Research Field : Chemical Engineering

Extended Abstract

Tungsten Trioxide (WO_3) Particles Synthesis as Photocatalyst Using Flame Spray Pyrolysis

Restu Mulya Dewa
Dept. of Chemical Engineering, ITS
mulyadewa@gmail.com

Sugeng Winardi
Dept. of Chemical Engineering, ITS
swinardi@chem-eng.its.ac.id

Widiyastuti
Dept. of Chemical Engineering, ITS
widi@chem-eng.its.ac.id

***Abstract*-The catalyst surface is a substantial component that must be exist to ensure the photocatalytic reaction take place. Tungsten trioxide (WO_3) can be an alternative source of catalyst material due to its excellent properties such as stability, inert, and good absorption of visible light. In this research, WO_3 material production was performed by flame spray pyrolysis method using ammonium tungsten pentahydrate (ATP) as the raw material. This method is chosen due to its simple system and ability to produce particles with high purity in a rapid time. To investigate the parameter affecting the crystallite/particle size, initial precursor concentration (0.01-0.02 M) was employed.**

***Index Terms* – Catalyst, flame spray pyrolysis, photocatalytic, tungsten trioxide**

INTRODUCTION

Photocatalytic is a light-assisted activation process of a substance called photocatalyst, which modify the chemical reaction rate without being directly involved in the chemical transformation. Photocatalytic reaction initiates when the excited electrons move from the filled valence band of semiconductor photocatalysts to the empty conduction band as the absorbed photon energy. Photon energy (hυ) that is equal to or greater than the band gap semiconductor photocatalysts leaves a hole in the valence band. This leads to formation of the electron and hole pairs (e^-, h^+).

The photocatalytic reaction rate highly depends on the absorption of radiation from photocatalysts [6]. Increase in the degradation rate is linear with the increase in light intensity during photocatalytic degradation [3]. An ideal photocatalyst for photocatalytic oxidation characterized by traits such as: photo-stability, chemically and biologically inert material, the sufficient availability, low cost, and have the ability to adsorb reactants under the activation of efficient photonic [2].

As a promising material, tungsten trioxide (WO_3) has received much interest because its anti-photo corrosion properties, stability in acid, and the potency to absorb the spectrum fractions of sunlight. WO_3 with the band gap range 2.4-2.8 eV especially absorb spectrum of sunlight in the near ultraviolet region and blue region [4].

The formation mechanism of WO_3 nanoparticles was produced by the flame spray pyrolysis method. Proper combustion of the precursor sprayed on the flame spray pyrolysis process is a promising technique for the rapid synthesis of nanoscale materials. The general step in all processes using liquid or solid reactants which dispersed in the liquid is the formation of droplets containing the precursor by using an aerosol generator. Droplets pass through a high-temperature environment, where the solvent evaporate and the precursors react to form products.

METHOD

Synthesis of WO_3 particles

WO_3 nanoparticles were produced from a precursor containing ATP (ammonium tungstate pentahydrate $(NH_4)_{10}W_{12}O_{41}.5H_2O$; purity 88-90%; Kanto Chemical Co., Inc., Japan) which was diluted into ultra-pure water (pH 5.7, PT. Otsuka Indonesia). To investigate the effect of concentration, precursor with concentration 0.01, 0.013, 0.015, 0.017, and 0.02 M were used. The precursor was put into *ultrasonic nebulizer* (OMRON NE-U17, Japan) which was used to generate monodispersed droplets. The generated droplets were then introduced into a glass reactor (D=9 mm and L=75 cm) with a burner using 1.5 L/min liquefied petroleum gas (commercial grade, PT Pertamina) as the fuel and 35.9 L/min compressed air as the oxidizer. The prepared WO_3 particles were then collected using an electrostatic precipitator.

Particles characterization

Crystallinity was analyzed using X-ray diffraction (XRD Philips 30Ma, 40kV). To investigate the photocatalytic activity, the photodegradation of 20 ml solution containing methylene blue (MB, $C_{16}H_{18}N_3SCl$, purity 69.6%, SAP Chemicals) at a concentration of 4 mg/L by the WO_3 particles (200 mg/L) under sunlight irradiation was examined. Before beginning the photodegradation reaction, the suspension was left in the dark place for 30 minutes to establish equilibrium between MB and the prepared WO_3 particles. The MB concentration after dark conditioning was recorded as the initial concentration. Changes in the MB concentration were measured every 10 minutes using a UV-vis spectrophotometer (Genesys 10S, Thermo Scientific).

