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APPENDICES

APPENDIX A
CALCULATION OF CATALYST PREPARATION

Calculation of the preparation of the silica modified titanium (IV) oxide

In this study, silica modified titanium (IV) oxide was prepared in each organic solvent have 6 different Si/Ti atomic ratios, Si/Ti = 0, 0.05, 0.1, 0.2, 0.3 and 0.5. Titanium (IV) tetra -tert –butoxide and tetraethyl orthosilicate is used as the reactant.

1.Titanium (IV) tetra –tert –butoxide ($Ti[O(CH_2)_3CH_3]_4$, TTB) has M.W. of 340.36 g
Titanium (Ti) has M.W. of 47.88 g

2.Tetraethyl orthosilicate ($Si(OC_2H_5)_4$, TEOS) has M.W. of 208.33 g
Silicon (Si) has M.W. of 28.085 g

Example: Calculation of preparation of titanium (IV) oxide with Si/Ti =0.05 is shown as follow:

TTB 25 g were used for preparation of all Si/Ti atomic ratio

TTB 25 g were consist of titanium equal:

$$\text{Titanium} = (47.88 / 340.36) \times 25 \text{ g} = 3.517 \text{ g} = 0.073454 \text{ mole}$$

So, Si/Ti = 0.05 has silicon equal:

$$\text{Silicon} = 0.05 \times 0.73454 = 0.003673 \text{ mole} = 0.10315 \text{ g}$$

From silicon = 0.10315g, TEOS is used equal:

$$(28.0855 / 208.33) \times \text{weight of TEOS} = 0.10315 \text{ g}$$

So, weight of TEOS = $(0.10315 / 28.0855) \times 28.33 = 0.7651$ g

The result of calculation of all Si/Ti atomic ratios is shown in Table A.1

Table A.1 Reagents used for the synthesis of silica modified titanium (IV) oxide

Si/Ti atomic ratio	Titanium (IV) tetra –tert – butoxide	Tetraethyl orthosilicate
0	25 g	0 g
0.05	25 g	0.7651 g
0.1	25 g	1.5303 g
0.2	25 g	3.061 g
0.3	25 g	4.591 g
0.5	25 g	7.652 g

$$\frac{P_bV}{273} = \frac{P_tV}{T} \qquad \text{(B.3)}$$

Where: V = constant volume

P_b = pressure at 0 °C

P_t = pressure at t °C

T = 273.15 + t, K

P_b = $(273.15 / T) \times P_t$ = 1 atm

Partial pressure

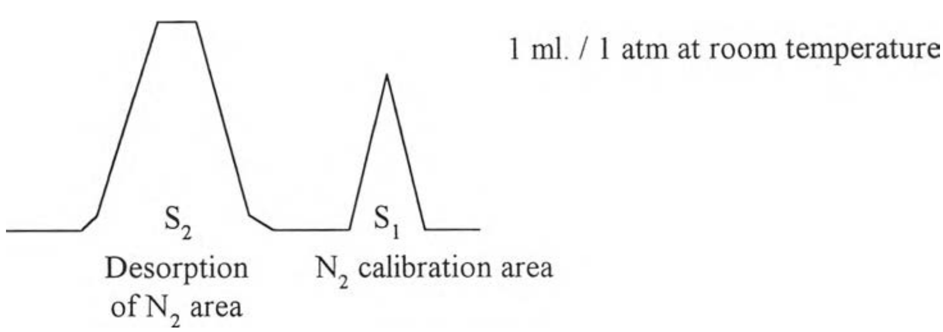
$$P = \frac{[\text{Flow of }(He+N_2) - \text{Flow of He}] \times P_b}{\text{Flow of }(He+N_2)} \qquad \text{(B.4)}$$

$= 0.3$ atm

N_2 saturated vapor pressure, P_o = 1.1 atm = 836 mm.Hg

$X = P / P_o = P / 1.1$

How to measure V



$$V = \frac{S_2}{S_1} \times \frac{1}{W} \times \frac{273.15}{T} \ \text{ml. / g of catalyst} \qquad \text{(B.5)}$$

Where: W = weight of sample

APPENDIX B

CALCULATION OF SPECIFIC SURFACE AREA

Calculation of BET surface area

From Brunauer-Emmett-Teller (BET) equation:

$$\frac{X}{V(1-X)} = \frac{1}{VmC} + \frac{(C-1)X}{VmC} \qquad \text{(B.1)}$$

Where: X = relative partial pressure of N_2, P/P_o

P = saturated vapor pressure of N_2 (or adsorbed gas) at the experimental temperature

P = equilibrium vapor pressure of N_2

V = volume of gas adsorbed at pressure P, ml. at the NTP / g of sample

V_m = volume of gas adsorbed at monolayer, ml. at the NTP / g of sample

C = $\text{Exp}\ (E_1 - E_2 / RT)$

Where: E_1 = heat of adsorption at the first layer

E_2 = heat of condensation of adsorbed gas on all other layers

Assume $C \rightarrow \infty$, then

$$\frac{X}{V(1-X)} = \frac{1(X)}{VmC} \qquad \text{(B.2)}$$

Let: $Vm = Vm'$

Vm = volume of gas adsorbed to form the N_2 complete monolayer

V = volume of gas adsorbed measured by GC

X = P / P_0

$S_b = 4.343\ V_m'$

So,

$$S_b = \frac{S_2}{S_1} \times \frac{1}{W} \times 0.7272 \times 4.343 \quad m^2\,g^{-1} \qquad (B.8)$$

