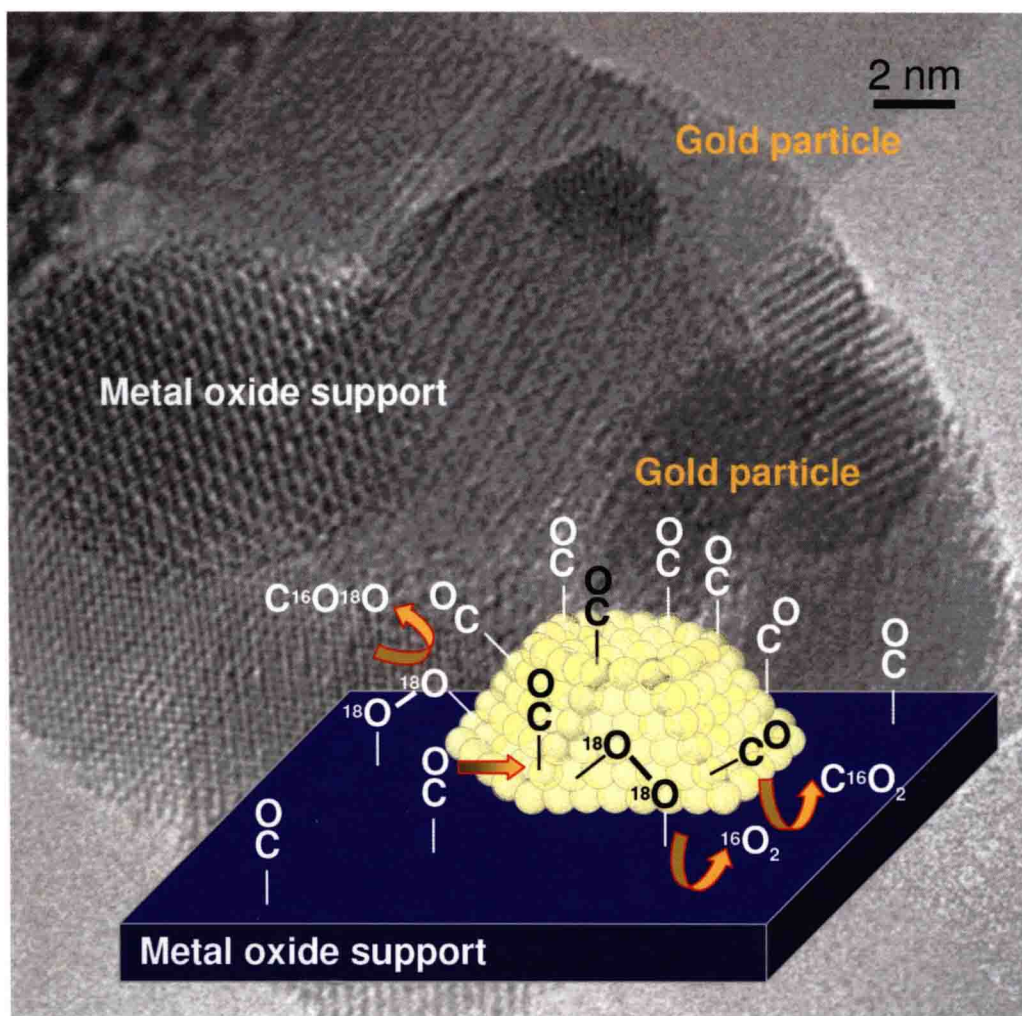


Edited by George Avgouropoulos and Tatyana Tabakova

Environmental Catalysis over Gold-Based Materials



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Preface

Gold, long regarded as poorly active catalyst, has become a subject of appreciable research for the last two decades. It has been demonstrated that gold-based catalysts, when prepared in an appropriate manner, are highly active and selective in oxidation, hydrochlorination, desulphurization and hydrogenation reactions, often at lower temperatures than existing commercial catalysts. Innovative recent research has also suggested that these materials could be effectively employed in hydrogen fuel processing reactions for fuel cell applications. Gold in a highly dispersed state can exceptionally catalyze water–gas shift and preferential CO oxidation reactions. Several factors control the activity and the selectivity of gold catalysts and can affect their efficiency in environmental catalytic processes. This book presents some recent advances in environmental catalysis over gold-based materials, featuring prominent authors, all experts in their respective fields. A general introduction to the field of environmental catalysis using supported gold and gold bimetallic alloy nanoparticles and the possibility of using sustainable feedstocks for the generation of chemicals are described in Chapter 1. Recent advances in the synthesis and physicochemical properties of gold-based materials are thoroughly summarized in Chapter 2, while Chapter 3 highlights the spectroscopic studies with respect to the nature of active gold species in several reactions, such as CO oxidation, water–gas shift and methanol reforming. Chapter 4 reviews most recent reports on preferential CO oxidation reaction over gold catalysts and highlights the key factors that control the activity/selectivity of these catalytic systems. A series of studies which have recently appeared in the literature investigating desulfurization reactions on novel gold–metal carbide catalysts are reviewed in Chapter 5, while Chapter 6 covers essential aspects regarding the physicochemical properties of gold, its interactions with hydrogen to dissociate this molecule and to perform selective

hydrogenation reactions. Current applications and future commercial opportunities are discussed in the last chapter (Chapter 7).

We thank all of the lead authors (G. J. Hutchings, C. Louis, F. Boccuzzi, J. A. Rodriguez, A. Corma and T. Keel) as well as their co-authors for their contribution and fruitful collaboration in the preparation of this book. We gratefully acknowledge the creativity and skills of M. Manzoli for designing the cover page. We hope that this book will be useful for readers and will stimulate new applications of gold-based materials. It can serve as a reference for researchers whose interest is attracted by the unusual catalytic properties of nanosized gold.

George Avgouropoulos
Tatyana Tabakova

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CHAPTER 1

