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# Iron Oxide-pillared Clay Catalyzed the Synthesis of Acetonides from Epoxides

Piyarat Trikittiwong<sup>a</sup>, Nipaka Sukpirom<sup>b</sup>, Shogo Shimazu<sup>c,\*</sup> and Warinthorn Chavasiri<sup>b,\*</sup>
<sup>a</sup>Program in Petrochemistry, Faculty of Science, Chulalongkorn University, Bangkok 10330, Thailand.
<sup>b</sup>Department of Chemistry, Faculty of Science, Chulalongkorn University, Bangkok 10330, Thailand.
<sup>c</sup>Department of Applied Chemistry and Biotechnology, Faculty of Engineering, Chiba University, Chiba-shi 263-8522, Japan.
# Corresponding author, e-mail: warintho@yahoo.com, Tel: 662-218-7625

#### # Corresponding aution, e-mail: warmino@yanoo.com, 1ei. 002-218

### Abstract

 $FeO_x$ -pillared clays (FeO\_x-pillared bentonite,  $FeO_x$ -pillared hectorite and  $FeO_x$ -pillared taeniolite) were synthesized by the intercalation of  $FeCl_3$  into clay interlayers and calcination. The synthesized catalysts were exploited for conversion of epoxides furnishing acetonides in good to quantitative yields with short time under mild conditions. The catalysts could be recovered and reused up to five times without appreciable loss of activity.

Keywords: Iron oxide pillared clays; Epoxides; 1,3-Dioxolanes

### 1. Introduction

The protection of a carbonyl functional group from nucleophilic attack is still challenging task. One of the most useful protecting methods for carbonyl compounds (aldehydes and ketones) includes acetalization, thioacetalization and ketalization [1]. 1,3-Dioxolanes are widely used to protect aldehydes and ketones. Epoxides are attractive intermediates due to their wide range of regio-, chemo- and stereoselective transformations with concomitant ring opening. The direct transformation of epoxides to acetonides was previously reported using several Lewis acid catalysts such as anhydrous CuSO<sub>4</sub> [2], BF<sub>3</sub>.OEt<sub>2</sub> [3], CH<sub>3</sub>ReO<sub>3</sub> [4], RuCl<sub>3</sub> [5], TiO(TFA)<sub>2</sub> and TiCl<sub>3</sub>(OTf) [6], Fe(TFA)<sub>3</sub> [7], K<sub>5</sub>CoW<sub>12</sub>O<sub>40</sub>·3H<sub>2</sub>O [8], Bi(TFA)<sub>3</sub> [9], TABCO [10], SnCl<sub>2</sub> [11], Sn(tpp)(OTf)<sub>2</sub>[12], Er(OTf)<sub>3</sub>[13], and LiBF<sub>4</sub>[14]. Some reported methods suffered from air- and moisture-sensitive catalysts, expensive and toxic catalysts, high refluxing temperature, long reaction time, difficulty in separation and recovery, less selectivity or yields and functional group intolerance. In recent years, inorganic solid acids catalyzed organic reactions have gained importance due to the proven advantage of heterogeneous catalysts such as simplified product isolation, easy work-up, mild reaction conditions, shorter reaction time, easy recovery and reusability of catalysts, better yield and a large reduction in the generation of wasteful products [15].

Previously, certain clays such as K10-montmorillonite [16] and natural kaolinitic clay [17] have been reported as an efficient catalyst in the synthesis of acetonides from epoxides. Nonetheless, no report on the utilization of  $FeO_x$ -pillared clays as catalysts has been addressed. Herein, we report a new and efficient procedure for the preparation of acetonides from epoxides by pillared clay catalysts under mild conditions.

#### 2. Experimental

#### 2.1. General

Two types of the 2:1 layered clays were used as starting materials. Hectorite (Volclay Siam Limited), with typical chemical analysis of (%wt): SiO<sub>2</sub> 61.78, Al<sub>2</sub>O<sub>3</sub> 1.58, MgO 20.32, Fe<sub>2</sub>O<sub>3</sub> 1.23, CaO 10.07, Na<sub>2</sub>O 2.80, K<sub>2</sub>O 0.33, was used as received. Bentonite clay (Cernic International Co., LTD) with typical chemical analysis of (%wt): SiO<sub>2</sub> 63.60, Al<sub>2</sub>O<sub>3</sub> 17.60, Fe<sub>2</sub>O<sub>3</sub> 3.10, CaO 3.00, Na<sub>2</sub>O 3.40, K<sub>2</sub>O 0.50, was purified by fractionated centrifugation in order to remove quartz and other dense impurities. De-ionized water was used throughout the study. For preparation of catalysts, FeCl<sub>3</sub> 6H<sub>2</sub>O (Riedel-de Haën) and NaOH (Merck) were of laboratory reagent grade. De-ionized water was used throughout the study. For catalytic study, all organic solvents were purified using standard distillation techniques. All reactants were purchased from Fluka as laboratory reagent grade.

