RAPPORTO FINALE SUI RISULTATI DEL PROGETTO COMUNE DI RICERCA
FINAL REPORT ON RESULTS OF JOINT RESEARCH PROJECT

1. Accordo /Agreement

CNR / HAS (Ungheria)

anni/ years 2016-2018

2. Titolo del progetto
Catalizzatori bimetallici AuCu e AuAg supportati per l'ossidazione selettiva di alcoli (benzilico, glicerolo): effetto del rapporto Au/Cu(Ag), della struttura e del supporto

2. Title of the project
Supported bimetallic AuCu and AuAg catalysts in selective alcohol (benzyl alcohol, glycerol) oxidation reactions: Au/Cu(Ag) atomic ratio, Au/Cu(Ag) structure and support effects

Parole chiave (massimo 3)
AuAg catalyst, AuCu catalyst, alcohol oxidation
Key words (max. 3)
Catalizzatori AuAg, catalizzatori AuCu, ossidazione di alcoli

(solo per parte italiana)
Area scientifica / Scientific area (tabella 1/ table1)
Scienze chimiche e tecnologie dei materiali (4)

3. Responsabili del progetto
Project leaders

Responsabile italiano	**Hungarian project leader**
Claudio Evangelisti	Andrea Vargáné Beck
Istituto di appartenenza Istituto di Scienze e Tecnologie Molecolari (ISTM-CNR) Indirizzo Via Golgi 19, 20133 Milano	Affiliation MTA Centre for Energy Research, Institute for Energy Security and Environmental Safety , Department of Surface Chemistry and Catalysis Address 29-33 Konkoly Thege Miklós út,1121 Budapest, Hungary

4. Obiettivi del progetto

I gruppi di ricerca ungherese e italiano avevano un background scientifico molto simile sulla catalisi ma diverse competenze sui campi di applicazione. Pertanto, hanno condiviso molto facilmente la loro esperienza con beneficio per entrambi.
Gli obiettivi principali del progetto erano:
1) Sintesi di catalizzatori altamente selettivi e stabili per l'ossidazione del glicerolo verso prodotti di alto valore aggiunto, impiegando ossigeno molecolare come ossidante. Infatti, solo l'uso di un ossidante ecocompatibile può rendere i processi sostenibili e alternativi ai processi stechiometrici o enzimatici.
2) Stabilire una correlazione diretta tra la struttura e la composizione della particella bimetallica (Au-Ag, Au-Cu) e la loro attività / selettività in dipendenza del supporto e del substrato (glicerolo o alcol benzilico). Questo studio consente un'applicazione più ampia del catalizzatore e promuove la progettazione di nuovi materiali catalitici.
3) Stabilire legami a lungo termine tra CNR-ISTM, Italia e MTA, Ungheria per ulteriori estese collaborazioni internazionali che potrebbero portare a formulare idee di progetti di ricerca comuni e richiedere borse di studio internazionali o dell'UE.

4. Aims of the project

Hungarian and Italian teams had similar scientific background on catalysis but different expertise of application. Therefore, they shared very easily their experience with benefit for both of them.
The main task of the project was:
1) Synthesis of highly selective and durable catalysts for glycerol oxidation tailored for targeted high-value products, able to operate with oxygen as the oxidant. Indeed only the use of environmentally friendly oxidant can make the processes sustainable and alternative to stoichiometric or enzymatic processes.
2) To establish a direct correlation between the bimetallic particle (Au-Ag, Au-Cu) structures and composition and their activity/selectivity in dependence of the support and the substrate. This study allows a wider application of the catalyst and promote design of new, targeted catalytic materials.
3) To establish long-term linkages between CNR-ISTM, Italy and MTA, Hungary for further extensive international collaborations possibly leading to formulate common research project ideas and to apply for international or EU research grants.

