak intensity. All prepared TiO<sub>2</sub> peaks referred to anatase structure. Correlation between particle and crystallite size as the effect of annealing temperature is shown in Figure 4b. Due to the sintering effect, particle size increased by increasing the annealing temperature. Over 600°C, generated TiO<sub>2</sub> over 1000 nm.

The photodegradation of RhB under UV irradiation continuously decreased over time by adding annealed TiO<sub>2</sub> (Figure 5a). Annealing temperature influencing the increase of photodegradation efficiency higher than that of raw TiO<sub>2</sub>. The remaining RhB decreased from 45 to 20% by increasing the annealing temperature from 200 to 1000°C. Photocatalytic activity of the annealed TiO<sub>2</sub> from 400 to 1000°C showing a similar efficiency. Even though the crystallinity of annealed TiO<sub>2</sub> increased proportionally with annealing temperature, the particle size also increased. The bigger particle size quite possibly affected decreasing the specific surface area resulted in the lower photocatalytic efficiency due to the surface-active site. This correlation phenomenon has been proven in our previous study [17].



Figure 5b and 5c show the morphology of raw  $\text{TiO}_2$  and annealed  $\text{TiO}_2$  at  $600^\circ\text{C}$ . The SEM images depicted that morphology change was hardly founded. This result inline with Figure 4b. Under different annealing temperatures, the optimum condition to prepare the best  $\text{TiO}_2$  was  $600^\circ\text{C}$  for 120 minutes.

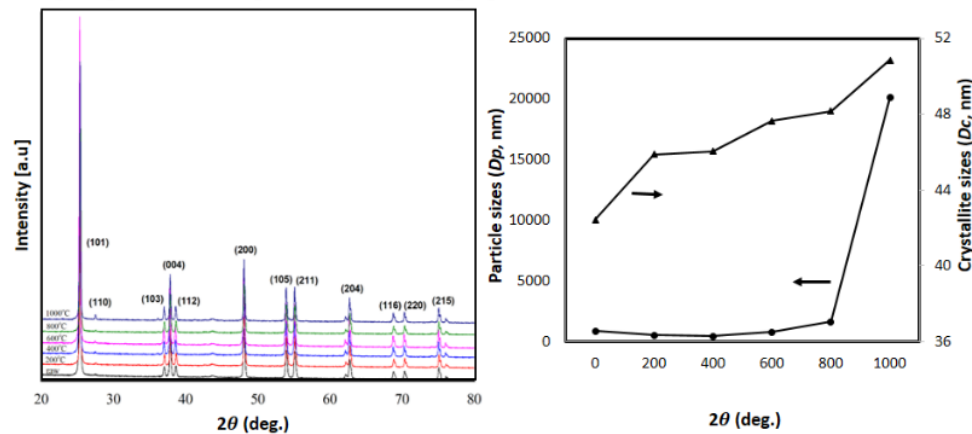


Figure 4. a) Effect of annealing temperature on the XRD patterns of the prepared  $\text{TiO}_2$ , and b) correlation between crystallite and particle size.

