# CRITICAL REVIEWS

*Ermelinda Falletta,*
*Cristina Della Pina*
*Dipartimento di Chimica Inorganica, Metallorganica*
*e Analitica "L. Malatesta"*
*Università di Milano, CNR-ISTM*
*ermelinda.falletta@unimi.it*

# GOLD NANOPARTICLES: THE POWER TO BE SMALL

**The present review reports some aspects of the catalysis by gold nanoparticles (preparation, methods and applications), discussing the properties of this catalytic system. The paper focuses on the oxidation reactions of some interesting starting materials as glycerol, glucose and amines.**

After the discovery of the extraordinary and surprising catalytic activity of gold nanoparticles (NPs), gold nanotechnologies have undergone a rapid growth [1]. In the last decade a lot of researchers directed their efforts towards new preparation methods of gold NPs and their applications in fundamental reactions for organic synthesis, especially oxidation and hydrogenation reactions [2]. Among them, Rossi's and Prati's research groups at Università di Milano have concentrated their attention in the selective oxidation of the alcoholic and aldehydic groups by using molecular oxygen in mild conditions [1].

Gold NPs-based catalysts allow to oxidize easily glycols to monocarboxylates [3], unsaturated alcohols to unsaturated aldehydes [4] and to hydrogenate unsaturated aldehydes and ketones to unsaturated alcohols [5] with selectivity up to 100%. Gold NPs find employment in a lot of important reactions as ethyne hydrochlorination [6], CO abatement [7], direct synthesis of hydrogen peroxide [8], carbohydrates oxidation [9], total combustion of volatile organic compounds (VOCs) [10-12], tertiary amines [13] and aminoalcohols [14] oxidation and many others.

Gold catalysts show exceptional properties as high selectivity, activity and durability, especially if compared to catalysts based on metals of platinum group. Beside these aspects, the biocompatibility, availability and possibility to work in "green conditions" make gold NPs environmental-friendly catalysts for sustainable processes.

In this review some peculiarities of gold catalysis are discussed, as the methods of preparation and some applications deriving from the experience recently matured in this field.

## Methods of preparation

Nowadays numerous methods exist for obtaining gold particles having nanometric dimensions. Overall, the most widespread preparation techniques include: co-precipitation [15], deposition-precipitation [16], vapour-phase deposition [17], co-sputtering [18], impregnation of phosphine complexes or clusters [19, 20] and deposition of colloidal gold (sol) on oxides and activated carbons [21].

### Co-precipitation method

A solution of $HAuCl_4$ and a metallic nitrate are added to a solution of $Na_2CO_3$. The co-precipitated products (hydroxides and carbonates) are washed, dried and calcined at T>250 °C.

This method can be used to prepare $Au/\alpha$-$Fe_2O_3$, $Au/Co_3O_4$, $Au/NiO$ and $Au/Be(OH)_2$ catalysts. In the presence of magnesium citrate this procedure can be followed also for the preparation of $Au/TiO_2$ catalyst.

### Deposition-precipitation

The pH value of a $HAuCl_4$ solution is increased to 6-10, then the support is added. After one hour $Au(OH)_3$ deposits on the surface of the support. This method can be used to deposit gold with high dispersion on MgO, $TiO_2$ and $Al_2O_3$. Every kind of support with a specific area (S.A.) >50 $m^2/g$ can be used. One of the most important parameter for this kind of preparation is the pH value. In fact, six species can be present in the solution $Au(OH)_nCl_{4-n}$ (n=0-4) and $Au(OH)_3$ that precipitates.

When the pH value is included between 6 and 10, the main species is $Au(OH)_3$. Because of the high pH values, this method cannot be used with acidic supports as $SiO_2$ or $SiO_2$-$Al_2O_3$.

Using carbon as the support, particles with 200 nm of diameter are obtained, probably due to the reductive carbon surface.

### Vapour-phase deposition

The vapours of an organic gold compound as dimethyl gold acetonate are adsorbed on a support previously evacuated and pyrolized. This method can be used for numerous supports with high specific area.