5. Risultati ottenuti per obiettivo (1 pagina)

I risultati ottenuti possono essere riassunti come segue:

Obiettivo 1):

Catalizzatori di bimetallici AuAg altamente attivi e selettivi supportati su allumina sono stati sintetizzati da due approcci diversi: Solvated Metal Atom Deposition (SMAD) e Sol Immobilization (SOL). Entrambi i sistemi bimetallici SOL e SMAD hanno presentato un aumento sinergico dell'attività rispetto alle rispettivi sistemi monometallici nella reazione di ossidazione del glicerolo, impiegando ossigeno molecolare come ossidante. Sono state osservate forti differenze nella selettività dei prodotti di reazione. Il catalizzatore AuAg SOL è risultato più attivo di quello di AuAg SMAD e, cosa più interessante, il campione SOL ha mostrato una diversa selettività rispetto a quella monometallica essendo il glicerato ulteriormente ossidato a tartronato.

Obiettivo 2):

-Due diverse metodologie (Sol e SMAD) sono state applicate per la preparazione di due diverse serie di catalizzatori di Au depositati su tre tipi di supporti in carbonio, cioè Vulcan, Norit e X40S, caratterizzati da diverse strutture e funzionalizzazioni. La massima attività è stata osservata con i sistemi di Au/Vulcan. Tali campioni mostravano l'esposizione più bassa di Au e la più piccola dimensione delle particelle. Inoltre, il campione SOL su Vulcan ha mostrato una stabilità piuttosto buona nei successivi ricicli contrariamente al campione SMAD che ha subito una forte disattivazione dopo il primo ciclo di reazione.

- Catalizzatori AgAg depositati su allumina, con diversi rapporti molari AuAg, sono stati sintetizzati seguendo due diverse metodologie, ovvero l'immobilizzazione SOL e la tecnica SMAD. Le dimensioni delle particelle nei campioni bimetallici sono risultate leggermente più grandi di quelle nei corrispondenti monometallici Au, mentre le particelle di Ag nel sistema monometallico sono più grandi. Questi ultimi catalizzatori avevano un'attività trascurabile rispetto a quelli contenenti Au, ed entrambi i sistemi bimetallici SOL e SMAD presentavano un aumento sinergico dell'attività. La presenza di nanoparticelle bimetalliche è stata rivelata in entrambi i casi dalle caratterizzazioni TEM, XPS ed EXAFS che hanno rivelato la presenza di un core ricco di Au ed una shell esterna ricca di Ag. Analisi XAS ha dimostrato che nella preparazione SOL Ag copre solo parzialmente Au, mentre nel caso dei campioni SMAD l'arricchimento di Ag sulla superficie è molto più marcato rispetto ai sistemi SOL. Sono state rivelate forti differenze nell'ossidazione catalitica del glicerolo in termini di attività e selettività. Il catalizzatore AuAg SOL è più attivo di quello di AuAg SMAD e, cosa più interessante, il campione SOL ha mostrato una diversa selettività rispetto a quella monometallica essendo il glicerato ulteriormente ossidato a tartronato. Il campione calcinato ha mostrato una maggiore selettività al tartronato e un prodotto di scissione inferiore a C-C probabilmente a causa dell'aumento della dimensione delle particelle metalliche.

- Catalizzatori bimetallici di AuCu depositati su allumina e diversi tipi di carbonio come supporto (Au / Cu = 1/1, 4/1, 1/4) ed i relativi sistemi monometallici, sono stati preparati con l'approccio SOL. Negli spettri UV-Vis e l'analisi TEM ha confermato la formazione di particelle bimetalliche. È stato inoltre valutato il ruolo del supporto: Alumina e Carbon Vulcan XC-72 hanno mostrato le migliori prestazioni in termini di assorbimento. Tali catalizzatori bimetallici hanno mostrato un'elevata attività nell'ossidazione dell'alcol benzilico a benzaldeide (0,3 M, metallo / substrato 1/500, 4 bar O_2, 120°C). Nonostante l'attività trascurabile del Cu/C monometallico equivalente, abbiamo osservato un forte effetto sinergico positivo. Ulteriori caratterizzazioni e test catalitici sono attualmente in corso.