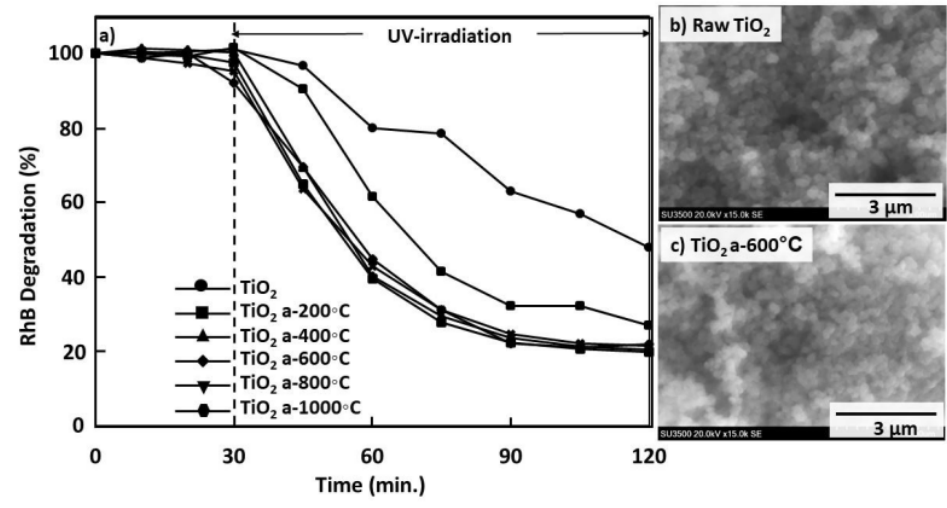


Figure 5. Effect of annealing temperature on the photodegradation of 8 ppm RhB under UV-irradiation with the SEM images of the morphology of low-grade  $\text{TiO}_2$  b) before and c) after annealing process at  $600^\circ\text{C}$  and 120 minutes.

### 3.3 Composite $\text{TiO}_2/\text{GO}$

XRD patterns of the prepared  $\text{TiO}_2/\text{GO}$  composite under various GO ratio from 0.6 to 1.2 wt% was performed. The peak results obtained for the composite are presented in Figure 6. All the diffraction peaks observed correspond to the characteristics  $\text{TiO}_2$  anatase. The expected GO peak (black line) was not found, probably due to their low concentration.

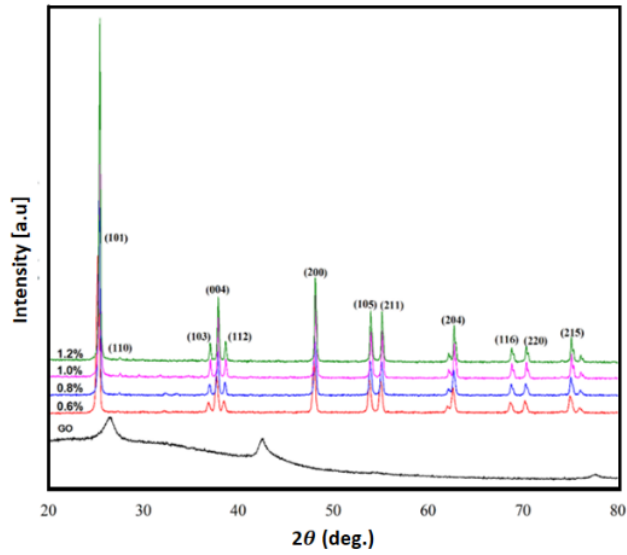
Figure 7 shows the Nitrogen adsorption-desorption isotherm of raw  $\text{TiO}_2$  and composite  $\text{TiO}_2/\text{GO}$ . Porosity was also identified and presented on the table insert the figure. The result shows that the  $S_{BET}$

of composite TiO<sub>2</sub>/GO was lower than raw TiO<sub>2</sub>, while all prepared composite TiO<sub>2</sub>/GO has a similar surface area. Based on BJH analysis, volume pores of raw TiO<sub>2</sub> was higher, possibly due to the cavity between aggregate TiO<sub>2</sub>. Composite of TiO<sub>2</sub> and small concentration GO block the porosity of composite particles. 11

