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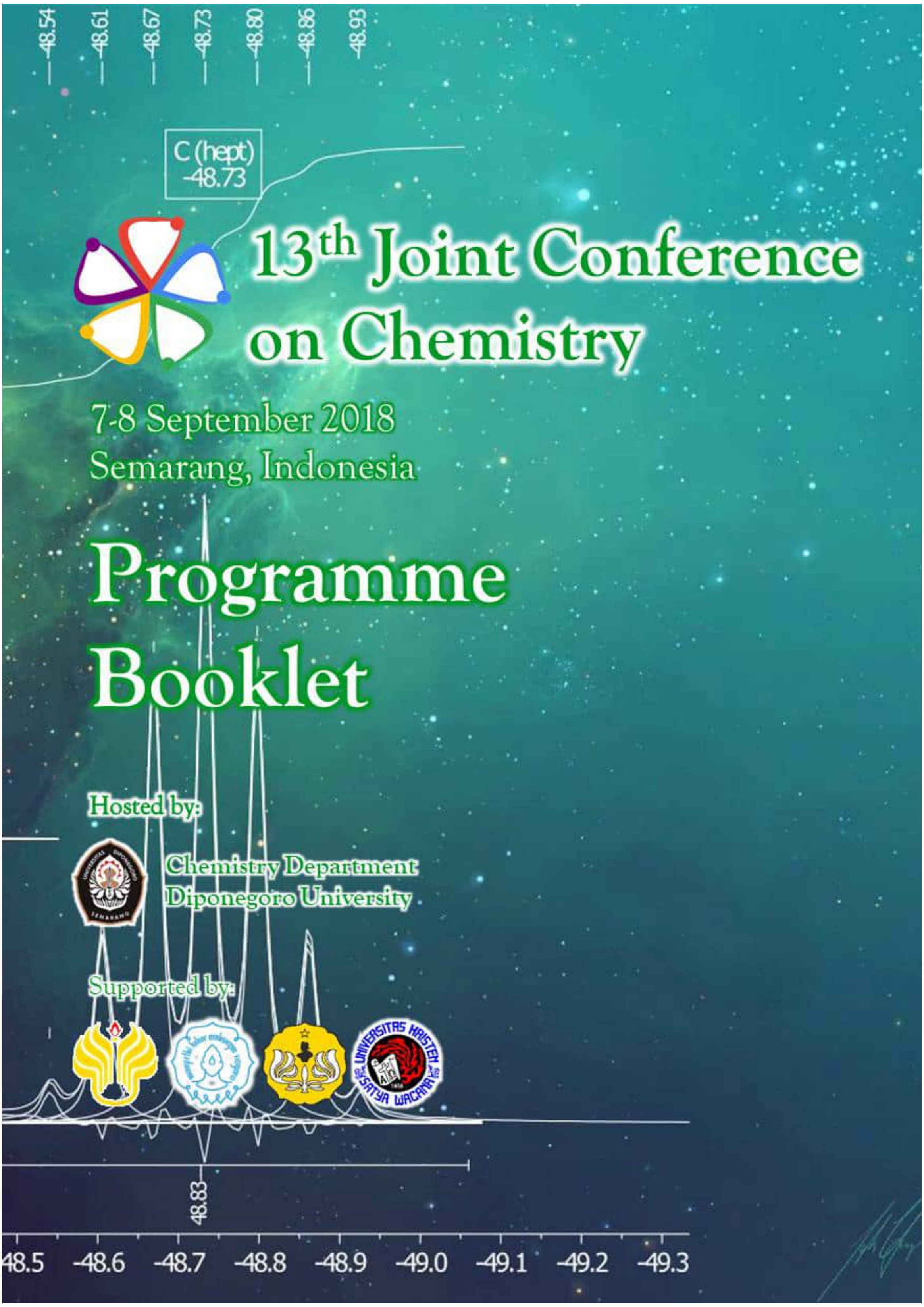