Data from experiment show below:

$S_1 = 0.028$ g

$S_2 = 0.2389$ g

W = 0.303 g

So that, $S_b = 89.6\ m^2\ g^{-1}$

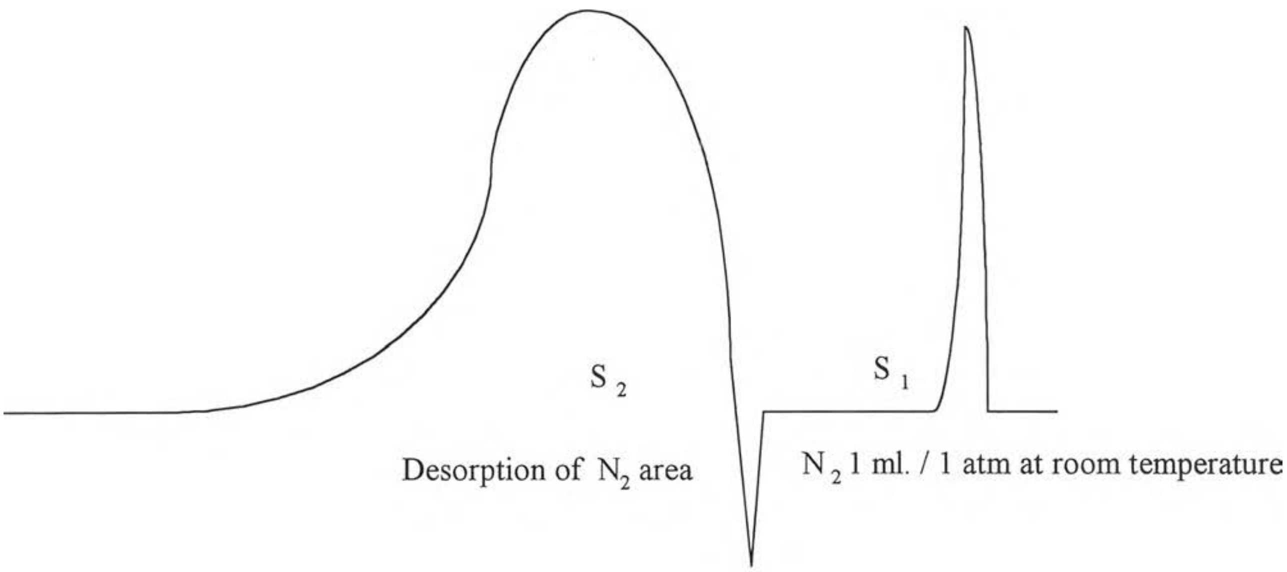
$$V_m{}' = \frac{V \times [1-[\text{Flow of (He+N}_2\text{) - Flow of He}] / 1.1]}{\text{Flow of (He+N}_2\text{)}} \quad \text{(B.6)}$$

Where: S = surface area from literature of N_2

= 4.373 m^2 / ml. of N_2

So that, $S_b = 4.373 \times Vm' \; m^2$ / g catalyst (B.7)

Example: The BET surface area of pure anatase titanium (IV) oxide prepared in 1,4 butanediol is calculated as follow:



From equation B.5 and B.6

$$V_m{}' = \frac{S_2}{S_1} \times \frac{1}{W} \times \frac{273.15}{T} \times \frac{[1-[\text{Flow of (He+N}_2\text{) - Flow of He}] / 1.1]}{\text{Flow of (He+N}_2\text{)}}$$

When T = 273.15 and

$$\frac{[1-[\text{Flow of (He+N}_2\text{) - Flow of He}] / 1.1]}{\text{Flow of (He+N}_2\text{)}} = 0.7272$$

APPEENDIX C
CALCULATION OF CRYSTALLITE SIZE

Calculation of crystallite size by Sherrer equation

Crystallite size was calculated from the half-height width of the 101 diffraction peak of anatase and 110 diffraction peak of rutile using the Sherrer equation. The value of the shape factor, K was taken to be 0.9 and KCl was used to be internal standard.