### Co-sputtering

Gold and a metal oxide in the atomic state are accelerated toward the vitreous support heated at 250 °C under oxygen at 0.4 Pa.
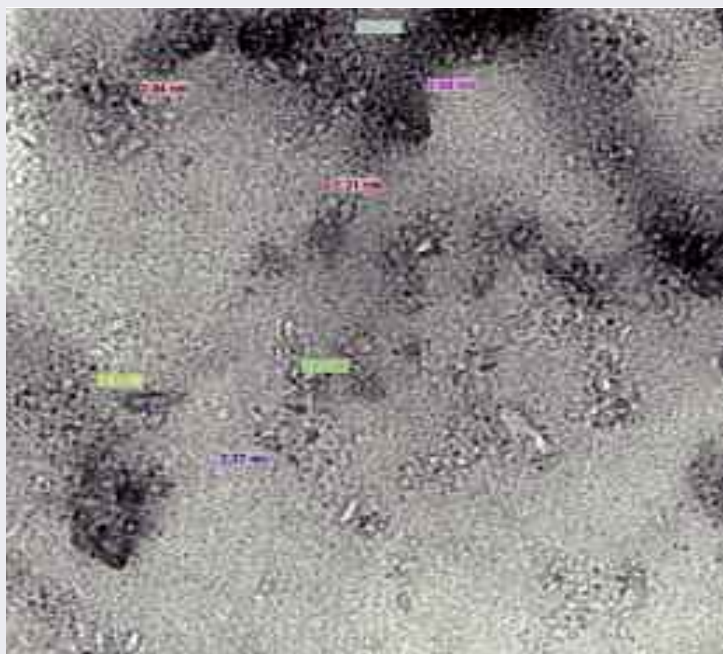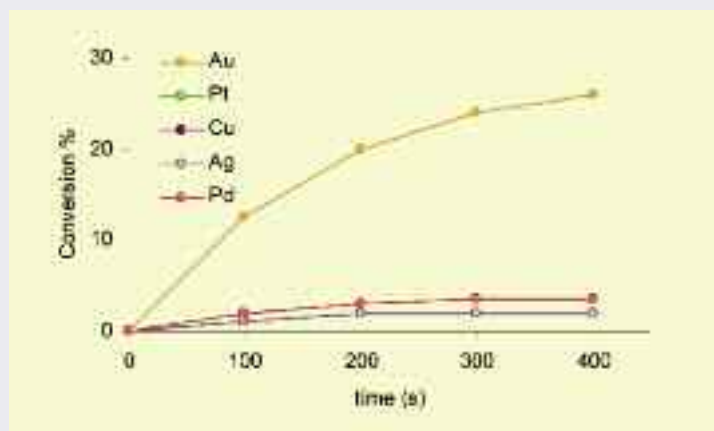


Fig. 1 - TEM image of 0.3% Au/C (X40S)



Fig. 2 - Comparison among colloidal metal catalysts in glucose oxidation

### Impregnation of phosphine complexes or clusters

Organometallic compounds are adsorbed on metal oxides and then are thermally decomposed.

Thermic or chemical (reduction) decompositions produce always metallic gold but the dispersion and the average diameter depend on the kind of support and treatment. The phosphinic ligands are particularly able to limit the growth of the metallic particles owing to the high tendency to bind metallic clusters.

### Deposition of colloidal gold (sol)

The advantage of this method consists in the possibility of controlling the nanoparticles size, before adsorbing them on the support. In this manner it is possible to obtain catalysts with specific characteristics.

This method allows to obtain particles with diameter between 1 and 20 nm. Small metallic particles are unstable in solution owing to agglomeration phenomena. In order to produce a stable colloidal dispersion, the reduction of a metal salt to the zero-oxidation state is performed in the presence of a protecting agent, which acts both as an electrostatic and/or steric stabilizer. The use of supporting agents can cause some problems. They could interfere in the reaction or, protecting the nanoparticles, prevent the contact of metal particles with the reagents.