### 2.2. General preparation of acetonides

The mixture of epoxide 1 mmol and acetone (3 mL) was added to round bottle flask with 10% wt of catalyst to epoxide. The solution was continuous stirred for desired time and temperature. After the specific time or the reaction was finished, the catalyst was filtered out of the mixture, and washed the product out with EtOAc. The solvent was evaporated to dryness under reduced pressure to afford the product. The reactions were monitored by TLC aluminium sheets precoated with silica gel (Merck's, Kieselgel 60 PF<sub>254</sub>). Products of the preparation of acetonides from epoxides were monitored by Shimadzu GC 14-B (FID, HP-5 column) and Shimadzu QP-5000 GCMS. The <sup>1</sup>H NMR spectra were obtained in CDCl<sub>3</sub> and determined by

Mercury plus 400 NMR spectrometer operated at 399.84 MHz. All products were identified by <sup>1</sup>H NMR.

2,2-*Dimethyl-4-phenyl-1,3-dioxolane*; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 1.48 (3H, s), 1.52 (3H, s), 3.67 (1H, t), 4.27 (1H, dd, J = 8.6, 6.4 Hz), 5.05 (1H, dd, J = 8.6 Hz), 7.31 (5H, m).

2,2-Dimethyl-hexahydro-benzo[1,3]dioxole; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 1.24-1.00 (4H, m), 1.35 (6H, s), 2.58-2.07 (4H, m), 4.34-4.05 (2H, m).

*4-Decyl-2,2-dimethyl-1,3-dioxolane*; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 0.81 (3H, t), 1.14 (16H, m), 1.29 (3H, s), 1.34 (3H, s), 1.56 (2H, m), 3.42 (1H, dd, J = 8.8, 7.2 Hz), 4.00 (2H, m).

*4-(Butoxymethyl)-2,2-dimethyl-1,3-dioxolane*; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 0.84 (3H, t), 1.30 (3H, s), 1.34 (3H, s), 1.48 (4H, m), 3.49 (3H, m), 3.67 (2H, m), 3.99 (1H, dd, J = 8.0, 6.8 Hz), 4.20 (1H, quin).

*4-(tert-Butoxymethyl)-2,2-dimethyl-1,3-dioxolane*; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 1.13 (9H, s), 1.31 (3H, s), 1.38 (3H, s), 3.25 (1H, dd, J = 9.0, 6.4 Hz), 3.41 (1H, dd, J = 9.0, 5.6 Hz), 3.68 (1H, dd, J = 8.0, 5.6 Hz), 4.01 (1H, dd, J = 8.0, 6.4 Hz), 4.13 (1H, quin).

*4-(Isopropoxymethyl)-2,2-dimethyl-1,3-dioxolane*; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 1.09 (6H, d), 1.28 (3H, s), 1.34 (3H, s), 3.31 (1H, dd, J = 9.6, 6.0 Hz), 3.44 (1H, dd, J = 9.0, 6.4 Hz), 3.53 (1H, septet), 3.65 (1H, dd, J = 8.0, 6.2 Hz), 3.98 (1H, dd, J = 8.0, 6.4 Hz), 4.16 (1H, quin).

2,2-Dimethyl-4-(phenoxymethyl)-1,3-dioxolane; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 1.38 (3H, s), 1.45 (3H, s), 3.89 (2H, ddd, J = 5.0, 9.2, 15.2 Hz), 4.01 (1H, dd, J = 9.0, 4.6 Hz), 4.12 (1H, dd, J = 14.4, 8.0 Hz), 4.45 (1H, quin), 6.91 (3H, m), 7.25 (2H, t).