General Introduction to the Field of Environmental Catalysis: Green Catalysis with Supported Gold and Gold Bimetallic Nanoparticles

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1.1 Introduction

The production of chemicals and energy is crucial to the way of life we have grown accustomed to over the recent decades of sustained growth. In some sense we take the availability of new materials and plentiful energy for granted and when there are disruptions to supplies this provides a major shock to the system. However, we now recognise that the resources available within the world are finite and consequently the desire to use sustainable resources other than fossil carbon reserves is now gaining momentum. Catalysis can and does play a major role with respect to developing the means by which bio-renewable

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feedstocks can be utilised. However, catalysis can play a wider role in environmental chemistry. In this respect green chemistry is now becoming a major driving force helping to shape the way in which chemical processes should be configured. The 12 principles of green chemistry embody the key tenets of what should be aimed for in new environmentally friendly processes:

- **Prevent waste:** Design chemical syntheses to prevent waste, negating end-of-pipe clean up.
- **Design safer chemicals and products:** Prepare effective chemical products with little or no toxicity.
- **Design less hazardous chemical syntheses:** Develop syntheses to use and generate substances with little or no toxicity to humans and the environment.
- **Use renewable feedstocks:** Utilise raw materials and feedstocks that are renewable rather than depleting.
- **Use catalysts, not stoichiometric reagents:** Minimise waste by using catalytic reactions.
- **Avoid chemical derivatives:** Avoid using blocking or protecting groups or any temporary modifications if possible, as these lead to waste.
- **Maximise atom economy:** Design syntheses so that the final product contains the maximum proportion of the starting materials.
- **Eliminate solvents or use safer solvents and reaction conditions:** Avoid using solvents, separation agents, or other auxiliary chemicals. If these chemicals are strictly necessary, then use environmentally friendly chemicals.
- **Increase energy efficiency:** Run chemical reactions at minimal temperature and pressure whenever possible as this is consistent with energy utilisation and recovery.
- **Design chemicals and products to degrade after use:** Where the applications permit such an approach, design chemical products that will break down into innocuous substances after use so that they do not accumulate in the environment.
- **Analyse in real time to prevent pollution:** Include in-process real-time monitoring and control during syntheses to minimise or eliminate the formation of by-products.
- **Minimise the potential for accidents:** Design chemical processes to minimize the potential for chemical accidents and releases to the environment.