Among these preparation methods, the latter one presents some advantages as the possibility of the nanoparticle-size control before the adsorption on the support.

Many methods are known to produce gold sols. Turkevich proposed the reduction by citrate ion [22], obtaining gold sols with an average diameter of 20 nm.

By experience, we can affirm that using $NaBH_4$ as a reductive agent it is possible to achieve smaller particles (average diameter 3 nm) (Fig. 1) [23].

However, despite the numerous methods of preparation, a lot of drawbacks are yet opened as the leaching of the metal at high pH values, the difficulty in maintaining small the average diameter of the gold NPs during the reaction and recycling, the ability to obtain a gold catalyst able to work at low pH values and in acidic conditions.
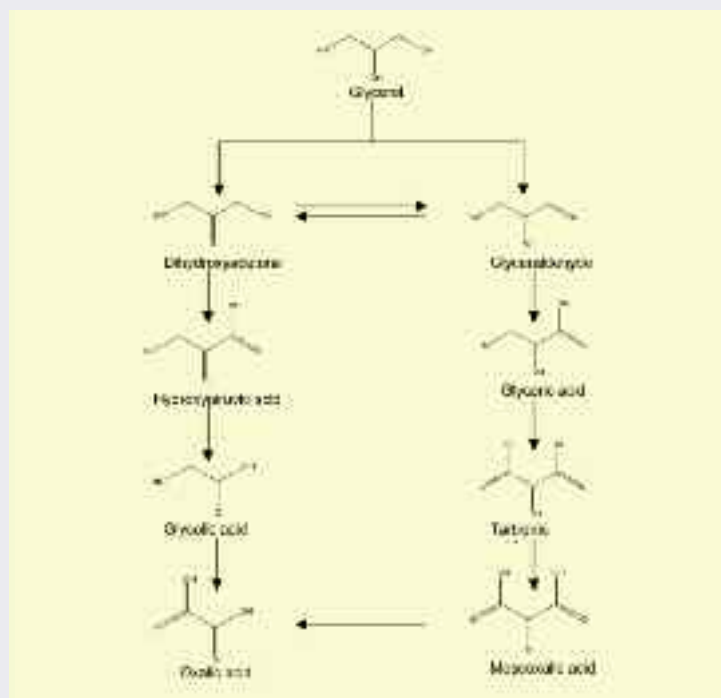
Scheme 1 - Reaction pathway for glycerol oxidation under basic conditions

### A brief comparison between gold nanoparticles and other metal catalysts

In comparison with other metal catalysts, especially Pt-metal catalysts, Au NPs show higher selectivity in the oxidation reactions. They distinguish among chemical groups and geometrical positions; for example, gold NPs oxidize alcoholic and aldehydic groups in the presence of double and triple bonds. In the reduction reactions, aldehydes and ketones are reduced to unsaturated alcohols. Fig. 2 shows a comparison among colloidal gold NPs and other colloidal metals in glucose oxidation. Gold nanoparticles show higher activity and selectivity than other metal catalysts.

Moreover, gold NPs do not undergo poisoning neither by amines, producing aminoacids from aminoalcohols (4.3.1.) and N-oxides from tertiary amines (4.3.2.), nor by molecular oxygen differently from Pt and Pd. However, gold NPs undergo a strong deactivation from sulphur compounds, which reduces their applications. Furthermore, the best performances of gold NPs have been observed at high pH values, producing carboxylates compounds instead of carboxylic acids. At the end of the reactions, the carboxylic acids are obtained by acidification of the reaction mixture containing the corresponding salts. This procedure causes the production of large amount of by-products that must be removed from the reaction mixture.

## Some applications

### Oxidation of alcohols and polyols

Alcohols and polyols are important starting materials for the production of numerous chemicals. They are economic and abundant products that can be obtained from natural and renewable sources.