RESULT AND DISCUSSION

Effect of Precursor Concentration on Particle Formation

WO_3 has some modifications (triclinic, orthorhombic, tetragonal, hexagonal, and cubic). Triclinic, monoclinic, orthorhombic, and tetragonal basically have the same arrangement WO_6 octahedra so all these phases can be transformed to one another. Therefore, WO_3 particles have only three completely different crystals settings; monoclinic, hexagonal, and cubic [5]. The XRD patterns show the produced particles have a monoclinic crystalline structure.

From this analysis it can be seen an increase in the peak intensity along with the increasing precursor concentration. It showed an increasing crystallinity degree of the particles at higher concentrations. The crystal diameters were 21.89, 34.42, 39.81, 31.12, and 26.26 nm, corresponding to precursor concentrations of 0.01, 0.013, 0.015, 0.017, and 0.02 M, respectively. Improved crystals diameter occurred only on the concentration 0.01 M to 0.015 M. Anomaly results were found for the case of precursor concentrations of above 0.015 M. The crystal diameter decreased drastically. It can be caused due to the solubility of ATP which get smaller at the higher precursors concentration. As a result, there are fewer particles dissolved in the ATP precursors that avoid the larger particles production.

Photocatalytic Performance Evaluation

MB absorbance gradually declined with increasing reaction time up to 30 minutes. After that, the adsorption reached equilibrium, as indicated by the relatively constant MB concentration.

The results showed the different quality of photodegradation on each WO_3 particle. WO_3 produced from the precursor 0.01 M had the best photodegradation quality compared to other precursors because it can degrade MB up to 65.75%. Meanwhile WO_3 particles from the precursor 0.013, 0.015, 0.017 and 0.02 M degraded MB by 75.02%; 79%; 70.65%; and 67.60%, respectively.

The ability of MB degradation by WO_3 improved with a reduction in crystal diameter. The smaller the WO_3 crystal diameter, the more MB degraded. Small crystals diameter also indicates the small particle diameter. At the same particles mass that are used to the photocatalytic activity test, the number of particles will increase with the reduction in particle size. This condition is followed by an increase in the total surface area of the catalyst, where a growing number of areas that can be used as a reaction place [1].

CONCLUSION

WO_3 particles can be obtained from ammonium tungstate pentahydate (ATP) using flame spray pyrolysis method. One of the parameter affecting the particle formation is initial precursor consentration. Methylene blue degradation ability increases with reduction in WO_3 crystal diameter.

REFERENCES

[1] Arutanti, O. et al. “Controllable crystallite and particle sizes of WO_3 particles prepared by a spray-pyrolysis method and their photocatalytic activity,” *American Institute of Chemical Engineers Journal*, vol. 60, no. 1, pp. 41-49, 2014.

[2] Colmenares, J.C. et al. “Nanostructure photocatalysts and their applications in the photocatalytic transformation of lignocellulosic biomass: an overview,” *Materials*, vol. 2, pp. 2228-2258, 2009.

[3] Karunakaran, C. & Senthilvelan, S. “Photooxidation of Aniline on Alumina with Sunlight and Artificial UV Light,” *Cataysis Communication*, vol. *6*, pp. 159–165, 2005.

[4] Liu, Y. et al. “Photoelectrochemical properties and photocatalytic activity of nitrogen-doped nanoporous WO_3 photoelectrodes under visible light,” *Applied Surface Sciences*, vol. 258, no. 12, pp. 5038-5045, 2012.

[5] Szilágyi, IM. et al. “WO_3 photocatalysts: influence of structure and composition,” *Journal of Catalysis*, vol. 294, pp. 119-127, 2012.

[6] Ustinovich, E.A. et al. “Heterogeneous photocatalysis in titania-stabilized perfluorocarbon-in-water emulsions: urea photosynthesis and chloroform photodegradation,” *Journal of Photochemistry and Photobiology A: Chemistry*, vol. 175, pp. 249–252, 2005.