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7-8 September 2018
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13th Joint Conference on Chemistry

7-8 September 2018

Semarang, Indonesia

Preface

On behalf of the Consortium of Chemistry Department in Central Java, Indonesia and the JCC Committee, I would like to thank you for your participation in the 13th Joint Conference on Chemistry which to be held from 7-8th September 2018 in Semarang, Indonesia. The Joint Conference on Chemistry is an annual conference organized by the consortium of Chemistry Department of five universities in Central Java: Diponegoro University (UNDIP), State University of Semarang (UNNES), Sebelas Maret University (UNS), Jenderal Soedirman University (UNSOED) and Satya Wacana Christian University (UKSW). The JCC has been held since 2006.

This conference provides an interactive international forum to provide for sharing and exchange information on the latest research on Chemistry and related sciences, to enhance the capacities for creating innovation system, to contribute in the formulation of global strategies in advancing science role as well as developing policy initiatives in community, to stimulate future collaborations among industries, researchers, governments and other stakeholders who apply science and technology for better live. The speakers and participants of the 13th JCC are up to 250 coming from various countries extending from Indonesia, Malaysia, Philippine, Australia, South Korea, Japan, Iran, Nigeria, UK and India.

We received nearly 200 papers submitted to be included in the proceedings of this conference and after the review and revision process we finally got 158 papers to be published

I would like to thank for the endeavour of committee from Chemistry Department - UNDIP and the consortium member. In addition, the conference committee acknowledges the technical and financial support from Diponegoro University.

Adi Darmawan, Ph.D
The Chair of 13th Joint Conference of Chemistry
Chemistry Department, Faculty of Science and Mathematics, Diponegoro University

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Synthesis of silver orthophosphate under dimethyl sulfoxide solvent and their photocatalytic properties

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Synthesis of silver orthophosphate under dimethyl sulfoxide solvent and their photocatalytic properties

Dyah Ayu Septiarini[1], **Mardiyah Kurniasih**[1], **Roy Andreas**[1], **Dadan Hermawan**[1], **Uyi Sulaeman**[1,*]

[1] Department of Chemistry, Jenderal Soedirman University, Purwokerto, 53123, Indonesia
* Corresponding author email: uyi_sulaeman@yahoo.com, Tel./Fax.: +62281638793

Abstract. The silver orthophosphate was successfully synthesized using the starting materials of $AgNO_3$, KH_2PO_4 under water and dimethyl sulfoxide (DMSO) solvents. The variation of DMSO in water was designed at 0, 5, 10, 15, 20 and 100% (v/v). The products were characterized by XRD, DRS, and SEM. The photocatalytic properties were evaluated under the blue light irradiation using the methyl orange degradation. The results showed that the DMSO significantly affected the morphology, particle size and bandgap energy of Ag_3PO_4. The addition of DMSO decreased the particle size of Ag_3PO_4 and changed the tetrahedron into an irregular shape. The bandgap energies of 2.33, 2.28 and 2.42 eV were observed in the sample prepared with the content of DMSO at 0, 15 and 100% respectively. The highest photocatalytic activity was found at 15% DMSO. This excellent photocatalytic activity might be due to the lower bandgap energy and the higher intensity ratio of [222]/[110] facet.

Keywords: Ag_3PO_4, DMSO, facet, methyl orange, photocatalyst.

1. Introduction

The water environment deterioration due to organic pollutants coming from the textile effluent has increased year by year. It needs effective technology to destroy these pollutants to support the health water environment in the future. The photocatalyst technologies of TiO_2, $SrTiO_3$, ZnO have been developed to answer this problem. However, these materials have high band gap energy that cannot be used effectively under sunlight. Some modification of them into iodine doped TiO_2 nanoparticles [1], Ta-N co-doped $SrTiO_3$ [2], Ag/ZnO [3] have been developed and improved the activity under visible light irradiation. However, their activities are still limited due to the low absorption in the visible region.