It is clear that environmental catalysis should seek to maximise atom and energy efficiency and should eliminate the use of solvent and encompass renewable feedstocks. In this introductory chapter we will give examples that encompass these two key features using supported gold and gold bimetallic nanoparticles. A key feature that is observed for gold catalysis is the high specificity that is found in many catalytic reactions, and this is of key importance for environmental catalysis.

1.2 Use of Renewable Feedstocks

It is generally recognised that oil supplies are both finite and will eventually become increasingly scarce. The geographical disposition of the major oil supplies has made the price of oil a political variable for the majority of recent years. However, it is also generally understood that there exist plentiful supplies of natural gas, particularly with the discovery of large deposits of shale gas. Despite this, there exist political drivers to diminish the dependency on materials production based solely on fossil fuels. In this respect the use of sustainable feedstocks is providing a driving force to discover new and improved chemical processes using these materials. However this drive to use sustainable feedstocks is not wholly embraced at present, as there are still widespread supplies of natural gas and coal. Indeed, several new Fischer–Tropsch plants based on natural gas have been commissioned, partly as a route to sulfur-free diesel and gasoline, and similar processes based on coal-derived syngas are expected to be commissioned in economies where coal is plentiful, *e.g.* India and China. The consumption of fossil fuels using existing pathways is therefore likely to continue for some time. Although the situation is complicated, the time is still right to consider the alternatives because in the longer term we can anticipate that the use of sustainable feedstocks will become politically expedient if viable catalytic chemistries can be developed.

1.2.1 Availability and Diversity of Biomass

More than 80% of the world's energy consumption and production of chemicals originates from fossil resources (*i.e.* oil, gas and coal). The finite nature of these fossil resources coupled with concerns regarding global warming have spurred a drive to develop new technologies for the generation of energy, chemicals and a materials supply from renewable resources. In this respect, it has recently been considered that biomass could become a major source for the production of energy and chemicals. Biomass derived from plants is generated from carbon dioxide and water using sunlight as the energy source while producing oxygen as an important by-product. The total current biomass production on the planet is estimated to be about 170 billion tonnes and consists of roughly 75% carbohydrates (sugars), 20% lignins and 5% of other substances in minor amounts in forms such as oils, fats, proteins, terpenes, alkaloids, terpenoids and waxes. Based on this biomass production, only 3.5% is presently being used for human needs, where the major part of this amount is used for human food (around 62%), 33% for energy use, paper and construction needs and the rest (about 5%) is used for technical (*i.e.* non-food) raw materials such as clothing, detergents and chemicals. The remaining 96.5% of biomass production is utilised within natural ecosystems.^{1–3}

At the current time, the available biomass, and, therefore, the renewable raw materials, are almost entirely provided by agriculture and forestry. Important renewable raw materials such as carbohydrates can be supplied by the sugar, starch and wood processing sectors. Specifically, plant raw materials, such as

sugar beet, sugar cane, wheat, corn, potatoes and rice can be used for the production of sucrose and starch, whereas cellulose and hemi-cellulose can be derived from the processing of wood. These basic products (starch, cellulose and hemi-cellulose) can in principle be further converted into a very broad range of products, by employing physical and chemical processes. These processes are based on partial or complete acid or enzymatic hydrolysis for breaking down the polysaccharides into sugar monomers. Starch and cellulose are polysaccharides having a glucose monomer unit and α -1,4 or β -1,4 glycoside linkages respectively; therefore hydrolysis will break them down into glucose, whereas sucrose hydrolysis will lead to the formation of fructose and glucose.⁴ Hemi-cellulose contains five different sugars, of which two are 5-carbon sugars (xylose and arabinose) and the other three are 6-carbon sugars (galactose, glucose and mannose). In addition, the most abundant building block of hemi-cellulose is xylan (a xylose polymer), which consists of xylose monomer units linked at the 1- and 4-positions. Therefore, hydrolysis of hemi-cellulose will provide a variety of 5-carbon and 6-carbon monomer sugars. In addition, other sugars, such as maltose and lactose, can be produced from biomass (barley and whey, respectively); their hydrolysis will produce glucose from the former and glucose and galactose from the latter. There is therefore a very wide range of sugars that can be made available from the utilisation of biomass. Many of these can be generated from biomass resources that cannot be utilised as foodstuffs and can be harvested on land not suitable for crop generation, and it is these starting materials that need to be the focus of future research attention.