Alcohols can be easily converted to the corresponding carbonylic or carboxylic derivatives which are strategic organic compounds. The possibility of oxidizing alcohols in water using molecular oxygen as the oxidant agent is an extraordinary conquest for the "green chemistry". The study on the liquid phase oxidation of the alcoholic group by gold catalysts started at Università di Milano at the end of the Nineties by Rossi's research group. The use of gold instead of palladium, platinum or copper in this kind of reactions allows to overcome some problems as metal leaching, low selectivity and deactivation. For all these reasons, together with the high chemoselectivity, gold is a good catalyst for the aqueous phase oxidation of alcohols. Among the most investigated alcohols, glycerol has recently known a growing interest due to its surplus as a consequence of biodiesel production, thus drastically dropping its price. Both industrial and academic worlds are searching for new applications for glycerine, which is already used in foods, cosmetics, detergents, pharmaceuticals, antifreeze and fine chemistry for the synthesis of a lot of important compounds. Scheme 1 shows the possible products of glycerol oxidation.

Glycerol oxidation is not an easy reaction at all, because of low selectivity and yield. That is why the market of the oxidized derivatives is not widely developed. The main oxidation products are dihydroxyacetone, used in tanning, glyceric acid, applied in medicine and in the synthesis of some aminoacids, and hydroxypiruvic acid, used in the synthesis of serine. Prati's research group at Università di Milano found the correlation between the NPs size and selectivity [24-27]. The catalytic behaviour greatly depends on the preparation method and on some parameters as the dispersion and the size of nanoparticles. Subsequently, Prati *et al.* showed that the bimetallic catalysts are more active than the corresponding monometallic systems. Activity and selectivity are influenced by numerous factors: particle size of the catalyst, kind of support, reaction conditions and, for the bimetallic systems, atomic ratio of the metals [28].

This exciting context infers a promising future application of gold NPs in glycerol oxidation on large scale, but until now no gold catalysed approach to this reaction has been applied in the industrial scale. The growing ability to obtain catalysts stable for long time in different conditions should hopefully open the way to this new, interesting application of gold NPs.

### Oxidation of aldehydes

Aldehydes can be easily converted to the corresponding carboxylated derivatives by catalysts based on the platinum group metals and carbohydrates, especially glucose, have attracted much interest.

Among these catalysts, gold-based ones show high activity and selectivity in water and they do not show deactivation on recycling.

### The case of glucose

Glucose is a cheap and renewable starting material for the production of chemicals (Scheme 2).

It is the constituent of starch, cellulose, sucrose and lactose. Glucose is industrially produced by enzymatic hydrolysis of starch from corn or biomass. The production of glucose from biomass is cheaper than corn and in the last years this kind of process has been improved.

Recently, because of the fast increase in petroleum cost, processes for the production of chemicals and new bioproducts by fermentation or chemical treatment of sugars have called researcher's attention. In some cases, however, only one route is applicable. No chemical processes are industrially applied for the production of gluconic acid and gluconates by glucose oxidation.

Owing to a low productivity of the glucose fermentation process and the difficulties in product and enzyme recovery, the use of clean and environmental friendly technologies is focusing a growing interest.

In the past years numerous efforts have been done to oxidize glucose using Pt-group metals as catalysts. In spite of high conversion and good selectivity, this kind of catalysts shows deactivation problems owing to leaching, self-poisoning and over-oxidation. In order to overcome these drawbacks, bi- and tri-metallic systems have been proposed. The best results have been obtained using Bi-based catalysts [29-30]. At present no industrial application of Pt-metal catalysts is known.