Currently, the silver orthophosphate (Ag_3PO_4) has been widely used in photocatalysis due to high absorption in the visible region. This photocatalyst could be prepared by coprecipitation method. Design of hybrid, doping of element and defects have been devoted by researchers to improve the catalytic activity. The PVA-Ag_3PO_4 hybrid design was successful to enhance the photocatalytic activity [4], the noble element doping of Pt, Pd and Au could successfully improve the catalytic activity [5]. The defect engineering both theoretically [6] and experimentally [7] has been applied to improve their catalytic activities. However, too complicated preparation and high cost might be an obstacle to an application; therefore, the simple and low cost of preparation should be found.

Many designs have been explored to find a low cost of preparation and excellent activity. The different starting material, concentration, and solvent could be used as the strategy of synthesis to generate the high photocatalytic activity. For instance, the high activity of tetrahedron and short tetrapod could be designed by different concentration of KH_2PO_4 aqueous solution with the $AgNO_3$ aqueous

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solution [8]. This tetrahedron could also be created using the starting material of $AgNO_3$ and H_3PO_4 in ethanol [9]. The saddle-like Ag_3PO_4 with high activity could be synthesized using the starting material of H_3PO_4 and Ag_3PO_4 under solvent of ethanol-water [10]. The trisodium citrate and acetic acid could be used to prepare the coral-like Ag_3PO_4 [11] that improve the catalytic activity. The microcubes of Ag_3PO_4 created using deposition-precipitation in the presence of ammonia could enhance the photocatalytic activity [12]. It is very interesting that the morphology could be controlled through the strategy of modification of starting material and concentration.

The modification of Ag_3PO_4 using the different starting material and concentration would affect the facet intensities. The changes of the facet in Ag_3PO_4 significantly affected the properties of Ag_3PO_4 and improved the photocatalytic activity. The facet of [111] could be created using the starting material of $AgNO_3$ and H_3PO_4 in ethanol that might be responsible for the high activity of tetrahedral Ag_3PO_4 [9]. It is because that the [111] facet has higher surface energy [13]. The facet of [110] could be designed by a facile precipitation through a reaction of silver-amino complex and Na_2HPO_4 in water that forming the tetrapod Ag_3PO_4 [14]. It also exhibits high photocatalytic activity. Therefore, it is very challenging to control the facet of Ag_3PO_4 for improving the photocatalytic activity. Here, the modification of Ag_3PO_4 properties was carried out by the coprecipitation method under the solvent of water and dimethyl sulfoxide (DMSO) mixture.

2. Experimental

2.1. Materials

Silver nitrate ($AgNO_3$), potassium dihydrogen phosphate (KH_2PO_4), dimethyl sulfoxide (DMSO), methyl orange (MO) are purchased from the Merck. These chemical reagents were used in the experiment without further purification.

2.2. Synthesis

The Ag_3PO_4 synthesized based on the method that developed by Wu *et al.* [15] with modification in the variation of DMSO. Typically, a 10 mL of 10 mM $AgNO_3$ was added to a 20 mL of DMSO aqueous solution with the percent of 0%, 5%, 10%, 15%, 20% and 100%. After mixing these solutions under the magnetic stirrer with the speed of 600 rpm for 10 minutes, five mL of 1.5 M KH_2PO_4 aqueous solution was added dropwise. The yellow solids were formed and mixed in the solution for 30 minutes. The precipitates were filtered, washed with water and dried at 60°C. The samples were named as DS-0, DS-5, DS-10, DS-15, DS-20, DS-100, respectively.

2.3. Characterization

The characterization of the samples was investigated by X-Ray Diffractometer (Shimadzu 7000) to identify the structure of Ag_3PO_4. Scanning Electron Microscope (JEOL JSM 6510LA) was used to identify the size and morphology. The absorption and band gap energy were analyzed using the Diffuse Reflectance Spectroscopy (JASCO V-670). The bandgap energies were determined by direct transition [16] using the formula of $\alpha h\nu = A(h\nu - E_g)^{n/2}$, where A is a constant, n depends on whether the transition is direct (n=1) or indirect (n=4), α is the optical absorption, hν is the photon energy and E_g is the bandgap energy.

