ELECTROCHEMISTRY WITH ULTRASOUND

University of Coventry. UK
University of Oxford. UK
University of Southampton. UK
Academy of Sciences of the Czech Republic. Czech Republic
University of Franché-Comte. France
University of Alicante. Spain
ELECTROCHEMISTRY WITH ULTRASOUND

Environmental applications
* Improved strategies for waste minimisation:
  - obviation of environmentally unfriendly systems in synthesis
  - sono-electrochemical reactor design
* Degradation of pollutants and enhanced environmental clean-up using sono-electrochemistry

New systems
* Novel electrosynthesis reactions with applications in organic and biochemistry
* Novel functional materials and their practical applications, including nanoparticles and conducting polymers.
* Development of new electrode materials and the understanding of surface processes in these processes

Technological applications
* Improved methods for electrodeposition, electrodissolution, including effects on morphology, hardness, microstructure...
* Scale-up from micro-scale to pilot-plant scale

Electroanalysis
* Development of enhanced electroanalytical procedures that are effective in real media, leading to improved sensors and biosensors
* Sensitive electroanalyses for metal ions and other deleterious electroactive species in the environment
WORK PROGRAMME FOR THIS TERM

Events

Kick off COST D-32 Meeting, held in Alicante, July 2004.

Kick off Working Group Meeting, held in Alicante, December 2004.

Annual Working Group Meeting, held in Prague, November 2005.

Annual Working Group Meeting, held in Hamburg, June 2006.

Annual Working Group Meeting, held in Madrid, September 2007.

Tasks

**General task:** Finding and development of common research interests by means of:
- Research interactions
- Short term scientific missions

**Specific tasks:**
- Sonoelectrochemical reactor design
- Degradation of pollutants using sonoelectrochemistry
- Development of new electrode materials
- Sensitive electroanalyses for metal ions
ELECTROCHEMISTRY WITH ULTRASOUND

*University of Alicante: Grupo de Electroquímica Aplicada y Electrocatálisis. Departamento de Química Física e Instituto de Electroquímica. Spain
  Modified electrodes for Environmental Clean up and Remediation. Scale-up

*University of Oxford: Physical and Theoretical Chemistry Laboratory. UK
  Sonoelectrochemical mechanisms and analyses, fundamental studies, polyphasic systems

*University of Coventry: School of Science and the Environment. UK
  Electroorganic synthesis, Functional Materials, conducting polymers, bioelectrochemistry

*University of Franche-Comté: Laboratoire de Chimie des Matériaux et des Interfaces. Site de l’IUT. Département Chimie. France
  Sonoelectrochemistry, corrosion, electroplating, electrode surface studies, scale-up

*Ecole Normale Supérieure: Département de Chimie. Paris. France
  High-speed studies, kinetics and mechanistic elucidation

*Academy of Sciences of the Czech Republic: J. Heyrovsky Institute of Physical Chemistry. Czech Republic
  Physical aspect of sonoelectrochemistry, molecular electrochemistry

*University of Southampton: Department of Chemistry. UK
  Sonoelectrochemical mechanisms and analyses, fundamental studies
Working group meetings

Kick off COST D-32 Meeting, held in Alicante, July 2004.
Kick off Working Group Meeting, held in Alicante, December 2004.
Annual Working Group Meeting, held in Prague, November 2005.
Annual Working Group Meeting, held in Hamburg, June 2006.
Annual Working Group Meeting, held in Madrid, September 2007.

Participations in another WG Meetings

“Physical aspects of Sono(electro)chemistry: Distribution of intensity of ultrasound”
J. Klíma, A. Frias-Ferrer, J. González-García, J. Ludvík, V. Sáez, J. Iniesta
COST WG 2+WG7 Workshop 9-10 June 2005, Oxford, UK
Meetings (WG labs met up)

Talks

1. “Contribution of electrochemistry to the study of power ultrasound applied to surfaces”
J.Y. Hihn (WG4)
ESS9th Conference April 2004. Badajoz. Spain

2. “Effects of ultrasonic irradiation on electroless coating and electroplating”
F. Touyeras, J.Y. Hihn, X. Bourgoin, B. Jacques, L. Hallez (WG4)
ESS9th Conference April 2004. Badajoz. Spain

3. “New reactor design for sonochemical applications”
R. Viennet, J.Y. Hihn, M. Jeannot, R. Berriet (WG4)
ESS9th Conference April 2004. Badajoz. Spain

4. “Characterization of a 20 kHz Sonoreactor. Part I: Analysis of Machanical effects by classical and numerical methods”
V. Sáez, A. Frías-Ferrer, J. Iniesta, J. González-García, A. Aldaz, E. Riera (WG4+WG11)
ESS9th Conference April 2004. Badajoz. Spain
General Meetings (WG labs met up)

5. “The Effect of Ultrasound on the voltammetry of Copper in strong alkali”
D. J. Walton, M. Plattes, T. J. Mason, J. P. Lorimer (WG4+WG2)
ESS9th Conference April 2004. Badajoz. Spain

Posters

1. “Visualisation of the actives zones near the transducer in an ultrasonic reactor using 40 kHz frequency”
A. Mandroyan, R. Viennet, Y. Bailly, L. Girardot, M. L. Douche, J.Y. Hihn, P. Nika (WG4)
ESS9th Conference April 2004. Badajoz. Spain