In our laboratories, Rossi firstly discovered the extraordinary ability of gold in promoting aerobic oxidation of glucose in aqueous solution under mild conditions [31]. In glucose oxidation gold catalysts show higher selectivity to sodium gluconates and higher resistance to poisoning than other catalysts. Great attention was addressed to the preparation of catalyst and experimental conditions obtaining TOF values up to 150,000 h$^{-1}$ at 50 °C. This exceptional TOF value is comparable with those of enzymatic systems [23, 32]. In order to define advantages and limits of gold catalysis in the aerobic glucose oxidation, a comparison was performed between a gold catalyst and a commercial enzyme, (Hyderase). The TOF value obtained in the enzymatic oxidation (600,000 h$^{-1}$) is higher than gold-catalysed oxidation one (90,000 h$^{-1}$). However, the achieved productivity is four times superior for the gold catalyst. Moreover, it is impossible to recycle Hyderase after the reaction owing to its solubility, whereas the solid gold catalyst can be easily filtered off for a new recycling. Fig. 3 reports the
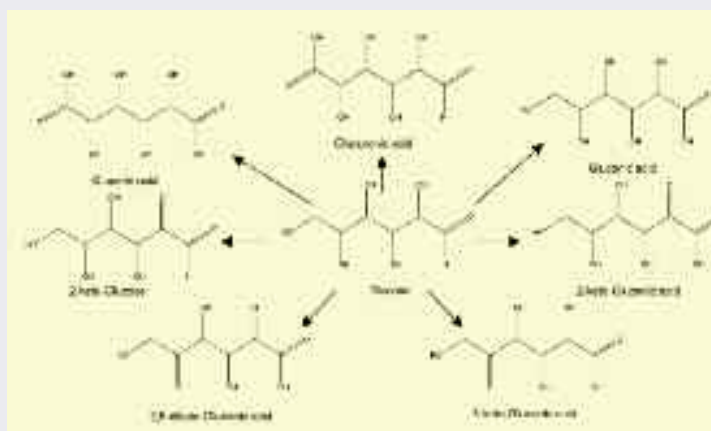
Scheme 2 - Chemicals obtained by glucose oxidation

results obtained after four recycling runs.

As showed in Fig. 3, the catalyst can be recycled four times maintaining a good conversion.

No direct method is known for the preparation of free gluconic acid. At present it is achieved by acidifying calcium gluconate with sulphuric acid and removing the by-product, $CaSO_4$. In fact, neither noble metal catalysts nor enzymes are able to produce free carboxylic acids at low pH. In order to overcome this difficulty, bi-metallic catalysts have been investigated and a strong synergistic effect between Au and Pt was observed using Au-Pt/C as a catalyst (Au:Pt = 2:1 w/w) as reported in Fig. 4.

Recently we have demonstrated that gold-catalysed aerobic oxidation of glucose occurs through a two-electrons mechanism leading to gluconate and hydrogen peroxide [33]. The possible role of $H_2O_2$ as a competitor has also been investigated. This study has demonstrated that gold catalyses the two-electrons reduction of dioxygen forming hydrogen peroxide. However, in the presence of alkali, $H_2O_2$ decomposes. Therefore, $H_2O_2$ lacks a sufficient concentration in the mixture of reaction able to oxidise glucose. Concerning the mechanism of molecular activation, Scheme 3 has been proposed.

Scheme 3 shows how the electron-rich gold species 1 activates molecular oxygen by nucleophilic attack. Intermediate 2 can subsequently evolve to produce gluconate, hydrogen peroxide and release the catalyst [33].

### Oxidation of amines

For long time the catalytic oxidation of amines using traditional metals has been studied without success, owing to the inhibition effect of the amino-group on this kind of metals. Gold catalysis partly overcomes the poisoning problem as reported in recent literature [13, 14, 34-36].

