CARBON MONOXIDE OXIDATION OVER MODIFIED TITANIUM DIOXIDE SUPPORTED GOLD CATALYSTS

John Achu Moma

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DECLARATION

I declare that this thesis is my own, unaided work. It is being submitted for the Degree of Doctor of Philosophy in the University of the Witwatersrand, Johannesburg. It has not been submitted before for any degree or examination in any other University.

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Signature of candidate

________ day of _________________ 2007

ABSTRACT

Highly dispersed gold nanoparticles on metal oxide surfaces have recently been reported to exhibit high catalytic activity for low-temperature carbon monoxide oxidation. Amongst the metal oxides, titanium dioxide, more often the commercial form Degussa P25, has been the most studied support for gold as a catalyst for CO oxidation because it yields some of the most active and stable catalysts. Physical and chemical modification of catalysts supports has been shown to affect their catalytic properties.

In this research, modified gold supported catalysts have been prepared, characterized and tested for CO oxidation. Their properties have been compared with those of the unmodified catalysts. Catalysts containing1wt% Au supported on M_xO_y and TiO_2/M_xO_y mixed oxide (M = Zn, Mg, Ni, Fe, Cr, Cu, Mn and Co; TiO_2:M_xO_y mole ratio of 5:1; TiO_2 = Degussa P25) were prepared by the single step borohydride reduction method and it was found that TiO_2 gave the most superior activity as support for gold for CO oxidation, followed by TiO_2/M_xO_y and the corresponding M_xO_y.

The specific activities for CO oxidation of Au/TiO_2 catalysts per unit of prepared in the range 0.05 to 1 wt% of Au indicates that for catalysts prepared by deposition precipitation, there is a significant decrease in specific activities with an increase with gold loading. For the single step borohydride reduction procedure, specific activities decrease less significantly with increasing gold content, implying that for economic and practical reasons, it would be advantageous to prepare gold catalysts with low gold loadings. Cyanide leaching of 1 wt% Au/TiO_2 catalysts at different Au:CN^- ratios, to selectively remove some of the gold in the catalysts, shows the activity per unit mass of gold to increases as more gold is removed from the catalyst. This is consistent with the idea that gold exists in more than one oxidation state in the systems and a significant fraction of the gold present in the catalysts do not contribute to catalytic activity.

A number of anions and cations have been incorporated into TiO_2 as support for gold catalysts and also into as-prepared Au/TiO_2 catalysts at levels ranging from 0.05 mol% to 2.5 mol% with respect to the support. The activities of the catalysts for CO oxidation reveal that at the highest concentration levels of the ions, in all cases, a decrease in activity compared with unmodified Au/TiO_2 is observed. However, addition of 0.05 to 0.4 mol% of the ions with respect to the support, prior to gold addition, in most cases, resulted in activity enhancement which increased with a decrease in the ion content. Similar addition of 0.05 to 0.4 mol% of the ions with respect to TiO_2 to Au/TiO_2 resulted in a decrease in activity. Attempts to understand the origins of these effects show that there is a degree of chemical interaction between the added ions and gold.

In memory of my grandmother

Abu Anna Gwan Lengah

PUBLICATIONS AND PRESENTATIONS RELATED TO THIS WORK

Publications

1. P. Mohapatra, John Moma, K.M. Parida and Mike S. Scurrell; "*Dramatic promotion of gold/titania for CO oxidation by sulphate ions" Chem. Commun.*, **2007**, 1044-1046
2. J.A. Moma, W.A. Jordaan and M.S. Scurrell; *"Effects of incorporation of ions into Au/TiO_2 catalysts for carbon monoxide oxidation" Topics in Catal.*, **44**, (2007), 167-172.
3. T. Ntho, J.A. Moma, M.C. Raphulu, T. Letswiti, A. Cannel and M.S. Scurrell; *"Catalysis by supported gold in carbon monoxide oxidation. Effect of gold loading and behaviour of catalysts with low gold loading"* in preparation 2007.

Oral Presentations

1. J.A. Moma and M.S. Scurrell; *Dramatic promotion of gold/titania for CO oxidation by sulphate ions"* in CATSA conference at Mossel Bay, 12-15 November 2006.
2. J.A. Moma and M.S. Scurrell; *"Effects of incorporation of ions into Au/TiO_2 catalysts for carbon monoxide oxidation"* in CATSA conference at Midrand, 13-16 November 2005.
3. J.A. Moma *"The effects of anions and cations on supported gold catalysts for carbon monoxide oxidation"* Guest lecture at Regional Research Laboratories, Bhubaneswar, India, 15 April 2005.
4. J.A. Moma *"The effects of anions and cations on supported gold catalysts for carbon monoxide oxidation"* Guest lecture at Indian Institute for Chemical Technology, Hyderabad, India, 8 April 2005.
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ABBREVIATIONS

ABBREVIATION	MEANING
AAS	Atomic absorption spectroscopy
AES	Atomic emission spectroscopy
B.E.	Binding energy
BET	Brunauer Emmet and Teller
CNT	Carbon nanotube
CVD	Chemical vapour deposition
DFT	Density functional theory
DP	Deposition precipitation
DR-UV/Vis	Diffuse reflectance ultraviolet visible spectroscopy
EELS	Electron energy loss spectroscopy
EXAFS	Extended x-ray absorption fine structure
FTIR	Fourier transform infrared spectroscopy
FWHM	Full width at half maximum
HRTEM	High resolution transmission electron microscopy
ICP	Inductively coupled plasma emission/absorption spectrometry
IEP	Isoelectric point
IMP	Aqueous impregnation
IR	Infrared spectroscopy
PEFC	Polymer electrolyte fuel cell
PEM	Proton exchange membrane
PO	Propene oxide
SIMS	Secondary ion mass spectrometry
SMAD	Solvated metal atom dispersion
SSBH	Single step borohydride
STM	Scanning tunelling microscopy
STS	Scanning tunneling spectroscopy
TEM	Transmission electron microscopy
TPD	Temperature programmed desorption

ABBREVIATION	MEANING
TPO	Temperature programmed oxidation
TPR	Temperature programmed reduction
UPS	UV-induced photoelectron spectroscopy
WAXS	Wide angle x-ray scattering
WGS	Water-gas shift
XANES	X-ray absorption near edge structure
XAS	X-ray absorption spectroscopy
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction