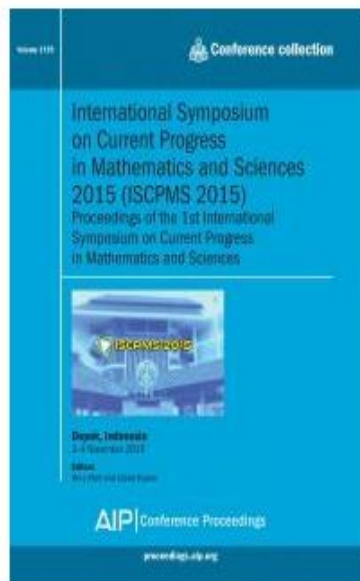




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## Preface From The Editors

The International Symposium on Current Progress in Mathematics and Sciences (ISCPMS) was successfully held in Depok from November 3<sup>rd</sup> to November 4<sup>th</sup>, 2015. In conjunction with the Third IndoMS International Conference on Mathematics and its Application (IICMA) the symposium attracted more than 148 participants coming not only from universities, but also from related research institutions. We are very delighted to present 82 papers in these proceedings after performing a standard review process to the presented papers and involving a number of leading scientists from Indonesia to this end.

The topics covered in this symposium include mathematics and natural sciences. These topics were intensively discussed in the parallel sessions of the symposium. In the plenary session a number of prominent scientists from Australia, Netherlands, Japan, Korea, Malaysia and Indonesia presented the latest findings of their cutting edge researches. We are very happy because these talks attracted almost all registered participants. We really hope that these presentations could inspire the participants to improve their researches as well as to find new bright ideas in their researches. It is also important to mention here that the symposium was aimed to provide a better communication medium among the scientists in Indonesia and surrounding countries.

We would like to thank the members of International Advisory Board and Scientific Committee for selecting the appropriate presentations as well as for selecting and asking the referees to review the selected papers. We are really indebted to the organizing committee for their hard work in preparing and executing this symposium. We also thank our generous sponsors, the Faculty of Mathematics and Natural Sciences, Directorate of Research and Community Engagement (DRPM) Universitas Indonesia, the Ministry of Research, Technology and Higher Education, for kindly supporting this symposium. Finally, we thank all participants for their contributions to this symposium.

Depok, March 2016

Editors  
Terry Mart  
Djoko Triyono

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# Modification of Cellulose Acetate Nanocomposite with TiO<sub>2</sub>-Organoclay as Nanofiller and Its Self-Photodegradation Study

Siti Zahrotul Luthfiah<sup>1</sup>, Yuni. K. Krisnandi<sup>1, a)</sup>, Kadek Andhika<sup>1</sup>,  
Riwandi Sihombing<sup>1</sup> and Adel Fisli<sup>2</sup>

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**Abstract** Nanocomposite cellulose acetate has been synthesized using organoclay nanofiller modified with TiO<sub>2</sub>. Tapanuli bentonite was previously subjected to purification and sodium exchange processes, then modified with TiO<sub>2</sub>, that was added as much as 0%, 1%, 3%, 5%, and 10% weight of the total composite. FTIR analysis showed intercalation with Hexadecyl Ammonium Bromide (HDTMABr) surfactant was successfully carried out, indicated by new absorption bands at 2636 cm<sup>-1</sup> and 2569 cm<sup>-1</sup>. XRD diffractogram shows the increase in basal spacing on the modification of bentonite from 15.7 Å to 19.7 Å after modification. Fabrication of nanocomposite film was carried out using acetone as solvent and through solvent casting method. Nanocomposite application in photodegradation test was carried out under direct sunlight irradiation, UV light, and without irradiation for six days. It is found that the greater the amount of TiO<sub>2</sub> in the composites, the more weight loss occurred, due to photodegradation. Percent weight loss in the UV light irradiation are 1.11%, 2.15%, 2.73%, 3.18%, and 3.96%, while under direct sunlight irradiation, the weight loss was 1.03%, 3.03%, 3.88%, 4.53%, and 5.57%. Modification of nanocomposite with the addition of photocatalytic TiO<sub>2</sub> has shown to give the nanocomposite the ability of self-photodegradation.

