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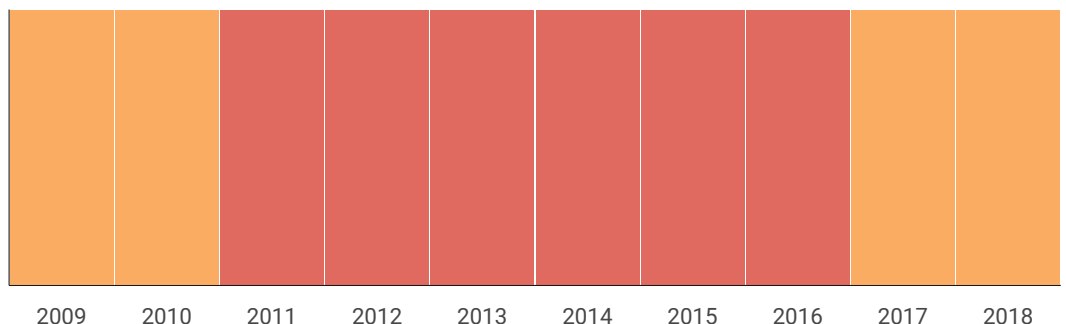
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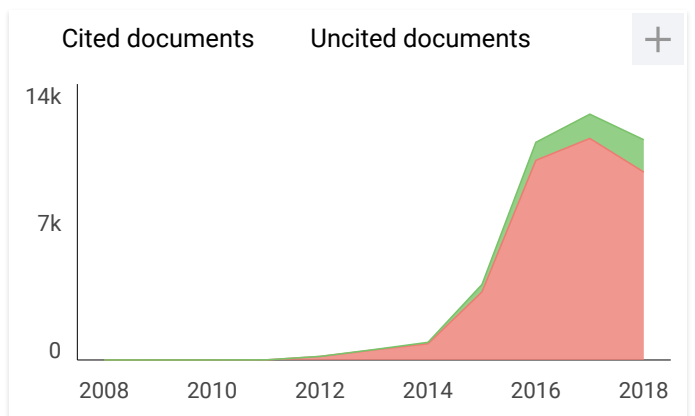
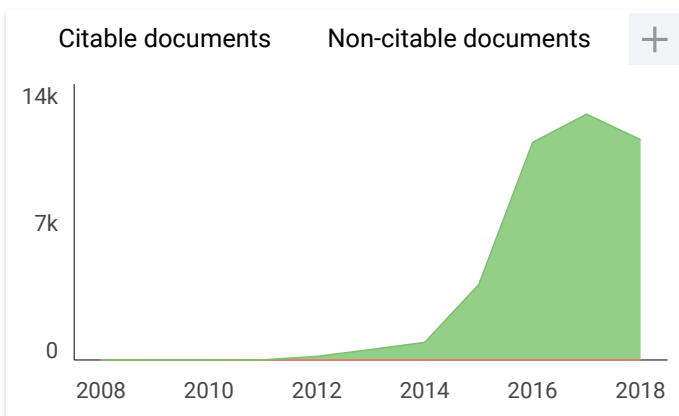
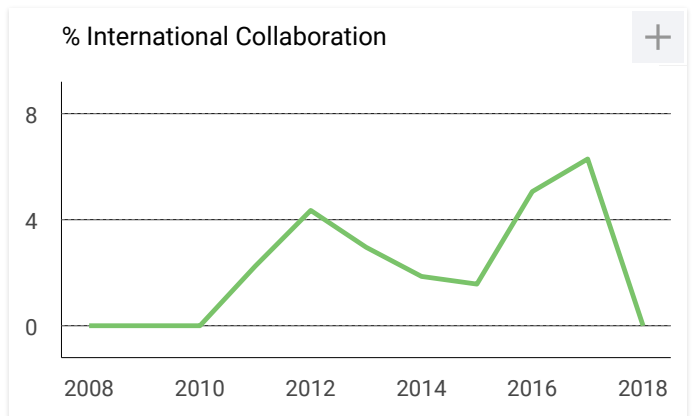
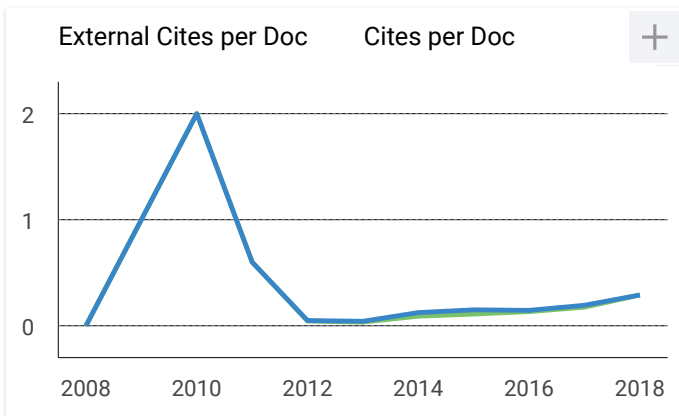
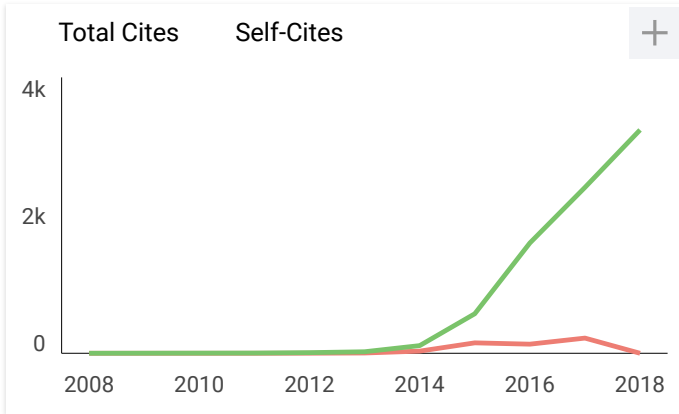
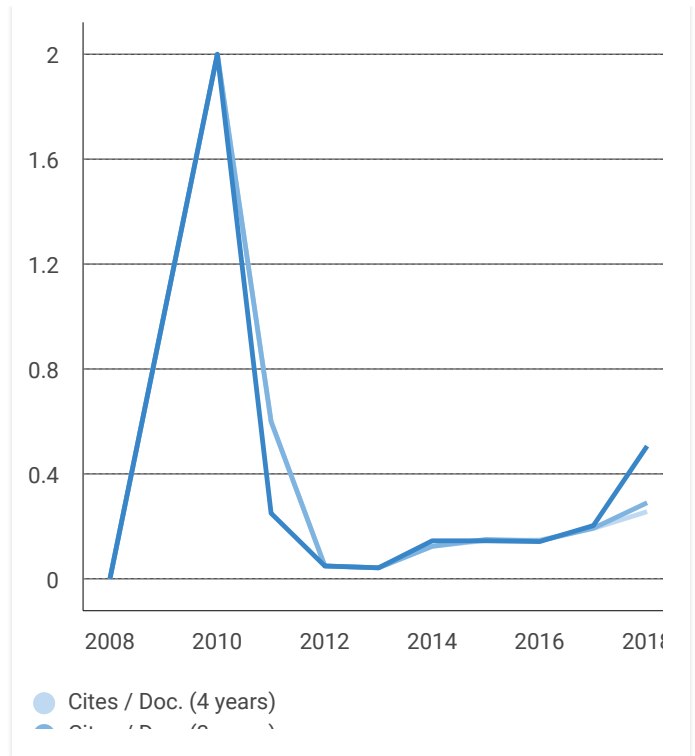
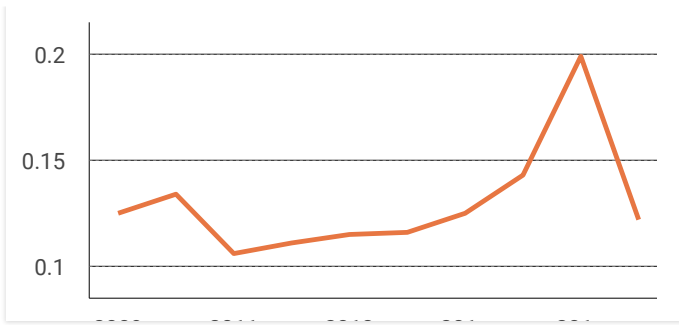