1.2.2 Pathways for Utilisation of Bio-renewable Feedstocks

As noted previously, throughout the world there are huge resources of bio-renewable feedstocks, *e.g.* starch, cellulose, vegetable oils, and attention has been turning to considering whether these materials can be utilised more effectively. Until now the production of liquid fuels from biomass has been based on the use of processes such as acid hydrolysis of biomass for sugar production, thermochemical liquefaction and/or pyrolysis for bio-oils production, and gasification of biomass to produce syngas ($\text{CO} + \text{H}_2$).⁴ Further treatment of the sugars produced, employing a fermentation process, leads to the production of ethanol, whereas the use of a dehydration process makes it possible to produce aromatic hydrocarbons.^{5,6} In the case of liquefaction and pyrolysis, further treatment of these products by refining allows one to generate liquid fuels.^{7,8} Finally, the Fischer–Tropsch process can be used for the synthesis of alkanes from syngas, whereas methanol production is also possible from syngas.⁹

An alternative strategy is the use of carbohydrates as a liquid fuel in fuel cell systems and this idea has recently attracted significant attention. For example, it has been demonstrated that carbohydrates can be used for the production of electricity in a fuel cell which comprises a vanadium flow battery.¹⁰

At the current time, the primary technology for the generation of liquid fuels using renewable biomass resources is based on the fermentation of

carbohydrates for the production of ethanol (bio-ethanol). However, this process is still not economically viable, owing to the high cost of the processing route. Consequently, research is now focused on the use of more plentiful and inexpensive sugars, such as ligno-cellulose, as this will enable a reduction in the cost. Production of bio-oils by liquefaction or pyrolysis, based on thermochemical treatment of biomass, is an inherently simpler process, but it produces a wide range of products (*e.g.* aromatic compounds, CO, CH₄, H₂, tars, chars, alcohols, aldehydes, ketones, esters and acids). Therefore, improvements in selectivity are considered essential for this latter approach. Research is currently being conducted with the goal of upgrading bio-oils to more valuable products.⁹ Finally, the gasification of biomass can produce syngas (CO and H₂); however, this process requires volatilisation of water, thus decreasing the overall energy efficiency.¹¹ It can be concluded that the majority of these methods have complex processing requirements and the efficiency of liquid fuel synthesis remains low.

There has been much attention to the concept of a bio-refinery that is capable of producing either syngas or a new raft of platform chemicals (Figure 1.1). Biomass from any source can indeed, in principle, be gasified, but this will incur an energy penalty, as noted earlier. The production of H₂ and CO by vapour-phase and aqueous-phase reforming of biomass-derived oxygenated hydrocarbons has recently been described.⁴ Vapour-phase reforming of biomass-derived oxygenated hydrocarbons having high volatility (such as methanol, glycerol, ethylene and propylene glycol) is advantageous over the use of alkanes derived from petrochemical feedstocks. However, for less volatile biomass-derived oxygenates such as glucose and sorbitol, it is advantageous to use the aqueous phase reforming process to avoid using excessively high temperatures.

