

STUDIES ON PREPARATION OF Fe(III)/TiO₂ BY SOL-GEL METHOD

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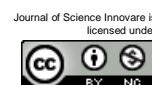
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Abstrak. In this study, Fe/TiO₂ powder was synthesized by sol-gel method using mixture of Titanium Tetraisopropoxide (TTIP) as precursor and FeCl₃ as iron source of 10% (w/w). The Fe/TiO₂ powder was calcined at 500 °C for 1 hour. The sample of Fe/TiO₂ was characterized using FTIR and DRS measurements. FTIR analysis showed that Fe was successfully doped on TiO₂ and then DRS analysis proved that the visible light was absorbed by Fe/TiO₂ with its band gap energy up to 2.3 eV..

Keywords: Iron; TiO₂; Titanium; Fe/TiO₂



I. INTRODUCTION

The problem of TiO₂ for photocatalytic processes is relatively large band gap (3.0 eV for the rutile phase and 3.2 eV for the anatase phase) [1]. Therefore, the photocatalytic occurred in the spesific region because it only absorbs under UV light at 388 nm. Properties of TiO₂ could be improved by expanding the work area of TiO₂ under visible region. Many researcher modify the surface area of TiO₂ makes the new properties of TiO₂ by doping various metals such as Fe, Mn, Cr and others [2]. Fe (III) is a transition metal ion that promises to support the purpose of this study because the radius size of Fe and Ti is almost the same. Therefore Fe atoms replace Ti atoms in the crystal lattice [3].

Modification TiO₂ with Fe (III) using Fe(NO₃)₃·9H₂O as a precursor has shown efficiency in the photocatalytic reduction process of nitrate by 65.97% for 360 minutes (Kobwittaya & Sirivithayapakorn [4]). In research conducted by Organisian, Hreniak, Sikora, Koniarek, Iwan [5] reported Fe (III) - Doped TiO₂ showed magnetic properties, namely supermagnetic. Thus, the main objective of this study was to synthesize TiO₂ photocatalyst material by doping Fe (III) transition metal ions sourced from FeCl₃ with an added concentration of 10% w / w using the solgel method. This purpose is for expanding the work area of TiO₂ form ultraviolet to visible light. To ensure this, the resulting Fe (III) -doped TiO₂ powder will be characterized by Fourier

Transform Infrared (FTIR) and Diffuse Reflectance Spectroscopy (DRS)

II. RESEARCH METHODS

Materials

Titanium (IV) Isopropoxide (TTIP) (97 %) and FeCl₃ reagent grade from Sigma Aldrich. Ethanol 96% from Merck, and aquades.

Synthesis of Fe (III)- doped TiO₂ Powders Titanium precursor was dissolved in ethanol. Briefly, 4.5 ml of TTIP and 21 ml of ethanol were mixed with 3.5 ml of distilled water and addition of FeCl₃ the obtaining sol with the molar ratio Fe/TTIP equal 10% w/w. The solution was stirred at room temperature for 4 h. During the stirring the titanium dioxide powder was formed, the sample is filtered and dried at room temperature for 24 hours, then the powder was annealed at 500°C for 1 h.

Material Characterization

Fe-TiO₂ powder characterized by Fourier Transform Infrared (FTIR) and Diffuse Reflectance Spectroscopy (DRS).

III. RESULTS AND DISCUSSION

Based on the results of the synthesis, Fe(III)-doped TiO₂ powders obtained by sol gel method. Fig. 1. shows the appearance of TiO₂ standard (A) and Fe(III)/TiO₂ (B) as the result of synthesis. Brown color indicates Fe was successfully doped onto titanium dioxide (B).



Figure 1. (A) TiO₂ Degussa P25, (B) Synthesis Result Fe(III)/TiO₂

The infrared spectra of both powder showed a strong absorption band at 3200-3600 cm⁻¹ characteristic of -OH group (Figure 2 and 3). The Ti-O-Ti band around 400-800 cm⁻¹ after calcination process was sharper indicating that Ti-

OH bonds was fully converted to TiO-Ti as characteristic of pure TiO₂. The change in colour of the powder before and after adding Fe from white to yellow expected for iron was successfully trapped in TiO₂ (Figure 2). This was interpreted from FTIR spectra which showed a new band at about 2200-2300 cm⁻¹ indicating loading of iron in TiO₂ (Figure 2).

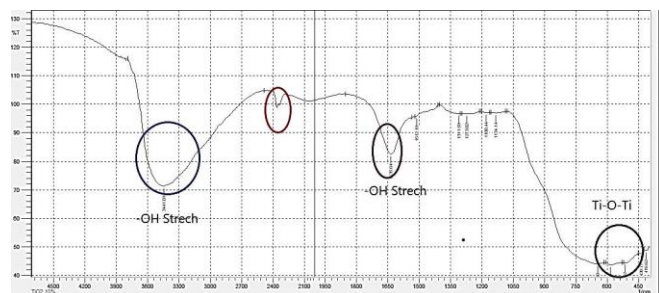


Figure 2. FTIR Spectra of Fe(III)-doped TiO₂ Calcined at 500°C

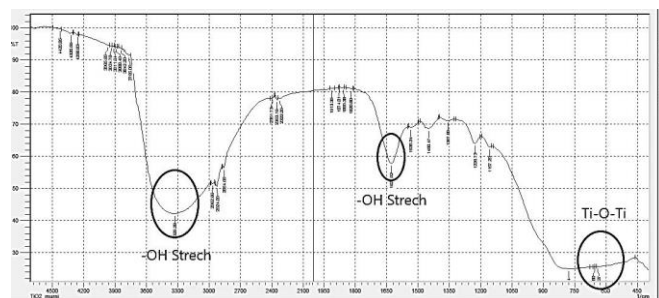


Figure 3. FTIR spectra of TiO₂ Degussa P25

Furthermore, after calcination, the samples were analyzed using a Diffuse Reflectance Spectrophotometer (DRS) to determine band gap of Fe(III)-TiO₂. DRS analysis showed the absorption maximum band of 329 nm. Moreover, weak absorption was observed in the range 400–600 nm and is caused by the presence of Fe in titanium dioxide. The percent (%) reflectance sample compared to a standard Degussa P25 (see Fig.4) were 3,2 eV. This value determined by calculating the Kubelka-Munk equation according to :

$$F(R) = \frac{K}{S} = \frac{(1-R)^2}{2R} \dots\dots\dots (1)$$

F(R) = is the Kubelka–Munk function R = the ratio of the intensities of radiation reflected in diffuse manner from the sample and from the known sample K = represents the absorption coefficient of radiation and S is the scattering factor.

Calculation results show samples synthesized TiO₂ has an energy gap is 2,30 eV. Meanwhile, the energy gap Degussa P25 standard is 3,2 eV.

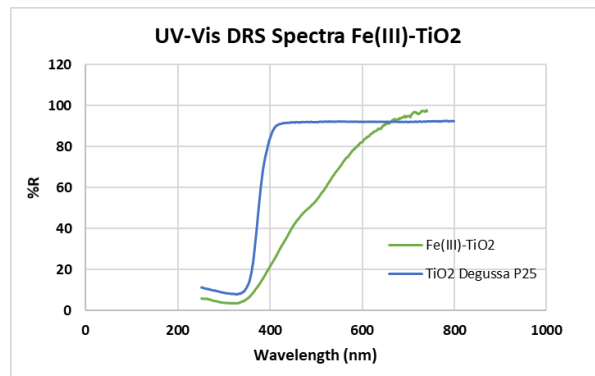


Figure 4. Diffuse Reflectance Spectra of the as prepared sample Fe(III)-TiO₂

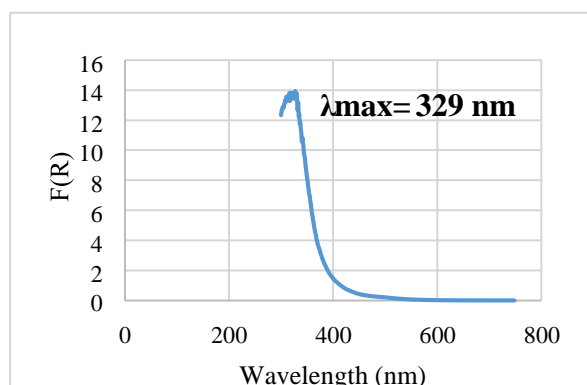


Figure 5. Spectra Wavelength Maximum of Fe(III)-TiO₂

IV. CONCLUSION

TiO₂ doped with Fe content were synthesized through sol gel method has the influence on the structure, was confirmed by FTIR. Band gap energy of TiO₂ is also broadening as well as the light absorption zone towards the red region. The calculation result of the band gap energy sample was decreased from 3.2 to 2.3 eV. It implies that Fe(III)-doped TiO₂ has potential as a photocatalyst material for degradation organic compounds in visible wave area. Additionally, the sol gel method is a simple, easy and cheap methods to obtain Fe-TiO₂.

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