### 2.3. General preparation of FeO<sub>x</sub>-pillared clay catalysts

 $FeO_x$ -pillared clays were synthesized by intercalation of iron precursors, following by calcinations at high temperature. Na-clays were dispersed in de-ionized water (10%wt) by vigorous stirring for 1 day at RT. Then 1 M FeCl<sub>3</sub>  $^{\circ}6H_2O$  was slowly added in the suspended Na-clay under stirring for 24 h at RT. The products were collected by centrifugation, and washed with de-ionized water until chloride ions were eliminated. The as-synthesized products, Fe-intercalated clays were dried followed by calcination at 300°C.

### 2.4. The characterization of $FeO_x$ -pillared clay catalysts

The prepared catalysts were characterized by XRD, surface area analyzer and NH<sub>3</sub>-TPD [18]. The XRD of raw clays and synthesized FeO<sub>x</sub>-pillared clays were measured by a Rigaku, Dmax 2200/utima+ X-ray powder diffractometer (40 Kv, 30 mA) with a monochromater and Cu K<sub> $\alpha$ </sub> radiation. The 2 $\theta$  was ranged from 2 to 30° with the scan speed of 3°/min and the scan step of 0.02°. The BET specific surface area of raw clays and synthesized FeO<sub>x</sub>-pillared clays were obtained from surface area anylasis using the Quantachrome Autosorb-1 nitrogen adsorptometer. Acidity of all catalysts was measured by temperature-programmed desorption (TPD) spectra of ammonia were recorded by Quantachrome Autosorb-1-C equipped with thermal conductivity detector.

Sample preparation for ICP: The 0.0400 g of a calcined catalyst, placed in a 100 mL Teflon beaker, was soaked with 10 mL concentrated HCl and subsequently with 10 mL of 48% HF in order to remove silica in the form of volatile  $SiF_4$  species. The mixture was heated but not boiled to dryness on a hot plate. The removal of silica was repeated three times. Then, 10 mL of 6 M HCl: 6 M HNO<sub>3</sub> mixtures in the ratio of 1:3 was added and further heated to dryness. 5 mL of 6

M HCl was added and the mixture was warmed for 5 min to complete dissolution. The solution was transferred to 50 mL polypropylene volumetric flask and made up its volume by adding deionized water. If the sample was not analyzed immediately, the solution was then transferred into a plastic bottle with a treated cap underlined with a polypropylene seal.

### 3. Results and discussion

### 3.1. Catalytic activities of FeO<sub>x</sub>-pillared clay catalysts

A mild and efficient method for the direct conversion of epoxides to acetonides using FeO<sub>x</sub>pillared clays at RT in acetone was disclosed. A synthesis of 1 from styrene oxide with acetone is displayed in Figure 1. Three FeO<sub>x</sub>-pillared clays (FeO<sub>x</sub>-pillared bentonite, FeO<sub>x</sub>-pillared taeniolite and FeO<sub>x</sub>-pillared hectorite) were screened compared with raw clays (Table 1). The quantitative yield of 1 was obtained using FeO<sub>x</sub>-pillared bentonite (10%wt) at RT for 5 min. When the reaction was performed without catalyst, no product was observed. The comparative examination on the effect of pillared clays was experimented. FeOx-pillared bentonite exhibited the highest yield. These results were well corresponded with N2 adsorption-desorption, NH3-TPD and ICP-OES experiments. The BET surface area, the amount of acidity and wt% of Fe content of all catalysts are summarized (Table 2). The acidic character of pillared clays derived from either Brønsted (weak acid sites) or Lewis site (strong acid sites). Weak acid site appeared to be associated with the coordination of water with iron in pillars, while strong acid site was attributed to Fe<sub>2</sub>O<sub>3</sub>. The nature and strength of acid sites were related to types of clays and pillared species. These results pointed out that type of clays strongly affected on the acidity of synthesized catalysts. FeO<sub>x</sub>-pillared clays gave much higher % yield than pure clays (Table 1) corresponding well with the acidity and BET surface area observed.

