International Journal of Applied Engineering Research

Country: India - SIR Ranking of India

Subject Area and Category: Engineering (miscellaneous)

Publisher: Research India Publications

Publication type: Journals

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Coverage: 2011-2017 (cancelled)

Scope: The International Journal of Applied Engineering Research (IJAER) is an international research journal, which publishes top-level work from all areas of Engineering Research and their application including Mechanical, Civil, Electrical, Computer Science and IT, Chemical, Electronics, Mathematics, Environmental, Education Geological etc. Researchers in all technology and engineering fields are encouraged to contribute articles based on recent research. Journal publishes research articles and reviews within the whole field of Engineering Research, and it will continue to provide information on the latest trends and developments in this ever-expanding subject.

Quartiles:
- 2009: Q3
- 2010: Q3
- 2011: Q4
- 2012: Q4

SJR:
- 2009: 25
- 2010: 25
- 2011: 25
- 2012: 25
- 2013: 25
- 2014: 25
- 2015: 25
- 2016: 25
- 2017: 25
- 2018: 25

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The SJR is a size-independent prestige indicator that ranks journals by their 'average prestige per article'. It is based on the idea that 'all citations are not created equal'. SJR is a measure of scientific influence of journals that accounts for both the number of citations received by a journal and the importance or prestige of the journals where such citations come from. It measures the scientific influence of the average article.

This indicator counts the number of citations received by documents from a journal and divides them by the total number of documents published in that journal. The chart shows the evolution of the average number of times documents published in a journal in the past two, three and four years have been cited in the current year. The two years line is equivalent to journal impact factor™ (Thomson Reuters) metric.

<table>
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<tr>
<th>Year</th>
<th>Cites / Doc. (4 years)</th>
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<td>2017</td>
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Total Cites

Self-Cites

Evolution of the total number of citations and journal's self-citations received by a journal's published documents during the three previous years. Journal self-citation is defined as the number of citations from a journal citing articles to articles published by the same journal.

<table>
<thead>
<tr>
<th>Year</th>
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<tbody>
<tr>
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External Cites per Doc

Cites per Doc

Evolution of the number of total citation per document and external citation per document (i.e. journal self-citations removed) received by a journal's published documents during the three previous years. External citations are calculated by subtracting the number of self-citations from the total number of citations received by the journal's documents.

<table>
<thead>
<tr>
<th>Year</th>
<th>Cit / Doc</th>
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<tr>
<td>2008</td>
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</table>

International Collaboration

International Collaboration accounts for the articles that have been produced by researchers from several countries. The chart shows the ratio of a journal's documents signed by researchers from more than one country; that is including more than one country address.

<table>
<thead>
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<th>International Collaboration</th>
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<tr>
<td>2009</td>
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</table>

Citable documents

Non-citable documents

Ratio of a journal's items, grouped in three years windows, that have been cited at least once vs. those not cited during the following year.

<table>
<thead>
<tr>
<th>Year</th>
<th>Uncited documents</th>
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<td>2010</td>
<td>0</td>
</tr>
<tr>
<td>2011</td>
<td>3</td>
</tr>
</tbody>
</table>

Cited documents

Uncited documents

Not every article in a journal is considered primary research and therefore "citable", this chart shows the ratio of a journal's articles including substantial research (research articles, conference papers and reviews) in three year windows vs. those documents other than research articles, reviews and conference papers.

<table>
<thead>
<tr>
<th>Year</th>
<th>Uncited documents</th>
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<td>2011</td>
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</tr>
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Area of Interest: Data Mining, VLSI Physical Design, Computer Networks, Embedded systems

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Catalytic Performance of Al-HDTMA Bentonite Impregnated Fe on Phenol Hydroxylation