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## Catalytic Performance of TiO<sub>2</sub>-Fe<sub>3</sub>O<sub>4</sub> Supported Bentonite for Photocatalytic Degradation of Phenol

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### Abstract

The capability of TiO<sub>2</sub>-Fe<sub>3</sub>O<sub>4</sub> supported bentonite for phenol photocatalytic degradation was investigated. The raw bentonite obtained from Pacitan, Indonesia was used as porous support for synthesis of the TiO<sub>2</sub>-Fe<sub>3</sub>O<sub>4</sub> based photocatalyst with varying ratio of TiO<sub>2</sub> and Fe<sub>3</sub>O<sub>4</sub>. The variations TiO<sub>2</sub>-Fe<sub>3</sub>O<sub>4</sub> of in this experiment were 2:1 and 3:1. The synthesis of the photocatalytic materials have been carried out by sol-gel method and followed by calcination. The temperatures of calcination was at 700°C. The physicochemical properties of TiO<sub>2</sub>-Fe<sub>3</sub>O<sub>4</sub> bentonite samples were characterised by X-ray diffraction. Photocatalytic activity of the materials was evaluated by phenol photodegradation using UV light. The titania content in the materials significantly influenced the physicochemical properties and catalytic activity.

**Keywords:** TiO<sub>2</sub>, anatase, bentonite, photocatalytic, phenol degradation

### 1. Introduction

The TiO<sub>2</sub> photocatalysts have been extensively studied [1]. As a semiconductor with wide band gap energy and nontoxicity, it is widely used as gas sensors [2], pigment [3], solar-energy conversion [4] and a promising photocatalyst for water and air purification [5–9], dyes adsorption [10] and degradation [11] because of its high oxidative power, chemical stability, low cost, and nontoxicity [12]. Unfortunately, one severe disadvantage of the TiO<sub>2</sub> semiconductor material is the large band gap of 3.0 or 3.2 eV in the rutile or anatase crystalline phase, respectively and only absorbs the UV light which accounts for merely 5% of the solar spectrum. Therefore, there have been many efforts to extend activity of TiO<sub>2</sub> into the visible region to improve

utilize efficiency of  $\text{TiO}_2$  [13–15]. In addition, the use of titania in bulk form suffers from catalyst recovery and activity reduction, which results in ineffective application of the catalyst. In some cases, fine titania particles could also block the penetration of light into the solution. To overcome the limitations, several approaches were proposed e.g. attaching titania particles onto stable inorganic supports [16-19]. In principle, inorganic materials with high specific surface area and chemical stability can be used as host matrix. Natural porous materials such as zeolites and clay minerals are also good and cheaper supports with eco-friendly properties.

Bentonite, one kind of clay minerals, possess high porosity, exchangeable cations and swellable properties. Previously, several researchers reported the preparation of titania immobilization on clay minerals via pillarization processes. However, during the preparation, highly acidic environment of titanium pillaring precursor damages the structure of clay minerals [20,21]. Accordingly, the low crystallinity of supported titania would have a reduced capability of catalyst in catalysis. Additionally, the preparation showed not very good reproducibility [22]. Recently, some investigations reported that the used of several metals, such as Al, Zr and Fe as pillaring precursors for preparation of pillared clay minerals for synthesis of supported  $\text{TiO}_2$  or  $\text{TiO}_2$  composites as catalysts, showed the catalysts have higher thermal stability and specific surface area [23-27].

This paper deals with the phenol degradation on the catalyst activity of  $\text{TiO}_2$ - $\text{Fe}_3\text{O}_4$  immobilized in bentonite. This paper also deals with the ratio of  $\text{TiO}_2$ - $\text{Fe}_3\text{O}_4$  loading.

## **2. Experimental**

The starting clay was a natural Pacitan bentonite, extracted from Pacitan region, East Java, Indonesia. The preparation of  $\text{TiO}_2$ - $\text{Fe}_3\text{O}_4$  based photocatalyst was based on modification of Massart method by sol-gel method. The material was prepared by mixing colloidal Ti (from  $\text{TiCl}_4$ , Merck) and colloidal Fe (from  $\text{FeCl}_2$  and  $\text{FeCl}_3$ , Merck) which has molar ratio of 2:1 and 3:1. Immobilization process was carried out by mixing this colloid with bentonite suspension. The mixture was heated at  $50^\circ\text{C}$ . After 24 hours, the mixture was cooled and washed with aquadest. The obtained solid was dried and calcined at  $700^\circ\text{C}$  for 4 hours with nitrogen and oxygen stream. All the materials were systematically characterized by powder X-ray diffraction (Shimadzu XRD 1000).

The catalytic performance of catalyst materials was carried out in a batch glass reactor equipped with thermometer for photocatalytic degradation of phenol. It was open to the atmosphere and thoroughly stirring with magnetic stirrer. The reaction was conducted at  $30^\circ\text{C}$  for 60 minutes. The initial molar ratio of phenol was 400 ppm; the weight of catalyst was 0.25 g. The course of the phenol degradation was followed by uv-vis spectrophotometer means of Genesis 10. Conversion is defined as the ratio of consumed phenol over the fed phenol for the reaction.



### 3. Result and discussion

The XRD patterns of montmorillonite showed the characteristic reflections at  $2\theta=6.3^\circ$  ( $d_{001}=14.9 \text{ \AA}$ ) and  $2\theta=19.9^\circ$  ( $d=4.5 \text{ \AA}$ ). Other reflections at  $21.8^\circ$  and  $26.6^\circ$  indicated crystoballite and quartz (Fig. 1). Reflections of titania on Bent-Ti:Fe (2:1 and 3:1) were observed. The titania phase was identified at  $2\theta$   $25.1^\circ$ ,  $37.7^\circ$ , and  $61.8^\circ$ . These reflections indicated tetragonal titania (anatase). In addition, the magnetite phase was identified at  $2\theta$   $35.6^\circ$  and  $30.3^\circ$ . However, the intensity of the anatase and magnetite phase was low. Additionally, the (101) reflection of rutile at  $27^\circ$  was found. The hydrolysis rate of  $\text{TiCl}_4$  may affect the titania and crystallization in the bentonite. Titanium and Fe would be fast deposited in gel form before it reached the bentonite pores [11]. Due to the gel form with larger molecular size,  $\text{TiCl}_4$  were difficult to penetrate into the pores of bentonite and the condition would favor the formation of both anatase and rutile during the crystallization process.