2. “Hydrodynamic and mass transfer measurements close to an ultrasonic horn vibrating at two frequencies”
A. Mandroyan, M.-L. Doche, J.Y. Hihn, R. Viennet (WG4)
ESS9th Conference April 2004. Badajoz. Spain

3. “Characterization of a 20 kHz Sonoreactor. Part II: Analysis of chemical effects by classical and electrochemical methods”
V. Sáez, A. Frías-Ferrer, J. Iniesta, J. González-García, A. Aldaz, E. Riera (WG4+WG11)
ESS9th Conference April 2004. Badajoz. Spain
General Meetings (WG labs met up)

4. “Ultrasound effects on the kinetics reaction step in the silver thiosulphate reduction on platinum electrodes”
B. Pullet, J. P. Lorimer, J.Y. Hihn, F. Touyeras, T. J. Mason (WG4+WG2)
ESS9th Conference April 2004. Badajoz. Spain
MEETINGS/5

General Meetings-internal WG contributions

Oral presentations/1

1. “Aplicación del método de salto de temperaturas inducido mediante irradiación con láser. Electrodomonocristalinos de oro y platino”
   V. Climent, R. G. Compton, J. M. Feliu (Alicante+Oxford)
   VII Iberic Meeting of Electrochemistry and XXVI Reunión del grupo de Electroquímica de la RSEQ. 2004. Córdoba. Spain

   V. Climent, R. G. Compton, J. M. Feliu, A. Aldaz (Alicante+Oxford)

3. “Sono electrochemical degradation of perchloroethylene at 850 kHz”
   V. Sáez, J. Iniesta, A. Frías-Ferrer, J. González-García and D. J. Walton (Alicante+Coventry)
   Electrochem 2005, Newcastle, UK

4. “Structure function studies on Electrosynthetically-modified Proteins-Nitration of tyrosine”
   XVIIIth International Symposium on Bioelectrochemistry and Bioenergetics. 3rd Spring Meeting. Bioelectrochemistry 2005 Coimbra. Portugal
5. “Energetic balance in an ultrasonic reactor using focused or plan high frequency transducers”
L. Hallez, F. Touyeras, J. Y. Hihn, J. Klima (Besançon+Prague)
ESS10th Conference June 2006. Hamburg, Germany

6. “Optimisation of Sonochemical and/or Sonoelectrochemical cell with the help of numerical simulation of ultrasonic intensity distribution”
J. Klima, J. González-García, A. Frías-Ferrer, J. Ludvík, V. Sáez, J. Iniesta (Alicante+Prague)
ESS10th Conference June 2006. Hamburg, Germany

7. “Electrochemical synthesis of hydrogen peroxide assisted by ultrasound”
J. González-García, C. E. Banks, B. Šljukić, R. G. Compton (Alicante+Oxford)
ESS10th Conference June 2006. Hamburg, Germany
MEETINGS/7

General Meetings-internal WG contributions

Oral presentations/2

8. “Small scale preparative electrolysis in sonoelectrochemical cell optimized with the help of simulation of ultrasonic intensity distribution”
   J. Klima, J. Ludvik, J. Urban, J. González-García, V. Sáez, A. Frías-Ferrer (Alicante+Prague)

9. “Electrochemistry with ultrasound: state of the research in the field”
   V. Sáez, P. Bonete, M. D. Esclapez, J. Iniesta, J. González-García, D. Walton
   (Alicante+Coventry)

10. “Degradation of chloroethenes in aqueous solution by ultrasound”
    V. Sáez, M. D. Esclapez, P. Bonete, E. Marchante, J. González-García, D. Walton; O.
    Louisnard (Alicante+Coventry)
    11th Meeting of the European Society of Sonochemistry, June 2008. La Grande-Motte. France

11. “Sonoelectrochemistry: microjets, microstreaming or acoustics streaming”
    J. Klima, J. González-García, O. Louisnard (Alicante+Prague)
    11th Meeting of the European Society of Sonochemistry, June 2008. La Grande-Motte. France
MEETINGS/8

General Meetings-internal and external WG contributions

Oral presentations/1

1. “Comparison of the chemistry of tetracyclones and tetraarylthiophenes-S-oxides”
   A. Miura, F. Matasaka, J. Iniesta, D. Walton, S. Mataka, T. Thiemann

2. “The chemistry of thiophene-S-oxides and related compounds”
   T. Thiemann, D. J. Walton, A. Oliveira Brett, J. Iniesta, F. Marken, Y.Q. Li. (WG4(2)+WG5+WG6)
   Kyushu International Symposium on Physical Organic Chemistry, KISPOC-XI.
   2005, Fukuoka, Japan

3. “Photochemical and electrochemical reactions of sulphoxide, tiophenes, benzo and dibenzotriophenes: applications in new processes of photochemical synthesis and desulforation”
   J. Iniesta, M. D. Esclapez, V. Sáez, P. Bonete, J. González-García, D. Walton, T. Thiemann, T.
   Matsumoto, Y. Q. Li
   XXVIII Real Sociedad Española de Química. 2006 La Coruña (Spain) (WG4(6)+WG6)

4. “Sonoelectrochemical deposition of catalytically active iron metal at boron doped diamond electrodes: application to electroreduction of chloroacetates”
   V. Sáez, J. González-García, M. Anbu Kulaandainathan, Frank Marken
General Meetings-internal and external WG contributions

Poster presentations/1

1. “Mass transfer measurements in small electrode-ultrasonic horn distances”
B. G. Pollet, J. Y. Hihn, M.-L. Doche, A. Mandroyan, J. P. Lorimer, T. J. Mason (WG4+WG2)
ESS10th Conference June 2006. Hamburg, Germany