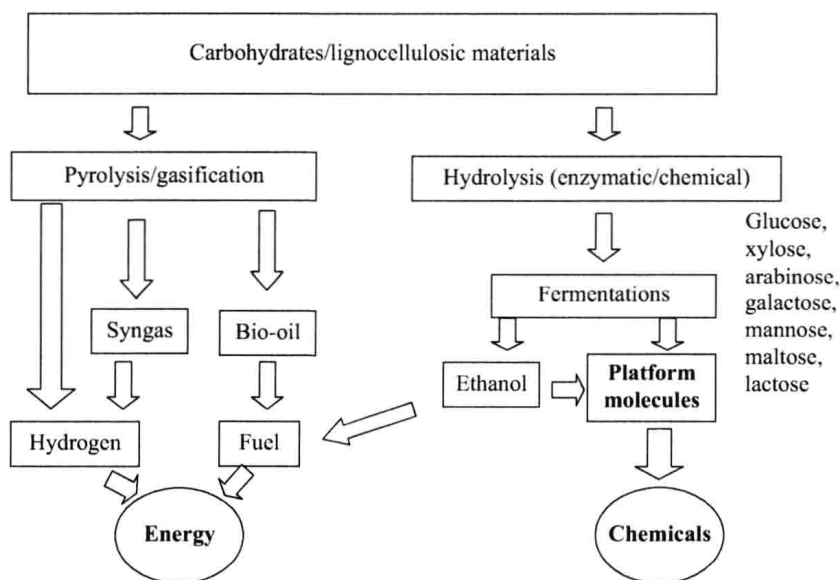


Figure 1.1 Scheme of bio-refinery processes.

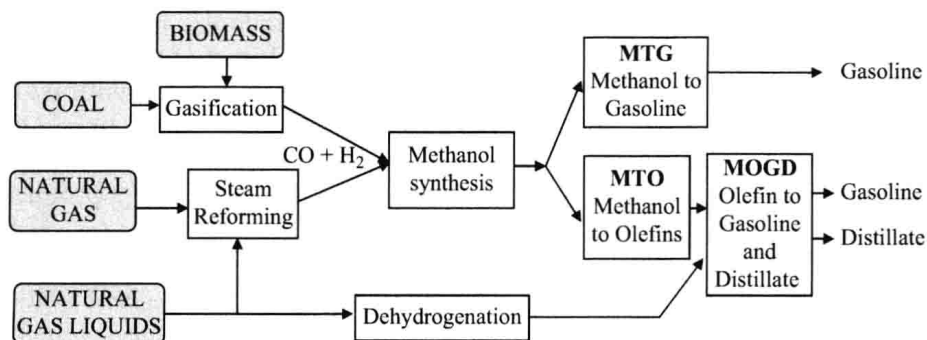


Figure 1.2 Routes to fuels and chemicals.

Routes using bio-renewable feedstocks that generate syngas can be used to interface with the need for the existing platform chemicals to be sustained. For the last 70 years the petrochemical and energy industries have focused their process technology on oil and hence huge processes have become entrenched for making key platform chemicals, *e.g.* ethene, propene, benzene, xylenes, from which many finished products are derived (Figure 1.2). There is therefore a desire to maintain this infrastructure and technology; after all it works very well. The initial new technologies based on methanol conversion, which sought to replace oil-derived approaches to chemicals and fuels,^{12,13} were also aimed at providing these platform chemicals, as can also be expected for the new coal-based Fischer–Tropsch processes. Hence there is a major driving force to convert everything to syngas and start from there. While technologically sound, such an approach loses all of the in-built complexity and functionality of bio-derived molecules. It has been the hallmark of the chemical industry for the last century to strip every initial feedstock down to, or almost to, its elemental components, thereby incurring massive energy penalties and potential loss of selectivity. For example, the synthesis of ammonia involves a catalytic pathway on an iron-based catalyst whereby the nitrogen–nitrogen triple bond is initially broken and then the atomic nitrogen hydrogenated. This should be contrasted with natural processes in which the bio-synthetic pathway to ammonia sequentially hydrogenates molecular nitrogen, requiring the final fission of a nitrogen–nitrogen single bond, and this represents a much lower energy pathway.¹⁴ It would be unfortunate if such a similar approach is now taken and the routes developed for the use of bio-renewable feedstocks are based on synthesis gas chemistry, because this would completely disregard all the chemical complexity intrinsic to the biological molecule.

An alternative to gasification and pyrolysis of biomass is fermentation. Using fermentation (see Figure 1.1) a broad range of carbohydrates are formed, principally sugars, as well as ethanol. The ethanol can be used as bio-renewable gasoline. A major feedstock produced from fermentation of biomass is glucose because, after all, cellulose is essentially polymeric glucose (see Figure 1.3). Glucose represents a highly functionalised molecule with a number of