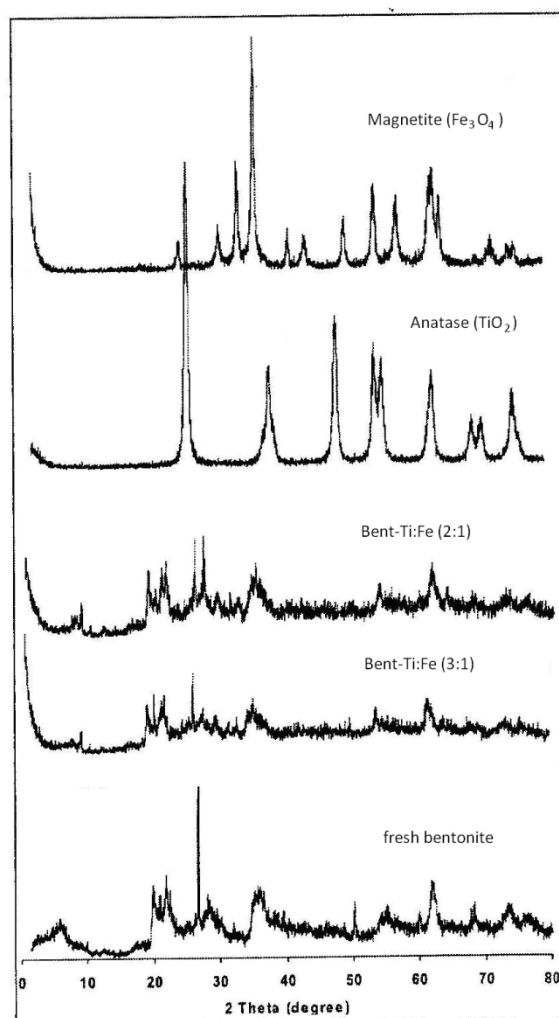


Fig 1. XRD diffractogram of bentonite  $\text{TiO}_2\text{-Fe}_3\text{O}_4$  in ratio molar variation

The photocatalytic activity of various catalysts in phenol photodegradation is presented in table 1. The result show that no reaction in absence of catalyst. Fresh bentonite showed the lowest degradation activity. The reducing of phenol in reaction using fresh bentonite as a result of adsorption process of phenol at the bentonite surface. The other materials exhibited varying activity depending on Ti loading and material phase. The catalytic activity followed the order magnetite > anatase > Bent Ti:Fe 1:2 > Bent Ti:Fe 2:1. This data showed very clearly that anatase and magnetite was responsible material for phenol photodegradation. When Ti and Fe was supported in bentonite, then the catalytic activity was lower than that of anatase and magnetite. This is probably due to the textural change and surface distribution of TiO<sub>2</sub> and Fe<sub>3</sub>O<sub>4</sub>. Loading TiO<sub>2</sub> and Fe<sub>3</sub>O<sub>4</sub> might be reduce the specific surface area, which brought about low adsorption of phenol on catalyst surface for the phenol to be contacted with TiO<sub>2</sub> and/or Fe<sub>3</sub>O<sub>4</sub> for photo reaction. In addition, this is also probably because of aggregation of TiO<sub>2</sub> and/or Fe<sub>3</sub>O<sub>4</sub>. it cause reducing TiO<sub>2</sub> and/or Fe<sub>3</sub>O<sub>4</sub> on bentonite making less amount of active sites than those of anatase and magnetite.

**Table 1. photocatalytic performance of bentonite TiO<sub>2</sub>-Fe<sub>3</sub>O<sub>4</sub>**

Catalyst	Phenol conversion (%)
Without catalyst	-
Fresh bentonite	10
Anatase	48
Magnetite	52
Bent Ti:Fe 1:2	18
Bent Ti:Fe 2:1	14

#### 4. Conclusion

In conclusion, the work has given a brief phenomenological frame to understand the action of bentonite supported Ti and Fe in photodegradation of phenol. It was found that anatase and magnetite have a role to catalyst the photodegradation of phenol. However, the photocatalytic activity of bent-Ti:Fe for phenol degradation was not very high. The synthesis of catalyst materials still need to be improved.

#### ACKNOWLEDGMENT

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