2. “Sonochemical degradation of volatile and non-volatile chlorinated compounds in aqueous solutions”
M. D. Esclapez, V. Sáez, P. Bonete, J. González-García, A. Fakouri, M. Schulz and A. Rehorek
(WG4(4)+WG2(3))
11th Meeting of the European Society of Sonochemistry, June 2008. La Grande-Motte. France
General Meetings (Individual labs contributions)

Oral presentations/1

1. “Characterization of bubble cavitation clouds by laser tomography”
A. Mandroyan, R. Viennet, Y. Bailly, M.-L. Doche, J. Y. Hihn (Besançon)
ESS10th Conference June 2006. Hamburg, Germany
General Meetings (Individual labs contributions)
Poster presentations/1

1. "Electrochemical measurements of a flow induced by low frequencies ultrasound. Influence of horn diameter and cell design”
M. –L. Doche, A. Mandroyan, J. Y. Hihn, R. Viennet (Besançon)
ESS10th Conference June 2006. Hamburg. Germany

2. "Effect of ultrasonic wave on the electropolymerization of conducting polymer films”
F. Lallemand, J. Y. Hihn, L. Hallez, F. Touyeras (Besançon)
ESS10th Conference June 2006. Hamburg. Germany

3. "Mapping flow velocities in an ultrasonic reactor working at 3 frequencies: 20, 40 and 60 kHz”
A. Mandroyan, R. Vinnet, Y. Bailly, L. Simonin, M.-L. Doche, J. Y. Hihn (Besançon)
ESS10th Conference June 2006. Hamburg. Germany
General Meetings (Individual labs contributions)

Poster presentations/2

4. “Effect of low frequency ultrasound irradiation on room temperature ionic liquid (RTIL’s) dehydration”
C. Costa, J. Y. Hihn, I. Bisel, P. Moisy, M. Rebetez, M. -L. Doche (Besançon)
ESS10th Conference June 2006. Hamburg, Germany

5. “500 kHz ultrasonic irradiation of a polymeric membrane during tangential filtration”
L. Hallez, H. Abourabia, F. Touyeras, J. Y. Hihn, P. Fievet, A. Szymczyk (Besançon)
ESS10th Conference June 2006. Hamburg, Germany

6. “Effect of spatiotemporal characterization of cavitation bubbles collapse nearby ultramicroelectrodes by means of chronoamperometry”
A. Mandroyan, M.-L. Doche, J. Y. Hihn, D. Chappe, J.M. Pothier (Besançon)
ESS10th Conference June 2006. Hamburg, Germany

7. “Sonoelectrochemical degradation of perchloroethylene in aqueous solutions”
V. Sáez, J. Iniesta, A. Frías-Ferrer, P. Bonete, J. González-García (Alicante)
ESS10th Conference June 2006. Hamburg, Germany
General Meetings (Special Session organization in International Congress)

1. Structured session “Sonoelectrochemical processes”
   Chairman: José González García and D. Walton

2. Special session “Sonoelectrochemistry”
   Chairman: José González García
   International Congress of Ultrasonics. Santiago de Chile (Chile) 11-17 January 2009.
Internal research interactions/1

**Financed by COST via STSM**

*Short term scientific mission STSM (financed by COST) one month.*
Verónica Sáez (University of Alicante) in Coventry University
Topic: Sono(electro)chemical degradation of PCE at 850 kHz.
Identification of final products. Comparison with 20 kHz results from Alicante

1016 euros

*Short term scientific mission STSM (financed by COST) two weeks*
Jiri Klima (J. Heyrovsky Institute of Physical Chemistry) in University of Alicante
Topic: Numerical simulation of the US intensity distribution
Optimized geometry. First results to be used as basis for further studies

1200 euros
Internal research interactions/2

- **Financed by COST via STSM**
  
  *Short term scientific mission STSM (financed by COST) one week.  
  Emmanuel Maisonhaute (University of Paris) in Alicante University  
  Topic: Ultrafast electrochemistry on Molecular wires.  
  First results to be used as basis for further studies  

  
  *Short term scientific mission STSM (financed by COST) two weeks  
  Jiri Klima (J. Heyrovsky Institute of Physical Chemistry) in University of Franché-Comte  
  Topic: Sonochemiluminiscence at the focal point of a HIFU transducer as a function of the transmitted power.  

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  *Short term scientific mission STSM (financed by COST) one week.  
  José González García (University of Alicante) in Coventry University  
  Topic: Degradation of chlorinated organocompounds by sonochemical methods at high frequency  
  Checking test for the experimental set-up. Planning work for future collaboration
Internal research interactions / 2

External financial support

* Mobility grant (financed by Ministerio Educación y Ciencia (Spain)) one year. 28400 euros
  José González-García (University of Alicante) in University of Oxford
  Topic: Electrochemical synthesis of hydrogen peroxide assisted by ultrasound
  Fundamental aspects and scale up to a 1000 W sono-electrochemical reactor

* Mobility grant (financed by Generalidad Valenciana) two months. 2500 euros
  Victor Climent (University of Alicante) in Ecole Normale Supérieure (Paris)
  Topic: Fabrication of platinum and gold ultramicroelectrodes with well-defined surface structures.
  Gold and platinum ultramicroelectrodes with (111) surface orientation were fabricated and applied to study the kinetics of fast electron transfer reactions

