SOL-GEL MATERIALS SYNTHESIS AND CHARACTERIZATION FOR OPTICAL SENSING

Rapports

Informations projet

SOLGELSENS

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Final Report Summary - SOLGELSENS (SOL-GEL MATERIALS SYNTHESIS AND CHARACTERIZATION FOR OPTICAL SENSING)

The project brings together four research groups from Spain (Universidad de Oviedo), France (Université Paul Sabatier in Toulouse, and Ecole Superieure de Physique et Chimie Industrielle) and Ukraine (Taras Shevchenko National University of Kyiv) to share the expertise and knowledge of the involved partners in development of sol-gel technology for preparation and investigation of nanostructured and nanosized materials for their use in analytical chemistry. Main attention was given to development of the materials that can act as adsorbents and sensors for determination of forbidden additives (antibiotics) in food and toxic volatile compounds (particularly formaldehyde) in air. The two main areas of training are concerned: 1) sol-gel synthesis and physico-chemical characterization of a) nanostructured hybrid organo-silica materials, and b) nanosized Fe3O4@SiO2 particles for selective determination of antibiotics in food (model analyte: tetracycline); 2) Sol-gel synthesis and physico-chemical characterization of dye-doped sol-gel materials for volatile organic compounds (VOCs) sensing (model analyte: formaldehyde). For both area the materials properties were optimised for their application in development of optical sensor technology.

The use of antibiotics has become integral to the livestock production industry as growth promoters and therapeutic agents. In the European Union, 5000 ton of antibiotics (70% for non-therapeutic purposes) were used in 1999 for veterinary therapy [A.K. Sarmah, M.T. Meyer, A. Boxall, Chemosphere 65 (2006) 725–759.]. Studies have shown that between 17–76% of antibiotics administered to animals are excreted via urine and feces in unaltered form and as metabolites. Land application of manure as a supplement to fertilizer is a common practice in many countries. It is therefore likely that when animal manure is applied to agricultural fields, antibiotic residues can find their way into the receiving environment (e.g. waters). The use of antibiotics in food animals and fertilizers has generated concern due to the potential for increasing antimicrobial resistance, in addition to hypersensitivity for some individuals and on January 1, 2006, the European Union banned the feeding of all antibiotics and related drugs to livestock for growth promotion purposes.

Tetracycline antibiotics (Tc) are widely used in veterinary practice and their residues in the dairy products caused serious health problems. According to the Commission Regulation (EU) №37/2010 the maximum allowable residue level of Tc in food for human consumption is 100 ppb. The determination of trace amounts of Tc in milk is an important task. Besides standard chromatographic methods, bioluminescent, microbial and colorimetric test-kits based on solid-phase enzyme immunoassay are proposed for rapid screening of Tc in food.

The determination of antibiotics and formaldehyde with optical sensors is a good alternative to expensive and time-consuming chromatographic method of analysis. So the project aim - development of new materials for optical sensors has positive socio-economic impacts since allows rapid monitoring of illegal use of antibiotics in food and drinks as preservatives.

For development of sensitive elements for optical sensors next approachs were used.

1. Impregnated by polyelectrolytes (PE) silica-based (SiO_2) hybrid films (SiO_2-PE) demonstrates complex of necessary requirements to sensitive membranes for optical sensors: a) they are transparent, b) mesoporous, c) have good adhesion to glass and less fragile than SiO_2.
2. For selective optical detection of antibiotics residuals, sensibilized luminescent of Eu (III) complexes with this analytes can be used. Such complexes demonstrates intense luminescent that have sharp emission line.

In the project training in sol-gel preparation of a set of SiO_2-PE films with different polyelectrolytes was given and optimal conditions for their application in sensor technology were found. To improve adsorption kinetic and the film capacity several surfactants was used during sol-gel synthesis as structure directed compounds. Conditions of Eu ion immobilisation, the membrane stability and its analytical characteristics (intensity of luminescence, selectivity) were studied. It was demonstrated that hybrid organo-silica films (SiO_2-PE) obtained in the presence of poly(vinylsulfonic) acid (PVSA) possessed 10-20 times higher adsorption capacity for cationic cyanine dyes compare to SiO_2 films. Triblock copolymers (TBC), have a significant effect on the silica films morphology and allows to obtain materials with pore diameter up to 15 nm. Sorbitanemonolaurates (Tween) were successfully used as structure directed agents for synthesis of mesoporous silica sol-gel materials. Significant increasing in pore diameter can be achieved by using mixtures of high and low molecular weight surfactants.

As important scientific conclusions for this Project, higlights are:

- Use of cation-exchange polyelectrolytes, poly(vinyl sulfonic acid) and poly(styrene sulfonic acid), in the course of synthesis favor a firmer fixation of oppositely charged molecules of the cyanine dye.
- A mixture of Tween 20 and triblock copolymers used as templates improves the structural characteristics of film coatings, with the molar ratio between the surfactants in the sol strongly affecting the resistance and sorption properties of the hybrid coatings.
- The composite materials synthesized may be promising as solid-phase analytical reagents and elements of optodes to be used, in particular, for a spectrophotometric determination of formaldehyde.
- Cyanine dyes on the surface of the silica matrix may be prepared using the developed preparation methodologies. It is possible to synthesize stable photoluminescent hydrid materials that can have

interesting potential applications as new optical and photoluminescent sensors for analytical purposes. Further refinement of the sensing capabilities of this class of chemosensors is in progress.
- Surface functionalized Fe3O4 magnetic nanoparticles were prepared, aiming to extract trace tetracycline from aqueous solution. Before modification, the Fe3O4 weak adsorbed tetracycline, whereas the functionalized Fe3O4/SiO2/AdPA and Fe3O4/SiO2/PVSA exhibited high adsorption affinity for them, resulting from chelating interaction by surface acidic group. Thus, we proposed combines the simplicity and selectivity adsorption with the easy magnetic separation from a solution with residual concentration of tetracycline without any preliminary filtration step.

Concerning management and dissemination:

-Most of the Training and Seminars where satisfactorily completed increasing the expertise of all partners and especially the young researchers and PhD students.
-The number of Reports and Publications produced indicates one good level of collaboration.
-New projects have been started as a consequence of this SOLGELSENS collaboration, in particular the FP7-MC-IRSES Action SICCATALYSIS – 319013 "Porous Silicon Carbide as a Support for Co Metal Nanoparticles in Fischer-Tropsch Synthesis”
-The interest of APMChem S.L. a local company in Asturias, interested in the commercialization of the materials produced within the SOLGELSENS Action.
-The number of participants and their involvement, as well as the scientific level, was very satisfactory.

Documents connexes

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