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APPENDICES

APPENDIX A

Calculation of Catalyst Preparation

The calculation of y wt% Palladium over MCM-41

The palladium compound used is Palladium acetate ($\text{Pd}(\text{OAc})_2$), which has a molecular weight of $224.49 \text{ g mol}^{-1}$ and the molecular weight of palladium is 106.4 g mol^{-1} .

In basis of 100 g catalyst, the catalyst is composed of

$$100 \text{ g of a wt\% Pd/MCM-41 catalyst has palladium} = y \text{ g}$$

$$100 \text{ g of a wt\% Pd/MCM-41 catalyst has MCM-41} = 100-y \text{ g}$$

$$\begin{aligned} \text{Based on 1 g of MCM-41, requires palladium} &= \frac{y}{(100-y)} \times 1 \text{ g} \\ &= \frac{y}{(100-y)} \text{ g} \end{aligned}$$

Assumed $\frac{y}{(100-y)}$ as z,

$$\text{Pd } 106.40 \text{ g, containing in Pd(OAc)}_2 = 224.49 \text{ g}$$

$$\text{Pd } z \text{ g, requires Pd(OAc)}_2 = \frac{z \times 224.49}{106.40} \text{ g}$$

Table A-1 Amount of palladium used per gram of MCM-41 support.

Catalysts	Amount Pd(OAc)₂ (g)
0.5 wt% Pd/MCM-41	0.0106
1.0 wt% Pd/MCM-41	0.0213
3.0 wt% Pd/MCM-41	0.0652

APPENDIX B

Calculation of Metal Active Sites

The example of calculation shown below is for 0.5wt% Pd/MCM-41 catalyst. The calculation of metal active sites of the catalyst by hydrogen chemisorption at room temperature has the procedure as follows:

Let the weight of catalyst used	=	X	g
The amount of palladium in catalyst	=	$X \times \left[\frac{0.5}{100} \right]$	mol
	=	106.42	
	=	P	mol
Average peak area of H ₂ after adsorption	=	A	unit
Peak area of H ₂ after the first pulse	=	B	unit
Total area of H ₂ after adsorption	=	A-B	unit
Volume of H ₂ adsorbed on catalyst each pulse	=	0.6859	mL
H ₂ concentration input each pulse	=	9.65	%
Reaction temperature	=	D	K
The amount of H ₂ input each pulse	=	$\frac{[9.65 \times 0.6859]}{100}$	mL
	=	0.0661	mL
H ₂ concentration at STP	=	$\frac{0.0661}{22.4 \times 1000}$	mol STP
	=	2.95×10^{-6}	mol STP
The amount of H ₂ input each pulse at D K	=	$\frac{2.95 \times 10^{-6} \times 273}{D}$	mol

$$\begin{aligned} &= Y \quad \text{mol} \\ \text{The amount of hydrogen uptake} &= \frac{Y \times [A - B]}{A} \quad \text{mol} \\ &= Z \quad \text{mol} \\ \text{Mole of palladium site over the support} &= Z \times 2 \quad \text{mol atom} \\ &= S \quad \text{mol atom} \\ \text{\% palladium dispersion} &= \frac{S}{P} \times 100 \quad \text{\%} \end{aligned}$$

APPENDIX C

BET Surface Area Determination

Brunauer, Emmett and Teller (BET) developed several models for gas adsorption on solids which have become the effective standard for surface area measurements. The models are valid for multiple layers on homogeneous surfaces [Brunauer, 1940]. This procedure for multipoint adsorption has been standardized by the American Society of Testing Materials (ASTM): Catalysts-D32, (ASTM: D3663-84, 1988) or Nitrogen adsorption at 77 K. The sample is first evacuated while heating to about 400° C, and then cooled to about 77 K with a container of liquid nitrogen. The partial pressure of N₂ above the sample is gradually increased and the amount of N₂ adsorbed at each pressure after allowing equilibrium to be reached is noted. Each adsorbed molecule occupies an area of the surface comparable to its cross-sectional area (0.162 nm²). By measuring the number of N₂ molecules adsorbed at monolayer coverage, the internal surface area can be calculated. According to this standard procedure, the sorption isotherms can be plotted similar to that of silica which is shown in Figure C-1.