Obiettivo 3):

Durante il progetto abbiamo avuto l'opportunità di collaborare a livello internazionale, di visitare il laboratorio ungherese, di collaborare, discutere con i colleghi ungheresi sulla possibilità di ulteriori estese collaborazioni internazionali per formulare idee di progetti di ricerca comuni e di presentare domanda per la progetti di ricerca di cooperazione internazionale o UE. Con questa ottica, è stata creata una nuova collaborazione sul tema del progetto (catalizzatori bimetallici AuAg) con l'Universidad Nacional Autónoma de México, Centro di Scienza Applicata e Sviluppo Tecnologico (CCADET) (prof. Rodolfo Zanella).

5. Achieved results (one page)

The results achieved can be resumed as follows:

Aim 1):

Highly active and selective bimetallic AuAg catalysts supported on alumina for glycerol oxidation were synthesized by two different approaches: Solvated Metal Atom Deposition (SMAD) and the Sol Immobilization (SOL). Both the SOL and SMAD bimetallic systems presented synergetic activity increase compared to their monometallic counterparts in glycerol oxidation reaction using oxygen as the oxidant. Strong differences in product selectivity were observed: AuAg SOL is more active than AuAg SMAD one and, most interestingly, the SOL sample showed a different selectivity compared to the monometallic one being glycerate further oxidized to tartronate

Aim 2):

-Two different methodologies (Sol and SMAD) were applied for preparing two different series of Au catalysts supported on three types of carbon supports i.e. Vulcan, Norit and X40S, characterized by different structures and functionalization. We found that the highest activity was shown by AuNPs on Vulcan, samples that showed the lowest exposure of Au but the smallest particle size. Moreover, SOL sample on Vulcan showed a pretty good stability on recycling differently from the SMAD sample which underwent strong deactivation after the first catalytic cycle.

-AuAg catalysts on Al_2O_3, with different AuAg molar ratios, have been synthesized following two different methodologies, namely SOL immobilization and SMAD techniques. The particle sizes in the bimetallic samples were slightly larger than that in the corresponding monometallic Au ones, while the Ag particles in the monometallic silver ones were larger. The latter catalysts had negligible activity compared to the gold containing ones, and both the SOL and SMAD bimetallic systems presented synergetic activity increase. The presence of bimetallic nanoparticles has been revealed in both cases by TEM, XPS and EXAFS characterizations with Au-rich core and Ag-rich shell. XAS showed that in SOL preparation Ag only partially covers Au whereas in the case of SMAD the Ag enrichment on the surface is much larger than in the SOL case. Strong differences were revealed in the catalytic oxidation of glycerol in terms of activity and selectivity. AuAg SOL prepared is more active than AuAg SMAD one and, most interestingly, the SOL sample showed a different selectivity compared to the monometallic one being glycerate further oxidized to tartronate. The calcined sample showed a higher selectivity to tartronate and a lower to C-C cleavage product probably due to the enlargement of the metal particle size.

- Bimetallic AuCu catalysts deposited on alumina and different types of carbon as support (Au/Cu = 1/1, 4/1, 1/4) and the relative monometallic systems, were prepared by SOL approach. In the UV-Vis spectra and TEM analysis confirmed the formation of bimetallic particles. The role of the support was also evaluated: Alumina and Carbon Vulcan XC-72 showed the best performance in terms of absorption. Such bimetallic catalysts showed a high activity in the benzyl oxidation (0.3 M, metal / substrate 1/500, 4 bar O_2, 120°C) despite the negligible activity of the equivalent monometallic Cu/C, we observed a strong positive synergistic effect. Further characterizations and catalytic tests are currently understudy.

Aim 3):

During the project we had the opportunity to co-operate on international level, to visit the Hungarian laboratory, to collaborate, discuss with Hungarian colleagues on the possibility further extensive international collaborations possibly leading to formulate common research project ideas and to apply for international or EU research grants. In this frame, a new collaboration on the project topic (bimetallic AuAg catalysts) with Universidad Nacional Autónoma de México, Centre of Applied Science and Technologic Development (CCADET) (prof. Rodolfo Zanella) was established.