2.4. Photocatalytic Activity

The amount of 0.1 gram photocatalysts was added to 100 mL of 10 mg/L methyl orange solution, mixed with the magnetic stirrer with the speed of 600 rpm for 30 minutes to achieve the equilibrium of adsorption-desorption. The photocatalytic reaction was conducted under visible light (Skyled, 3 Watt)). Every 5 minutes, 5 ml of solution was taken out and centrifuged at 2000 RPM to separate it from the catalyst. The decrease of methyl orange was monitored by the UV-Visible spectrophotometer. The decreased photocatalytic activity was studied using the pseudo-first-order kinetic, using the formula of ln(Co/C)=kt, where Co is the initial concentration (mg/L), C is concentration at t time of photodegradation (mg/L), k is the rate constant (min^{-1}), and t is the time of degradation (min) [17].

3. Results and Discussion

The structures of Ag_3PO_4 in the samples of E-0, E-15, and E-100 were investigated using the XRD, the results are shown in Fig. 1. They have a similar structure of cubic (JCPDS No. 06-0505) without the presence of other secondary phases. The reaction of formation can be expressed as follows:

$$3AgNO_{3(aq,DMSO)} + KH_2PO_{4(aq)} \rightarrow Ag_3PO_{4(s)} + KNO_{3(aq)} + 2HNO_{3(aq)} \quad (1)$$

The different ratio of facet was observed among the samples. The addition of DMSO changed the intensity ratio of [222]/[110] and [222]/[320]. The highest ratio of [222]/[110] was found in the sample of E-15. The facet of Ag_3PO_4 is very important to identify because the facet of the surface significantly affected the activity of a catalyst. The intensity ratio of [222] to [110] peaks for DS-0, DS-15 and DS-100 are 1.27, 1.38, 1.29, respectively. All these intensity ratios are higher than the standard ratio of 1.00 [14]. The highest ratio of [222]/[110] intensity was found in the sample of DS-15.

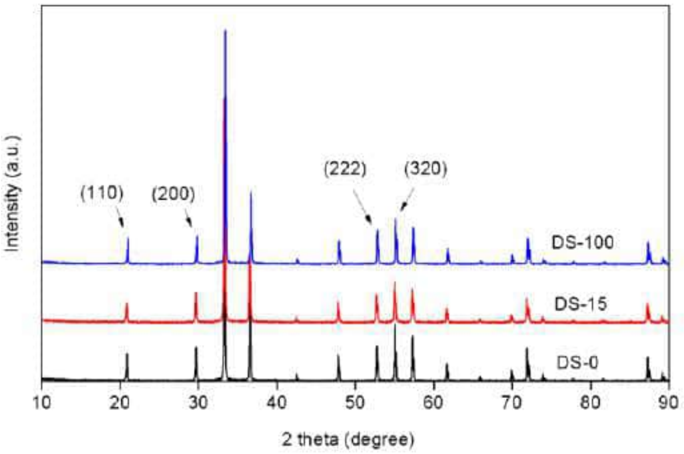

Figure 1. XRD profile of Ag_3PO_4 synthesized using starting material of $AgNO_3$ in water (DS-0), 15% of DMSO (DS-15) and 100% of DMSO (DS-100) reacted with the KH_2PO4 in water.