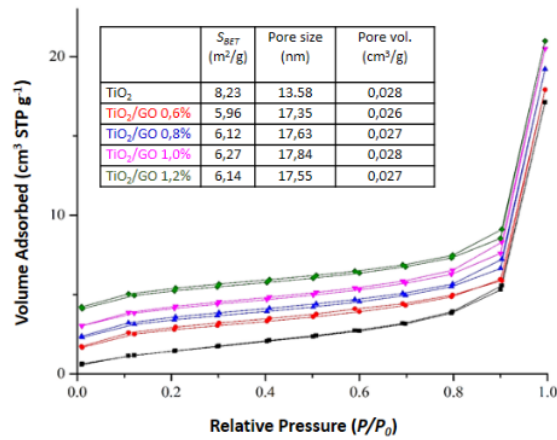
The investigation of the photocatalytic activity of composite TiO<sub>2</sub>/GO through the degradation of RhB under UV and Xe irradiation is plotted in Figure 8a and Figure 8b, respectively. A systematic study was performed on the adsorption process, confirming the prepared photocatalyst's complete saturation in the first 30 minutes under dark condition. Compared to pure TiO<sub>2</sub>, TiO<sub>2</sub>/GO showed better adsorption of RhB. Since the specific surface area of composite TiO<sub>2</sub>/GO and raw TiO<sub>2</sub> was almost comparable, the enhancement of the adsorption process by adding GO was due to electrostatic interaction between RhB and the catalyst surface[8]. Under UV irradiation, composite TiO<sub>2</sub>/GO shows a greater photocatalytic performance than that pure TiO<sub>2</sub> (81%). Regarding the remaining of RhB, 1.0 wt% of GO contributing to the highest efficiency of RhB degradation was 93% after 120 minutes. The presence of sufficient GO concentration increase the life charge carriers resulted in the improvement of interface charge transfer to the photocatalyst surface. Here, additional a higher GO concentration at 1.2 wt% showed the lowest photodegradation or similar to pure TiO<sub>2</sub> at 80%. Heterojunction TiO<sub>2</sub>-GO promotes the charge separation avoiding the recombination between electron and hole. However, the excessive GO concentration generates over states influencing the faster recombination process[18]. Under Xe irradiation, the efficiency of RhB degradation proportional to the increase of GO concentration. 1.2 wt% of GO resulted in the highest efficiency was of 31%, three times higher than that of pure TiO<sub>2</sub>. Additional of GO was effectively improved the photodegradation of RhB under Xe-light. A comparison of photodecomposition rate (*k*) under UV and Xe irradiation is shown in Figure 9. Even though the degradation efficiency of RhB under UV irradiation showed better efficiency than that of under Xe irradiation, the *k* rate of composite TiO<sub>2</sub>/GO over pure TiO<sub>2</sub> slightly increased. The *k* rate of composite TiO<sub>2</sub>/GO over pure TiO<sub>2</sub> was of 1 and 3 times under UV and Xe irradiation, respectively. Since TiO<sub>2</sub> has an energy band gap of around 3.2 eV, which can be activated under UV irradiation, the GO influences the composite catalyst surface activity. The presence of GO enhanced interaction between RhB and the photocatalyst. The presence of GO responsible for introducing new C 2p and O 2p energy states below the conduction band of TiO<sub>2</sub> resulted in the transition of O 2p and Ti 3d as a new state[10,19,20]. Additional energy states influenced the TiO<sub>2</sub>/GO composite photoexcitation of electron and the visible adsorb ability of light. In simply, the presence of sufficient number of GO accommodate facilitates the transportation of electron due to the conductive structure of composite photocatalyst [21]. The summary of comparison of RhB photodegradation by composite TiO<sub>2</sub>/GO during 2020 is proposed in Table 1. A view of paper is just for overview of the previous works related to the composite TiO<sub>2</sub> and GO to decompose RhB. The comparison showed that current result has the lowest photodegradation activity. The different of presentation degradation was due to the different of characteristic from the produced photocatalysts which influenced by material source, experimental method, kind of co-catalyst used. Different from the previous works, here, TiO<sub>2</sub> technical grade priced at 0.6 USD per kG has been used. To enhance the photocatalytic performance, more optimization is still required, but still focus on the utilization of a cheaper material resources. 4

This research proposed the idea of improving the photocatalytic activity of low-grade TiO<sub>2</sub> under visible light. Photoabsorption activity was influenced by physical and electronic properties. The physical properties can be determined by the specific surface area, morphology, particle and crystallite size [17]. Meanwhile, the electronical properties can be verified by electronic band structure and valence state which can be analysed by DR-UV and XPS, respectively [10]. Further optimization is inevitable to reach the optimum photodegradation efficiency.





**Figure 6.** XRD patterns of the prepared photocatalyst TiO<sub>2</sub>/GO composite with different GO ratios.



**Figure 7.** Nitrogen adsorption-desorption isotherm with the insert table of porosity characteristic based on BJH analysis result.