* Research visit (financed by Université de Franché-Comté, Besançon) two weeks 500 euros
  A. Mandroyan (Université de Franché-Comté, Besançon) in Ecole Normale Supérieure (Paris)
  Topic: High speed voltammetry on Ultramicroelectrodes.
  Comparison of results with the ultrasonic generators form Besançon at 3 frequencies (20, 40 and 60 kHz). Better comprehension of asymmetric cavitation
External (with other D32 labs) research interactions/1

Financed by COST via STSM

*Short term scientific mission STSM (financed by COST) two weeks
D. Bogdal (Politechnika Krakowska, Poland) (WG10) in University of Coventry
Topic: Sonoelectrochemical studies on polycarbazoles
Polycarbazole films on indium-tin oxide coated (ITO) glass

1720 euros

*Short term scientific mission STSM (financed by COST) three months 2006
V. Sáez (University of Alicante, Spain) in University of Bath (WG5)
Topic: “Detection and degradation of tetrachloroethylene and triclosan by sonoelectrochemical methods”
Comparison between results obtained with tetrachloroethylene and triclosan

???? euros

*Short term scientific mission STSM (financed by COST) three months 2008
M. D. Esclapez (University of Alicante, Spain) in University of Cologne (WG2)
Topic: Degradation of chlorinated organocompounds by sonochemical methods at high frequency”
Comparison between results obtained with PCE and TCAA

???? euros
RESEARCH INTERACTIONS /5

External (with other D32 labs) research interactions/1

External financial support

Research visit (financed by EPSRC (UK)). six months. 7500 euros
D. Walton (University of Coventry) in University of Kyushu
Topic: Synthesis of new dibenzothiophene derivatives
Obtention of initial products

Research visit (financed by Ministerio Educación y Ciencia (Spain)). One month. 1500 euros
Jesús Iniesta Valcárcel (University of Alicante) in University of Kyushu
Topic: Synthesis of new dibenzothiophene derivatives
Obtention of initial products
EXTERNAL FINANCIAL SUPPORT/1

Recently completed projects

1. School of Science and the Environment. University of Coventry, United Kingdom
   “Electrochemical and photochemical reactions of thiophene-S-oxides”. D. J. Walton
   Financed by EPSRC Postdoctoral Assistantship. Dr. J. Iniesta
   82500 euros

2. Department of Physical Chemistry. University of Alicante
   “Sonoelectrochemical degradation of chlorinated organocompounds”. J. González-García
   financed by University of Alicante. Spain
   16200 euros

3. School of Science and the Environment. University of Coventry. United Kingdom
   “The electrosynthetic modification of bioactive species”. Professor D. J. Walton
   (two years: 01.09.2001-31.08.2003)
   Financed by EU. Marie Curie Fellowship: Dr. J. Iniesta
   120000 euros
EXTERNAL FINANCIAL SUPPORT/2

Individual WG004 members ongoing research projects

1. Laboratory of Molecular Electrochemistry, Department of Electrochemistry, Prague
   “Sonoelectrochemistry-physical mechanism and application in molecular electrochemistry”.
   Ministry of Education, Youth and Sports of the Czech Republic
   47000 euros

2. Laboratory of Molecular Electrochemistry, Department of Electrochemistry, Prague
   “Sonoelectrochemistry-physical mechanism and application in molecular electrochemistry”.
   J. Heyrovsky Institute of Physical Chemistry, Czech Republic
   88000 euros

3. Department of Physical Chemistry, University of Alicante
   “Design and characterization of sonoelectrochemical reactors”.
   Ministry of Industry, University and Science, Comunidad Valenciana, Spain
   33539 euros

4. School of Science and the Environment, University of Coventry
   “Grant support for COST D32 Working Group 004”.
   (three years: 01.11.2005-31.10.2008).
   EPSRC, United Kingdom
   22500 euros
EXTERNAL FINANCIAL SUPPORT/3

Individual WG004 members ongoing research projects

5. Department of Physical Chemistry. University of Alicante
   “Structural and functional effects of the electrochemical modification of proteins on diamond
   electrodes: nitration of tyrosine”. Dr. J. Iniesta
   Financed by University of Alicante.

   14224 euros

6. Department of Physical Chemistry. University of Alicante
   “Developments of novel materials, processes and devices for the photochemical, photoelectrochemical and sonochemical desulphurisation of organosulphur compounds in fuels
   and wastes”. Dr. J. Iniesta
   (five years: 01.03.2005-28.02.2010).
   Financed by Ministry of Education and Science. Spain

   12000 euros

7. Department of Physical Chemistry. University of Alicante
   “Surface Electrochemistry” Dr. V. Climent
   Financed by Ministry of Education and Science. Spain

   6000 euros
EXTERNAL FINANCIAL SUPPORT/4

Joint ongoing research projects

1. School of Science and the Environment. University of Coventry
   “Framework 6 STREP Network “Selectnano” coordinated by University Israel (with other D32 labs)
   (From September 2005) 150000 euros. European Union

2. School of Science and the Environment. University of Coventry
   “Marie Curie Transfer of Knowledge “Sopholides” coordinated by National Hellenic Institute, Athens, Greece. (with others D32 labs)
   (From May 2005 date) 30000 euros. European Union
Publications jointed between WG laboratories/1

1. “Coulostatic potential transients induced by laser heating of platinum stepped electrodes: influence of steps on the entropy of double layer formation”