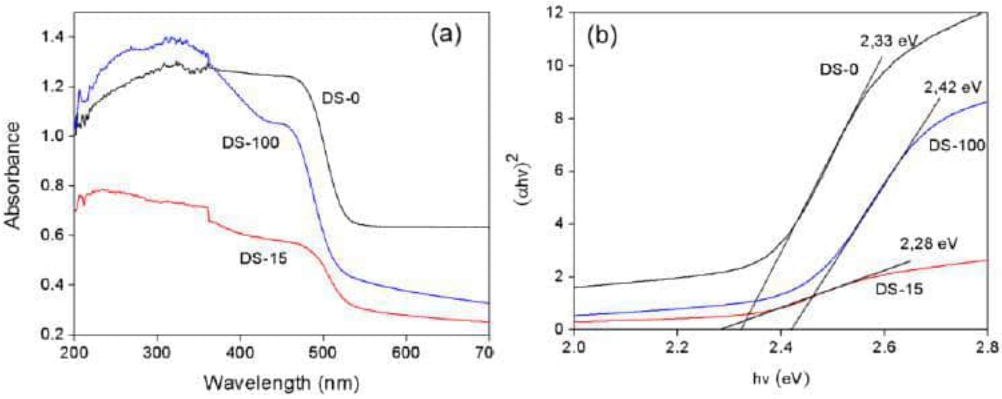

Figure 2. DRS of Ag3PO4 of the sample DS-0, DS-15 and DS-100 (a) with the calculation of bandgap energy using the plot of hν vs $(\alpha h\nu)^2$ (b).

Fig. 2 showed the absorption of DS-0, DS-15, and DS-100. The addition of DMSO clearly influenced the optical properties. The broad absorption at visible region (above ~530 nm) decreases by increasing the content of DMSO, indicating that the DMSO might reduce the defect in the surface of Ag_3PO_4. The most decreasing of this absorption could be found in the sample of DS-15. The high broad absorption in the visible region reflected that the high defect was created in the surface which suppresses the photocatalytic activity. The bandgap energy was also calculated using the direct transition and the results showed that the bandgaps of 2.33 eV, 2.28 eV, and 2.42 eV were observed in the samples of DS-0, DS-15, and DS-100 respectively. The lower of bandgap energy was found in the sample of DS-15.

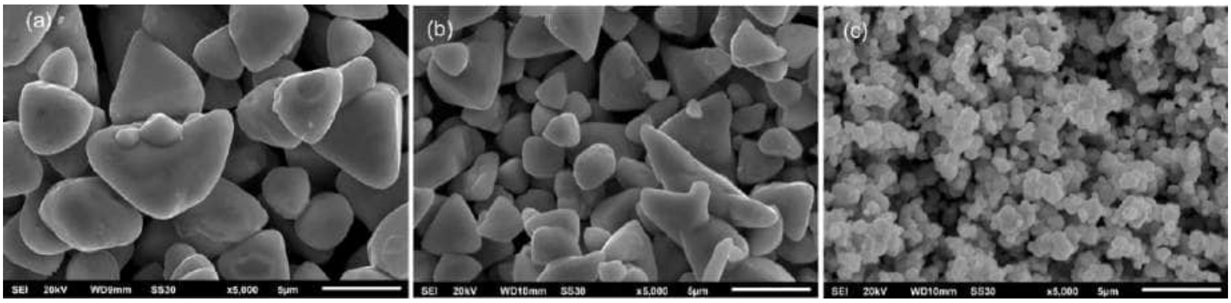

Figure 3. SEM images of the samples of DS-0 (a), DS-15 (b) and DS-100 (c).

The morphology of DS-0, DS-15, and DS-100 was investigated using Scanning Electron Microscope. The results were shown in Fig. 3. The tetrahedron with the particle size of 2.5-6.0 μm was observed in the sample of DS-0. These morphologies changed under synthesis using DMSO as found in the sample of DS-15. It showed that the part of the tetrahedron particle changed into irregular shape with the particle size of 2.0-4.0 μm. There is no tetrahedron found in the sample of DS-100 which has the diameter of 0.5-1.0 μm. It was interesting that the addition of DMSO significantly decreases the particle size of Ag_3PO_4 (table 1). The DMSO might have a significant role to control crystal growth. The smaller particle size could be found in the sample of DS-100.

Table 1. Intensity ratio of facet, bandgap energy and particle size of Ag_3PO_4.