The developed iron oxide-pillared bentonite provided the excellent yield within 5 min revealing its remarkably efficient catalyst for the conversion of styrene oxide to its acetonide. This process was found to be comparable with that previously reported using homogeneous catalyst [19]. Nonetheless, the easy work-up protocol and reusability of exploited clay system would lead this procedure more advantage.

iron oxide-pillared bentonite RT, 5 min quantitative yield FeCl<sub>3</sub> RT, 12 min Figure 1. Synthesis of acetonides from epoxides 92% yield

7

### Table 1

Conversion of styrene oxide to 1 catalyzed by different  $FeO_x$ -pillared clays catalysts

Entry	Catalysts	Yield (%)
1	FeO <sub>x</sub> -pillared bentonite	quant <sup>a</sup>
2	FeO <sub>x</sub> -pillared hectorite	83 <sup>b</sup>
3	FeO <sub>x</sub> -pillared taeniolite	77 <sup>b</sup>
4	bentonite	trace <sup>c</sup>
5	hectorite	trace <sup>c</sup>
6	taeniolite	trace <sup>c</sup>
7	none	$0^{c}$

Reaction conditions: styrene oxide (1 mmol), acetone (3 mL), catalyst 10 wt% to styrene

oxide at 30°C for <sup>a</sup>5 min; <sup>b</sup>30 min; <sup>c</sup>24 h.

### Table 2

### The amount of acidity and surface area

Entry	Catalysts	Wt% Fe	Surface	acidity (mmol/g)		Total amount
		content	area	weak	strong	of acidity
	2		(m <sup>2</sup> /g)	acid sites	acid sites	(mmol/g)
1	FeO <sub>x</sub> -pillared bentonite	10.14	155.52	0.62	3.11	3.73
2	FeO <sub>x</sub> -pillared hectorite	9.54	122.30	0.73	1.43	2.16
3	FeO <sub>x</sub> -pillared taeniolite	4.22	20.33	0.61	0.91	1.52
4	bentonite	3.49	65.05	0.14	1.12	1.26
5	hectorite	0.95	57.11	0.04	0.51	0.55
6	taeniolite	0.35	3.53	0.43	-	0.43

When the amount of catalyst increased from 2 to 10 wt%, the yield of product was increased. Almost quantitative yield of product was achieved when 10 wt% of iron oxide-pillared bentonite to styrene oxide was employed. The lower amount of catalyst was used, the lower yield was detected. Using 30 wt% of catalyst, the yield of product was comparable with that using 10 wt% of catalyst. Thus, the suitable amount of iron oxide-pillared bentonite was 10 wt% (Table 3).

### Table 3

Entry	Fe-pillared bentonite	% Yield	% Styrene oxide
	(wt%)		(recovered)
1	2	83	17
2	5	91	8
3	10	94	0
4	30	95	0

Effect of the amount of iron oxide-pillared bentonite

Reaction conditions: styrene oxide 1 mmol, acetone in excess (3 mL), iron oxide-pillared

bentonite varied at RT (30°C) for 5 min.

High to excellent yields of acetonides were obtained in all cases (Table 4). Styrene oxide was stirred in the presence of 10% wt FeO<sub>x</sub>-pillared bentonite and acetone at RT for 5 min to produce acetonide in quantitative yield. For cyclic epoxide (cyclohexene oxide) and terminal aliphatic epoxide (1-dodecene oxide), 94 and 92% yields were achieved after 2 h (entries 2-3,

respectively). The FeO<sub>x</sub>-pillared bentonite could also catalyze the efficient reaction of the epoxides with ethereal linkage on the epoxide ring such as butyl glycidyl ether, *tert*-butyl glycidyl ether, 2,3-epoxypropyl *iso*propyl ether and 2,3-epoxypropyl phenyl ether with acetone to give their corresponding 1,3-dioxalones in high yields (entries 4-7). All products were identified by GC and <sup>1</sup>H NMR compared with authentic samples.

### Table 4

Conversion of epoxides to acetonides catalyzed by FeOx-pillared bentonite

Entry	Epoxides	Acetonides	Yield (%)
1	<u> </u>		quant
2	0		94
3	<u></u>		92
4	~ <u>Q</u>		88
5	$\rightarrow^{0}$	$\rightarrow 0 \rightarrow 0$	63
6	Yo-Q	$\gamma_{0}$	90
7			92

*Reaction conditions:* epoxide (1 mmol), acetone (3 mL), 10 wt% FeO<sub>x</sub>-pillared bentonite to epoxide at  $60^{\circ}$ C for 2 h.

Table 5 reveals the scope of the reaction with other carbonyl compounds such as acetophenone, cyclohexanone and benzaldehyde. The reactions could proceed well with these selected carbonyl compounds (entries 2-4). Without FeO<sub>x</sub>-pillared bentonite, no reaction occurred.