Lazar and Angelici report the reaction of isocyanides with primary amines and molecular oxygen to produce carbodiimide under mild conditions in the presence of bulk gold particles (about 1,000 nm) [34]. In another paper,
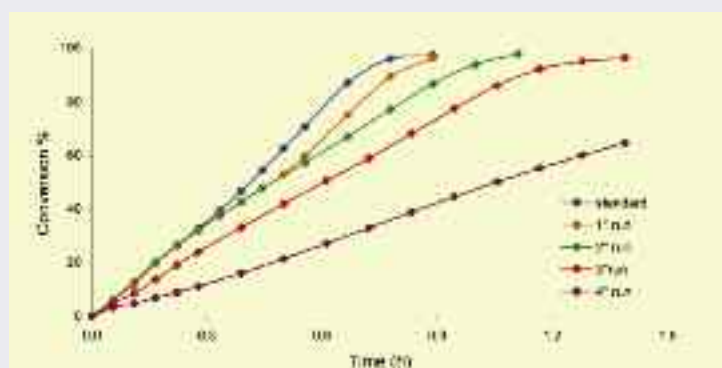
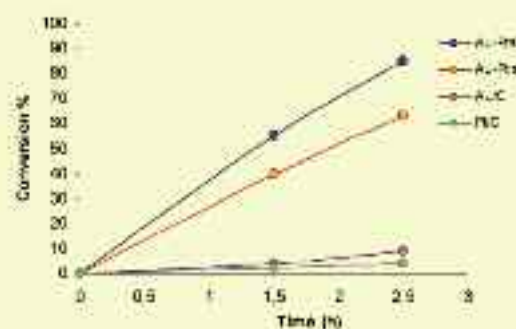Fig. 3 - Recycling runs for the 0.5% Au/C catalysed glucose oxidation

Fig. 4 - Comparison of catalytic activity of monometallic and bimetallic nanoparticles in the glucose oxidation to free gluconic acid

Zhu and Angelici indicate the preparation of ureas from carbon monoxide, primary amines and molecular oxygen in the presence of the same catalyst [35]. Secondary amines can be dehydrogenated to imines under mild conditions in the presence of bulk gold, while cyclic amines lead to oxidative dimerization by metal-catalyzed carbon-nitrogen coupling [36]. Among the different amines, aminoalcohols and tertiary amines represent two important starting materials for the production of fundamental chemicals.

### The case of aminoalcohols

Aminoacids are key constituents of many natural compounds as proteins. They are organic molecules containing in their structure two important groups: amino group and carboxylic group. Some of them are strategic materials for industry of foods and pharmaceuticals or as intermediates for the synthesis of chemicals.

In fact, glycine, sarcosine and N,N-dimethylglycine are important intermediates in organic chemical industry. They are mainly employed to produce surface-active agents. Sodium glutamate is used in food industry as a sapidity exalter. L-hydroxyphenylalanine (L-DOPA) is a drug applied in Parkinson's disease, 5-hydroxytriptophane (5-HTP) is used for the treatment of neurological symptoms associated to phenylketonurie, while $\gamma$-aminobutyrric acid (GABA) carries on many physiological functions (neurotransmission, hypotension, diuretic effects, soothing effect, prevention of diabetes) and it serves also as drug and integrator.

Presently, aminoacids are produced by amination of halo-acids [37] or by Strecker synthesis [38], which have a high environmental impact.

An alternative method for the synthesis of aminoacids is the selective oxidation of aminoalcohols. A bi-metallic Pt-Cu catalyst has been claimed to be active for the dehydrogenation of aminoalcohols [39], while Au/C catalyst has been shown to catalyse the oxidation of ethanolamines to aminocarboxylates in aqueous NaOH [40]. The presence of the basic amino group in aminoalcohols suggests the possibility of oxidising the alcoholic group in the absence of added alkali by means of gold catalysts. Rossi *et al.* have investigated the selective oxidation of aminoalcohols by noble metals and compared the obtained results using different noble metals as Au, Pd and Pt [14].

It has been observed that alkali promotes the oxidation rate and,



Scheme 3 - Mechanism of molecular activation of gold catalyst in the glucose oxidation in the presence of alkali

among the catalysts used for this reaction, 1% Au/C gives the best performances [14].

However, the catalytic activity strongly depends on the nature of the substrate and support used to prepare the catalysts. Therefore, the preparation of aminoalcohols needs drastic conditions and industrial applications are very far from applications.