Samples	[222]/[110]	[222]/[320]	Band gap (eV)	Particle size (μm)
DS-0	1.27	0.61	2.33	2.5-6.0
DS-15	1.38	0.68	2.28	2.0-4.0
DS-100	1.29	0.76	2.42	0.5-1.0

Table 2. Pseudo first order kinetic of photocatalytic reaction.

Sample	Rate Constant (min^{-1})	R^2
DS-0	0,0897	0,9736
DS-5	0,0925	0,9825
DS-10	0,1156	0,9700
DS-15	0,1730	0,9342
DS-20	0,1358	0,9514
DS-100	0.0091	0.9989

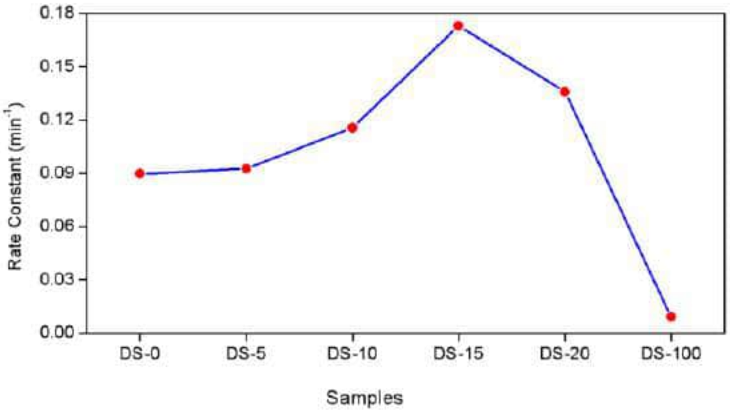

Figure 4. Photocatalytic activity of Ag_3PO_4 for the samples of DS-0, DS-0, DS-5, DS-10, DS-15, DS-20, DS-100 analysed using the pseudo-first-order kinetics.

The photocatalytic activities of DS-0, DS-5, DS-10, DS-15, DS-20, DS-100 were evaluated using the methyl orange degradation under blue light irradiation. The results can be seen in Fig. 4. These photocatalytic activities followed the pseudo-first-order kinetics (table 2). The activity increases by the increasing of DMSO up to the sample of DS-15 where the highest activity achieved. However, by adding more of DMSO, the activity decreases, and the lowest activity was found in the sample of DS-100. The different photocatalytic activity might be due to the different morphology, facet, particle size, and band gap energy. The highest of photocatalytic activity was observed in DS-15. Around 1.9 times higher of photocatalytic activity was observed at DS-15, compared to the sample of DS-0. The higher ratio of [222]/[110] could have higher surface energy that enhances activity in the surface. The lower band gap energy of the sample of DS-15 might also increase the photocatalytic activity. With the lower bandgap energy, more energy at visible light could be used for the excitation leading to enhanced photocatalytic activity. It is well known that the smaller particle size the higher activity could be obtained. However, the smallest particle size of DS-100 did not show high photocatalytic activity. It might be due to the changes of morphology from tetrahedron to irregular form.

It is also well known that the high photocatalytic activity of silver phosphate was generated by visible light irradiation. Under irradiation, the photocatalyst would generate the holes in valence band and electron in conduction band in the surface. The hole would react with hydroxide ion resulting in hydroxyl radical whereas the electron could react with oxygen resulting in the superoxide radical. These species are responsible for the photocatalytic activity. This mechanism is highly affected by defect sites on the surface. The high defect would increase the recombination of electron and holes pair leading to low catalytic activity. In this experiment, DMSO has a significant role to decrease the broad absorption in the visible region indicating the high defect might be suppressed.

4. Conclusions

The silver orthophosphate was successfully synthesized using the starting materials of $AgNO_3$, KH_2PO_4 under water and dimethyl sulfoxide (DMSO) solvents. The DMSO significantly affected the morphology, particle size and band gap energy of Ag_3PO_4. The highest photocatalytic activity was found in DMSO at the sample of 15% DMSO. The high of photocatalytic activity might be due to the low band gap energy and the high ratio of [222]/[110] intensities.

