

7th International Gold Conference

Recent developments in gold research from heterogeneous catalysis to biomedical imaging

Reviewed by Nicoleta Muresan* and Agnes Raj

Johnson Matthey Technology Centre, Blounts Court, Sonning Common, Reading RG4 9NH, UK

*Email: muresn01@matthey.com

Introduction

The 7th International Gold Conference takes place every three years and in 2015 was hosted by the Cardiff Catalysis Institute at Cardiff City Hall, UK, from 26th to 29th July. Scientists from all over the world gathered to discuss the latest advances in gold chemistry. Over three days, the conference covered different aspects from homogeneous and heterogeneous catalysis to biomedical imaging. The conference had more than 200 attendees from academia and industry and was divided into three parallel sessions with 72 talks and nearly 100 posters.

This review will focus mainly on the industrially relevant heterogeneous catalysis and to some extent biomedical applications.

Gold in Catalysis

Catalysis by Au has rapidly increased importance in chemistry since Masatake Haruta (Osaka National Research Institute (ONRI), Japan) and Graham Hutchings (Cardiff University) found that Au can be

used as a catalyst in various reactions. Historically, Au was known to be a transition metal rich in coordination numbers but compared with other metals, bulk Au was thought to be chemically much less reactive. However, this changed in 1973 when Bond *et al.* (1) reported the hydrogenation of olefins over supported Au catalysts; subsequently Haruta and Hutchings independently found that supported nanosized Au catalysts are surprisingly active for low temperature carbon monoxide oxidation and for the hydrochlorination of acetylene to vinyl chloride monomer (2, 3). The most important aspects of Au catalysts are the high activity at low temperatures, promotion by water and unique selectivity. The catalytic activity of Au nanoparticles (NP) can be further tuned by careful choice of the support material. There are many examples of Au NP loaded on semiconductor materials to exhibit high CO oxidation activity at temperatures below 0°C, activity which has not been reached by other metals (4–8).

Low Temperature CO Oxidation by Gold

At the beginning of the 1980s, Haruta reported that Au NP deposited on semiconducting oxides such as titanium dioxide (TiO₂), iron(III) oxide (Fe₂O₃), cobalt(II,III) oxide (Co₃O₄) and nickel oxide (NiO) exhibit markedly high catalytic activity for CO oxidation even at temperatures as low as 200 K (7). The contributing factors to this high activity are still disputed. Haruta studied low-temperature CO oxidation with Au and platinum NP supported on TiO₂ prepared by different methods and found that activity is dependent on the

Plenary and Keynote Speakers

Masatake Haruta (Tokyo Metropolitan University, Japan)	Simple Chemistry Catalyzed by Gold Nanoparticles	Mostafa A. El-Sayed (Georgia Institute of Technology, USA)	Solution Metallic Catalysis on the Nanoscale
Luis M. Liz-Marzán (Bionanoplasmonics Laboratory, CIC biomaGUNE, Spain)	Crystalline Facets in Gold Nanocrystals	Tatsuya Tsukuda (The University of Tokyo, Japan)	Precise Synthesis and Non-scalable Oxidation Catalysis of Gold Clusters
Catherine J. Murphy (University of Illinois at Urbana-Champaign, USA)	A Golden Age for Colloidal Nanoparticles	Christopher Kiely (Lehigh University, USA)	Population Distributions and Catalytic Hierarchy of Active Species in Gold on Iron Oxide Catalysts for Low Temperature CO Oxidation
Dean Toste (University of California, USA)	Homogeneous Gold Catalysts for Selective Organic Synthesis	Vivian Wing-Wah Yam (The University of Hong Kong, Hong Kong)	Luminescent Gold-Containing Molecular Photofunctional Materials
Peter Johnston (Johnson Matthey Chemical Catalysts, UK)	The Development and Commercialisation of Gold Catalysts for VCM Production	Mona Tréguer-Delapierre (University of Bordeaux, France)	Gold Nanoparticles of Unusual Morphologies
Robert T. Tshikhudo (DST/Mintek Nanotechnology Innovation Centre, South Africa)	Gold Nanotechnology Innovation at Mintek: Creating and Capturing Value in the Biotechnology Sector		
Malcolm Kadodwala (University of Glasgow, UK)	Biosensing with a Twist: Detection and Characterization of Biomaterials with Sculpted EM Fields		
Chris J. Ackerson (Colorado State University, USA)	Jahn-Teller Effects in Gold Nanoclusters		
Ewa Kowalska (Hokkaido University, Japan)	Visible Light-induced Photocatalysis Through Surface Plasmon Excitation of Gold on Titania Surfaces		
Cristina Nevado (University of Zürich, Switzerland)	On Gold Mechanistic Studies and Beyond		
Antonio M. Echavarren (Institute of Chemical Research of Catalonia, Spain)	Synthetic Transformations via Gold-Carbenes		
A. Stephen K. Hashmi (Universität Heidelberg, Germany)	Efficient Homogeneous Gold Catalysis		

preparation method. Interestingly, hemispherical Au particles adhering to the support, prepared by deposition-precipitation methods, exhibited greater activity than spherical particles having little interaction with the support prepared using impregnation and photochemical methods. Pt catalysts did not show such variation in activity with the preparation method, indicating that only the metal is involved in CO conversion. These data indicate that metal-support interactions play a crucial role in Au-catalysed CO oxidation at low temperature (9). Another important aspect is the size of the NP. It has been found that the optimal inside diameter range for maximum activity is between 0.5 and 5 nm (9), supporting Kubo's prediction five decades ago that small NP would behave differently from the bulk (10). Metal NP smaller than 2 nm show a discrete energy band structure similar to those of semiconductors, **Figure 1**.

The promoting effect of moisture has been intensively studied and it has been found that it promotes CO oxidation at room temperature but is not indispensable at temperatures below 273 K, depending on the support. Semiconducting supports such as TiO₂, manganese(IV) oxide (MnO₂), Fe₂O₃, Co₃O₄, NiO, zinc oxide (ZnO), zirconium dioxide (ZrO₂) and cerium(IV) oxide (CeO₂) exhibit high activity at temperatures below 273 K in the absence of water. However, insulating, non-reducible metal oxides such as aluminium oxide (Al₂O₃), silicon dioxide (SiO₂) or titanium carbide (TiC) show moderate

activity and require a significant amount of moisture (11). A detailed study on the influence of moisture and NP size on CO oxidation over supported Au catalysts, including kinetic studies has been published (11).

As an extension to low-temperature CO oxidation over supported Au catalysts, Professor Bert Chandler (Trinity University, USA) presented a new process for eliminating CO from hydrogen feeds with minimal hydrogen oxidation, the so-called preferential oxidation (PROX) process with Au/TiO₂ and Au/Al₂O₃ catalysts. CO PROX over Au catalysts has been investigated in two ways: firstly, the fundamentals of CO oxidation are studied in the absence of H₂ (at low conversion); secondly the CO oxidation reaction is studied at industrially useful conversions and temperatures (close to 80°C) in the presence of H₂ (PROX). In both cases, the reaction is greatly affected by the presence of water in the feed and formation of carbonates on the catalyst surface. In a recently published study, the Chandler group established that the CO oxidation reaction is co-catalysed by weakly adsorbed water at the NP perimeter (12, 13).

In situ characterisation of CO oxidation over bimetallic Au-Ag/TiO₂ catalysts was presented by Catherine Louis (Centre National de la Recherche Scientifique (CNRS)-University Pierre et Marie Curie (UPMC), France). Bimetallic Au-Ag NP supported on TiO₂ showed higher activity and stability for CO oxidation at room temperature than monometallic Au and Ag catalyst. The best performances were obtained by Au-Ag/TiO₂ catalysts with an Au:Ag ratio of 1:0.8 after activation in

H₂ at 550°C. Transmission electron microscopy (TEM) showed that the average metal particle size increases with the temperature of activation and micro-energy dispersive spectroscopy (EDS) performed on individual particles indicated that the metal particles become more bimetallic. Ultraviolet (UV)-visible spectroscopy performed during *in situ* reduction indicated that the bimetallic particles formed gradually as temperatures increased. The evolution of the surface composition of the metal particles studied by X-ray photoelectron spectroscopy (XPS) and CO diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) revealed an increasing proportion of silver atoms during activation under H₂ (14).

Other Industrially Relevant Processes with Gold Catalysts

Peter Johnston presented the latest progress in the development and commercialisation of Au catalysts for vinyl chloride monomer (VCM) production. He showed that Au supported on activated carbon as catalyst for the hydrochlorination of acetylene shows superior activity to the current mercury(II) chloride (HgCl₂) on carbon. He explained the importance of the migration to the new, mercury-free process taking into consideration the environmental and handling problems due to the toxic nature of the Hg catalyst. He described the progress in developing ultra-low Au catalysts for this application (3, 15, 16).

Another important industrial process is glycerol oxidation. In recent years, the catalytic transformation of alcohols into fuels and other valuable chemicals has played an important role in the quest to more sustainable processes. It is well known that oxidation of glycerol is a complex process leading to a complex mixture of products including six potential C₃ oxygenated products. Chunli Xu (Shaanxi Normal University, China) presented the effect of the acid/base nature of supports on the selectivity of glycerol oxidation over supported Au-Pt bimetallic catalysts. Various supports, including acidic (TiO₂ and CeO₂), basic (magnesium oxide (MgO), magnesium hydroxide (Mg(OH)₂), magnesium carbonate (MgCO₃), Mg(OH)₂, ZnO and calcium carbonate (CaCO₃)) and amphoteric (Al₂O₃ and hydrotalcite), were reported. The results showed that the acid-base properties of the supports significantly affected product selectivity. The selectivity to glyceraldehyde was inversely proportional to the strength of basic sites on the supports while the

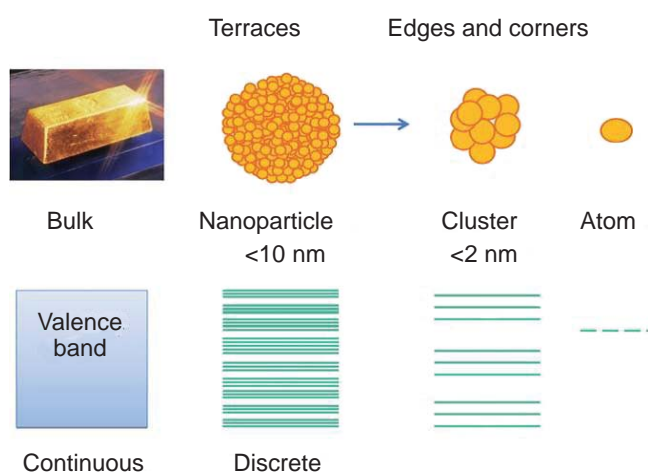


Fig. 1. Major surface states and valence band structures of bulk, NP, clusters and atoms (9) (Reproduced by permission of The Royal Society of Chemistry)

selectivity to tartronic acid, glycolic acid and glyoxalic acid was directly proportional to the strength of basic sites of supports. In contrast, the selectivity to glyceric acid was neither proportional to the strength of basic sites of supports nor proportional to the strength of acid sites of supports (17).

Two other talks on alcohol oxidation were presented by Emiel J. M. Hensen (Eindhoven University of Technology, The Netherlands) studying the synergy between Au NP and Cu⁺ in MgCuCr₂O₄-spinel supports in the selective aerobic oxidation of alcohols and olefins. He showed that Au/MgCuCr₂O₄ catalyst is highly active and selective for the aerobic oxidation of ethanol to acetaldehyde. TEM, XPS and extended X-ray absorption fine structure (EXAFS) characterisation results show that the synergy involves close interaction between metallic and surface Cu⁺ species stabilised in the support. MgCuCr₂O₄ is more stable under reaction conditions than MgCuAl₂O₄. The use of MgCuAl₂O₄ leads to segregation of Cu and formation of copper(II) oxide (CuO). Au/MgCuCr₂O₄ is also an active catalyst for the selective oxidation of propylene to acrolein (selectivity close to 90%). Density functional theory (DFT) study of the reaction mechanism explained the dynamic nature of Au atoms at the interface of the Au clusters with the support. Adsorption of propylene leads to the formation of an isolated Au atom that strongly binds propylene. The reaction proceeds by activation of the allylic C–H bond by an adsorbed O₂ molecule. Detailed analysis of the electronic structures of the reaction intermediates in the catalytic cycle show that the critical role of Cu is the decrease of the desorption energy of acrolein (18–21).

On the same subject of selective oxidation of alcohols to aldehydes or ketones, Feng Wang (Waterloo Institute for Nanotechnology, Canada) discussed the effect of crystallinity of Au NP supported on CeO₂ for the conversion of alcohols to aldehydes or ketones and amines to imines *via* oxidative dehydrogenation. Both types of reactions achieve moderate to excellent yields. Importantly, regardless of Au size and surface area of CeO₂ support, the study reveals that the active plane for C–H and N–H bond activation is the {110} plane on nanorod ceria. Characterisations by Raman and XPS show that positively charged Au dominates in nanorod Au/CeO₂ possibly *via* the interaction with oxygen vacancy sites. Au NP supported on CeO₂(110) crystal planes were more reactive than on CeO₂(111) and (100) in the oxidative dehydrogenation of alcohols.

Kinetic analysis and a Hammett plot suggest that hydride transfer is involved and the cationic Au is catalytically active. The Au/CeO₂ catalysts were initially evaluated in the oxidation of benzyl alcohol to benzaldehyde showing around 99% selectivity for benzaldehyde (22, 23).

Another important catalytic process is low-temperature C–C bond activation. Hidehiro Sakurai (Osaka University, Japan; and Japan Science and Technology Agency) has shown that bimetallic Au/Pd alloy nanoclusters stabilised by a hydrophilic polymer, poly(*N*-vinylpyrrolidone) (PVP), have unusual catalytic activity towards the activation of the C–Cl bond at room temperature under aqueous conditions. The reaction does not occur with either Au or Pd single metal clusters, indicating that a synergistic alloy effect plays a key role. In addition, alloy clusters exhibited unique reactivity when aryl bromide was used as coupling partner. With Pd-rich catalyst the reaction occurs through conventional catalysis by leaching of Pd from alloy clusters (24–26).

The selective hydrogenation of acetylene to ethylene over SiO₂-supported Au alloyed Pd single-atom catalyst (SAC) was discussed by Tao Zhang (Chinese Academy of Sciences, China). This industrially important process uses mainly Pd-based catalysts but suffers from poor selectivity. The speaker showed that conversion of acetylene to ethylene can be achieved with high selectivity using Au alloyed Pd SAC on SiO₂ support. *In situ* DRIFT spectra of CO adsorption and EXAFS results give evidence of the formation of Au-Pd interaction. The data fitting results gave only Pd-Au interactions in the AuPd_{0.025}/SiO₂ catalyst, providing direct evidence for the isolation of Pd atoms. This new catalyst improved the catalytic performance of the acetylene conversion by 95% compared to the monometallic Au/SiO₂ catalyst and ethylene selectivity by 1000% compared to the monometallic Pd catalyst (27).

Two other relevant talks were delivered by Florencia C. Calaza (Fritz-Haber-Institut der Max-Planck-Gesellschaft, Germany) and Wilm Jones (The UK Catalysis Hub, UK; and Cardiff University, UK) on carbon dioxide activation by negatively charged two-dimensional (2D) Au clusters; and photocatalytic H₂ production using core-shell AuPd promoters with controlled shell thickness, respectively. The adsorption and activation of CO₂ on various Au clusters grown on Ag(001)/supported MgO films were reported using

XPS, temperature programmed desorption (TPD) and infrared reflection absorption spectroscopy (IRAS). It has been found that the adsorption of CO₂ in the presence of 2D Au clusters with negatively charged perimeters is reversible and that these species are stable up to slightly above room temperature. When heated beyond 380 K, the clusters appear to take on more three-dimensional (3D) morphologies. The negative charge on the 2D clusters is responsible for the activation of CO₂. Furthermore, isotope labelled compounds were used (¹³CO₂, ¹⁸CO₂ and ¹⁸O₂ for labelling the oxide surface) to identify the presence of different species on the surface (28). Au NP supported on TiO₂ followed by the deposition of thin layers of one and two monolayer equivalent of Pd was studied for photocatalytic H₂ production. Deposition of Pd was performed by a photodeposition method as well as sodium borohydride (NaBH₄) reduction (SR). The photocatalytic performance was evaluated using the reforming of ethanol as a model. TEM analysis demonstrates that all metal NP were deposited on the TiO₂ homogeneously for all samples. Analysis by XPS confirmed the presence of metallic Au and Pd with the lack of any significant PdO. The absence of PdO suggests the Pd is associated with Au, as segregated Pd would likely oxidise. The association of Pd with Au was also supported by EXAFS results which show Pd existing in a Au-rich environment with no significant Pd–O and Pd–Pd interactions (29).

