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**International Conference on Life and Applied Sciences for Sustainable Rural Development (ICLAS-SURE)** is an annual international event organized by Institute of Research and Community Service, Universitas Jenderal Soedirman (Unsoed), Indonesia. Universitas Jenderal Soedirman (Unsoed) is one of the outstanding National University in Indonesia, which is located in Purwokerto, Central Java, Indonesia. This university was established by Minister of Higher Education and Science, Republic Indonesia, based on Presidential Decree No. 195 dated September 23, 1963. Since 1963, Universitas Jenderal Soedirman has been experiencing on rural resource development as well as community services.

Following the success of the 1<sup>st</sup> and 2<sup>nd</sup> **ICLAS-SURE**, this year, the **Institute of Research and Community Service, Universitas Jenderal Soedirman**, organize **The 3rd ICLAS-SURE**. The vision of Jenderal Soedirman University is to be **globally recognized as a university that focuses on sustainable rural and local wisdom development**. Hopefully, this core competence in sustainable rural development shall initiate the university to be nationally and internationally renowned as the center of rural community empowerment. To achieve this vision and cope with the COVID 19 pandemic, this year, we bring the particular theme, "Interdisciplinary approaches and applied technologies for sustainable rural-environmental resources based on local wisdom before and during COVID-19 pandemic". COVID-19 has led to a significant loss of output, employment, and income, affecting rural development. To develop a sustainable rural development, we must fulfil three basic needs, i.e. people welfare improvement, protection of natural, landscape, and cultural resources, and food security through a sustainable farming production.



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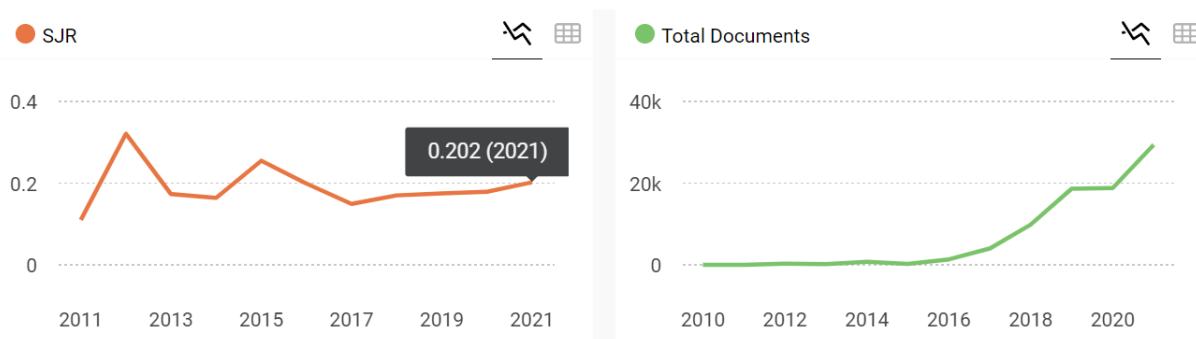


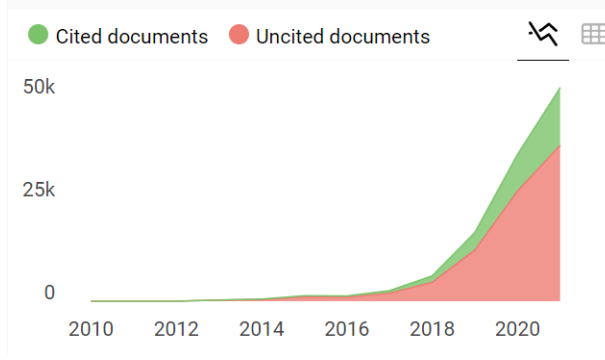
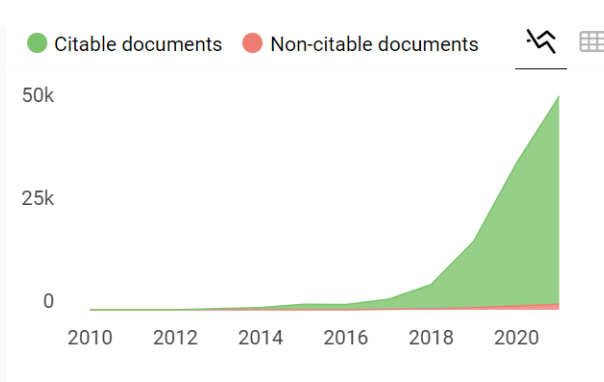
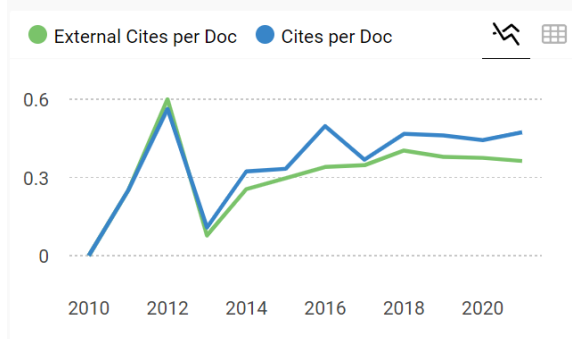
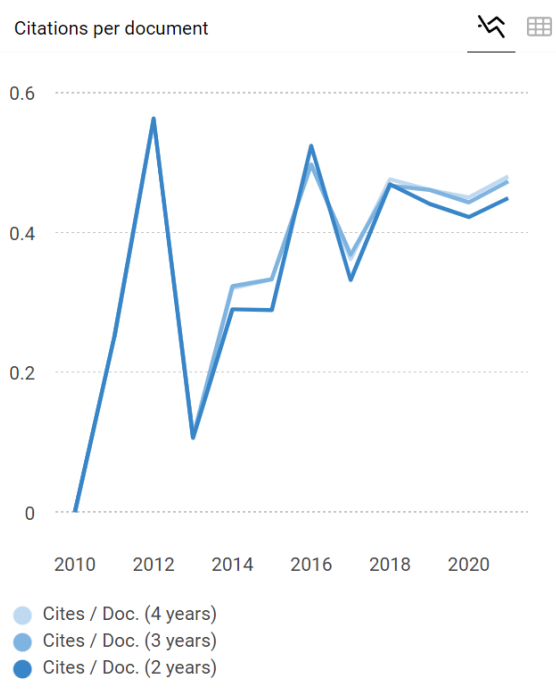
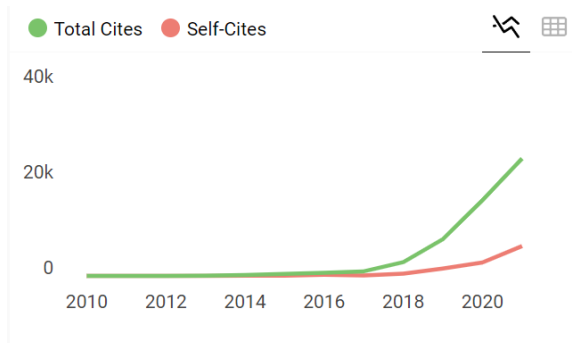
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## The synthesis of $\text{Ag}_3\text{PO}_4$ under graphene oxide and hydroxyapatite aqueous dispersion for enhanced photocatalytic activity

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# The synthesis of $\text{Ag}_3\text{PO}_4$ under graphene oxide and hydroxyapatite aqueous dispersion for enhanced photocatalytic activity

U Sulaeman\*, R D Permadi, H Diastuti

Department of Chemistry, Jenderal Soedirman University, Purwokerto, 53123  
Indonesia

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**Abstract.** The development of  $\text{Ag}_3\text{PO}_4$  photocatalyst for organic pollutant degradation is very challenging due to excellent activity under visible light exposure. The research aims to synthesize  $\text{Ag}_3\text{PO}_4$  under graphene oxide (GO) and hydroxyapatite (HA) as a phosphate ion source for Rhodamin B degradation. The  $\text{Ag}_3\text{PO}_4/\text{GO}$  was prepared using the precipitation method with the starting material of graphene oxide aqueous dispersion,  $\text{AgNO}_3$ , and hydroxyapatite suspension. The structure, absorption, morphology, and element composition of photocatalysts were studied using XRD, DRS, SEM, and EDX. Photocatalytic abilities of the samples were tested using RhB oxidation under blue light exposure. The results exhibited that GO improves the crystallinity and visible absorption spectrum of  $\text{Ag}_3\text{PO}_4$ . Incorporating GO on  $\text{Ag}_3\text{PO}_4$  decreases the ratio of O/Ag and O/P leading to a defect formation. The reaction mechanism on the surface of the photocatalyst was mainly run by holes and superoxide radical ions. The modification of  $\text{Ag}_3\text{PO}_4$  using hydroxyapatite and GO improved photocatalytic activity.