6. Prodotti del progetto / Results obtained

	n./no.
Pubblicaz. scient. su riviste internaz./ scientific publications on international reviews con IF 4 senza IF	4
Pubblicaz. in atti congressi internaz./ publications in international congress proceedings	4
Pubblicazioni in atti congressi nazionali / publications in national congress proceedings	
Pubblicazione libri nazionali / Publication of national books	
Pubblicazione libri internazionali / Publication of international books	
Altre pubblicazioni / other publications	
Brevetti / Patents	
Prototipi / Prototypes	
Strumentazione / Equipment and /or Devices	
Programmi software / Software	
Banche dati / Data bases	
Protocolli / Protocols	
Nuovi Materiali / New Materials	
Nuovi processi / New processes	
Cataloghi/inventari/repertori / Catalogues/Inventories	
Atlanti/Carte/Mappe / Atlases/Charts/Maps	
Progetti di ricerca / Reserch project	
Trasferimento innovazioni / Knowledge transfer	
Laboratori congiunti / Joint laboratories	
Alta formazione / Training	
Altro / Other	1

7. Informazioni dettagliate sui risultati indicati sub 6

La collaborazione in oggetto ha portato alla presentazione di dei seguenti contributi scientifici:

Articoli su riviste internazionali in lingua inglese:

1) A. Jouve, M. Stucchi, I. Barlocco, C. Evangelisti, F. Somodi, A. Villa, L. Prati, "*Carbon-Supported Au Nanoparticles: Catalytic Activity Ruled Out by Carbon Support*", Top. Catal. (2018), *in press*; DOI: 10.1007/s11244-018-1001-7.

Abstract
The catalytic oxidation of glycerol produces high added value chemical products. Gold-based catalysts showed activity and selectivity depending on particle size and specific preparation method. Moreover, the support plays a fundamental role in modulating the stability of the catalytic system. However, the literature is still lacking of a precise disclosure of these important relationships. Herein, we synthesized two series of gold catalysts on different carbon supports (Vulcan-XC72R, X40S and Norit GSX), the first synthesized by solvated metal atom deposition (SMAD) and the second by Sol Immobilization technique (SOL). First of all, the specific physico-chemical properties of the supports and the synthesis procedure influenced the dispersion and the size of Au NPs making a direct comparison among the different carbon difficult. In particular, on Vulcan-XC72R, AuNPs showed narrow size and good dispersion, whereas on Norit GSX and X40S-Camel a notably wider size distribution has been revealed. XPS analyses showed Au exposure changed accordingly to the presence of oxygen species, lower O content corresponding to lower Au exposure. Unexpectedly however this does not correspond to a lower activity, being the lowest Au/C (%at), the Vulcan ones, the most active catalysts. Comparing SOL and SMAD prepared samples on the same carbon, the SOL ones always result more active.

2) L. Prati, A. Villa, A. Jouve, A. Beck, C. Evangelisti, A. Savara, "*Gold as a modifier of metal nanoparticles: effect on structure and catalysis*" (2018), Faraday Discuss. (2018), 208, 395-407; DOI: 10.1039/c7fd00223h.

Abstract
Bimetallic gold based catalysts have been prepared by sol immobilisation technique. Despite a very similar metal dispersion different structures are revealed depending on the secon metal, being alloyed systems preferred in the case of Pd , Pt and Cu while core-shell in the case of Ru. A positive synergistic effect between the metals have been revealed only in the case of Pd and Cu in the oxidation of benzyl alcohol. AuPd/C has been also studied in the hydrogenation of benzaldehyde where the bimetallic catalyst revealed also a different selectivity compared to the monometallic counterpart.

3) J. Karolyi, M. Nemeth, C. Evangelisti, G. Safran, Z. Schay, A. Horvath, F. Somodi, F. "*Carbon dioxide reforming of methane over Ni-In/SiO2 catalyst without coke formation*", J. Ind. Eng. Chem. (2017), 58, 189-201; DOI: 10.1016/j.jiec.2017.09.024.