### Table 5

	F	Reaction of	f carbon	yl compounds	with styrene	oxide catalyzed	l by Fe	O <sub>x</sub> -pillared	bentonite
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Entry	Carbonyl compounds	1,3-Dioxolane	Yield (%)
1	H <sub>3</sub> C CH <sub>3</sub>	$H_3C$ $O$ $Ph$ $H_3C$ $O$	>99
2	Ph CH <sub>3</sub>	$Ph \xrightarrow{O} Ph$ $H_3C O$	83
3		C C Ph	77
4	Ph H	Ph O Ph H O	85

*Reaction conditions*: carbonyl compound (2 mmol), styrene oxide (1 mmol),  $CH_2Cl_2$  (3 mL), 10 wt% FeO<sub>x</sub>-pillared bentonite to styrene oxide at refluxing  $CH_2Cl_2$  for 2 h.

The proposed mechanism is presented (Scheme 1). The Lewis acid-mediated reaction of an epoxide with a carbonyl compound was reported to proceed *via* the coordination of a metal oxide

12

of the catalyst with the epoxide oxygen [13], followed by nucleophilic ring-opening of the epoxide by the carbonyl oxygen and finally 1,3-dioxolane ring formation.



Scheme 1. The Proposed Mechanism

### 3.2 Catalytic activities of reused of FeO<sub>x</sub>-pillared bentonite

 $FeO_x$ -pillared bentonite could be easily recovered, simply by filtering the catalyst, washing with EtOAc, drying at 100°C in oven for 2 h and calcination. The recovered  $FeO_x$ -pillared bentonite was used in the next run. The results are summarized (Table 6).

### Table 6

Activity of regenerated  $FeO_x$ -pillared bentonite by calcination at 300°C for the conversion of styrene oxide to acetonide

Entry	Times	% Yield of 1
1	1	99
2	2	99
3	3	88
4	4	76
5	5	74

*Reaction conditions*: styrene oxide (1 mmol), acetone (3 mL), catalyst 10 wt% to styrene oxide at 30°C for 5 min.

In order to investigate for the reusability of the prepared  $FeO_x$ -pillared bentonite, the reused catalyst was employed in the next run. After reusing twice, 88% of acetonide was detected. The regenerated  $FeO_x$ -pillared bentonite exhibited lower activity than the original catalyst. This may derive from the collapse of  $d_{001}$  spacing owing to the calcination process. The analysis of the BET specific surface areas of the regenerated  $FeO_x$ -pillared bentonite exhibited the lower value than the original catalyst: 155.52 and 114.04 m<sup>2</sup>/g, respectively. There are two reasons: (i) the recalcination process at 300°C was not enough to remove all organic residues that blocked the pores of the catalyst, and (ii) the collapse of the d-spacing also caused the smaller pores. However, re-calcination process at higher 300°C may cause the pillared structure change or collapse.

The attempt to utilize this catalyst was carried out. The new protocol was accomplished by simply filtering  $FeO_x$ -pillared bentonite from the reaction mixture, drying at 100°C in oven for 2 h and not calcinating. The process opened the good opportunity to exploit this catalyst at least five times without appreciable loss of activity as summarized (Table 7).

### Table 7

Activity of regenerated FeO<sub>x</sub>-pillared bentonite by not calcination for the conversion of styrene oxide to acetonide

Entry	Times	% Yield of 1
1	1	99
2	2	93
3	3	96
4	4	92
5	5	98

*Reaction conditions*: styrene oxide (1 mmol), acetone (3 mL), catalyst 10 wt% to styrene oxide at 30°C for 5 min.

### 4. Conclusion

 $FeO_x$ -pillared bentonite as a highly effective catalyst for the conversion of epoxides to acetonides was described. The reaction furnished 2,2-dimethyl-4-phenyl-1,3-dioxolane in quantitative yield with short reaction time under mild conditions. The reaction worked smoothly for a wide range of epoxides and carbonyl compounds. The calcination step was not requited for the reuse of catalyst. Up to five times of this catalytic system could be achieved without appreciable loss of activity.

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### Highlights

•FeO<sub>x</sub>-pillared bentonite efficiently catalyzed the conversion of epoxides.

•The reaction furnished acetonides in excellent yields under mild reaction conditions.

•The quantitative yields was obtained using 10wt% of catalysts at RT for 5 min.

•Up to five times of this catalytic system could be achieved without loss of activity.