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ABSTRACT

This paper describes the addition of Fe into modified-bentonite layer by impregnation method. Natural bentonite (from Pacitan, Indonesia) was intercalated with HDTMA-Br 1.5% solution before pillared with Al metal to give Al-HDTMA bentonite forms. The ratio of bentonite and intercalating agent or pillaring agent was 1 gr/50 ml. The mixture was agitated, and then the solid phase was washed with distilled water. Then it was dried and calcined at 723 K for 4 hours. This modified bentonite was then impregnated with Fe solution. The Fe concentrations were 0.01 M, 0.05 M, and 0.1 M. All the materials were characterized using FT-IR and X-ray diffraction. X-ray diffractogram showed that the higher Fe penetrates into bentonite, the lower the crystallinity of bentonite. Their catalytic activity and selectivity were studied for phenol hydroxylation using H₂O₂ (30%). The reaction conditions of this reaction were as follows: ratio of phenol/H₂O₂ = 1:1 (molar ratio), concentration of phenol = 1 M, reaction temperatures were 333 K, and ratio of catalyst/phenol was 1:10. The best catalytic performance to convert phenol and produce hydroquinone by phenol hydroxylation reaction is on PILB HDTMA-Al,Fe 0.05 M.

Keywords: bentonite, impregnation, catalyst, phenol hydroxylation

INTRODUCTION

Clay is one of the most abundant material found in nature. Bentonite, the material which is used in this research, is natural bentonite from Tulakan, Pacitan in east Java. Bentonite consists of montmorillonite mineral as the major component. Up to now, bentonite can be utilized as filler, ion exchanger, catalyst, adsorbent etc.
Bentonite has characteristics such as moisture content as high as 30-40 %, and easy to be swelling. The characteristics cause unstable structure and make low selectivity for both adsorbent and catalyst. Otherwise, the interlayer spacing of the swollen clay is sufficient to receive the Al and Al/Fe polyoxocations [1]. Because of that, bentonite has to be modified to get more stable structure. One of the ways to modify bentonite is by pillarisation process [2]. The application fields of these substrates are very broad because of the possibility to intercalate different types of pillars making them suitable for specific adsorption and catalytic processes.

The introduction of inorganic pillars in the natural bentonite improves its resistance and stability, increases its micro porosity and provides larger surface area and accessibility to its acid sites (Brönsted and Lewis sites) [3,4]. Pillared bentonite can be utilized as catalyst because of the reason that pillared bentonite has active sites such as silica group (SiO$_4$) and alumina group (AlO$_4$). The presence of active metal which is penetrated into bentonite structure during pillarisation process can enhance the catalytic performance of pillared bentonite. Otherwise, the presence of pore which has definite size will give the ability to select both of reactants and products.

To increase its selectivity, some modifications of bentonite have been done. One of the modifications is clay intercalation using organic molecules (anionic, cationic and non-ionic molecules). This process cause the distance between clay molecules becomes longer, so that the pore size becomes bigger. As a result, the bigger molecules (organic molecules usually have big molecular size) can be laid on the bentonite surface where the adsorption and/or catalytic process is occur [5-7]. In addition, the other modification is clay pillarisation using metal oxides [5,6,8]. Various works have been reported on the catalyst synthesis [9-13]. They show that catalytic activity depends on several factors such as chemical composition, preparation method, calcinations temperature and characteristic of catalyst surface.

In environmental catalysis there is a great interest in obtaining pillared clays containing iron (oxides or oxyhydroxides) species use in the catalytic wet peroxide oxidation of toxic organic compounds since many industrial processes yield a variety of organic contaminants which are poured into natural water sources with negative impact on ecosystem and humans (toxicity, carcinogenic and mutant properties) [14]. Among these pollutants contained in industrial residual water we can highlight phenol, substituted phenols (chlorophenol, nitrophenol), oxalic acid, acetic acid, pesticides and herbicides [14]. Phenol is particularly considered as one of the most toxic organic contaminants and it is commonly chosen as model molecule on catalytic oxidation of organic compounds studies in diluted aqueous medium [15]. Several processes of catalysis industrial importance have been reported over pillared clays [16–18].

This work shows the synthesis of solid catalyst, which is bentonite was intercalated using Cetyl-Tetramethylammonium Bromida, CTMA-Br or HDTMA then pillared by Al. Al-HDTMA bentonite was then pillared with Fe by impregnation method, in which iron is introduced as active phase (activation sites for hydrogen peroxide as well as organic molecules) in order to develop the catalyst material to the complete oxidation of organic molecules.
EXPERIMENTAL SECTION

Materials
Clay (natural Pacitan bentonite), FeCl₂ (Merck), Cetyl-Tetramethylammonium Bromida, CTMA-Br or HDTMA-Br (Merck), NaOH (Merck), Al(OH)₃ (Merck).