### The case of tertiary amines

Hydrogen peroxide converts primary amines into nitro-compounds, whereas secondary amines are oxidised to hydroxylamines. Tertiary amines are converted by hydrogen peroxide into the corresponding hydrated N-oxides that are transformed to N-oxides by heating under vacuum. Aromatic amines can be oxidised by peroxodisulfuric and trifluoroperacetic acids to nitroderivatives [41].

In the last years the interest in the preparation of N-oxides has grown and several new preparation methods have been published using $H_2O_2$ as an oxidant in the presence of catalysts, like tungsten-containing compounds [42] and titanosilicates [43].

N-oxides are occupying an important place in the realm of synthetic and biological chemistry, because of their unique property to exist as stable free radicals. They are finding application as probes in materials [44], spin labels in biochemistry [45], magnetic resonance imaging [46], electron spin resonance imaging [47], in polymer synthesis [48] and more widely as metal free oxidants in organic synthesis [49].

In our laboratories we have investigated the aerobic oxidation of aqueous solutions of tertiary amines by nanometric gold-based mono- and bimetallic catalysts. In some cases we obtained N-oxides with 100% yield under mild conditions [13].

This appears as a new application of gold catalysis in organic synthesis and the possibility to obtain N-oxides under "green" conditions is a very exciting goal. However the most interesting N-oxides, used in detergent industry, are compounds with a long carbon chain. This kind of compounds is insoluble in water and until now we are not able to oxidize them. In the next future the development of new preparation methods of gold NPs, for example in organic solvents, and novel approaches for this kind of reaction could open further stimulating achievements.

## Conclusions

The observation that nanometric gold can be surprisingly active as a catalyst has fast revolutionized both the world of catalysis and the oxidative systems. Gold-catalyzed oxidation of alcohols and aldehydes, in particular, is such a successful way that has become a key topic. Catalysis by gold is now a maturing field and we are moving from the time of catalyst discovery, using relatively non-complex methods of synthesis, to more specific preparations and design, aided by detailed understanding of how nanoparticles and complexes can be prepared, supported by collaborations between material scientists and chemists, as well as the insights gained from model and theoretical studies. New methods are being refined and so this will fuel further progress towards the remaining challenges.
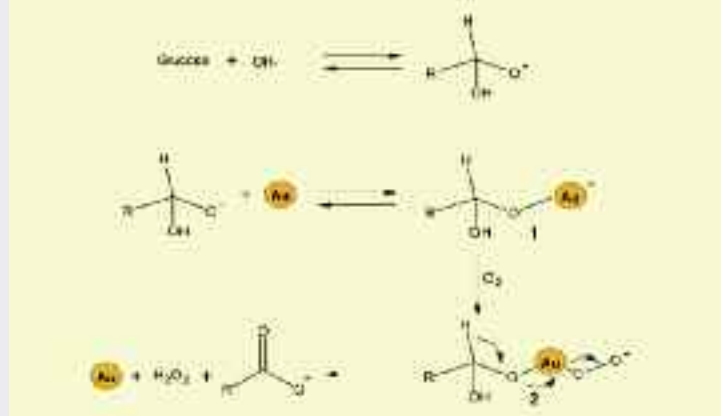
Ermelinda Falletta got her master degree in Chemistry (2004) at the Università di Palermo. In 2005 she joined Prof. Rossi's research group by a scholarship of CIMAINA at the Università di Milano. She is presently involved in the research of gold catalysis in the reactions of oxidation of organic compounds

Cristina Della Pina received the master degree at Università di Milano and awarded the Ph.D. degree in Industrial Chemistry in 2006.
Her work focuses on the development of heterogeneous catalysts.

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## ABSTRACT

### *Nanoparticelle di oro: il potere del piccolo*

*Questo articolo illustra alcuni aspetti e proprietà della catalisi mediante nanoparticelle di oro (preparazione, metodi e applicazioni). Il lavoro si focalizza sulle reazioni di ossidazione di alcuni materiali di partenza interessanti, come il glicerolo, il glucosio e alcune ammine.*

CRITICAL REVIEWS