## 1. Introduction

Recently, the utilization of graphene oxide (GO) on the synthesis of silver phosphate-based photocatalyst has greatly developed. This modification has significantly improved the performance of photocatalysts. GO has potential applications due to good thermal stability, flame resistance, and mechanical performance [1]. The application of GO on  $\text{Ag}_3\text{PO}_4$  can increase adsorption performance [2,3], expand the visible light absorption [4,5], enhance the photogenerated charge separation efficiency [6–8], and improve the charge collection efficiency [9]. The immobilization of  $\text{Ag}_3\text{PO}_4/\text{GO}$  composite on the nickel foam improves the adsorption ability [2]. This design bringing the photogenerated electrons is highly transferred away, leading to a stable and efficient photocatalyst. The design of  $\text{Ag}_3\text{PO}_4/\text{graphene oxide}$  aerogel composites using the hydrothermal method increases the specific surface area that improves the adsorption performance [3]. The incorporation of GO into  $\text{Ag}_3\text{PO}_4$  can influence the absorption properties, such as redshift absorption [5]. GO can improve both the visible region's absorption and adsorption properties after coupling with  $\text{Ag}_3\text{PO}_4$  [4]. GO is also a good electron acceptor that can capture photoexcited electrons and enhance the electron transfer and charge separation [6]. In the composite of  $\text{ZnO}/\text{GO}/\text{Ag}_3\text{PO}_4$ , GO can act as a bridge between ZnO and  $\text{Ag}_3\text{PO}_4$  that can increase the transmission rate [7]. This composite showed higher adsorption, a more effective separation of hole and electron, and a higher rate of electron transfer. This phenomenon was



also found in GO-Ag<sub>3</sub>PO<sub>4</sub>/Bi<sub>2</sub>O<sub>3</sub>, GO can serve as a facilitator to transfer the photoexcited electrons from the CB (conduction band) of Bi<sub>2</sub>O<sub>3</sub> to the VB(valence band) of Ag<sub>3</sub>PO<sub>4</sub>, generating the Z-scheme reaction [8].

The improvement of Ag<sub>3</sub>PO<sub>4</sub> photocatalyst can also be supported by hydroxyapatite (HA). The Ag<sub>3</sub>PO<sub>4</sub>/HA composite design generated a redshift and high absorption in visible and UV regions that lead to improved catalytic properties [10]. The catalytic improvement was also provided through a synergistic effect of HA, carbon dots, and Ag<sub>3</sub>PO<sub>4</sub> as found in the composite of HA/N-doped carbon dots/Ag<sub>3</sub>PO<sub>4</sub> [11]. This modification successfully increased active sites. Coupling the Ag<sub>3</sub>PO<sub>4</sub> and HA enhanced catalytic performance through a vacancy of HA that was created under irradiation leading to a Z-scheme reaction [12]. Ag<sub>3</sub>PO<sub>4</sub>/HA composites can also have adsorption properties for Pb(II) [13]. This phenomenon could be applied for Pb(II) immobilization, which was very beneficial for water treatment. Interestingly the HA can be utilized as a phosphate source of Ag<sub>3</sub>PO<sub>4</sub> [14]. This preparation successfully enhances the absorption spectrum in the visible region, decreases the particle size, and changes the mechanism of active species.

Based on the above reports, coupling GO on Ag<sub>3</sub>PO<sub>4</sub> using hydroxyapatite is very promising. The GO can improve the separation of photoexcited electrons and holes, and hydroxyapatite can enhance absorption in the visible region. The experiment aims to incorporate the graphene oxide on Ag<sub>3</sub>PO<sub>4</sub> that is synthesized under hydroxyapatite suspension. This method is new in the application of GO and hydroxyapatite for Ag<sub>3</sub>PO<sub>4</sub> preparation. It has not yet been reported by other researchers. The results showed that the simultaneous design using GO and hydroxyapatite increased the crystallinity and visible absorption of Ag<sub>3</sub>PO<sub>4</sub>. This modification might induce defect formation in Ag<sub>3</sub>PO<sub>4</sub>. These phenomena improve the efficiency of separation of hole and electron, leading to high catalytic activity.

## 2. Materials and Methods

### 2.1. Materials

The materials of AgNO<sub>3</sub> (Merck), CaCl<sub>2</sub> (Merck), KH<sub>2</sub>PO<sub>4</sub> (Merck), ethylenediamine (Merck), and graphene oxide aqueous dispersion (5 mg/mL) (Goographene, USA), were used in the synthesis of the photocatalyst. The Rhodamine B (Merck) was used as a dye for the analysis of photocatalytic activity.

### 2.2. Synthesis

The Ag<sub>3</sub>PO<sub>4</sub>/GO was synthesized using the starting material of AgNO<sub>3</sub>, graphene oxide aqueous dispersion, and hydroxyapatite (Ca<sub>10</sub>(PO<sub>4</sub>)<sub>6</sub>(OH)<sub>2</sub>) as a source of silver and phosphate respectively. The hydroxyapatite (HA) was synthesized using CaCl<sub>2</sub> and KH<sub>2</sub>PO<sub>4</sub> at pH 8 adjusted using ethylenediamine [14,15]. The co-precipitation method was applied to prepare the photocatalyst of Ag<sub>3</sub>PO<sub>4</sub>/GO. The quantity of 0.45 g of graphene oxide aqueous dispersion (5 mg/mL) was added to the AgNO<sub>3</sub> solution (1 g of AgNO<sub>3</sub> in 10 mL of water). This mixture was added to the hydroxyapatite suspension (0.3 g of HA in 20 mL of water), mixed under a magnetic stirrer for 30 minutes. The precipitates were filtered, washed with water three times, and dried at 105°C for 5 hours. The Ag<sub>3</sub>PO<sub>4</sub> without graphene oxide was also prepared with a similar procedure.

### 2.3. Characterization

The structure of Ag<sub>3</sub>PO<sub>4</sub> and Ag<sub>3</sub>PO<sub>4</sub>/GO were characterized using the XRD (Rigaku Miniflex 600), operating at 40 kW, 15mA, using Cu. The morphology and atomic composition were analyzed using SEM-EDX (JEOL, JSM-6510). The morphology magnification of 15000 times was set at 20 kV. The composition was analyzed using ZAF Method Standardless Quantitative Analysis at 20 kV, with a magnification of 3000 times, a counting rate of 3232 cps, and an energy range of 0-20 keV. Absorptions were analyzed using UV-vis DRS (JASCO V-670) with a wavelength range of 320-700 nm.



#### 2.4. Photocatalytic Activity

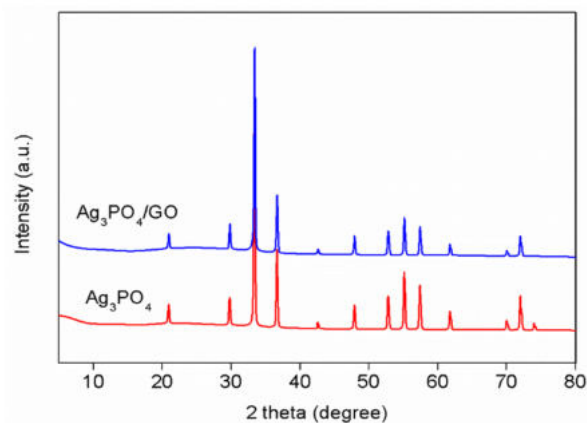
The photocatalytic ability of  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$  were examined using RhB oxidation under the blue LED lamp (Duralux, 3 Watt) [14,16]. The catalyst (0.1 g) was mixed with RhB solution (100 mL, 10 mg/L). The dark treatment and photocatalytic reactions were set at 10 and 8 minutes, respectively. The solution (5 ml) was taken out every 2 minutes and separated from the catalyst using centrifugation. The RhB concentration was monitored by the spectrophotometer. The catalytic recyclability was evaluated up to 3 cycles of 1<sup>st</sup>, 2<sup>nd</sup>, and 3<sup>rd</sup> reactions.

### 3. Results and Discussion

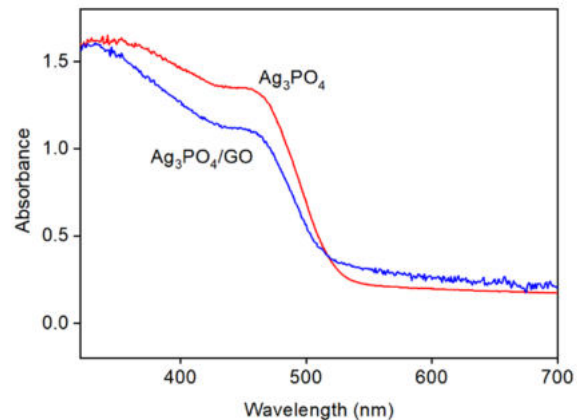
The  $\text{Ag}_3\text{PO}_4$  was successfully designed using  $\text{AgNO}_3$ , hydroxyapatite, and graphene oxide. The body-centered cubic structure was created in both  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$  (JCPDS No. 06-0505) [17] (figure 1). Figure 2 showed the absorption of  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$  at 320-700 nm. The broad absorption above 520 nm was observed in  $\text{Ag}_3\text{PO}_4/\text{GO}$ . This phenomenon might be originated from the formation of the defect site. The absorption coefficient and the band-gap can follow the direct transition of Tauc's relation [18,19]:

$$(\alpha h\nu)^2 = B(h\nu - E_g) \quad (1)$$

where  $E_g$ ,  $h$ ,  $\alpha$ ,  $\nu$ , and  $B$  is a bandgap, Planck constant, absorption coefficient, light frequency, and a constant, respectively. The optical bandgap of the two samples was similar (2.44 eV).



**Figure 1.** XRD profile of  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$



**Figure 2.** Absorption spectra of  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$  measured using DRS instrument

The diffraction peak of graphene oxide is not detected due to a very small GO impregnated on the surface of  $\text{Ag}_3\text{PO}_4$ . The addition of GO did not change the structure, however, it can affect the intensity of diffraction. The higher intensity was observed in  $\text{Ag}_3\text{PO}_4/\text{GO}$  suggested that the GO can improve the crystallinity. It is also found that the FWHM and 2 theta of  $\text{Ag}_3\text{PO}_4/\text{GO}$  are higher than that of  $\text{Ag}_3\text{PO}_4$  (Table 1). The three highest peaks at  $33.358^\circ$ ,  $36.624^\circ$ , and  $55.112^\circ$  could be found in the sample of  $\text{Ag}_3\text{PO}_4$  for (210), (211), and (320) diffractions, respectively. After incorporating the GO, the 2 theta shifted to  $33.378^\circ$ ,  $36.669^\circ$ , and  $55.130^\circ$ . The distance of shift was found at  $0.020^\circ$ ,  $0.045^\circ$  and  $0.018^\circ$  for (210), (211), and (320) diffractions, respectively. Among these shifts, the crystalline plane of {211} is more affected, suggesting that the defect might be higher created on this plane. This phenomenon occurred because the defect can affect the crystalline planes [20].

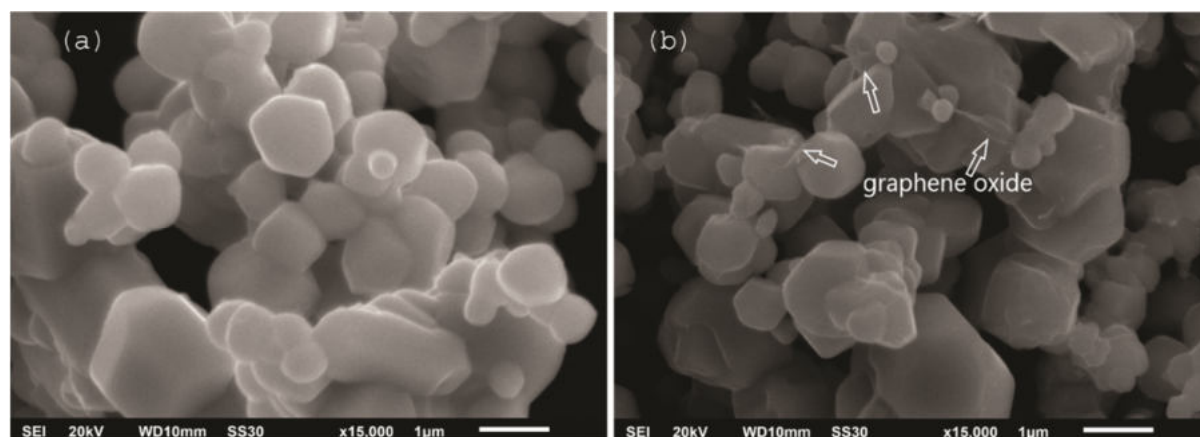
The morphology of  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$  were investigated, the results can be seen in figure 3a and figure 3b. The morphology of the two particles was not significantly changed after incorporating

GO. The crystal shape of samples is irregular ranging from 0.42  $\mu\text{m}$  to 2  $\mu\text{m}$ . A thin layer of graphene oxide was observed on the surface of  $\text{Ag}_3\text{PO}_4/\text{GO}$ . The GO forms a super thin layer that is strongly attached to the  $\text{Ag}_3\text{PO}_4$ . Due to the hydroxyl and epoxide, a bond bridge between the GO and the cubic  $\text{Ag}_3\text{PO}_4$  might form [21].

**Table 1.** Comparison of XRD data from the sample of  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$

Sample	2 $\theta$	d ( $\text{\AA}$ )	FWHM	Height (Counts)
$\text{Ag}_3\text{PO}_4$	33.358(3)	2.6839(2)	0.2043(19)	1270(36)
$\text{Ag}_3\text{PO}_4/\text{GO}$	33.378(3)	2.6823(3)	0.2174(17)	1520(33)

The elements of the sample were successfully analyzed using SEM-EDX and the atomic composition can be seen in table 2. A large impurity of carbon was formed in the precipitate of the samples. This impurity might be originated from the carbonate in the solution. Incorporation of GO on  $\text{Ag}_3\text{PO}_4$  decrease the carbon impurity. Interestingly, the calcium ion from hydroxyapatite was not observed in SEM-EDX, indicating that the  $\text{Ca}^{2+}$  could not be precipitated and easily dissolved in water, whereas phosphate ion was successfully co-precipitated with silver forming  $\text{Ag}_3\text{PO}_4$ .



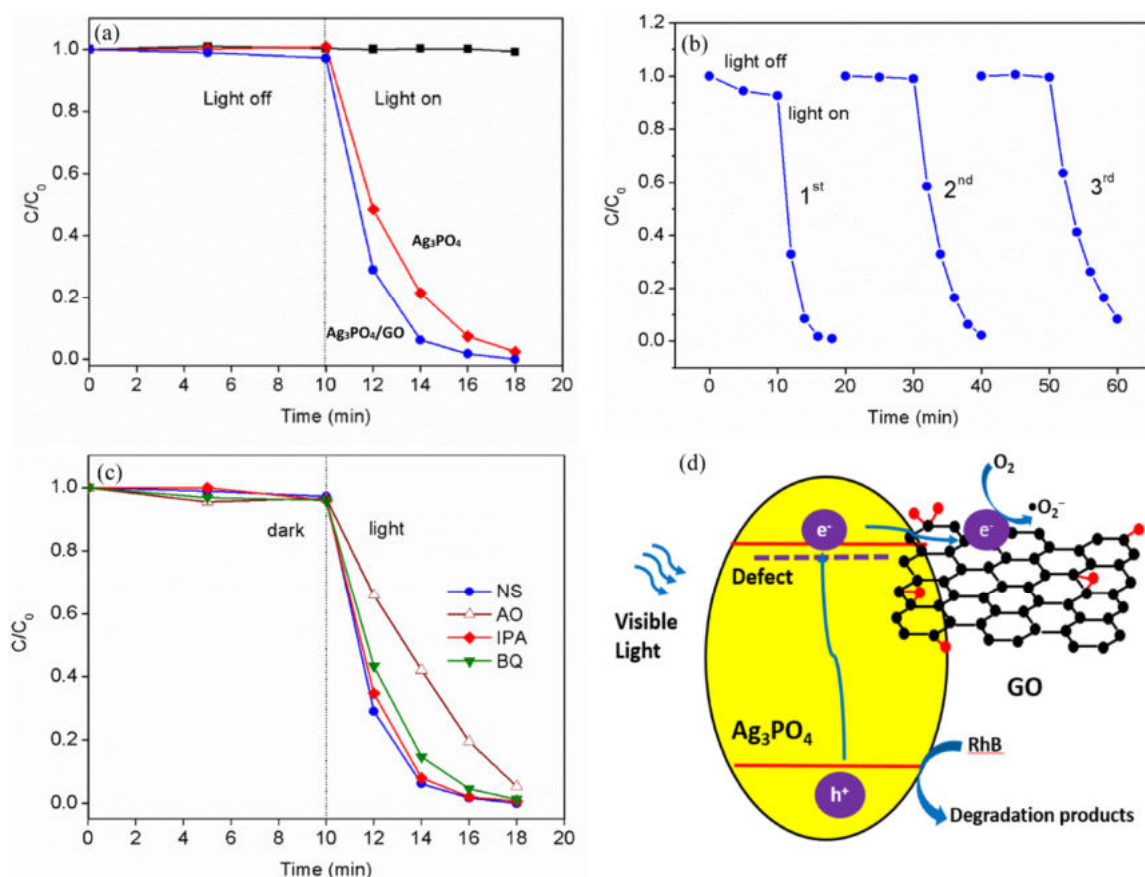
**Figure 3.** SEM images of  $\text{Ag}_3\text{PO}_4$  (a) and  $\text{Ag}_3\text{PO}_4/\text{GO}$  (b)