Gold in Sensing Applications

One of the sessions focused on sensing applications using Au as the active material. Malcolm Kadodwala (Glasgow University, UK) delivered a particularly interesting talk titled 'Biosensing with a Twist: Detection and Characterization of Biomaterials with Sculpted EM Fields', on ultrasensitive detection and characterisation of biomolecules using Au to create plasmonic nanomaterials (30–32). 'Metal Oxide Supported Gold Nanoparticles for Gas Sensing Applications' delivered by Katia Fajerweg (Paul Sabatier University, France), focused on Au/ZnO and Au/TiO₂ for CO and propane detection (33, 34). Another presentation on sensing with Au was delivered by Emilie Lebon-Tailhades (Laboratoire de Chimie de Coordination, France; and Sciences et Technologies pour l'Aéronautique et l'Espace, France). She introduced a new strategy for nitrate monitoring in aqueous media using Au nanostructured electrodes (35). 'Gold-decorated

MW-CNT Modified Electrodes for Glycerol Electroanalytical Detection and Transformation' was presented by Laura Prati (University of Milan, Italy). She mentioned that carbon nanotubes could be decorated with Au NP using sol immobilisation or deposition precipitation. More details can be found in the literature (36).

Gold in Clinical Diagnostics and Treatments

Using nanotechnology-based systems for diagnosing and treating specific diseases is increasing in popularity in recent decades and it is particularly relevant for personalised medicine, allowing a disease to be detected at an early stage and a suitable therapy to be directed toward the target tissue. The unique properties of Au play an important role in this area. A series of presentations were delivered by experts including Laura Maiore on synthesis, characterisation and evaluation of Au complexes as antimicrobial compounds (37) and Meike Roskamp (Midatech Pharma Plc, UK) on Au anticancer drugs. Midatech Pharma are using ultra-small targeted sugar-coated Au NP as delivery agents and have their own manufacturing facility for large scale NP under good manufacturing practice (GMP) conditions (38). In the same area of drug delivery, Federica Scalleti delivered a talk on the fabrication of Au nanorods for incorporation into nanocapsule systems (39). Another presentation in the same field was by Sebastian Kopitzki (Midatech Pharma Plc) on targeted treatment of liver diseases. This study focuses on attaching the targeting and chemotherapeutic agents to ultra-small glycol-coated Au NP. It has been shown that after administration and circulation in the body the targeted Au NP accumulate in the liver, where the small NP show increased tumour penetration potential (40). In the diagnostics area, Yasuro Niidome (Kyushu University, Japan) presented a study on liver tissue mass spectrometry imaging using Au NP. He showed that the highly stable chemical nature of Au and the high sensitivity of mass spectrometry enabled the Au nanorods to serve as a mass marker in body tissue (41, 42).

Conclusion

This review presents only a fragment of the presentations and posters disclosed. The increasing popularity of the conference, combined with the large number of key stakeholders present, highlighted the

importance of Au catalysis. It has been shown that the most important characteristics of Au catalysts: the high activity at low temperatures, promotion by water and unique selectivity can be further tuned by careful choice of support, different promoters or catalysis conditions. The range of applications in the conference, from replacing mercury in VCM production to diagnosing and treating medical conditions, presents an optimistic message that Au catalysis and applications will play a crucial role in a sustainable future.

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The Reviewers



Nicoleta Muresan is a Senior Scientist working in Core Science studying the development of precious metal precursors and catalyst preparations at Johnson Matthey Technology Centre, Sonning Common, UK. She completed her degree in Chemistry at the University of Bucharest, Romania, and PhD in Organometallic Chemistry at the University of Bochum, Germany, followed by Post-Doctoral Research positions in Organometallic and Coordination Chemistry at the Max Planck Institute for Bioinorganic Chemistry, Germany, and Renewable Energy at the University of Cambridge, UK.



Agnes Raj is a Senior Scientist, compressed natural gas (CNG) project leader and global coordinator at Johnson Matthey Emission Control Technologies, Sonning Common, UK. She obtained MSc and MPhil in Chemistry from the University of Madras, India and PhD in Material Science from Imperial College London, UK. Since joining Johnson Matthey in 2007, her research activities have been focused on platinum group metal-based catalysts for diesel/CNG aftertreatment systems.