Effect of Fe Incorporation on the Photocatalytic Activity of TiO₂ by Sol-Gel Method

Siti Aida Ibrahim¹²* and Srimala Sreekantan²
¹Faculty of Mechanical & Manufacturing, UniversitiTun Hussein Onn Malaysia, Parit Raja, 86400 BatuPahat, Johor, Malaysia.
²School of Materials & Mineral Resources Engineering, Engineering Campus, UniversitiSains Malaysia, 14300 Nibong Tebal, Penang, Malaysia.

*Email: saida@uthm.edu.my

Abstract
Nanostructured Fe-TiO₂ and TiO₂ with anatase structure were synthesized via combination method of sol-gel and peptization process. The samples were characterized by X-ray diffraction (XRD), Transmission emission microscopy (TEM) and UV-Vis spectroscopy (UV-Vis). The presence of Fe³⁺ ion shifted the absorption profile of TiO₂ to the longer wavelength side of the spectrum, indicating an obvious photocatalytic activity under visible irradiation. Photocatalytic activity of the samples were evaluated by methyl orange (MO) discolouration under UV-light irradiation. Compared with the pure TiO₂ nanoparticles, the Fe-TiO₂ nanoparticles exhibited higher photocatalytic activity with 95% discolouration within 2 h.

Keywords: TiO₂, photocatalyst, sol gel method, cation doping

Introduction
TiO₂ is one of the most semiconductors metal oxides used in many applications covering various field such as optoelectronics, medicine, sensor devices and catalyst [1]. As photocatalyst, TiO₂ has a large band gap (3.2 eV for anatase TiO₂) and thus it limits its ability to function well in visible region as only 4-5% of UV light presence in solar spectrum [2]. To improve this limitation, metal doping on TiO₂ can be used. In this work, Fe-TiO₂ was successfully prepared via combination methods of sol-gel and peptization process. In this study, the presence of Fe³⁺ ion towards the formation of TiO₂ and its performance in photocatalytic activity is investigated using methyl orange (MO) as model compound.

Experimental Methods
Titanium (IV) isopropoxide (Ti(OH)₃)₄ and iron (III) nitrate nonhydrate (Fe₃O₉·9H₂O) from Sigma Aldrich were used as titanium and iron sources, respectively. The pure TiO₂ was synthesized using procedure in previous study [2]. In a typical preparation of Fe-TiO₂ particles, a 0.4M TTIP was added drop wise to hydrolysis medium of 500 ml iron (III) nitrate solution (0.8mmol Fe) with pH adjusted of 3. The white mixture was stirred for 2 hours at room temperature and peptized in HNO₃ at 85°C for 8 hours. The resultant mixture was clear and transparent in colour and characterized by XRD, TEM, and UV-VIS spectrophotometer. Photocatalytic activity (PCA) was determined by MO discolouration test under UV light irradiation for 5h.

Results and Discussion
Fig.1 shows the XRD patterns of Fe-TiO₂ and TiO₂ prepared by combined method of sol-gel and peptization process. It is noticed that both samples adopt anatase structure. With Fe incorporation, the degree of crystallinity of anatase is declined due to the increased surface disorder on TiO₂ [3]. Meanwhile, TEM image of Fe-TiO₂ is illustrates in Fig.2. The average particle size is about 4-6 nm and in accordance with result obtained from XRD. The light absorption of Fe-TiO₂ is obtained via UV-Vis diffuse reflectance and the wavelength
was extended to 547nm, suggesting an obvious photocatalytic activity under visible irradiation. This result indicates that Fe-TiO$_2$ is capable to absorb both UV light and visible light as maximum visible light absorption being at 547 nm.

![XRD patterns of (a) pure TiO$_2$ and (b) Fe-TiO$_2$. (A: anatase)](image)

**Fig. 1.** XRD patterns of (a) pure TiO$_2$ and (b) Fe-TiO$_2$. (A: anatase)

![TEM images of Fe-TiO$_2$](image)

**Fig. 2.** TEM images of Fe-TiO$_2$

The PCA performance is evaluated by MO discoloration under UV light exposure and is shown in Fig. 3. As observed, PCA of Fe-TiO$_2$ shows the highest MO discoloration percentage with 95% after 2 hours irradiation. The order of MO discoloration percentage could be followed as Fe-TiO$_2$ $>$ TiO$_2$ $>$ Blank. Based on this result, it is believed that the synergistic effect of Fe and TiO$_2$ provide good photocatalytic activity and promotes the discoloration reaction.

**Fig. 3.** MO discoloration under UV light exposure for 5 h by (a) Fe-TiO$_2$, (b) TiO$_2$ and (c) Blank.

**Conclusion**

Fe-TiO$_2$ nanoparticle (4-6nm) was successfully prepared via combined method of sol gel and peptization process at 85°C for 8 hours. The result demonstrates that the Fe$^{3+}$ ions incorporation retains anatase structure. However, the presence of Fe tuned the light absorption characteristic, extending the wavelength towards visible region and enhanced the PCA of Fe-TiO$_2$ as 95% of MO was removed within 2 h.

**References**