**Table 2.** Atomic composition (%) from the SEM-EDX measurement of  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$ .

Atom (%)	$\text{Ag}_3\text{PO}_4$	$\text{Ag}_3\text{PO}_4/\text{GO}$
Ag	10.35	18.78
P	3.61	6.64
O	36.78	46.40
C	46.43	23.01
Cu	0.52	1.07
Zn	0.35	0.89
Cd	0.82	1.28
Ar	1.14	1.94

Due to high carbon impurity, the precise investigation of the sample differences should be in the atomic ratio. The atomic ratios of P/Ag, O/Ag, and O/P in  $\text{Ag}_3\text{PO}_4$  can be estimated at 0.35, 3.55, 10.2, respectively, whereas in the  $\text{Ag}_3\text{PO}_4/\text{GO}$ , they were 0.35, 2.47, and 6.99, respectively. The sample of  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$  has a similar atomic ratio of P/Ag but the atomic ratio of O/Ag and O/P in  $\text{Ag}_3\text{PO}_4/\text{GO}$  is lower than that of  $\text{Ag}_3\text{PO}_4$ , indicating that the incorporation of GO might influence the environment of co-precipitation. The lower ratio of O/P in  $\text{Ag}_3\text{PO}_4/\text{GO}$  might be originated from the oxygen vacancy phenomenon.

The photocatalytic abilities of  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$  were investigated using RhB oxidation. The results can be seen in figure 4a. The pseudo-first-order reaction was utilized to investigate the profile of photocatalytic activity with the equation of  $\ln(C_0/C_t) = kt$ ,  $C_t$  and  $C_0$  are concentration at  $t$  time and initial concentration of photocatalytic reaction,  $k$  is the rate constant [16]. The pseudo-first-order reaction occurred in both  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$  with the rate constant of  $0.455 \text{ min}^{-1}$  and  $0.670 \text{ min}^{-1}$ , respectively. The  $\text{Ag}_3\text{PO}_4/\text{GO}$  showed faster reaction activity (1.5 times faster than the  $\text{Ag}_3\text{PO}_4$ ). Many results showed that the utilization of GO increased the adsorption [2,3], however, due to the low amount of GO impregnated on  $\text{Ag}_3\text{PO}_4$ , the adsorption in the dark condition is not so high.



**Figure 4.** Photocatalytic activity of  $\text{Ag}_3\text{PO}_4$  and  $\text{Ag}_3\text{PO}_4/\text{GO}$  (a), Photocatalytic cycling of  $\text{Ag}_3\text{PO}_4/\text{GO}$  (b), the effect of scavenger to photocatalytic in  $\text{Ag}_3\text{PO}_4/\text{GO}$  (c) NS=no scavenger, AO=ammonium oxalate, IPA=isopropyl alcohol, BQ=benzoquinone, and the proposed mechanism of photocatalytic activity in  $\text{Ag}_3\text{PO}_4/\text{GO}$  (d).