From Sherrer equation:

$$t = \frac{0.9\lambda}{B \cos \theta_B} \qquad (C.1)$$

Where: t = crystallite size

K = shape factor = 0.9

λ = X-ray wavelength, Cu Kα : λ = 1.5418 A°

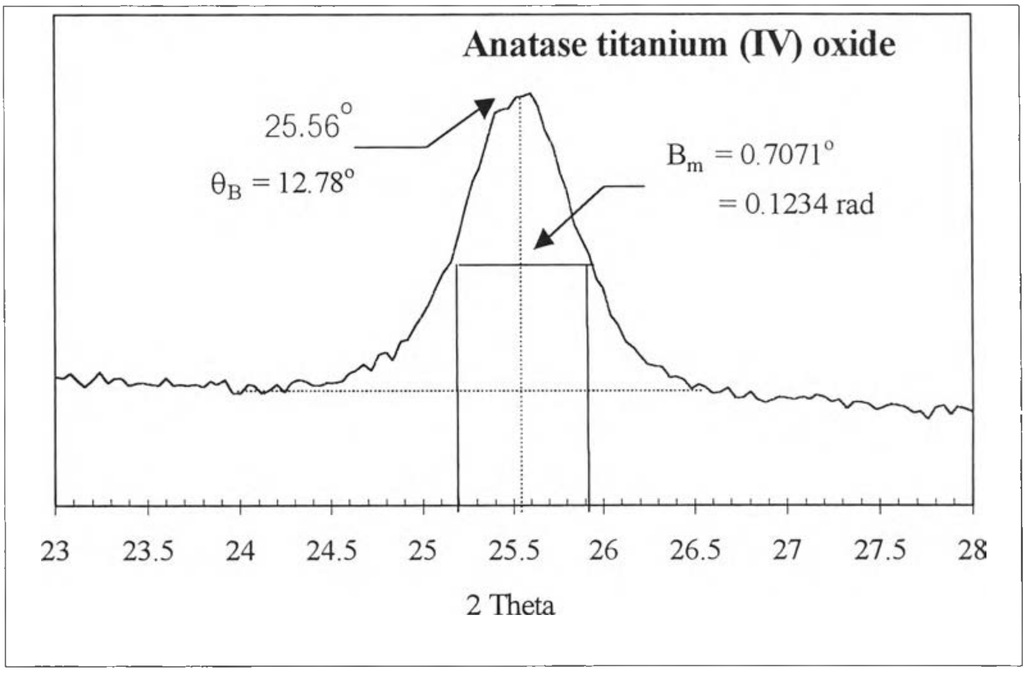
θ_B = the Bragg angle

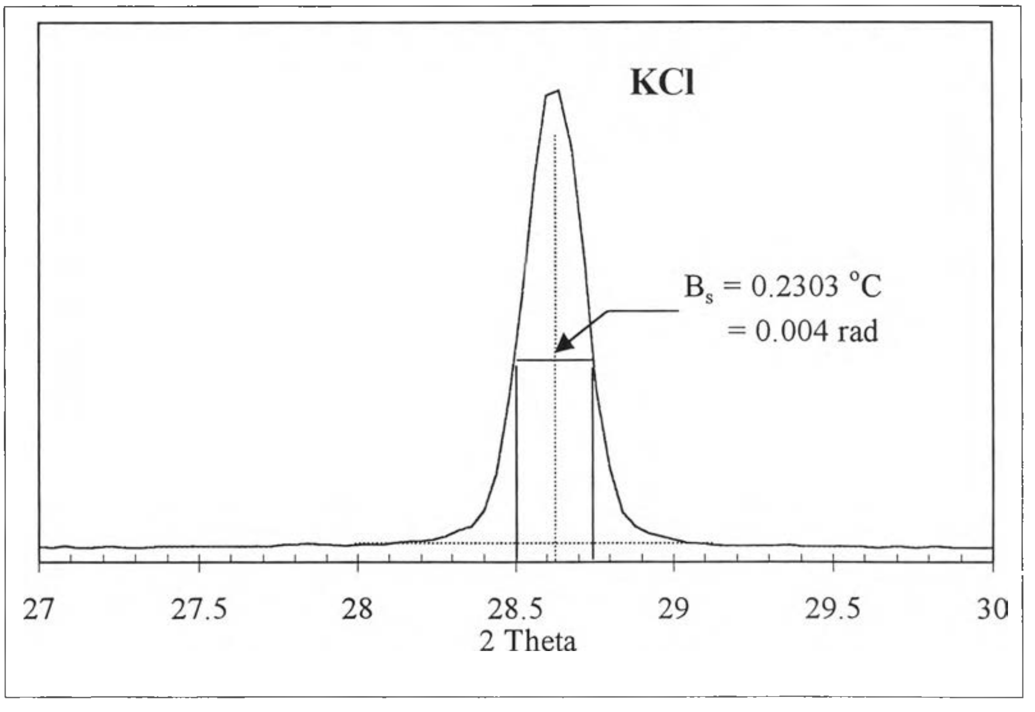
$B = B_M - B_S$

B_M = the measured peak width in radians at half peak height.

B_S = the corresponding width of a standard material

Example: The crystallite size of pure anatase titanium (IV) oxide prepared in 1,4 butanediol is calculated as follow:





From $B = B_M - B_S$

$= 0.01234 - 0.00402 = 0.00832$

$\theta_B = 12.78\,^{o}$

$\lambda = 1.5418\ A^{o}$

So, $t = (0.9 \times 1.5418) / (0.00832 \times \text{Cos } 12.78)$

$= 170\ A^{o} = 17\ nm$

VITA

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