## INTRODUCTION

In the modern era, the use of plastics and other polymers has increased. Plastic has some advantages such as good durability, lightweight, easily manufactured, as well as cheap. These advantages in turn have a negative impact when it is not managed properly. The increasing amount of plastic waste that difficult to be biologically recycled has posed new problems needed to be solved. Photocatalytic oxidation is recommended as an environmental friendly way to degrade plastic waste [1]. Cellulose and its derivatives can be used to form more environmental friendly and biodegradable plastic polymers [2].

However, until now, the natural polymer still could not match the quality of the synthetic polymer. This problem can be circumvented by doing modifications of the natural polymer. Natural polymers can be formed into a nanocomposite [3] which is a combination of the polymer with the filler as a reinforcing network. The size of the composite is in nanometer thus the name nanocomposite. Based on dispersion of nanoparticles in a matrix composites, it is expected to form the desired physical properties of the composite.

Clay in Indonesia contained many montmorillonites, which are the main content of the bentonite. The characteristics of a montmorillonite is the high value of cation exchange capacity, good level of swelling, large surface area, and easily decompose in nature [4]. Based on those characteristics, montmorillonite has the right characteristics to form a nanocomposite. The present of AlO<sub>4</sub><sup>5-</sup> in SiO<sub>4</sub><sup>4-</sup> silica layers has given the structure a formal charge of -1, which is balanced by cations Ca<sup>2+</sup> and Na<sup>+</sup> cations [5].

Clay initially has hydrophilic properties [6]. Therefore, in order to be 'compatible' with organic compounds such as polymers, it needs to be modified so that it has organophilic properties [7]. Modification is often done by inserting a cationic surfactant into intergallery of bentonite to produce organoclay [8].

In our work, the organoclay was then modified with anatase TiO<sub>2</sub> in order to add photocatalytic properties to the composite. Until now, titanium dioxide still dominate photocatalytic applications. Generally, TiO<sub>2</sub> is added to the bentonite; as a pillar in intergallery of clay [1].

## EXPERIMENTAL

Montmorillonite was intercalated using surfactant Hexadecyl Ammonium Bromide (HDTMABr). Photocatalytic agent used in this study is TiO<sub>2</sub>. TiO<sub>2</sub> used is Degussa P25 which is a combination of rutile and anatase phases. All chemicals were used without further purification unless otherwise indicated resources. Acetone, NaOH (Sodium Hydroxide), NaCl (Sodium Chloride), Cellulose Asetate, TiO<sub>2</sub> (Titanium Dioxide).

Organoclay Synthesis. Tapanuli Bentonite was purified using pH 6 acetate buffer then carried cation exchange with 1M NaCl to form Na-bentonite purification. Na-bentonite purification was dispersed into aquabidest and surfactant (HDTMABr) by 1 CEC. The suspension was then stirred for 1 h at a temperature of 60 °C. After stirring was completed, the suspension was sonicated for 3 minutes and decanted. The precipitation was then dried at a temperature of 70 °C.

Fabrication of TiO<sub>2</sub>-organoclay-Cellulose Acetate. TiO<sub>2</sub> with variation of weight 1%, 3%, 5%, and 10% from the total composite was dispersed with 7% organoclay from the total composite in acetone and stirred for 1 hour at 60 °C and then sonicated for 3 minutes at room temperature. TiO<sub>2</sub>-organoclay that has been dispersed into acetone was then mixed into the cellulose acetate and stirred for 1 hour at room temperature followed by sonication process for 2 minutes. The suspension was poured into the bioplastics mold using stacked petri dishes and dried with a hotplate at 40 °C for 45 minutes. Synthesized TiO<sub>2</sub>-organoclay/cellulose acetate labeled as in Table 1 below.

Self-Photodegradation Test. The synthesized TiO<sub>2</sub>-organoclay/cellulose acetate was irradiated with two light sources which are UV light and direct sunlight. Each irradiation was performed 6 hours a day for six days. Irradiation with direct sunlight performed at 9:00 a.m to 4.00 p.m. Irradiation using UV lamps carried in a closed (wavelength range 280 nm - 320 nm and 8 W power) was carried in a closed reactor with the distance between the lamp and the sample was 17 cm. The irradiation was taken place at room temperature. Sample weighing tests were performed everyday. Irradiation test control was done by weighing six days composite without irradiation.

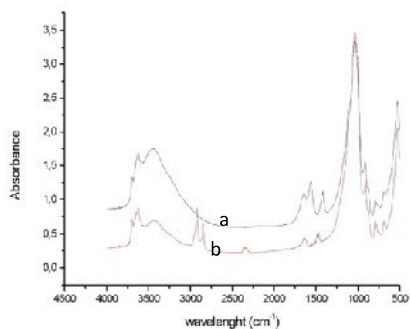
## RESULTS AND DISCUSSION

Intercalation of Surfactant and TiO<sub>2</sub>. Surface modification of clay minerals with surfactants is aimed to change the nature of the hydrophilic surface of the silicate into organophilic surface. This action is a strategic step in the preparation of clay to create a mineral that can absorb or react with organic pollutants [9].

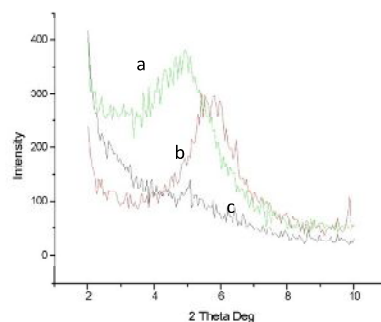
In Figure 1, it can be seen that there is a new absorption band indicating that the surfactants HDTMABr been intercalated into the bentonite to form organoclay. The new absorption band is stretching vibration absorption band of symmetry and asymmetry CH<sub>2</sub> on the wave of 2928 cm<sup>-1</sup> and 2851 cm<sup>-1</sup> and CH<sub>2</sub> scissoring vibration absorption band at wave number 1409 cm<sup>-1</sup> to 1538 cm<sup>-1</sup>.

TABLE 1. Variation of Composite

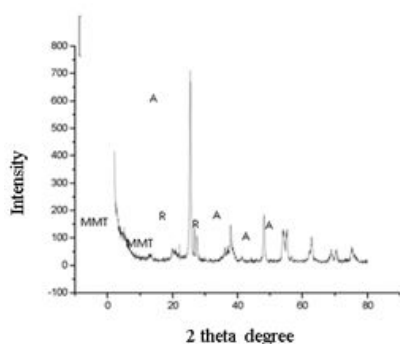
No	Composite Name	Composition
1	0% CA-TiO <sub>2</sub> -organoclay	0.35g Organoclay + 4.65g CA
2	1% CA-TiO <sub>2</sub> -organoclay	0.35g Organoclay + 0.05g TiO <sub>2</sub> + 4.55g CA
3	3% CA-TiO <sub>2</sub> -organoclay	0.35g Organoclay + 0.15g TiO <sub>2</sub> + 4.5g CA
4	5% CA-TiO <sub>2</sub> -organoclay	0.35g Organoclay + 0.25 gr TiO <sub>2</sub> + 4.4g CA
5	10% CA-TiO <sub>2</sub> -organoclay	0.35g Organoclay + 0.5g TiO <sub>2</sub> + 4.15g CA



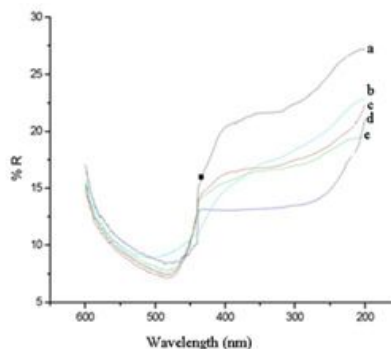
**FIGURE 1.** FTIR spectra pattern in (a) Purification of Bentonite Na (b) organoclay



