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APPENDICES

APPENDIX A

CALCULATION OF CATALYST PREPARATION

Calculation of the second element modified metal oxide

In this study, silica-modified metal oxide were prepared in each organic solvent have different Si/M molar ratio, M represented as Ti, Fe and Zn, representively. Si/M = 0, 0.005, 0.08

1. Titanium (IV) tert-butoxide (TNB, $Ti[O(CH_2)_3CH_3]_4$) 97% has a molecular weight of 340.36 g/mol
2. Iron (III) acetylacetonate ($Fe[CH_3COCH{=}C(O\text{-})CH_3]_3$) 97% has a molecular weight of 353.18 g/mol
3. Zinc (II) acetylacetonate ($Zn[CH_3COCH{=}C(O\text{-})CH_3]_2$ 95% has a molecular weight of 263.59 g/mol
4. Tetraethyl orthosilicate (TEOS, $Si(OC_2H_5)_4$) 98% has a molecular weight of 208.33 g/mol

Example : Calculation of preparation of silica-modified titania with Si/Ti = 0.08 are as follows :

Fifteen gram of TNB was used for the preparation of Si/Ti = 0.08

TNB 15 g was consisted of pure TNB equal to : $(15/340.36) \times 0.97 = 0.0427$ mol

TNB 1 mol has Ti 1 mol, so that, Ti has 0.0427 mol

To get the product with molar ratio Si/Ti of 0.08 ;

Silicon = 0.08×0.0427 mol = 3.416×10^{-3} mol

Tetraethyl orthosilicate required is equal to : $208.33 \times 3.416 \times 10^{-3} = 0.7117$ g

APPENDIX B

CALCULATION OF THE CRYSTALLITE SIZE

Calculation of the crystallite size by Debye-Scherrer equation

The crystallite size was calculated from the half-height width of the diffraction peak of XRD pattern using the Debye-Scherrer equation.

From Scherrer equation:

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad \text{(B.1)}$$

where D = Crystallite size, Å

K = Crystallite-shape factor = 0.9

λ = X-ray wavelength, 1.5418 Å for CuKα

θ = Observed peak angle, degree

β = X-ray diffraction broadening, radian

The X-ray diffraction broadening (β) is the pure width of a powder diffraction free of all broadening due to the experimental equipment. Standard α-alumina is used to observe the instrumental broadening since its crystallite size is larger than 2000 Å. The X-ray diffraction broadening (β) can be obtained by using Warren's formula.

From Warren's formula:

$$\beta^2 = B_M^2 - B_S^2 \qquad \text{(B.2)}$$

$$\beta = \sqrt{B_M^2 - B_S^2}$$

Where B_M = The measured peak width in radians at half peak height.

B_S = The corresponding width of a standard material.

Example: Calculation of the crystallite size of titania

The half-height width of 101 diffraction peak = 0.93125^{o}

= 0.01625 radian

The corresponding half-height width of peak of α-alumina = 0.004 radian

The pure width = $\sqrt{B_M^2 - B_S^2}$

= $\sqrt{0.01625^2 - 0.004^2}$

= 0.01577 radian

B = 0.01577radian

2θ = 25.56^{o}

θ = 12.78^{o}

λ = 1.5418 Å

The crystallite size = $\dfrac{0.9x1.5418}{0.01577\cos 12.78}$ = 90.15Å

= 9 nm

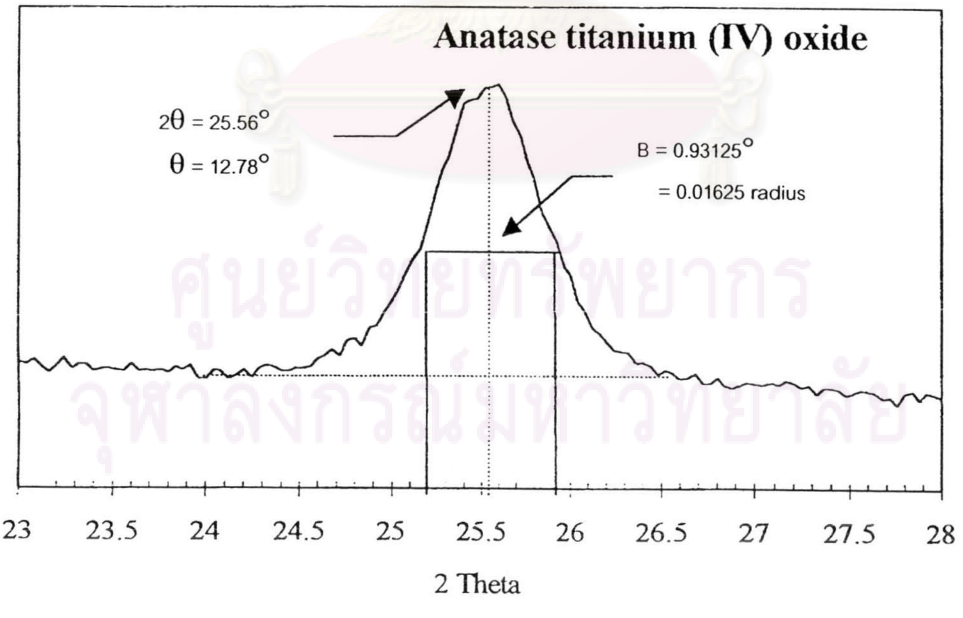

Figure B.1 The 101 diffraction peak of titania for calculation of the crystallite size

APPENDIX C

CALCULATION OF SPECIFIC SURFACE AREA

The method for surface area calculation is using the crystallite size from XRD line broadening and density of each metal oxide.

$$\text{Surface area} = \frac{6}{\rho d}$$

Where, ρ = density of metal oxide

d = crystallite size calculated from Scherrer equation

Table C.1 Density of transition metal oxide

Metal oxides	Density (g/cm^3)
TiO_2	4.2
Fe_2O_3	5.2
ZnO	5.6

Example Specific surface area calculation for titanium oxide synthesized at 300°C

Crystallite size = 12.94 nm

Density of titanium oxide = 4.2 g/cm^3

$$\text{Surface area} = \frac{6}{4.2\times10^{6}\times12.94\times10^{-9}}$$

$$= 110.40\ m^2/g$$

VITA

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