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Appendices

APPENDIX A

Gas chromatography analyzer was used to determine products of cyclohexane oxidation. Cyclohexanone and cyclohexanol products were identified using standard addition method.

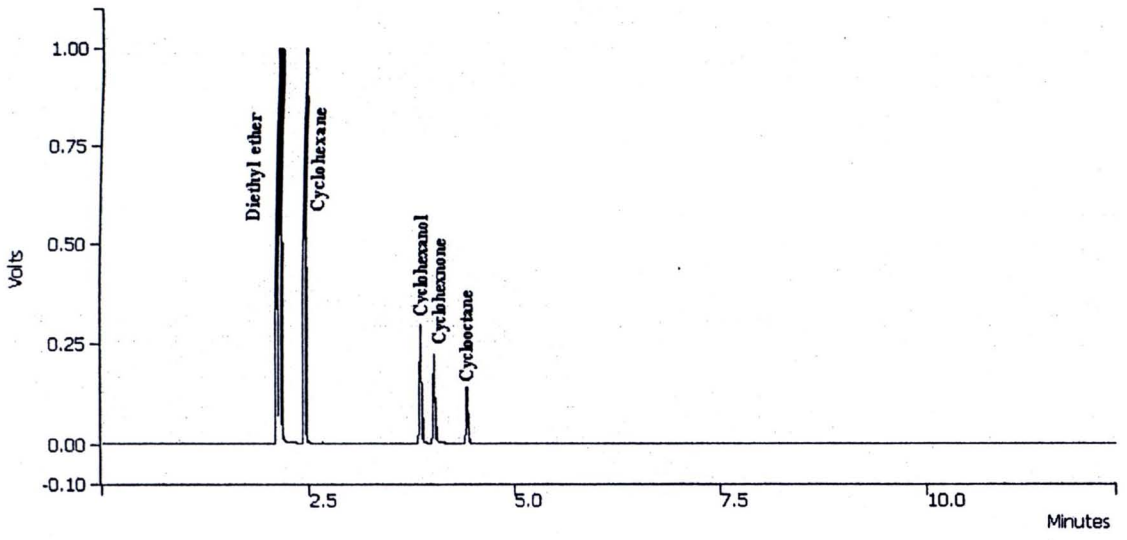


Figure A-1 A gas chromatogram of liquid products from oxidation of cyclohexane.

Calculation of the correction factor

The correction factor was calculated based upon the results obtained from gas chromatographic analysis (see also the experimental section). Cyclooctane was used as and internal standard.

Example:

A:exact amount of desired product prepared (mmol)

B: total volume of the reaction (ml)

C: peak area of the desired product

D: peak area of the internal standard

E: exact amount of substrate (mmol)

F: exact amount of internal standard was added (mmol)

The calculation of the correction factor can be described as follows:

The amount of the product from the reaction mixture

= (F x C/D) = G

The amount of the product in B ml (tatal volume of the reaction)

= G x B = H

Thus, the correction factor of the product can be calculated as:

= A/H = I

The % product can be calculated as:

%Yield of product = (H x I / E) x 100

The correction factors of chemicals are listed as follows:

Cyclohexane = 3.2

Cyclohexanone = 5.4

Cyclohexanol = 4.2

Calculation of %selectivity of cyclohexanone and cyclohexanol

Using peak areas obtained from GC analysis

% Selectivity of cyclohexanone

= (%yield of cyclohexane/%yield of product) x 100

%Selectivity of cyclohexanol

= (%yield of cyclohexanol/%yield of product) x 100

Determination of cyclohexyl hydroperoxide

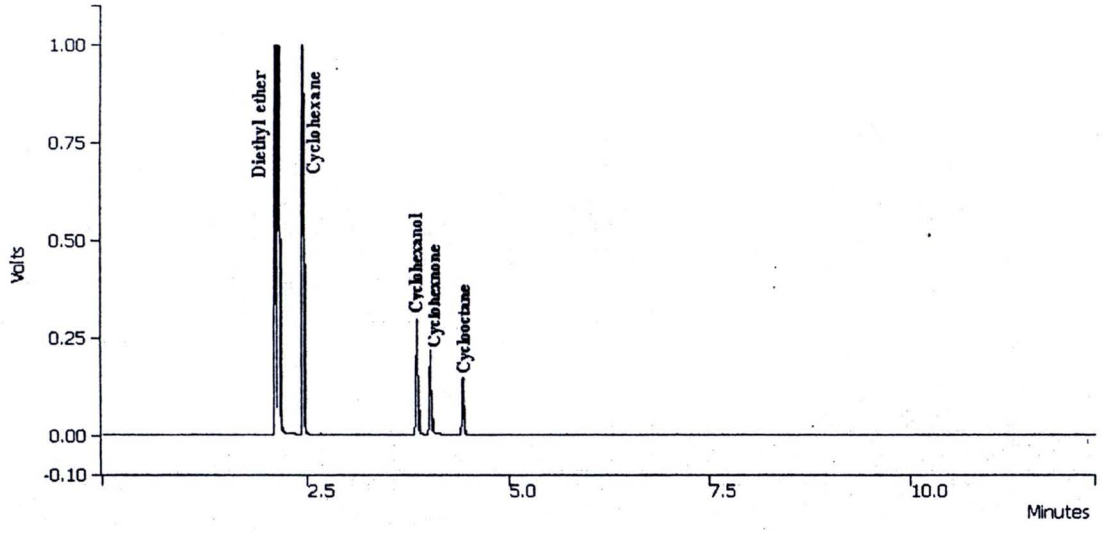


Figure A-2 GC diagram of cyclohexane oxidation and their oxygenated products using FePOM-MCM-41

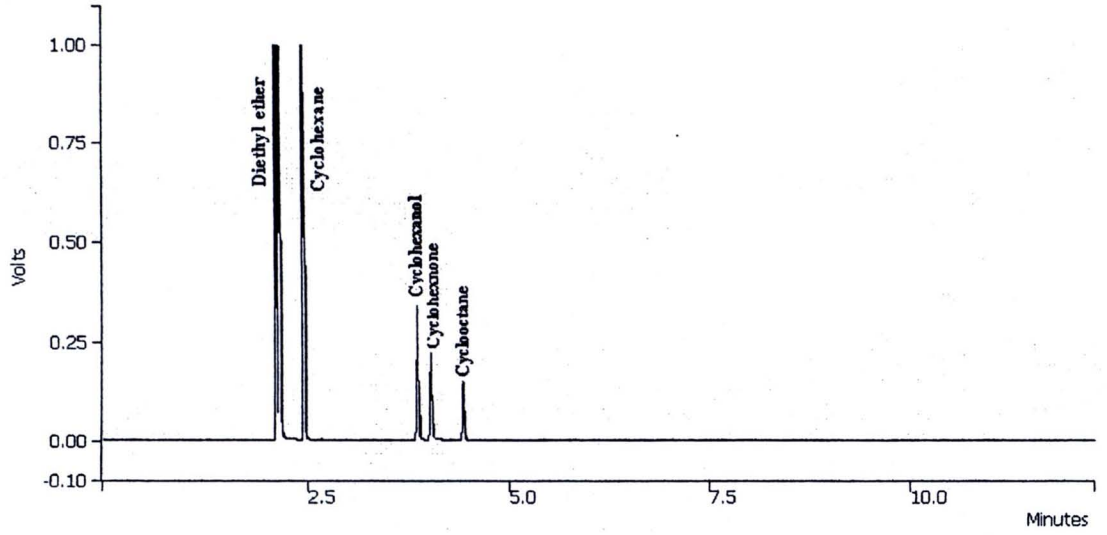


Figure A-3 GC diagram of cyclohexane oxidation and their oxygenated products using FePOM-MCM-41 after adding PPh_3.

APPENDIX B

Adsorption and desorption isotherms of 20%CoPOM/MCM41, 20%FePOM/MCM-41 and 20%CuPOM/MCM-41.

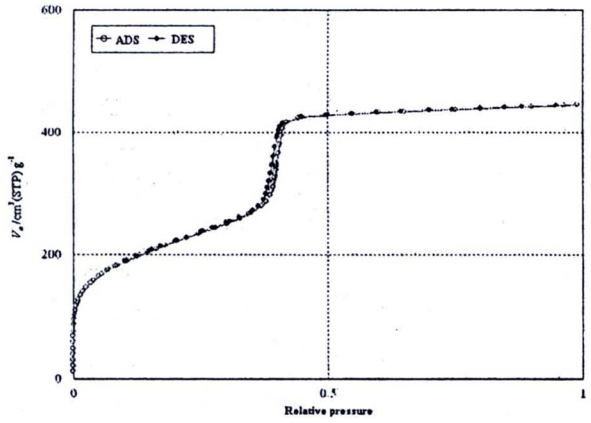


Figure B-1 Adsorption and desorption isotherm of the 20%CoPOM/MCM-41.

