PREPARATION OF S DOPED TIO

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PREPARATION OF S-DOPED TiO_2 VIA SOL-GEL/ HYDROTHERMAL METHOD AND ITS ACTIVITY AS PHOTODEGRADATION OF RB05

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ABSTRACT

S-doped TiO_2 photocatalyst has been prepared by the hydrothermal method at 180°C that assisted by the sol-gelel method and its photocatalytic activity was evaluated for reactive black 05. The obtained photocatalyst was characterized by XRD and SEM. The phase composition of S-doped TiO_2 was influenced by the amount of sulfur.

Keywords: TiO_2, Doping process, Sulfur, Photocatalyst, Reactive Black 05.

INTRODUCTION

TiO_2 is classified as semiconductor materials that have excellent property as a catalyst, especially in the photodegradation of organic pollutants, i.e. dyes, phenolic compounds, etc. The previous researches that focused on the photoactivity of TiO_2, mostly work under UV light due to it shows high activity and stable under UV light.[1–3]

The issues that were found during its applications, photoactivity of TiO_2 is not fully effective when it utilized under solar light. Several methods have been developed to improve its performance under solar light.[4–6] One of these efforts is through the doping technique. The doping technique also influences the bandgap of the doped materials with a range in the visible light area. As the dopant, either metal or non-metal elements can be incorporated with the TiO_2, it depends to the atomic radius between dopant and oxygen atom of TiO_2.[7–10] The previous work about the doping process of TiO_2 has reported results on the substitution of several elements, i.e. nitrogen, oxygen, and sulfur.[10,11] The incorporating of those elements into TiO_2 showed a significant change in the photoactivity of materials, especially sulfur.

S-doped TiO_2 in the previous work has been prepared using calcination technique, sol-gel, hydrothermal and in the supercritical condition in CS_2/ethanol.[12,13] From those several methods, the combination method between sol-gel and hydrothermal provides a simple and best method to obtain nanosized of doped TiO_2 photocatalyst. Also, the photocatalyst of TiO_2 that prepared using the hydrothermal method can have a different ratio between retail and amateurs depend on the preparation of raw material and temperature during hydrothermal. Based on the literature review, there have been few reports on the preparation of S-doped TiO_2 using a hydrothermal method that assisted by the sol-gel method. In the present work, the S-doped TiO_2 was prepared using a hydrothermal method that assisted by the sol-gel method. The photoactivity of the obtained S-doped TiO_2 was evaluated for degradation of reactive black 05.

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EXPERIMENTAL

Materials

Sulfur and TTIP (97%) were obtained from Merck and Sigma Aldrich, respectively. Absolute ethanol was provided by Guangdong Guanghua Sci-Tech Co. Ltd.

Preparation of S-doped TiO_2

The doping process was performed using the method that has been described previously.[14] The as-prepared S-doped TiO2 then was characterized using several instruments, i.e Bruker D8 Advance X-ray diffractometer for crystallography analysis, scanning electron microscopy TM4000 Hitachi for morphological analysis and UV-Vis Diffuse Reflectance Spectrophotometry Shimadzu UV-2600 Series for determine the band gap value.

Photoactivity Measurements

The photoactivity of S-doped TiO_2 was performed in a cylindrical glass that equipped with a condenser to control the temperature. The treatment was performed using a method that has been described as previously[15]. The obtained aliquots were characterized using UV-Vis spectrophotometer to determine the final concentration of imitated wastewater after treatment.

RESULTS AND DISCUSSION

The crystallographic of doped TiO_2 is shown in Fig-1. The obtained S-doped $TiO_2$with the incorporation of 0.5% S only consisted of anatase phase and rutile, no other phase can be found. However, when the amount of incorporated S increases the phase of TiO_2 was dominated by anatase. This result indicated the ratio of anatase, or rutile phase depends on the amount of S that doped to TiO_2. The phase composition of $TiO_2$was obtained using Match! software version 3.8.0.137 and the 0.5% S-doped TiO_2 showed a combination signal of JCPDS 96-900-1681 and JCPDS 96-720-6076, while 1.0% S-doped TiO_2has similar signal to JCPDS 96-720-6076.