Instrumentation
Fourier Transform Infra Red Spectrophotometer (FTIR, Bruker Tensor 27, Germany), X-ray diffraction (XRD, Shimadzu XRD 1000)

Procedure
The starting clay was a natural Pacitan bentonite, extracted from Pacitan region, East Java. Cationic polyoxy Fe solution is prepared by dissolve and stir Fe salt and NaOH in water. Clay suspension is prepared by dissolve 1 gr of bentonite in water. Polyoxy solution and suspension are mixed and stirred. The solid is washed and then dried in the oven at 373 K. Intercalation is carried out by adding 1 gr bentonite suspension into 50 ml surfactant (Cetyl-Tetramethylammonium Bromida, CTMA-Br or HDTMA) solution. The materials were calcined at 723 K for 4 hours.

The pillaring agent solution was prepared by mixing NaOH and Fe which has molar ratio OH to Al = 0.8. Pillarisation process of the clay was carried out by mixing HDTMA-bentonite and pillaring agent solution with ratio [gram bentonite/volume of solution] = 1 gram/50 mL. The mixture was heated at 343 K. After 5 hours, the mixture was cooled, and washed with aquadest. The obtained solid was dried in the oven at 373 K. This modified bentonite was then impregnated with Fe solution. The Fe concentrations were 0.01 M, 0.05 M, and 0.1 M After impregnation process, Al-HDTMA bentonite was calcined at 773 K for 4 hours with nitrogen and oxygen stream.

The chemical analysis of the solids was carried out by Fourier Transform Infra Red Spectrophotometer (FTIR, Bruker Tensor 27, Germany) to characterize bentonite structure. The X-ray diffraction (XRD) study was done in Shimadzu XRD 1000 device. It identified crystalline structure and d₀₀₁ spacing of pillared bentonite.

The catalytic oxidation reaction of phenol in a diluted aqueous medium was carried out in a batch glass reactor equipped with thermometer, and reflux condenser. It is open to the atmosphere, and thoroughly stirring with magnetic stirrer. Firstly, the reactor was loaded with phenol solution and catalyst and heated up until 333 K. The reaction was conducted at 333 K for 4 hours. The hydrogen peroxide solution was added stepwise during a reaction time of 4 hours. The molar ratio of phenol to hydrogen peroxide = 1:1. The experiment variables are the weight ratio of catalyst to phenol = 1:10, and 5:10. The course of the phenol conversion and selectivity was followed by high performance liquid chromatography (HPLC) by means of Knauer.

RESULTS AND DISCUSSION

Characterization

FTIR spectra
Characterization was conducted to Al pillared HDTMA-bentonite (PILB HDTMA Al)
without Fe impregnation and with Fe impregnation in various Fe concentration. The experiments were conducted in Fe concentrations 0.01 M (PILB HDTMA Al, Fe 0.01M), 0.05 M (PILB HDTMA Al, Fe 0.05M), and 0.1 M(PILB HDTMA Al, Fe 0.1M). The results were shown by figure 1.

Fig. 1. FTIR Spectra of PILB HDTMA-Al dan Fe impragnated PILB HDTMA-Al in various Fe concentration

Fig. 1 displays the absorption peak in the region 1035-1050 cm$^{-1}$ which is indicated that Fe has introduced into Al-HDTMA pillared bentonite. It can be observed by the change of the peak in that region. The higher Fe was impregnated into Al pillared bentonite, the lower the intensity of the absorption. It shows that more Fe was introduced into pillared bentonite, the crystallinity of it was decrease (amorph). It was caused that pillar structure in previous material (O-Al-O) becomes disorder because of the formation of Fe oxide.

The absorption peak in the range 526-650 cm$^{-1}$ of PILB HDTMA Al-Fe in various concentration was appeared different. The absorption peak at 526 cm$^{-1}$ of PILB
HDTMA Al-Fe 0.05 was appeared narrower than that of PILB HDTMA Al-Fe in other concentration. It is proved that the number of double range structure of PILB HDTMA Al-Fe 0.05 M is relatively higher and more stable than that of other pillared bentonite. It suggests that the number of created pores of PILB HDTMA Al-Fe 0.05 M was higher and the pore was bigger than those of other pillared bentonite. The form of the absorption peak in that region was shown smooth in line with increasing Fe concentration. The absorption peak in the region 910 – 920 cm⁻¹ show that the amount of Fe impregnated was higher result in the peak was getting smaller and weaker. It was occurred because the octahedral structure TO₆ (AlO₆) was pushed by Fe which is introduced into bentonite structure. It caused O-Al-O bonding in octahedral structure TO₆ broken down. The damage of octahedral structure results in decreasing of crystalinity of bentonite structure.