**FIGURE 2.** Low angle XRD pattern of (a) raw bentonite (b) 10% TiO<sub>2</sub>-organoclay (c) organoclay



**FIGURE 3.** XRD pattern of 10% TiO<sub>2</sub> Organoclay (\*A= Anatase, R = Rutile, MMT = Montmorillonite)



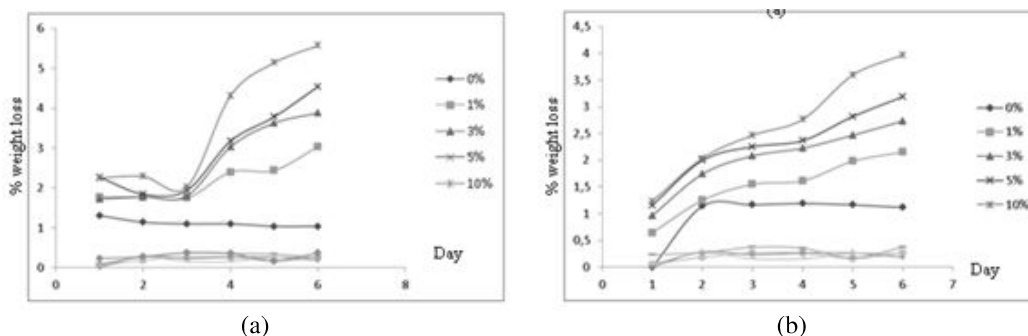
**FIGURE 4.** UV spectra DRS (a) 10% TiO<sub>2</sub>-organic clay (b) 0% TiO<sub>2</sub>-organoclay (c) 5% TiO<sub>2</sub> organoclay (d) 3% TiO<sub>2</sub>-organoclay (e) 1% TiO<sub>2</sub>-organoclay

**TABLE 2.** Energy band gap TiO<sub>2</sub> photocatalyst after dispersion on Cellulose Acetate Plastics

Photocatalyst	TiO <sub>2</sub>	1% TiO <sub>2</sub> Film	3% TiO <sub>2</sub> film	5% TiO <sub>2</sub> Film	10% TiO <sub>2</sub> film
$\lambda$ (nm)	413	434	428	409	402
E <sub>g</sub> (eV)	3	2.85	2.89	3.03	3.08

In Fig. 2, XRD diffractogram was shown in 2 theta range of 2 °C to 10 °C to determine changes in the structure of bentonite as a result of treatment. In accordance with Jovicic *et al.* [10] characteristic peak of montmorillonite is in 5°, a (100) lattice plane. Purification and cation uniformity do not cause damage to the structure of the bentonite. This is indicated by the significant changes that occur in the XRD pattern of MMT. Furthermore, when HDTMABr cationic surfactant was added, there was a shift in the basal spacing of the characteristic peak of bentonite into two smaller theta. By using the Bragg equation, it can be obtained that the shift come from the increase in the size of basal spacing of 15.1 Å (raw bentonite) to 19.7 Å (organoclay).

At 10% TiO<sub>2</sub>-organoclay (Fig. 3), the characteristic peak of montmorillonite at 5° almost disappears. This indicates the organoclay has been covered by TiO<sub>2</sub> that has far more than the amount of montmorillonite. Figure 3 shows the diffractogram according to Degussa P25 TiO<sub>2</sub> diffractogram [11]. Figure 4 shows the UV DRS spectra of modified cellulose acetate composite and acetic sellose composite. It can be seen that the TiO<sub>2</sub>-organoclay-/cellulose acetate 1%, 3%, 5%, and 10% have a value of  $\lambda_g$  nm at 434 nm, 428 nm, 409 nm and 402 nm, respectively. It can be seen that there has been a decreasing value in  $\lambda_g$  (blue shift). Decreasing of  $\lambda_g$  value caused increasing of E<sub>g</sub> value. E<sub>g</sub> value for each of the plastic composite photocatalyst TiO<sub>2</sub>-organoclay-Cellulose Acetate were summarized in Table 2.



**FIGURE 5.** Graphic of (a) Composite Irradiation with Direct Sunlight (b) Irradiation with UV-B at room temperature

Figure 5(a) displays a graph of percent weight loss of the cellulose acetate composite with variation of the addition of TiO<sub>2</sub>-organoclay over time with irradiation using UV-B light having wavelength of 280-315 nm. There is an increase in percent weight loss from the first day until the sixth day of irradiation for each composite sample. From the graph it is shown that time of radiation is directly proportional with percent weight loss caused by photodegradation. In Figure 5(b), solar radiation are strongly influenced by the intensity of sunlight on the day of testing. From looking at the pattern of radiation with UV light and sunlight, there was a common pattern that the increase of TiO<sub>2</sub> concentration in composite will cause the increase in reduction of the percent weight of the composite.

## CONCLUSIONS

Fabrication of TiO<sub>2</sub>-organoclay modified cellulose acetate composite has been carried out. The presence of TiO<sub>2</sub> in the composite causes severe reduction in the composite after irradiation with sunlight and UV light. This shows there is apotential of TiO<sub>2</sub>-nanocomposite to become self-photodegradation composite.

## ACKNOWLEDGMENTS

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