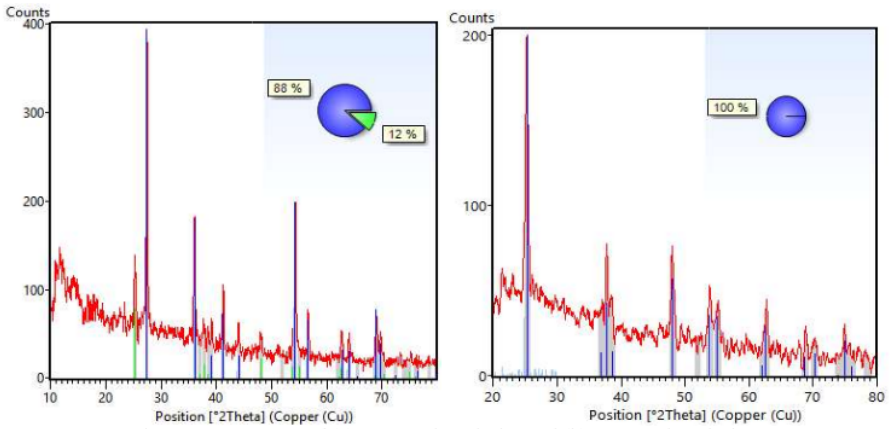

Fig.-1 XRD Spectra of (a) 0.5% S-doped $TiO_2$and (b) 1.0% S-doped TiO_2

Figure-2 shows the SEM image of doped TiO_2 obtained by hydrothermal assisted by sol-gel method. The effect of the amount of S that doped to TiO_2 can be seen in the particle size of the photocatalyst. The particle size of TiO_2 was reduced by the increase of the amount of S, the particle size of the TiO2. The nanoparticle of TiO_2 with particle size in the range > 1 µm was changed to 40-60 nm when the concentration of S increases.

The photodegradation of RB05 was evaluated by determining the concentration of dyes in the system at a specific time. In Fig-3, the photodegradation of RB05 by amateurs, 0.5% S-doped TiO_2 and 1.0% S-doped

TiO_2 were 83.10; 20.73 and 98.31%, respectively. The as-prepared 1.0% S-doped TiO_2 showed to be more photoactive than undoped TiO_2 with a rate constant of 4.03×10^{-2}. The 0.5% S-doped exhibited the lowest photoactivity with a rate constant of 2.24×10^{-3}.

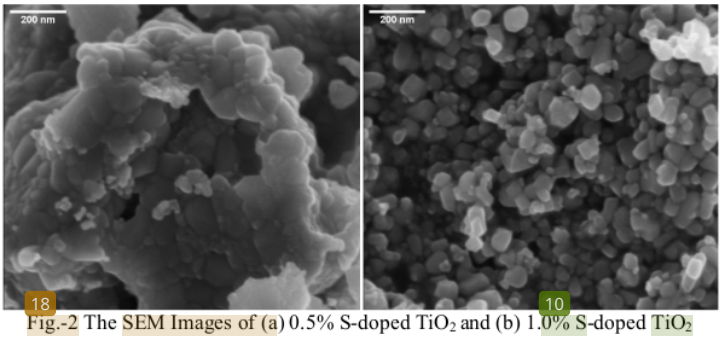

Fig.-2 The SEM Images of (a) 0.5% S-doped TiO_2 and (b) 1.0% S-doped TiO_2

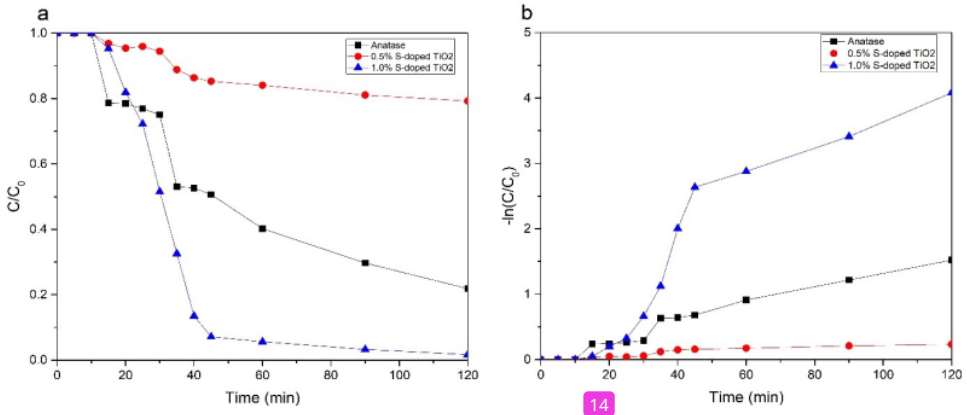

Fig.-3 (a) Photocatalytic Activity of doped TiO_2 and (b) Rate of Photocatalytic Activity of doped TiO_2

The process of dyes photodegradation could be said as a complicated process that cannot be only viewed from one perspective, but it should be observed from all perspectives, i.e. particle size, morphology, and phase/ chemical composition of photocatalysts[16–20]. In this study, the photoactivity of doped TiO_2 was influenced by two factors, i.e. the particle size of doped TiO_2 and phase composition of doped TiO_2. Based on kinetic theory, particle size has a big influence on the reaction rate. Smaller particle size will have a larger surface area and it can facilitate the dyes photodegradation be more effective as the impact of the increase of photogenerated electrons. The other perspectives, the phase composition of doped TiO_2has an important role on the dye photodegradation process. The ratio of rutile phase in the 0.5% S-doped TiO_2 was 88%, the high rutile phase content in S-doped TiO_2 influenced to the lower photocatalytic activity. When the amount of S was enhanced, the doped TiO_2 phase was dominated by anatase and improved the photocatalytic activity. This result also supported by the previous work, the ratio of rutile and anatase phase has an important role to the photocatalytic activity, when the rutile ratio is high the photocatalyst will show lower activity and vice versa.[4,16,21,22]

CONCLUSION

S-doped TiO_2 was successfully synthesized using hydrothermal assisted sol-gel method. The morphological and photocatalytic activity of doped $TiO_2$was totally dependent on the S amount that incorporated into TiO_2.

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