Based on the analysis, it can be suggested that increasing number of Fe will cause the damage of bentonite crystalline structure and decreasing the number of pores in bentonite structure. It is validated by the absorption peak of PILB HDTMA Al-Fe in various concentrations at 525.8 cm⁻¹ was smoother and narrower than that of PILB HDTMA Al.

**X-Ray Diffraction characterization**

The success of pillarisation and impregnation process of bentonite by using Al and Fe metal can be characterized by X-ray diffraction. By observing the shifting of the diffraction peak and the change of d₀₀₁ spacing as shown by figure 2, it can be suggested the size of created pores of bentonite.

![Fig. 2. X-ray diffractogram of PILB HDTMA in various Fe concentrations](image-url)
The main peak of fresh bentonite is appeared at \( 2\theta = 5.828^\circ \) and \( d_{001} \) spacing = 15.1524. The smaller the value of \( 2\theta \), the bigger the layer distance of bentonite is. It suggests the created pillars are higher and the pores are bigger. Figure 2 shows that the peak at \( 2\theta \) of PILB-HDTMA-Al shifts from 5.828\(^\circ\) to 6.25\(^\circ\) with the value of \( d_{001} \) spacing = 14.1302, it means that interlayer spacing of bentonite is getting smaller. Al metal which is penetrated into interlayer of bentonite will be crowded horizontally and results in creating low pillar. Figure 2 shows that last three of impregnated bentonite almost has no main peak. It is suggested that most of the crystalline structure of Al pillar bentonite was broken. The higher Fe concentration used as impregnating agent, the lower the main peak in diffractogram. If the amount of Fe in bentonite structure is higher, the existing pillars because of pillarisation process become damage after calcinations. The phenomenon is occurred because Fe is easier to attack oxygen atom of –O-Al-O-group. It is proved by the evidence that the \( I^- \) count value of main peak is getting lower in line with increasing Fe concentration (Table 1).

Table 1. The effect of Fe concentration to layer distance of PILB HDTMA-Al

<table>
<thead>
<tr>
<th>Sample</th>
<th>( 2\theta ) (degree)</th>
<th>( d_{001} ) spacing (Å)</th>
<th>( I^- ) count</th>
<th>The amount of Fe (mg/gr)</th>
<th>The amount of penetrated Fe (mg/gr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh bentonite</td>
<td>5.828</td>
<td>15.152</td>
<td>695</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PILB HDTMA-Al</td>
<td>6.25</td>
<td>14.130</td>
<td>295</td>
<td>1.2679</td>
<td>-</td>
</tr>
<tr>
<td>PILB HDTMA-Al,Fe 0,01 M</td>
<td>6.38</td>
<td>13.842</td>
<td>194</td>
<td>2.0412</td>
<td>0.7734</td>
</tr>
<tr>
<td>PILB HDTMA-Al,Fe 0,05 M</td>
<td>6.283</td>
<td>14.055</td>
<td>192</td>
<td>4.2095</td>
<td>2.9416</td>
</tr>
<tr>
<td>PILB HDTMA-Al,Fe 0,1 M</td>
<td>6.213</td>
<td>14.213</td>
<td>95</td>
<td>5.9258</td>
<td>4.6579</td>
</tr>
</tbody>
</table>

PILB-HDTMA-Al and PILB-HDTMA Al-Fe has lower crystallinity than fresh bentonite. Fresh bentonite has \( d_{001} \) spacing = 15.1524 while PILB-HDTMA-Al has \( d_{001} \) spacing = 14.1302. It means that the existing pillar has broken and decreases the crystallinity of bentonite. It also occurs on Fe-impregnated bentonite. The higher Fe penetrates into bentonite, the lower the crystallinity of bentonite. It is validated by IR spectrum in the range 900-1100 cm\(^{-1}\). The absorption peak is getting weaker in line with increasing Fe concentration.

