



UNIVERSITI PUTRA MALAYSIA

**CHARACTERISATION OF BISMUTH OXIDE
VIA PRECIPITATION METHOD**

NOR HIDAYATY BT. KAMARULZAMAN

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By

NOR HIDAYATY BT. KAMARULZAMAN

**Thesis Submitted to the School of Graduate Studies,
Universiti Putra Malaysia in Fulfilment of the Requirement
for the Degree of Master of Science**

February 2006



Special Appreciation to.....my family

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

CHARACTERISATION OF BISMUTH OXIDE VIA PRECIPITATION METHOD

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NOR HIDAYATY BT. KAMARULZAMAN

February 2006

Chairman: Irmawati Ramli, PhD

Faculty : Science

A systematic study was carried out in order to investigate the effect of bismuth concentration, precipitating agent concentration, and type of precipitating agent on the microstructural properties of Bi_2O_3 . The formation and properties of the precursors and Bi_2O_3 were monitored by X-ray powder diffraction (XRD), FTIR spectroscopy, scanning electron microscopy (SEM) and temperature programmed reduction of hydrogen (H_2 -TPR).

Monoclinic α -phase Bi_2O_3 was precipitated as a single phase in high purity form directly from synthetic route. Upon heat treatment, the morphology shows non-agglomerated small needles in size between 10.8 and 30.5 μm . This was obtained when using NaOH as precipitating agent. Whereas, tetragonal β -phase Bi_2O_3 was obtained when using NH_3 solution as precipitating agent. Nevertheless, this was only successful when using low concentration of bismuth, i.e. 0.1 M, because above that β -phase transformed into α -phase. Metastable β -phase was achieved through multiphase precursors, which upon

calcinations at 723 K managed to eliminate all of the unwanted species. From SEM images β -phase Bi_2O_3 was revealed as plate like particles organised into rosette clusters.

The surface area measurement indicated that NH_3 solution series of samples give higher value than NaOH series of samples. These values which in accordance to the materials particle size that have an influence on reduction activity. Higher reducing reactivity was displayed by NH_3 solution samples due to smaller particle size which activity as an induction factor. Total amount of oxygen removed are about the same in all samples with more than a monolayer oxygen has been removed. Thus, with high reduction activity suggested that the oxygen may originate from sublattice or lattice of the oxide.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

**PENCIRIAN BISMUT OKSIDA MELALUI
KAEDAH PEMENDAKAN**

Oleh

NOR HIDAYATY BT. KAMARULZAMAN

Februari 2006

Pengerusi: Irmawati Ramli, PhD

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Satu kajian yang sistematik telah dijalankan bagi mengkaji kesan kepekatan bismut, kepekatan agen pemendakan dan jenis agen pemendakan yang digunakan keatas sifat mikrostruktur Bi_2O_3 . Pembentukan dan sifat bagi prekursor dan partikel Bi_2O_3 dilakukan dengan menggunakan kaedah analisis terma-gravimetri (TGA), Pembelauan X-Ray (XRD), Pengukuran Luas Permukaan BET, Mikroskopi Pengimbas Elektron (SEM), Spektroskopi Inframerah (FTIR) dan Penurunan Berprogram Suhu dengan hidrogen (TPR).

Monoklinik fasa $\alpha\text{-Bi}_2\text{O}_3$ telah dapat dimendakkan secara terus melalui kaedah sintesis, dengan terhasilnya fasa tunggal berketulenan tinggi. Semasa proses pemanasan dilakukan, morfologinya menunjukkan jejarum kecil yang tidak bergumpal dengan saiz di antara 10.8 and 30.5 μm . Ianya terhasil dengan menggunakan NaOH sebagai agen pemendakan. Manakala, tetragonal fasa $\beta\text{-Bi}_2\text{O}_3$ telah terhasil apabila larutan NH_3 digunakan sebagai agen pemendakan. Walaubagaimanapun ianya hanya berlaku apabila

kepekatan bismut yang rendah digunakan, 0.1 M, ini kerana pada kepekatan bismut yang lebih tinggi, fasa β ini akan berubah kepada fasa α .

Metastabil β - Bi_2O_3 terhasil melalui multifasa prekursor dimana semasa proses kalsinasi pada 723 K, spesies yang tidak dikehendaki telah berjaya disingkirkan. Melalui imej SEM pula, β - Bi_2O_3 dilihat terdiri daripada plat yang tersusun membentuk 'rosette cluster'. Pengukuran luas permukaan mendapati sampel bagi siri larutan NH_3 telah memberikan nilai yang lebih tinggi berbanding sample dari NaOH . Nilai yang tinggi ini adalah berdasarkan kepada saiz partikelnya dimana ianya telah memberikan kesan kepada aktiviti penurunannya. Aktiviti penurunan yang tinggi juga telah diperolehi daripada sampel NH_3 , disebabkan oleh saiz partikel yang kecil dan telah bertindak sebagai faktor induksinya. Jumlah keseluruhan oksigen yang disingkirkan adalah sama bagi kesemua sampel dimana lebih dari satu lapisan oksigen telah disingkirkan. Maka, dengan peningkatan aktiviti penurunan ini, oksigen yang terlibat dicadangkan adalah dari sublatice atau latice oksida itu.

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For the Blessing and Strength*

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I certify that an Examination Committee met on **14th February 2006** to conduct the final examination of Nor Hidayaty Binti Kamarulzaman on her Master of Science thesis entitled “Characterisation of Bismuth oxide via Precipitation Method” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

NOR HIDAYATY BT. KAMARULZAMAN

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