2. “Ultrasound effects on the kinetic reaction step in the silver thiosulphate reduction on platinum electrodes”
   B. G. Pollet, J. P. Lorimer, J. Y Hihn, F. Touyeras, T. J. Mason, D. J. Walton (Besançon+Coventry)
   Ultrasonics Sonochemistry 12 (2005) 7-11

3. “Effect of deposited bismuth on the potential of maximum entropy of Pt(111) single crystal electrode”

4. “Mass spectrometry in demonstrating the site-specific nitration of hen egg lysozyme by an improved electrochemical method”
   D. Matters, J. Helen Cooper, L. McDonnell, J. Iniesta, J. Heptinstall, P. Derrick, D. Walton, I. Peterson (Alicante+Coventry)
Publications jointed between WG laboratories

5. “Optimisation of 20 kHz sonoreactor geometry on the basis of numerical simulation of local ultrasonic intensity and qualitative comparison with experimental results”
J. Klíma, A. Frias-Ferrer, J. González-García, J. Ludvík, V. Sáez, J. Iniesta (Alicante+Prague)
Ultrasonics Sonochemistry 14 (2007) 19-28

6. “Energetic balance in an ultrasonic reactor using focused or plan high frequency transducers”
L. Hallez, F. Touyeras, J. Y. Hihn, J. Klíma (Besançon+Prague)
Ultrasonics Sonochemistry 14 (2007) 739-749

7. “Electrosynthesis of hydrogen peroxide via reduction of oxygen assisted by power ultrasound”
J. González-García, C. E. Banks, B. Šljukić, R. G. Compton (Alicante+Oxford)
Ultrasonics Sonochemistry 14 (2007) 405-412

8. “At point of use Sono-electrochemical generation of Hydrogen Peroxide for Chemical synthesis: The Green Oxidation of Benzonitrile to Benzamide”
J. González-García, L. Drouin, C. E. Banks, B. Šljukić, R. G. Compton (Alicante+Oxford)
Ultrasonics Sonochemistry 14 (2007) 113-116

9. “Simulation of a sono-reactor and the effects of boundaries”
O. Louisnard, J. González-García, I. Tudela, J. Klíma V. Sáez, Y. Vargas (Alicante+Prague)
Ultrasonics Sonochemistry submitted
ACHIEVEMENTS/3

Publications jointed with laboratories from others D32 WG labs/1

1. “The photochemistry of thiophene-S-oxides”

2. “Characterization of a 20 kHz sonoreactor: Part I: Analysis of mechanical effects by classical and numerical methods”
V. Sáez, A. Frías-Ferrer, J. Iniesta, J. González-García, A. Aldaz, E. Riera (WG4+WG11)
Ultrasonics Sonochemistry 12 (2005) 59-65

3. “Characterization of a 20 kHz sonoreactor: Part II: Analysis of chemical effects by classical and electrochemical methods”
V. Sáez, A. Frías-Ferrer, J. Iniesta, J. González-García, A. Aldaz, E. Riera (WG4+WG11)
Ultrasonics Sonochemistry 12 (2005) 67-72

4. “Microwave-enhanced electrochemical processes in micellar surfactant media”
M. A. Ghanem, F. Marken, B. A. Coles, R. G. Compton (WG4+WG5)
Publications jointed with laboratories from others D32 WG labs/2

5. “Microwave activation of the electro-oxidation of glucose in alkaline media”
M. A. Ghanem, R. G. Compton, B. A Coles, A. Canals, A. Vourema, P. John, F. Marken (WG4+WG5)
Physical Chemistry Chemical Physics 7 (2005) 3552-3559

6. “Microwave enhanced electroanalysis of formulations: processes in micellar media at glassy carbon and at platinum electrodes”
M. A. Ghanem, R. G. Compton B. A. Coles, A. Canals, F. Marken (WG4+WG5)
Analyst 130 (2005) 1425-1431

7. “Microwave activation of electrochemical processes at glassy carbon and boron-doped diamond electrodes”
Electroanalysis 17 (2005) 385-391

8. “Microwave activation of processes in mesopores: the thiourea electrooxidation at mesoporous platinum”
M. A. Ghanem, B. A Coles, R. G. Compton, F. Marken (WG4+WG5)
Electroanalysis 18 (2006) 793-800
Publications jointed with laboratories from others D32 WG labs/3

9. “Focused microwaves in electrochemical processes”
F. Marken, U. K. Sur, B. A. Coles, R. G. Compton (WG4+WG5)

10. “Electrochemical oxidation of tetracyclones and Tetraphenylthiophene-S-oxide”
J. Iniesta, H. Alcock, D. J. Walton, M. Watanabe, S. Mataka, T. Thiemann (WG4(2)+WG6)

11. “Electrodeposition and Stripping of catalytically active Iron Metal Nanoparticles at Boron-Doped Diamond Electrodes”
V. Sáez, J. González-García, M. Anbu Kulandadinathan, F. Marken (WG4+WG5)

12. “New Developments in solvent reduced wittig olefination reactions with stabilized phosphoranes”
T. Thiemann, M. Watanabe, J. Iniesta (WG4+WG6)
Sogo Rikogaku (Kyshu Daigaku Daigakuin 28(4) (2007) 379-389
ACHIEVEMENTS/6

Relevant publications/1

1.“Sonoelectrochemical processes: A review”
R. G. Compton, J. C. Eklund, F. Marken (Oxford)
Electroanalysis 9 (1997) 509-522  
111 cites