Recycled catalytic activity was also investigated (figure 4b). The catalytic activity decreased after cyclic reaction up to three times. The rates of photocatalytic reaction are  $0.684 \text{ min}^{-1}$ ,  $0.377 \text{ min}^{-1}$ , and  $0.243 \text{ min}^{-1}$  for the reaction of 1<sup>st</sup>, 2<sup>nd</sup>, and 3<sup>rd</sup>, respectively. The decreased activity might be caused by the photoreduction of  $\text{Ag}^+$  to  $\text{Ag}^0$ . It suggested that although the photogenerated electrons can be highly separated through the GO, they have still reduced  $\text{Ag}^+$  ions leading to photo-corrosion. Another reason is due to lower adsorption in the 2<sup>nd</sup> and 3<sup>rd</sup> reactions. The 1<sup>st</sup> reaction showed the adsorption in the dark condition, whereas 2<sup>nd</sup> and 3<sup>rd</sup> did not show the adsorption. This problem might be generated by the reaction 1<sup>st</sup> that can break the bond of GO from the  $\text{Ag}_3\text{PO}_4$  leading to low adsorption on the surface.

The mechanisms of photocatalytic in  $\text{Ag}_3\text{PO}_4/\text{GO}$  were studied using BQ (benzoquinone), AO (ammonium oxalate), and IPA (isopropyl alcohol) to scavenge the species of  $\cdot\text{O}_2^-$ ,  $h^+$ , and  $\cdot\text{OH}$ ,

respectively[14]. The results were shown in figure 4c. The AO addition in reaction significantly quenched the photocatalytic reaction, showing that the reaction in the surface of  $\text{Ag}_3\text{PO}_4/\text{GO}$  mostly runs via the  $\text{h}^+$ . The mechanism runs in the following order:  $\text{h}^+ > \cdot\text{O}_2^- > \cdot\text{OH}$ . The high role of  $\text{h}^+$  in the mechanism might be generated by highly transferring a photogenerated electron to GO. When the  $\text{Ag}_3\text{PO}_4/\text{GO}$  was exposed by the light, the electron in the VB of  $\text{Ag}_3\text{PO}_4$  can be excited to the CB, producing a hole in the VB. The photogenerated electron in the CB transfers to GO, therefore the hole acts more efficiently to oxidize the RhB. The proposed mechanism in the surface reaction is shown in figure 4d.

The high role of the reaction mechanism is also through a superoxide radical ion. Because GO is a powerful electron acceptor, it can easily capture the photoexcited electrons. The photoexcited electron on the surface of GO could create a reduction reaction to produce a superoxide radical ion[22]. The GO on the surface of  $\text{Ag}_3\text{PO}_4$  improved the separation of photoexcited electron and hole pair, leading to enhanced photocatalytic activity. The lattice defects generated by GO can serve as traps for electron trapping, which will also improve the separation of electrons and holes [6,23].

The role of  $\cdot\text{OH}$  is not significant in the photocatalytic reaction mechanism. The  $\cdot\text{OH}$  could be highly produced when water or hydroxyl ion ( $\text{OH}^-$ ) adsorbed in the surface and reacted with hole producing  $\cdot\text{OH}$ . However, in this case, the adsorbates (RhB) might stronger be trapped by a hole under irradiation leading to decreased  $\cdot\text{OH}$  formation on the surface of  $\text{Ag}_3\text{PO}_4$ .

#### 4. Conclusion

The co-precipitation of  $\text{Ag}_3\text{PO}_4$  using the starting material of  $\text{AgNO}_3$ , graphene oxide, and hydroxyapatite was successfully synthesized. The graphene oxide improves the crystallinity, decreases the impurity, and forms the defect in the surface of  $\text{Ag}_3\text{PO}_4$ . The photocatalytic reaction of  $\text{Ag}_3\text{PO}_4/\text{GO}$  runs faster than the  $\text{Ag}_3\text{PO}_4$ . The enhanced photocatalytic activity was caused by improving the separation of photoexcited electrons and holes in the surface. The mechanism of the photocatalytic reaction was carried out by hole as a main role, and superoxide radical ion as a second role.

#### 5. Acknowledgment

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