Acknowledgments

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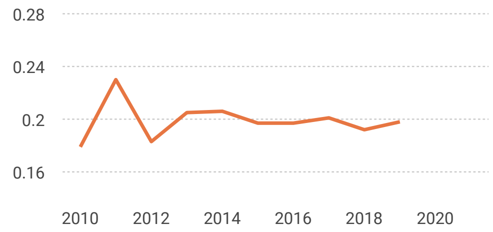

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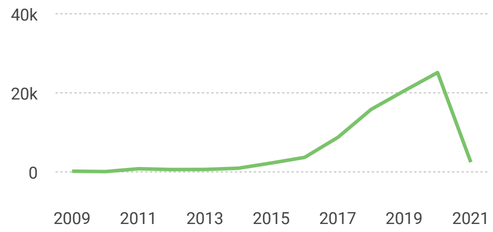

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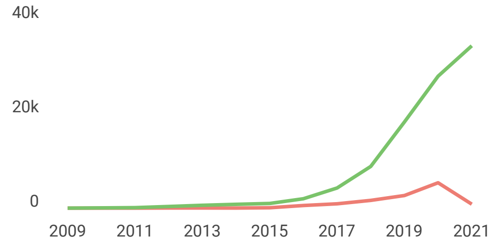

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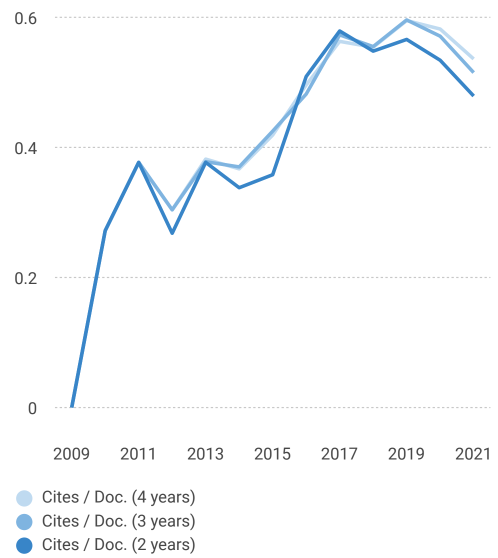

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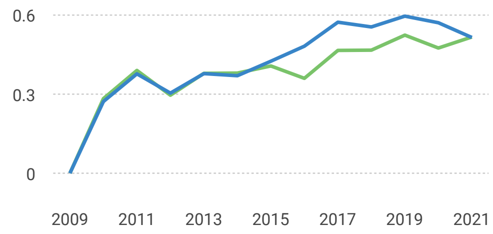

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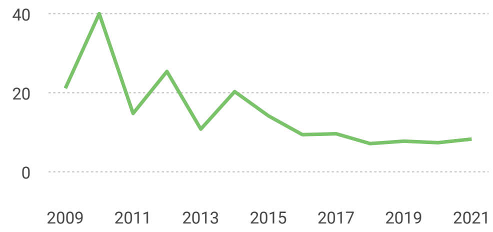

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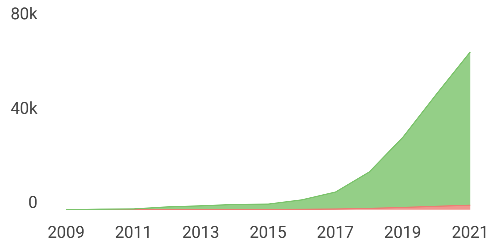

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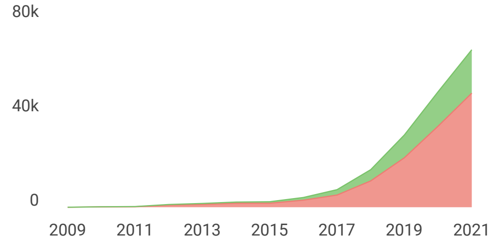

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