2.“Sonoelectrochemistry”
T. J. Mason, J. P. Lorimer, D. J. Walton (Coventry)
Ultrasonics 28 (1990) 333-337  
69 cites

3.“Voltametry in the presence of ultrasound: mass transport effects”
53 cites

4.“Ohmic drop compensation in cyclic voltametry at the scan rates in megavolt per second range: access to the nanometric diffusion layers via transient electrochemistry”
C. Amatore, E. Maisonhaute, G. Simonneau (Paris)
42 cites

5.“Effect of ultrasound on the electrodeposition of lead dioxide on glassy carbon electrodes”
J. González-García, J. Iniesta, A. Aldaz, V. Montiel (Alicante)
19 cites
**ACHIEVEMENTS/7**

**Relevant publications/2**

6. “Electrochemical behaviour of zinc in 20 kHz sonicated NaOH electrolytes”  
M. -L. Doche, J.-Y. Hihn, F. Touyeras, J. P. Lorimer, T. J. Mason, M. Plattes (Besançon+Coventry)  
11 cites

7. “Sonoassisted electrooxidation polymerisation of salicylic acid: role of acoustic streaming and microjetting”  
J. Klima, C. Bernard (Prague)  
6 cites

**Relevant awards**

1. Richard Compton (Oxford University) awarded Tilden Medal (Royal Society of Chemistry) 2006  
Professor Compton was awarded the medal for his innovative, quantitative investigations of the kinetics and mechanisms of reactions at solid and liquid interfaces.

2. J. González-García (Universidad de Alicante) awarded Carl Wagner Award 2005 (Working Party on Electrochemical Engineering of the European Federation of Chemical Engineering)  
Dr. González-García was awarded the medal for his well-recognised scientific contribution in Europe in various areas of the Electrochemical Engineering.
COST ACTION D32 MID TERM EVALUATION MEETING

Oral presentations

   J. González-García

Poster presentations

1. “Optimization of sonochemical and/or sonoelectrochemical cell with the help of numerical simulation of ultrasonic intensity distribution”
   J. Klima, J. González-García, A. Frías-Ferrer, J. Ludvik, V. Sáez, J. Iniesta (Alicante+Prague)

2. “Research in the group "New Technological Development in Electrochemistry: Sonoelectrochemistry and Bioelectrochemistry" Alicante University”
   V. Sáez, A. Frías-Ferrer, P. Espí, M. D. Esclapez, P. Bonete, J. Iniesta, J. González-García

3. “Selective modification at tyrosine residues by the electrochemical, sonoelectrochemical and sonochemical nitration of lysozyme”
   D.J.Walton, J. Heptinstall, J. Iniesta (Alicante+Coventry)
Poster presentations/2

4. “Effect of low frequency ultrasound irradiation on room temperature ionic liquid (RTIL’s) dehydration”
C. Costa, J. Y. Hihn, I. Bisel, P. Moisy, M. Rebetez, M. –L. Doche (Besançon)

5. “Electrochemical measurements of a flow induced by low frequencies ultrasound. Influence of horn diameter and cell design”
M. –L. Doche, A. Mandroyan, J. Y. Hihn, R. Viennet (Besançon)
FURTHER ONGOING ACTIVITIES/1

Individual WG004 members activities

1. Université de Franché-Comté. Besançon. France (three years): PhD. Grant in Besançon for Cedric Costa
Title: Electrochemistry in RTIL’s under ultrasound irradiation
Financed by the Université de Franché-Comté 33000 euros

2. Université de Franché-Comté.-IMASONIC-ITT industries (three years): Postdoctoral grant for Loic Hallez related to research project in Besançon
Topic: ultrasonic irradiation of polymer film
Financed by Frech Goverment 180000 euros
Further cooperations internal WG

*Human mobility*

1. Postdoctoral position A. Mandroyan (Besançon) in Coventry University (financed by *******)
   Topic: Electrochemical studies under ultrasound
Further ongoing activities/3

Further cooperations external WG

Human mobility

1. Research visit V. Sáez (Alicante) in Bath University (WG4+WG5) 1980 euros
   Topic: Electrochemical studies under ultrasound
   Financed by University of Alicante
FURTHER PLANNED ACTIVITIES/1

Individual WG004 members activities

Topic: Development of sonoelectrochemical reactors for the dry-cleaning waste treatment
Financed by Spanish Ministry of Environment
FURTHER PLANNED ACTIVITIES/2

Further planned cooperations internal WG

*Human mobility*
Research visit D. J. Walton (Coventry) in Alicante University
Topic: “Comparison of sono electrochemical and magnetoelectrochemical effects on a model electroorganic systems”
financed by University of Alicante

*Joint research project proposal in Prospect via D32*
University of Alicante-University of Coventry (D. J. Walton)
Topic: Selective chlorination of proteins at tyrosine residues
Spanish support

COST D32 WG004 and other labs from Action D32
Topic: “AERO” Advanced Electrochemical Reactor operation”
Framework European Network Grant
FURTHER PLANNED ACTIVITIES/3

Further cooperations external WG

Human mobility

1. STSM (University of Coventry-Technical University Krakow, Poland)
   Topic: Sonoelectrochemical effect in conducting polymers
   financed by COST

Joint research project proposal in Prospect via D32

Université de Franché-Comté-University of Coventry (T. J. Mason)
Topic: Nanoparticles synthesis by sonoelectrochemistry
EGIDE programs

14000 euros
### ACTIVITY SUMMARY

<table>
<thead>
<tr>
<th></th>
<th>Inner WG</th>
<th>Jointed WGs (WG4 lab +..)</th>
<th>Subject/9th ESS (labs met up)</th>
<th>General</th>
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<td>1 WG4 talk</td>
<td>4 lab talk</td>
<td>6 jointed-inner talks</td>
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<td>3 jointed WGs talk</td>
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<td><strong>Research Interactions (STSM, visits…)</strong></td>
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DETAILED EXPLANATION OF THE ACTIVITIES
Kick off Working Group Meeting

Kick off Working Group Meeting, held in Alicante, 3rd-4th December 2004.