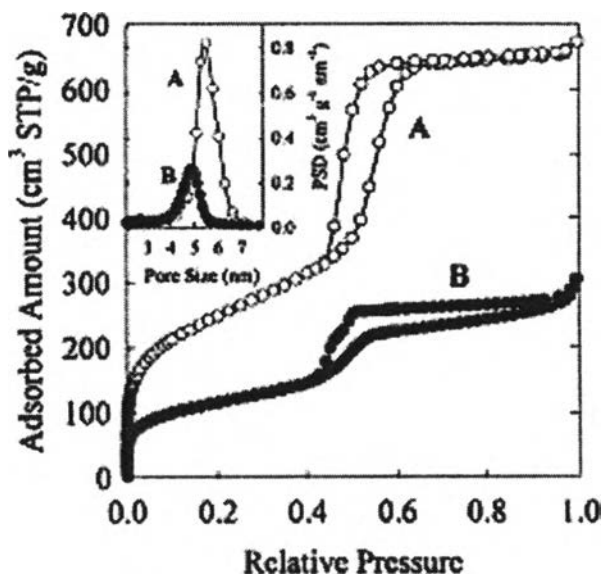


Figure C-1 Nitrogen adsorption isotherms at 77 K and pore size distributions for two MCM-41 materials (a) ordered MCM-41 and (b) disordered MCM-41 [Kruk and Jaroniec, 1999].

The BET equation describes the relationship between the volume adsorbed at a given partial pressure and the volume adsorbed at monolayer coverage is shown as the following.

$$\frac{x}{v(1-x)} = \frac{1}{cV_m} + \frac{(c-1)x}{cV_m} \quad \dots\dots\dots(\text{C-1})$$

Where $x = P/P_o$; P is the partial pressure of N_2 ; P_o is the saturation pressure at the experimental temperature; V is the volume adsorbed at P ; V_m is the volume adsorbed at monolayer coverage and c is a constant.

The surface area of porous solid can be determined by using N_2 adsorption data. The BET plot between $x/[v(1-x)]$ versus x following equation C-1 provides a straight

line in order to obtain a slope of $(c - 1)/cV_m$ and intercept $1/cV_m \cdot V_m$, the volume adsorbed at monolayer coverage can be calculated from Equation C-2.

$$V_m = \frac{1}{(c \times \text{intercept})} \dots\dots\dots(C-2)$$

The BET surface area is obtained using the widely accepted assumption that each N₂ molecule occupies an area of 0.162 nm². This following equation is commonly used to calculate the surface area of porous solid in m² g⁻¹.

$$S_A (m^2 g^{-1}) = \left[\frac{V_m \times 6.02 \times 10^{23} \text{ molecules mol}^{-1} \times 16.2 \times 10^{-20} m^2 \text{ per molecule}}{22400 cm^3 mol^{-1}} \right] \times g^{-1} \text{ sample}$$

BIOGRAPHY

Miss Siriluk Chiarakorn, was born on November 25, 1976 in Bangkok, Thailand. She finished her secondary school from Suksa Nari school, Bangkok. After that, she joined and graduated in major of Polymer Science, Faculty of Science at Chulalongkorn University and graduated in 1997. Afterwards, she enrolled in Master's degree in Applied Polymer Science and Textile Technology at Chulalongkorn University and graduated in 2000. In the same year, she started her Ph.D. degree in International Programs in Environmental Management, Chulalongkorn University and completed the program in May 2004.

During her Ph.D. study, she was accepted as an oral presentation in the 9th APPChE Congress and CHEMECA 2002, New Zealand and won the first prize honor in the 2nd Thailand Materials Science and Technology Conference, Bangkok in the same year. Recently, she also attended the 3rd National Environmental Conference, Songkhla, 2003.

She also had an opportunity to carry out her research at Washington State University from May to August, 2003 in the collaboration with Department of Chemical Engineering and Center for Multiphase Environmental Research. Before she accomplished her Ph.D. degree, her research work was published in the international journal; Korean Journal of Chemical Engineering, Volume 20, Issue 5 (2003).