Abstract
It was found that coke formation on Ni/SiO2, used in dry reforming of methane, can be eliminated completely by addition of indium to the catalyst. The interaction of the two metals was suggested by the results of TPR, CO-TPD and XPS measurements. The presence and uniform distribution of NiIn and Ni_2In alloy nanoparticles after reduction at 700°C of the calcined catalyst was shown by HAADF-STEM, HRTEM and EDX analysis. The indium atoms dilute the nickel surface inhibiting the formation of multiplebonded carbon species thereby hindering the development of carbon nanostructures on the catalyst surface during dry reforming.

4) A. Jouve, G. Nagy, F. Somodi, C. Tiozzo, A. Villa, A. Balerna, A. Beck, C. Evangelisti, L. Prati, "*Gold-Silver Catalysts: Effect Of Catalyst Structure On The Selectivity Of Glycerol Oxidation*", J. Catal., *submitted, under review*.

Abstract
The real structure of bimetallic AuAg particles and its impact on their catalytic efficiency has been investigated. Glycerol oxidation has been chosen as reaction, due to the importance of glycerol as a platform molecule but also because of the complex reaction pathway which allows to disclose a correlation between structure and activity/selectivity. Alumina supported bimetallic AuAg catalysts have been fabricated by two different preparation methods, namely Solvated Metal Atom Deposition (SMAD) and the Sol Immobilization (SOL) which provided different distribution of the metals. A detailed characterization of the morphology and surface properties of the catalysts, have been performed by transmission electron microscopy techniques, X-ray photoelectron spectroscopy (XPS) and X-ray absorption spectroscopy (XAS). AuAg SOL prepared showed a higher activity than AuAg SMAD one probably due to a higher interaction between Au and Ag. Interestingly, the SOL sample showed a different selectivity compared to the monometallic one being glycerate further oxidized to tartronate.

Comunicazioni a convegni nazionali ed internazionali:

1) A. Jouve, C. Evangelisti, A. Villa, L. Prati, "Gold-Silver catalysts for glycerol oxidation" (poster), winter school "Green Catalysis by Design" Scientific Meeting, 22-23 February 2017, Padova.

2) L. Prati, C. Evangelisti, A. Villa, A. Jouve, C. Tiozzo, G. Nagy, F. Somodi, G. Sáfrán, A. Beck, "*Gold-Silver catalysts: effect of catalyst structure in the selectivity of glycerol oxidation*" (oral presentation), 13th European Congress on Catalysis – EuropaCat-XIII, Florence, Italy, 27-31 August 2017, Abstract Book, chapter 3, p. 38-39.

3) G. Nagy, F. Somodi, G. Sáfrán, Z. Schay, T. Gál, A. Beck, L. Prati, C. Evangelisti, C. Tiozzo, "*Sol derived alumina and silica supported Au-Ag bimetallic catalysts: structure and activity in aerobic selective oxidation of benzyl alcohol*" (poster presentation), 33rd European Conference on Surface Science, Szeged, Hungary, 27 August – 1 September (2017) Abstract Book, p. 326

4) M. Marelli, A. Jouve, A. Villa, L. Prati, V. Dal Santo, A. Balerna, C. Evangelisti , "Structural *characterization of carbon-supported bimetallic AuCu nanoparticles derived from metal vapors*" (Oral Communication), MCM17, Rovinj (HR) - September 24-29, (2017) Abstract Book p. 583.

7. Detailed information on results indicated under point 6

The bilateral cooperation led to the presentation of the following scientific contributions:

Publications on International ISI Journals:

1) A. Jouve, M. Stucchi, I. Barlocco, C. Evangelisti, F. Somodi, A. Villa, L. Prati, "*Carbon-Supported Au Nanoparticles: Catalytic Activity Ruled Out by Carbon Support*", Top. Catal. (2018), *in press*; DOI: 10.1007/s11244-018-1001-7.

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because of the complex reaction pathway which allows to disclose a correlation between structure and activity/selectivity. Alumina supported bimetallic AuAg catalysts have been fabricated by two different preparation methods, namely Solvated Metal Atom Deposition (SMAD) and the Sol Immobilization (SOL) which provided different distribution of the metals. A detailed characterization of the morphology and surface properties of the catalysts, have been performed by transmission electron microscopy techniques, X-ray photoelectron spectroscopy (XPS) and X-ray absorption spectroscopy (XAS). AuAg SOL prepared showed a higher activity than AuAg SMAD one probably due to a higher interaction between Au and Ag. Interestingly, the SOL sample showed a different selectivity compared to the monometallic one being glycerate further oxidized to tartronate.