Participants and talks

University of Alicante
Welcome J. González-García
“Sonoelectrochemistry: fundamental and applied studies” V. Sáez
“Optimization of Hydrodynamics in Electrochemical Reactors: Use of numerical and experimental methods” A. J. Frías-Ferrer

University of Oxford
“Sonoelectroanalysis” R. G. Compton
“A hand held probe for the sonoelectroanalysis of arsenic”. A. Simm
“Materials for the sonoelectrosynthesis of hydrogen peroxide” C. Banks

University of Bath (Invited speaker)
“Microwave Effects and Applications in Electrochemistry” F. Marken
Kick off Working Group Meeting (II)

Participants and talks (II)

University of Coventry
“Electrooxidation and Photooxidation of Thiphene-s-Oxides” D. Walton

University of Franche-Comté
“Electrochemistry under ultrasound at the interface” J.-Y. Hihn
“Design of sonoreactors and application in surface treatment” M. L. Doche

J. Heyrovsky Institute of Physical Chemistry, Czech Republic
“Intensity of ultrasound in sonochemical and sonoelectrochemical measurements” J. Klima
“Electrochemical reduction and dimerization of halogenated benzothiophenes and benzothiophene-2-oxazolines. Use of sonication” J. Ludvik

Université de Paris
“Ultrafast sonoelectrochemistry” E. Masionhaute
Other activities
Visit to the pilot plant. The existence of this facility is an important aspect of the Operation at Alicante, which is not available at other laboratories in the network.

Achievements and objectives
* Talks reviewed the active research in the working group labs
  Alicante: Electrodeposition, pollutant degradation assisted by ultrasound
  Design and Characterization of (sono)electrochemical reactors

  Oxford: Sono-electroanalysis in real practical applications
  Sono-electrosynthesis, in particular hydrogen peroxide from oxygen

  Coventry: Electroorganic and photoelectroorganic synthesis focused on the oxidation of thiophene-S-oxides
Kick off Working Group Meeting (IV)

Objectives and achievements (II)

Prague: Physical background in sonochemistry and sonoelectrochemistry

Besançon: Better modelling of sonoelectrochemical systems
Electroplating assisted by ultrasound
Open to new emerging fields: Room temperature Ionic liquids polymer synthesis under US

Paris: Cavitation analysis with nanosecond time base equipment
Fundamental approach to the cavitation event

*Relevant points which can fit WG objectives
Definition of reproducibility and characterization of operating conditions
Identification of problems in applications or phenomena needing theoretical understanding
Sonoelectrochemical reactor design
Objectives and achievements (III)

*As a first step, development of STSMs in order to establish bilateral collaborations for:
  Mutual exchange of expertise
  Mutual training
  Finding specific collaborating targets
  Definition of the tasks and contributions in a future research project

*After, applications for the 7th European Framework will be considered looking at:
  Complementarity among laboratories
  Possibility to open the applicant list to other COST laboratories
Degradation of pollutants and enhanced environmental clean-up using sonoelectrochemistry

Chlorinated hydrocarbons are usually used as industrial degreasing agents: CCl₄, CHCl₃, C₂Cl₄, C₂Cl₃, among others. Withspread chemical contaminants in the subsurface aquatic environment, which are difficult to treat by conventional technologies.

Perchloroethylene (C₂Cl₄) is widely used as an industrial dry cleaning solvent and metal degreaser.
Short Term Scientific Mission: V. Sáez (Alicante) in Coventry

Objectives
Study of the degradation of a chlorinated organic compound using sonochemical, electrochemical and finally the exploitation of the combination of both methodologies at 850 kHz (equipment not available in UA).

Degradation of perchloroethylene as a model molecule in aqueous solution.

Perchloroethylene
Short Term Scientific Mission: V. Sáez (Alicante) in Coventry

Experimental set-up

Sonoreactor 850 kHz/140 W by Meinhardt
Ultraschalltechnik, K80-5
Conclusions

-Sonochemical treatment:
  * Higher yields with lower concentration of PCE
  * Higher yields with higher frequencies
  * Higher yields with higher ultrasonic intensities
  * Radical mechanism
  * After 5-hours treatment, no PCE, CF and CT were found

-Electrochemical treatment
  * Yield not influenced by increasing current densities
  * The mechanism does not seem to follow by radical soluble intermediates

Sono-electrochemical treatment
  * Yield higher than electrochemical ones for the same current density
  * A maximum yield is obtained for ultrasonic intensities series (work in progress)
**Conclusions (II)**

- Reaction products obtained by the three treatment are different:
  
  * Sonocidal: PCE and saturated chlorinated compounds ($C_1$ and $C_2$) even higher than $C_3$
  
  * Electrochemical: PCE, TCE and DCE (insaturated) No chlorinated compounds higher than $C_3$
  
  * Sonoelectrochemical: PCE, TCE and DCE (insaturated) and only $C_1$ saturated chlorinated compounds
  