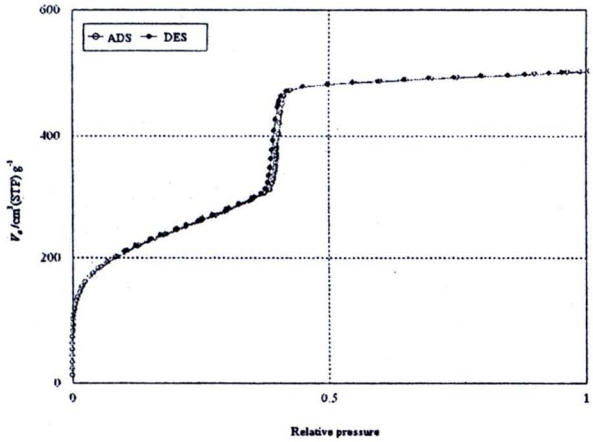


Figure B-2 Adsorption and desorption isotherm of the 20%FePOMMCM-41

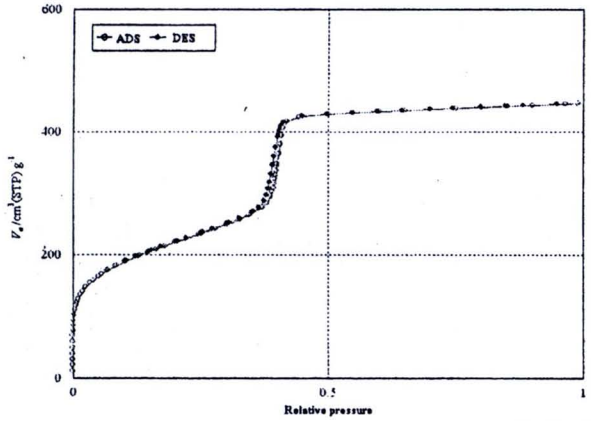


Figure B-3 Adsorption and desorption isotherm of the 20%CuPOMMCM-41

APPENDIX C

Thermo gravimetry analysis (TGA)

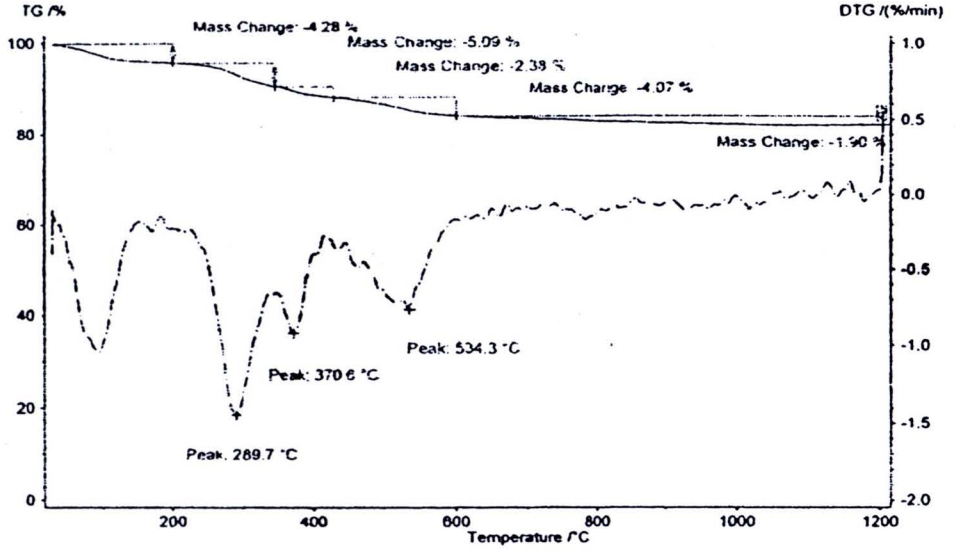
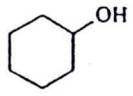
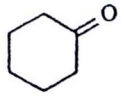
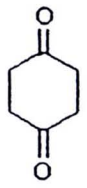
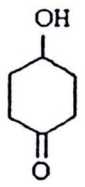


Figure C-1 TGA profile of the 20%CoPOMMCM-41

APPENDIX D
GC-MS

Table D-1 GC-MS of cyclohexane oxidation reaction

Condition: 20%CoPOM-MCM-41 5.1wt% , cyclohexane 92 mmol(10ml), H_2O_2/cyclohexane molar ratio = 1, 100°C, 8 h.

Formula	Structure	% Selectivity
cyclohexanol		35.71
cyclohexanone		52.55
1,4 cyclohexadione		4.08
4-hydroxy cyclohexanone		1.53
Hexanoic acid		6.12

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Operator   : SK
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Instrument :   Instrumen
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Misc Info  : CYCLOHEXANE EXTRACT
Vial Number: 1
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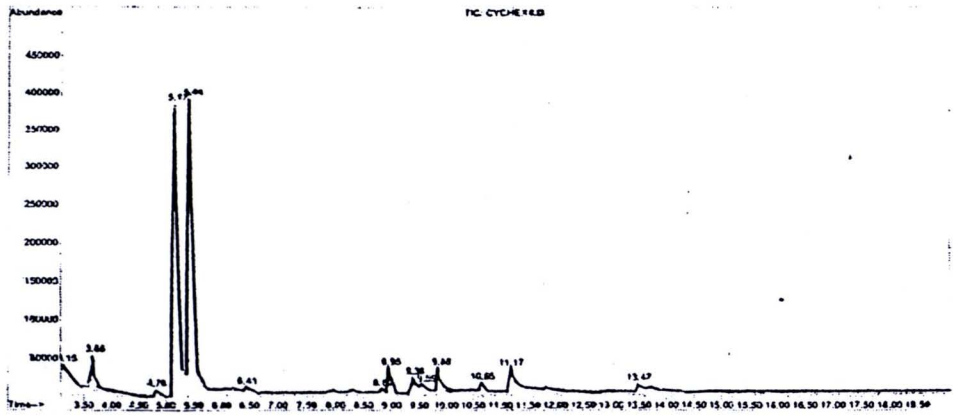

Area Percent Report

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Sample    : CYCLOHEXANE EX                         Inst    : Instrumen
Misc      : CYCLOHEXANE EXTRACT                    Multiplr: 1.00
                                                   Sample Amount: 0.00

MS Integration Params: autoint1.e

Method    : C:\MSDCHEM\1\METHODS\CIF065-2553.M (Chemstation Integrator)
Title     :

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	3.149	5	24	138	BV	15534	1253585	5.74%	2.291%
2	3.658	168	207	324	VB 4	46793	2198711	10.07%	4.019%
3	4.777	539	609	655	BB 7	6269	284044	1.30%	0.519%
4	5.166	677	749	825	BV 3	379542	21839725	100.00%	39.918%
5	5.436	825	846	959	VV	383216	20803407	95.25%	38.024%
6	6.415	1163	1198	1253	BB 5	5380	324566	1.49%	0.593%
7	8.797	2031	2054	2092	BV 5	5716	243491	1.11%	0.445%
8	8.945	2092	2107	2158	VV 4	33176	1021706	4.68%	1.867%
9	9.376	2158	2262	2321	PV 4	20173	1728770	7.92%	3.160%
10	9.587	2321	2338	2414	VV 4	11084	962053	4.41%	1.758%
11	9.863	2414	2437	2516	VB 3	31930	1224678	5.61%	2.238%
12	10.650	2691	2720	2780	BV 3	12749	507051	2.32%	0.927%
13	11.173	2857	2908	3014	BB 4	28900	1922590	8.80%	3.514%
14	13.471	3691	3734	3791	BB 6	8737	396608	1.82%	0.725%

Sum of corrected areas: 54710985

Figure D-1 GC diagram of cyclohexane oxidation and their oxygenated products using 20%CoPOM/MCM-41 at 100°C.

APPENDIX E

X-ray fluorescence spectrometry (XRF)

Table E-1 X-ray fluorescence spectrometry of 20%CoPOM/MCM-41

Sample	(% by wt.)				
	SiO_2	P_2O_5	Cl	CoO	WO_2
20%CoPOM/MCM-41	84.20	0.33	1.24	0.38	13.85

Calculation of %POM on MCM-41

From Table E-1 CoO = 0.38% so Co = 0.29%

From theory 20%CoPOM/MCM-41 have Co = 0.32%

If 20%CoPOM/MCM-41 has Co 0.32%

So X%CoPOM/MCM-41 has Co 0.29%

X= (0.29 x 20)/0.32

X= 18.12%

So the amount of CoPOM on the supported MCM-41 calculated by XRF technique was 18.12 % based on MCM-41.

VITAE

Mr. Jiraroj Jatupisarnpong was born on Mach 16, 1986 in Maehongson Thailand. He received a Bachelor Degree of Engineering, major in Petrochemicals and Polymeric Materials from Silpakorn University in 2008. Since 2008 he has been a graduate student in the Program of Petrochemistry and Polymer Science, Faculty of Science, Chulalongkorn University and graduated in 2010.

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1. ISAMAP at Chulalongkorn University 17-18 September 2009
2. PACCON 2010 at Ubonratchathani 21-23 January 2010.

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