Communications to National and International Conferences:

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8. Formazione di giovani ricercatori

Nel gruppo di ricerca italiano uno studente di dottorato (Dr. Andrea Jouve) ha lavorato a tempo pieno su questo progetto mentre un post-doc è stato parzialmente coinvolto (Dr.ssa Cristina Tiozzo, 1 anno). Entrambi sono stati coinvolti in tutte le parti sperimentali del progetto, come preparazione, caratterizzazione e test catalitici. All'interno del progetto hanno avuto l'opportunità di cooperare a livello internazionale, visitare il laboratorio ungherese, collaborare, discutere con i colleghi ungheresi.
Dal lato ungherese, uno studente di dottorato (Gergely Nagy) è stato coinvolto nell'intero argomento (compresi gli studi catalitici sull'ossidazione del alcol benzilico, la preparazione del catalizzatore, la caratterizzazione mediante UV-vis e la spettroscopia DRIFT). Un secondo studente (Johanna Károlyi) ha partecipato alle misurazioni TPO-TPR.

8. Training of young researchers

In the Italian team, one PhD student (Dr. Andrea Jouve) was full time involved on this project while one Post-Doc was partially involved (Dr. Cristina Tiozzo,1 year). They were involved in all the projects parts such as preparation, characterization and catalytic tests. They had the opportunity to co-operate on international level, to visit the Hungarian laboratory, to collaborate, discuss with Hungarian colleagues.
From Hungarian side one PhD student (Gergely Nagy) was involved in the entire subject (including benzyl-alcohol oxidation catalytic studies, catalyst preparation, characterization by UV-vis and DRIFT spectroscopy). Another one (Johanna Károlyi) participated to TPO-TPR measurements.

9. Motivazione degli sviluppi della collaborazione negli anni successivi

(eventuali estensione ad altri paesi, collaborazioni multilaterali, contratti nazionali o internazionali)

La collaborazione e la conoscenza delle reciproche infrastrutture, capacità, specialità e l'approccio alla ricerca ha assicurato le basi per formulare idee di progetti di ricerca comuni e richiedere sovvenzioni di ricerca internazionali o dell'UE e per estendere la nostra cooperazione ad una rete più ampia che utilizzi entrambe le nostre relazioni nazionali e internazionali. In questa prospettiva, grazie alle nostre visite al laboratorio ungherese, abbiamo avuto la possibilità di aggiungere nuovi partecipanti alla nuova proposta. Questo consentirà, come previsto nel nuovo progetto di ricerca, di investigare la morfologia dei materiali preparati a livello atomico, grazie ad analisi effettuate mediante un microscopio elettronico in trasmissione con aberrazione corretta, acquisito recentemente dal gruppo ungherese.

9. Reasons for cooperative project developments in the following years, if any

(extension to other countries, multilateral collaboration, national or international contracts

The collaboration, learning each other's infrastructure, capabilities, specialities, research approach in more detailed way laid to foundation for the formulation of common research project ideas and to apply for international or EU research grants for extension of our co-operation maybe in a wider network utilizing both our own national and international relations. In this view, during our visits to Hungarian laboratory, we had the possibility to add new participants taking part to the new proposal. This will allow, as envisaged in the new research project, to analyze the morphology of materials at the atomic level, by analysis carried out with an aberration-corrected transmission electron microscope, recently acquired by the Hungarian group.

(firma del responsabile italiano del progetto)

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date: Milano 11.09.2018

TABELLA 1

1	Scienze del sistema Terra e tecnologie per l'ambiente
2	Scienze bio-agroalimentari
3	Scienze biomediche
4 X	Scienze chimiche e tecnologie dei materiali
5	Scienze fisiche e tecnologie della materia
6	Ingegneria, ICT e tecnologie per l'energia e i trasporti
7	Scienze umane e sociali, patrimonio culturale