**Catalytic Activity Test and Selectivity for Phenol Hydroxylation Reaction**

Catalytic activity test for synthetic catalysts is conducted for phenol hydroxylation reaction. Table 2 shows phenol conversion and selectivity of hydroquinone, catechol, and benzoquinone. It describes that by using modified bentonite catalyst in the system, % conversion of phenol is higher than those of fresh bentonite and without catalyst system. It happens because the number of active site and suitable pore size is increase, and it gives effect on the amounts of phenol which can be converted into
product. Percentage conversion and reaction selectivity were determined by HPLC analysis

The reaction scheme as shown below:

![Reaction Scheme](image)

The catalytic reaction produced hydroquinone (HQ), cathecol (CAT) and benzoquinone (BEQ). The most expected product is HQ but it is produced in small amount. It is shown that the catalyst has not been selective enough to produce the expected product. The system also produced isomer product (CAT) in high amount and very small amount of benzoquinone. Phenol hydroxylation has been known to proceed via redox mechanism involving Fe$^{3+}$/Fe$^{2+}$ redox pair. It also required generation of OH$^{-}$ radicals by decomposition of H$_2$O$_2$. Transition metal ion was needed to initiate the decomposition of H$_2$O$_2$ [17,18]. Because of the fresh bentonite was devoid of any transition metal ions, it was inactive in hydroxylation. Table 2 shows that transition metal ion Fe was successfully loaded in catalyst. Fe$^{2+}$ might coordinate to H$_2$O$_2$ strongly enough to decompose it. However, it seems that surface area also plays an important role in the catalytic activity for phenol hydroxylation. It can be seen in table 2 that the intercalation samples (PILB HDTMA-Al) which has lower surface area values than that of impregnated bentonite (PILB HDTMA Al, Fe), was less active for phenol hydroxylation.

### Table 2 Hydroxylation of phenol to hydroquinone and cathecol

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>Conversion (%)</th>
<th>Selectivity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>hydroquinone</td>
<td>cathecol</td>
</tr>
<tr>
<td>Without catalyst</td>
<td>1.2</td>
<td>49.1</td>
</tr>
<tr>
<td>Fresh bentonite</td>
<td>4.4</td>
<td>47.0</td>
</tr>
<tr>
<td>PILB HDTMA-Al</td>
<td>58.8</td>
<td>8.4</td>
</tr>
<tr>
<td>PILB HDTMA-Al,Fe 0.01 M</td>
<td>72.8</td>
<td>23.9</td>
</tr>
<tr>
<td>PILB HDTMA-Al,Fe 0.05 M</td>
<td>73.8</td>
<td>27.6</td>
</tr>
<tr>
<td>PILB HDTMA-Al,Fe 0.1 M</td>
<td>69.7</td>
<td>27.5</td>
</tr>
</tbody>
</table>

Reaction condition: Catalyst/Phenol/H$_2$O$_2$ = 1/10/10
The results also show that the concentration of impregnation of Fe into modified bentonite plays the important role in the catalytic activity for phenol hydroxylation. Table 2 indicates that adjusment of Fe concentration causes variation of phenol conversion. Table 2 also depicts that the most excellent Fe concentration for impregnation of modified bentonite is 0.05M. This is supported by the results that phenol conversion is as high as 73.8%, and the selectivity of HQ reaches 27.6 %, and on contrary benzoquinone selectivity is very low. This result also states that pore size of Fe pillared bentonite has not been appropriate for HQ molecule but it is suitable for CAT molecule. HDTMA is expected to increase pore size and be able to support two bentonite layers before penetrating pillaring agent, but the experiment did not get optimal condition of material. It seems that the calcinations process was not successful to remove the organic surfactant molecules. Hence it blocked the porosity.

CONCLUSION
The intercalation of bentonite using HDTMA and followed by impregnation with Fe is successfully presented. However, method for preparing the catalytic material with large pores, namely mesopores, is still lacking. This paper showed that the pillaring bentonites have good potential to catalyze phenol hydroxylation reaction. The best catalytic performance to convert phenol and produce hydroquinone by phenol hydroxylation reaction is on PILB HDTMA-Al,Fe 0.05 M.

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REFERENCES
Catalytic Performance of Al-HDTMA Bentonite Impregnated