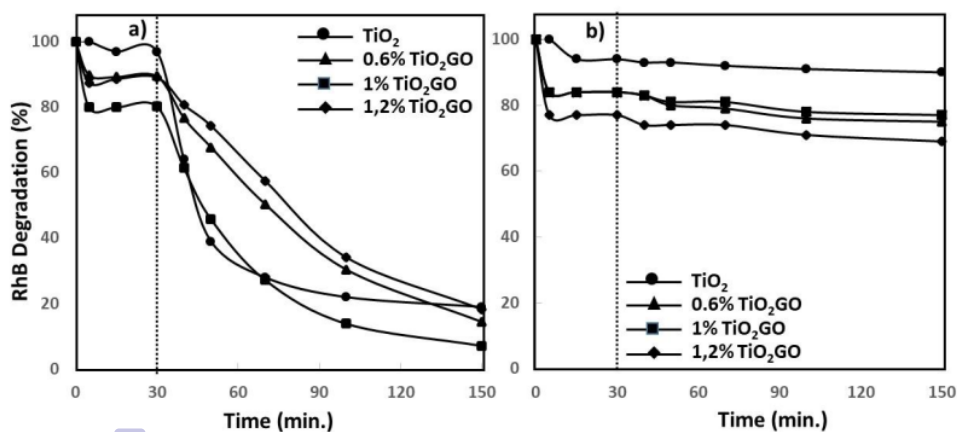


Figure 8. Photocatalytic activity of the prepared  $\text{TiO}_2/\text{GO}$  composite under a) Xe-light, and b) UV-light irradiation.

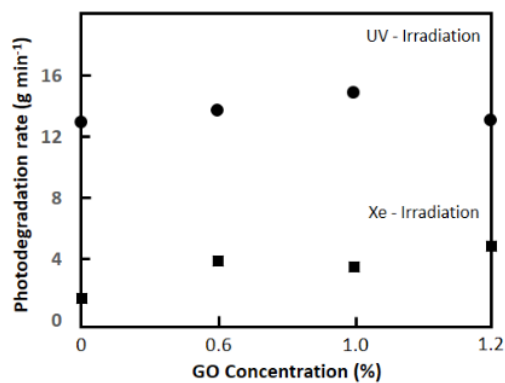


Figure 9. Photodecomposition rate under UV- and Xe- irradiation.

Table 1. Summary of photodegradation of RhB.

No.	Material	Light source	C, V, catalyst	%, t	Ref.
1.	$\text{TiO}_2/\text{GO}/\text{H}_2\text{O}_2$	UV-light	10 ppm, 250 ml,	100 %, 80 min	[22]
2.	$\text{TiO}_2/\text{GA}$	Xe-lamp	20 mg $\text{L}^{-1}$ , 25, 5 mg	90%, 60 min	[23]
3.	$\text{AgNw}/\text{TiO}_2/\text{GO}$	Xe-lamp	12 mg $\text{L}^{-1}$ , 30 mg	90%/, 100 min	[24]
4.	$\text{TiO}_2/\text{GO}$	Xe-Lamp	8 ppm, 250 mL, 40 mg	30%, 120 min	current

#### 4. Conclusion

A comprehensive study related to the improvement of photocatalytic activity of low-grade  $\text{TiO}_2$  under UV and Visible light has been proposed. Additional GO in small concentration successfully enhanced

the photodegradation efficiency and photodecomposition rate. The existence of GO under UV irradiation influencing the active surface sites of photocatalyst TiO<sub>2</sub>/GO. Where 1.0 wt% is the optimum condition to increased efficiency one time over pure TiO<sub>2</sub>, almost 100% of Rhb succeed to decompose. In comparison, GO's presence could enhance composite TiO<sub>2</sub>/GO three times over than that of pure TiO<sub>2</sub>. Under Xe irradiation, the presence of GO dominantly influenced the electronic properties of the prepared composite TiO<sub>2</sub>/GO. Unfortunately, the efficiency of photodegradation under visible light is not optimum. Therefore, further optimization and study related to surface active sites of the composite TiO<sub>2</sub>/GO is required.

### 5. Acknowledgment

This research was financially supported by KURITA Water and Environment Foundation No. 19Pid007, and ELSA LIPI – Indonesian Institute of Sciences.

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PAGE 1

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PAGE 2

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PAGE 3

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PAGE 4

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PAGE 5

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PAGE 6

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PAGE 7

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PAGE 8

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PAGE 9

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PAGE 10

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PAGE 11

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