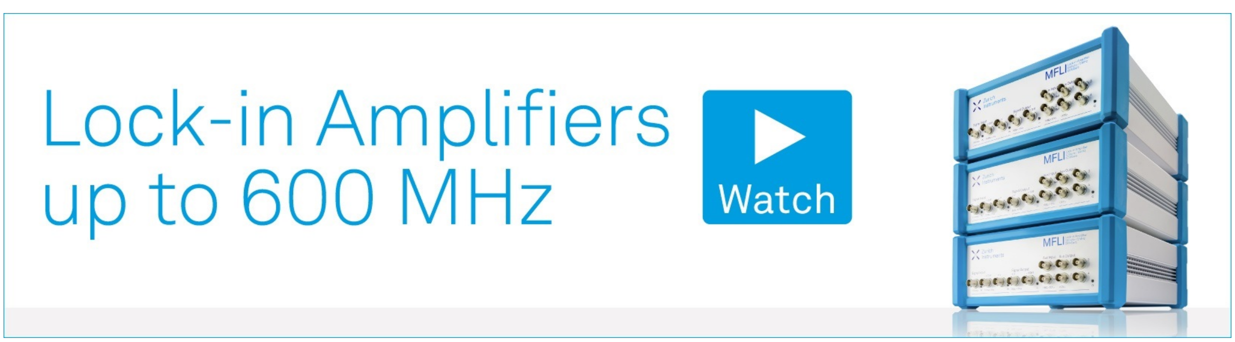
AIP Conference Proceedings

Effect of addition octadecyltrimethoxysilane (OTMS) on morphology ZSM-5-TiO_2 composite

Cite as: AIP Conference Proceedings **2243**, 020011 (2020); https://doi.org/10.1063/5.0001723
Published Online: 04 June 2020

A. Iryani, R. Kurniawati, D. Hartanto, M. Santoso, and H. Nur

View Online
Export Citation



© 2020 Author(s).

Effect of Addition Octadecyltrimethoxysilane (OTMS) on Morphology ZSM-5-TiO_2 Composite

A. Iryani[1, 2], R. Kurniawati[1], D. Hartanto[1, a], M. Santoso[1], and H. Nur[3]

[1]Department of Chemistry, Institut Teknologi Sepuluh Nopember, Surabaya, 60111, Indonesia.
[2]Departement of Chemistry, Pakuan University, Bogor, 16143, Indonesia
[3]Centre for Sustainable Nanomaterials, Ibnu Sina Institute for Scientific and Industrial Research, Universiti Teknologi Malaysia, Johor Bahru, 81310, Malaysia

[a]Corresponding author: djokohar@chem.its.ac.id

Abstract. ZSM-5-TiO_2 composites are usually used as adsorbents as well as photocatalyst in waste treatment. However, these composites are less effective insoluble waste treatment, so it needs to modify by adding organic substances that can react with the waste. In this study, Octadecyltrimethoxysilane (OTMS) was added to the surface of the ZSM-5-TiO_2 composite. The effect of addition OTMS on morphology ZSM-5-TiO_2 composite was studied. ZSM-5-TiO_2-OTMS composite was characterized by X-Ray Diffraction (XRD), Fourier Transformation Infrared Spectroscopy (FTIR), and Energy Dispersive X-ray Scanning Electron Microscopy (SEM-EDX). ZSM-5-TiO_2-OTMS shows a carbon-related peak at 2900 and 1470 cm^{-1} that can be ascribed as C-H stretching and C-O stretching vibration respectively from FTIR spectra. ZSM-5-TiO_2-OTMS composite retains the hexagonal shape surrounded by round-shaped. The addition of OTMS affects the morphology of the ZSM-5-TiO_2 composite. The more OTMS is added, the morphology of the ZSM-5-TiO_2-OTMS composite to be more irregular than composite ZSM-5-TiO_2. It also affects the amount of carbon content that is distributed on the surface of the ZSM-5-TiO_2 composite. This provides information material that can be used in synthesize catalysts in the insoluble waste treatment.

INTRODUCTION

The textile industry is one of the producers of wastewater from the coloring process [1]. Dye waste produced from the textile industry is generally non-biodegradable organic compounds, which can cause environmental pollution especially the aquatic environment [2]. Therefore, technology development is needed as an effort to reduce these impacts which can make the environment-friendly in a sustainable manner.

Some waste treatment techniques include sedimentation [3], biodegradation [4] and adsorption [5] but the process only transfers waste from one place to another without degrading the contaminant. At present, the photodegradation removal technique is an environmentally friendly technique [6].

One of the materials commonly used as adsorbents as well as photocatalysts is the ZSM-5-TiO_2 composite [7,8]. However, these composites have high hydrophilic properties so they are less effective in insoluble waste treatment. The addition of organic compounds such as alkylsilane is one method to reduce hydrophilic properties in some composites and can react with dye waste. Modified Ti-NaY composites with the addition of octadecyltrichlorosilane (ODS) so that the resulting composite is amphilic for the epoxidation reaction of the alkene reaction [9]. The silica surface can be modified by adding octadecyltrichlorosilane (OTCS), octadecyltrimethoxysilane (OTMS) and octadecyltriethoxysilane (OTES) [10]. In addition, the silica surface can be modified with addition (N, N-dim ethyl-amino) -alkyl-dimethylsilanes on SBA-15 and SBA 16 hydrophobic for alcohol adsorption [11]. The addition of OTMS on the silica surface and composites can change the surface properties to be hydrophobic, but the addition of OTMS to zeolite composites has not been carried out. This study focuses on the effect of addition OTMS on morphology ZSM-5-TiO_2 composite as providing information that can be used synthesize catalysts in the insoluble waste treatment.

The 8th International Conference of the Indonesian Chemical Society (ICICS) 2019
AIP Conf. Proc. 2243, 020011-1–020011-5; https://doi.org/10.1063/5.0001723
Published by AIP Publishing. 978-0-7354-2002-1/$30.00

EXPERIMENTAL

Materials

The materials used in this study were titanium (IV) isopropoxide (TiIP) (97%, Aldrich) as precursors TiO_2, ZSM-5 with Si/Al = 50 (Pingxiang Naike Chemical), *Octadecyltrimethoxysilane* (OTMS) (90%, Aldrich), isopropanol, ethanol, and toluene.

Preparation of ZSM-5-TiO_2-OTMS Composite

Synthesis ZSM-5-TiO_2 composites were carried out with the same procedure as the previous study [12]. The ZSM-5-TiO_2 composite with 10% TiO_2 was synthesized with precursor titanium(IV) isopropoxide (source TiO_2), ZSM-5, and isopropanol. Addition OTMS on the surface of the ZSM-5-TiO_2 composite was based on previous literature with the following steps [9]. OTMS was added to toluene drop by step with stirring until homogeneous. ZSM-5-TiO_2 composites were added to the sol to form a gel. Furthermore, the gel was centrifuged with ethanol solvent with 400rpm for 15 minutes. This treatment was repeated three times. The solids were then heated in an oven at 100° C for 24 hours.

Characterization Methods

X-Ray Diffraction (XRD)

Identification of the composite crystal phase of ZSM-5-TiO_2-OTMS using XRD. Solids are placed in the sample holder and put into XRD diffraction equipment at 2Θ 5-50°. XRD used Cu Kα radiation (λ = 1.5406 Å), with a voltage of 40 kV and a current of 30 mA.

Fourier transform infrared (FTIR) Spectroscopy

The functional groups in the ZSM-5-TiO_2-OTMS composite were analyzed using FTIR. FTIR spectra were recorded at 8400 Shimadzu from KBr pellets at room temperature, which was measured in the range of wavenumber 4000 - 500 cm^{-1} in transmittance mode.

Energy Dispersive X-ray Scanning Electron Microscopy (SEM-EDX)

SEM was used to observe the surface morphology of the ZSM-5-TiO_2 composite before and after the addition of OTMS. The sample were prepped with conductive tape to produce a conductive path. An electron detector was placed in the sample chamber.

RESULT AND DISCUSSION

XRD analysis

Based on previous research, XRD patterns of ZSM-5-TiO_2 composite showed peak characteristics of MFI structures (7-10 ° and 22-35 °) and anatase (25,3°). Other anatase TiO_2 at around 37.5 ° and 47 ° appear small peaks caused by the presence of ZSM-5 suppressing anatase crystal growth during synthesis [12].

XRD patterns of composite ZSM-5-TiO_2-OTMS can be seen in Figure 1. From diffractogram XRD, it shows that ZSM-5-TiO_2-OTMS composites have a similar pattern with the ZSM-5-TiO_2 composite. ZSM-5-TiO_2-OTMS composites have a lower intensity MFI peak (7-10 ° and 22-35 °) than ZSM-5-TiO_2 composites. OTMS covers the surface of the ZSM-5-TiO_2 composite so that the peak intensity of the MFI is reduced.

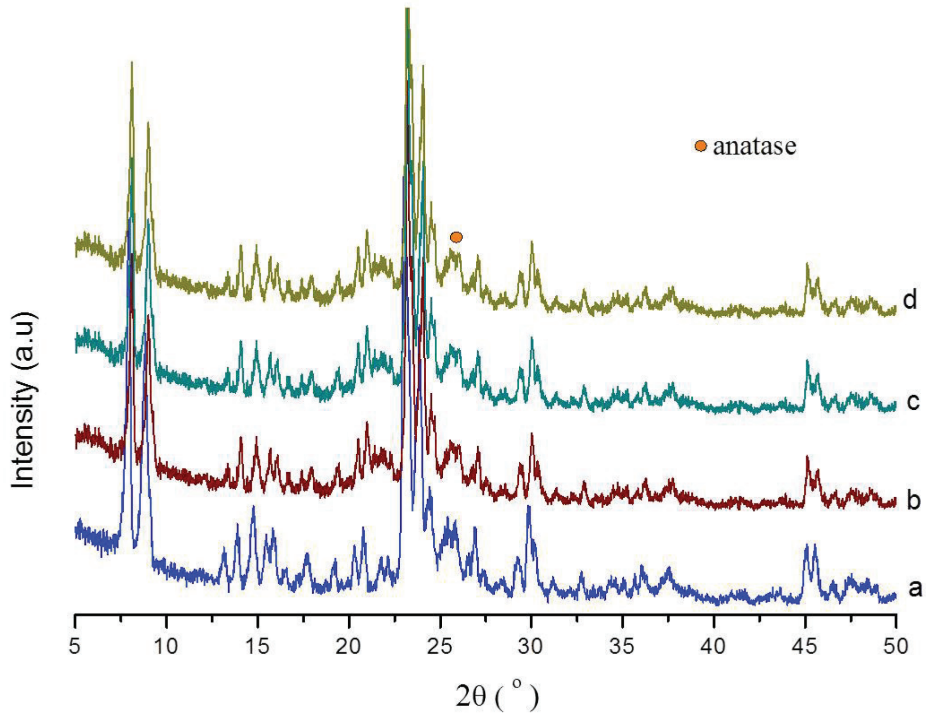


FIGURE 1. XRD patterns of (a) ZSM-5-TiO_2, (b) 0,53 ZSM-5-TiO_2-OTMS, (c) 1,06 ZSM-5-TiO_2-OTMS, and (d) 2,12 ZSM-5-TiO_2-OTMS Composite

FTIR analysis

Figure 2 shows a new ZSM-5-TiO_2-OTMS composite absorption band at wavenumbers around 1470 and 2900 cm^{-1}. The 2900cm^{-1} absorption band shows the C-H stretching vibration. The 1470 cm^{-1} absorption band shows the C-O stretching vibration with small transmittance. Based on these results, ZSM-5-TiO_2-OTMS composites have more carbon content than ZSM-5-TiO_2 composites. More OTMS were added, the more the carbon content in the composite. The addition of OTMS not only can increase the bonding with organic compounds but also it can reduce the wettability of the material.

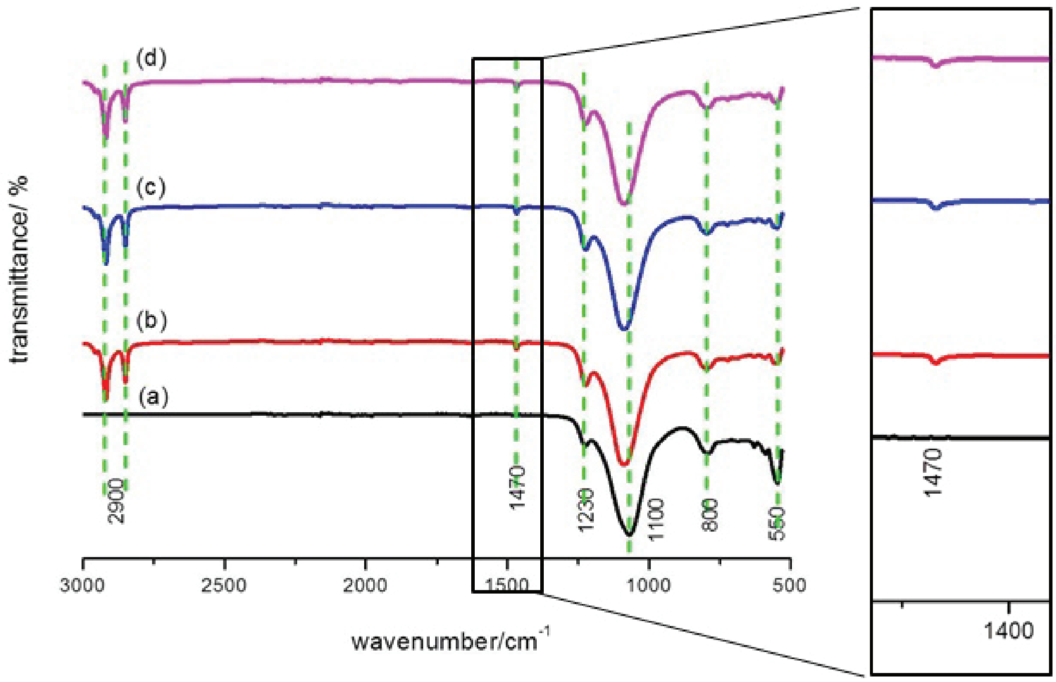


FIGURE 2. Spectra IR of (a) ZSM-5-TiO_2, (b) 0,53 ZSM-5-TiO_2-OTMS, (c) 1,06 ZSM-5-TiO_2-OTMS, and (d) 2,12 ZSM-5-TiO_2-OTMS Composite

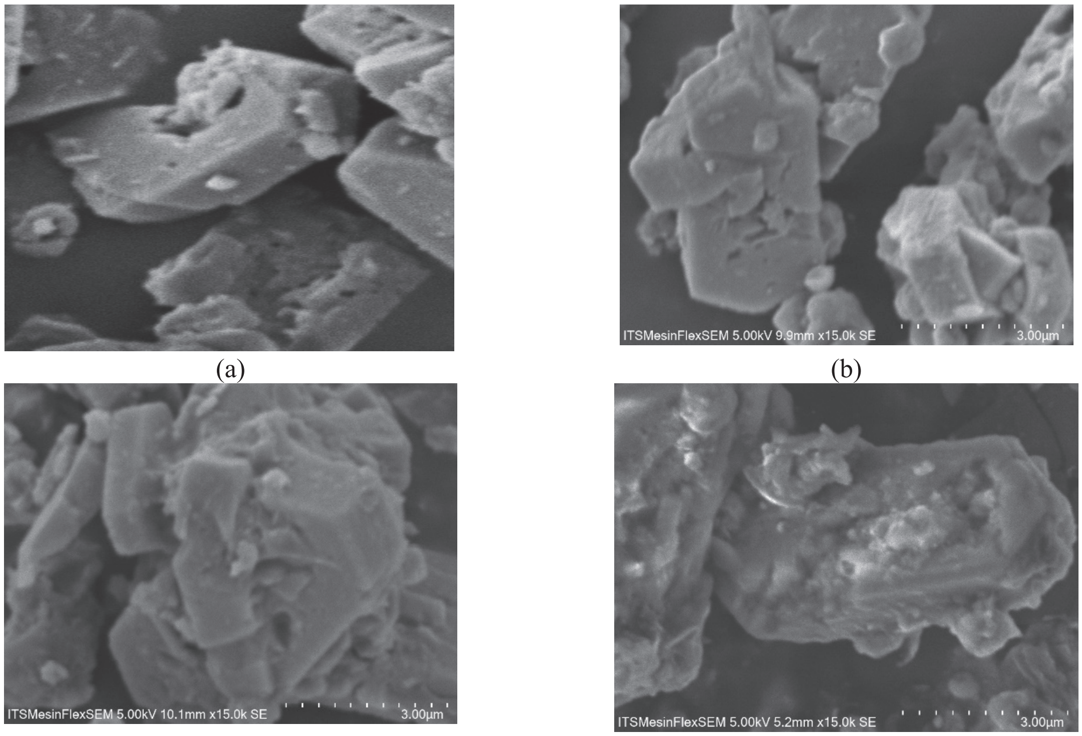
Scanning Electron Microscopy (SEM)

SEM-EDX is used to analyze the morphology and distribution of ZSM-5-TiO_2 composite before and after the addition of OTMS, which can be seen in Figure 3. Composite ZSM-5-TiO_2 has a hexagonal shape (ZSM-5) surrounded by a round shape (TiO_2), which maintains the shape of each constituent (3a). TiO_2 is uniformly distributed on the surface of ZSM-5. The addition of OTMS to the surface of the ZSM-5-TiO_2 composite can be observed in figure 3b-d. The more OTMS is added, it more covers the surface of the ZSM-5-TiO_2 composite. It causes the morphology of the ZSM-5-TiO_2-OTMS composite to be more irregular than composite ZSM-5-TiO_2. This result also supports the XRD pattern.

The EDX analysis results are shown in Table 1. Some amounts of titanium, silica, oxygen, and carbon elements in the ZSM-5-TiO_2-OTMS composite sample were detected. When 0.53 OTMS is added, 14.34% weight of the carbon element is found in the composite sample. If the amount of OTMS has doubled, the amount of carbon elements in the composite sample also increases. However, 2.12 OTMS is added, the amount of carbon in the composite is lower than 1.06 OTMS. This study shows that the distribution of 1.06 OTMS was better than the others. More OTMS were added, the more the carbon content in the composite.

Table 1. EDX analysis results of ZSM-5-TiO_2-OTMS composite

Variation of ZSM-5-TiO_2-OTMS Composites	% weight				
	Si	**Al**	**O**	**Ti**	**C**
0.53	31.96	1.94	45.95	1.95	14.34
1.06	33.58	1.96	47.27	2.84	18.20
2.12	33.57	2.25	43.57	2.44	18.17



(a) (b) (c) (d)

FIGURE 3. SEM images of (a) ZSM-5-TiO_2, (b) 0,53 ZSM-5-TiO_2-OTMS, (c) 1,06 ZSM-5-TiO_2-OTMS, and (d) 2,12 ZSM-5-TiO_2-OTMS Composite

CONCLUSION

ZSM-5-TiO_2-OTMS composite has been successfully synthesized via the sol-gel method. The results from X-Ray Diffraction analysis revealed that ZSM-5-TiO_2-OTMS composites have a lower intensity MFI peak than ZSM-5-TiO_2 composites. OTMS covers the surface of the ZSM-5-TiO_2 composite so that the peak intensity of the MFI is reduced. ZSM-5-TiO_2-OTMS composite has an absorption band at wavenumbers around 1470 and 2900 cm^{-1}. The 2900cm^{-1} absorption band shows the C-H stretching vibration. The 1470cm^{-1} absorption band shows the C-O stretching vibration with small transmittance. The addition of OTMS affects the morphology of the ZSM-5-TiO_2 composite. The more OTMS is added, the morphology of the ZSM-5-TiO_2-OTMS composite to be more irregular than composite ZSM-5-TiO_2. It also affects the amount of carbon content that is distributed on the surface of the ZSM-5-TiO_2 composite. This provides information material that can be used in synthesize catalysts in the insoluble waste treatment.

ACKNOWLEDGMENTS

This work was financially supported by ITS Local Grant Program 2018.

REFERENCES

1. A. Kumar, P. Choudhary, and P. Verma, Journal of Chemical and Pharmaceutical Research **4** (1), 763–771 (2012).
2. S. Khan and A. Malik, *Environmental and Health Effeects of Textile Industry Wastewater (*Springer, Dordrecht, 2013)
3. C. N. Mulligan, R. N. Yong, and B. F. Gibbs, Journal of Hazardous Materials **85**, 145–163 (2001).
4. O. J. Hao, H. Kim, and P. C. Chiang, Critical Reviews in Environmental Science and Technology **30**, 449– 505 (2000).
5. M. Jibril, J. Noraini, L. S., Poh, and A. M. Evuti, Jurnal Teknologi **60**, 15–19 (2013).
6. S. Zi, S. Chandren, L. Yuan, R. Razali, C. Ho, and D. Hartanto, Solid State Sciences **52**, 83-91 (2016).
7. X. Hu, K. Zhou, B. Chen and C. Chang, Applied Surface Science **362**, 329-334 (2016).
8. A. Tawari, W. Einicke, and R. Gläser, Catalysts **6**, 31 (2016).
9. H. Nur, S. Ikeda, and B. Ohtani, Catalysis **20**, 402–408 (2001).
10. V.V. Naik, R. Städler, and N. D. Spencer, Langmuir **30**, 14824-14831 (2014).
11. L Giraldo, M. J. Barranco, and J. C. M. Pirajan, Adsorption **22**, 813-824 (2016).
12. R. Kurniawati, A. Iryani, and D. Hartanto, D. “The effect of 2-propanol on the shifting b)and gap of ZSM-5/TiO2 Composite repared via sol gel method,”. AIP Proceeding (2018), Vol. 2049, pp. 020089.