  Compounds > $C_3$ only at higher ultrasound intensities

**Publications**

“Sonoelectrochemical degradation of perchloroethylene at 850 kHz”
V. Sáez, J. Iniesta, A. Frías-Ferrer, J. González-García and D. J. Walton
Electrochem 2005, Newcastle, UK
Short Term Scientific Mission: J. Klima (Prague) in Alicante

Improved strategies for waste minimisation by *sonoelectrochemical reactor design*

**Effects-events in sono(electro)chemistry**
- sonoluminescence
- sonolysis (thermolysis)
- mass transfer enhancement
- surface activation
- surface destruction
- hot spots
- hot spots
- turbulence
- acoustic streaming
- microstreaming
- microjets
- microjets
- microjets

**no sonochemical effect without cavitation !!!**
Short Term Scientific Mission: J. Klima (Prague) in Alicante

Objectives

Knowledge of local distribution of ultrasonic intensity is necessary for measurement of intensity:

\[ I = W_c = \frac{1}{2} \rho c \omega^2 A^2 = \frac{P_0^2}{2\rho c} \]

\[ I = \frac{P_{US}}{S} \]

only for progressive wave calculation
Short Term Scientific Mission: J. Klima (Prague) in Alicante

Procedure

resolution of wave equation using a FEM with Femlab Package:

\[
\nabla \left( \frac{1}{\rho} \nabla P \right) - \frac{1}{\rho c^2} \frac{\partial^2 P}{\partial t^2} = 0
\]

boundary conditions

\[ P(r, t) = p(r)e^{i\omega t} \]

\[
\nabla \left( \frac{1}{\rho} \nabla p \right) - \frac{\omega^2}{\rho c^2} p = 0
\]
Short Term Scientific Mission: J. Klima (Prague) in Alicante

Procedure: First with a well-known system with analytical resolution

\[ f = 1.1 \text{MH} \]
\[ z \]
\[ r = 8 \text{mm} \]
\[ p_0 \approx \sin \left( \frac{k}{2} \left( \sqrt{x^2 + r^2} - x \right) \right) \]
Short Term Scientific Mission: J. Klima (Prague) in Alicante

Procedure: moving to more realistic systems

\[ x = 2r \rightarrow I_{US} < 3\% I_0 \]

\[ V(I_{US} > 3\%I_0) < 1\% \]
Short Term Scientific Mission: J. Klima (Prague) in Alicante

Procedure: moving to more realistic systems

\[ R = 45, \ H = 25, \ D = 77 \text{ mm} \]
Short Term Scientific Mission: J. Klima (Prague) in Alicante

Procedure: moving to more realistic systems
Short Term Scientific Mission: J. Klima (Prague) in Alicante

Conclusions
- numerical solution of the wave equation is in good agreement with experiment
- the optimal geometry of experimental arrangement can result in a strong increase of intensity in specific part of the cell

Advantages of optimised geometry:
- the cell is simple and cheap;
- whatever cell can be used;
- the ultrasonic power necessary for cavitation is low;
- low ultrasonic power results in weak heating of the solution
  => no cooling is necessary;
- the fraction of reactor volume where intensity is high is large.

In sonoelectrochemistry:
- electrode does not need be placed in a small area near the horn surface

Publications
"Physical aspects of Sono(electro)chemistry: Distribution of intensity of ultrasound"
J. Klíma, A. Frias-Ferrer, J. González-García, J. Ludvík, V. Sáez, J. Iniesta
COST WG 2 Workshop 9-10 June 2005, Oxford, UK
One-year visit: J. González-García (Alicante) in Oxford

Objectives

Identification of practical electrocatalysts capable of acting as electrode materials in a sonoelectrochemical reactor for the reduction of oxygen (air) to hydrogen peroxide.

The determination of the stability of the electrocatalysts in respect of insonation to identify the optimal sonoelectrocatalysts for hydrogen peroxide formation.

Design, development and optimisation of a reliable and efficient laboratory bench scale sonoelectrochemical reactor.
One-year visit: J. González-García (Alicante) in Oxford

Results

\[ \text{Reactions: } +2H^+ + 2e \]

\[ \begin{array}{c}
\text{Reaction 1: } \\
\text{Reaction 2: }
\end{array} \]
One-year visit: J. González-García (Alicante) in Oxford

Results

![Graph showing electrochemical results with various markers indicating different conditions: no-modified, Reused, PAQ Chem, AL chem, GG chem, AL EC, GG EC, Vitreous carbon, and new. The x-axis represents E/V vs SCE, and the y-axis represents j/mA cm².](image-url)
One-year visit: J. González-García (Alicante) in Oxford

Conclusions and further work
9-10 phenantraquinone is a good electrocatalysts capable of acting as electrode materials in a electrochemical reactor for the reduction of oxygen (air) to hydrogen peroxide

Work in progress to analyze the stability as a electrode material in a sonoelectrochemical reactor

Design, development and optimisation of a reliable and efficient laboratory bench scale sonoelectrochemical reactor
Interactions between Working Groups
Short Term Scientific Mission: D. Bogdal WG10 in Coventry

Objectives
Carbazole electropolymerization in acetonitrile/ammonium system. Influence of the ultrasound and a magnetic field

Results
Polycarbazole films on indium-tin oxide coated (ITO) glass

Further work
Characterization of the luminiscent properties in Politechnika Krakowska
Further actions

Annual Working Group Meeting, to be held in Prague, November 2005.