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2nd International Conference and Expo on

# Separation Techniques

September 26-28, 2016 Valencia, Spain

## Scientific Tracks & Abstracts

## (Day 1)

Day 1 September 26, 2016

# Applications of Separation Techniques

**Session Chair**
**Morten Lykkegaard Christensen**
Aalborg University, Denmark

**Session Co-chair**
**Chun-Yi Chen**
Tokyo Institute of Technology, Japan

## Session Introduction

**Title: Performance of the deep bed filter at its loading with particles and microorganisms**
**Leon Gradon,** Warsaw University of Technology, Poland

**Title: Impact of sludge properties on solid-liquid separation of activated sludge**
**Morten Lykkegaard Christensen,** Aalborg University, Denmark

**Title: Nanofiltration for the Separation of polivalent and monovalent ions in high saline solutions**
**Raquel Ibanez Mendizabal,** University of Cantabria, Spain

**Title: Identification of Inhibitors of the Fe-2OG dependent oxygenases by capillary electrophoresis with laser-Induced fluorescence**
**Svetlana M Krylova,** York University, Canada

**Title: Separation of heath compounds in Goji (Lyceum barbarum) aqueous extracts by membrane technology**
**Conidi Carmela,** University of Calabria, Italy

Leon Gradon, J Chromatogr Sep Tech 2016, 7:6(Suppl)
http://dx.doi.org/10.4172/2157-7064.C1.019



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# Performance of the deep bed filter at its loading with particles and microorganisms

**Leon Gradon**
Warsaw University of Technology, Poland

The deep-bed filtration technique is one of the most effective available water purification methods. The real fibrous filter performance emphasizes during its loading, when the deposits gradually change a filtration space. The evolution of temporary filtration efficiency, pressure drop and dust capacity characterizes filter quality. This work is focused on the behavior of filter loading with abiotic and biotic objects presented in the water stream. Multilayer gradient and composite micro- and nanofibrous cartridges were produced for testing using the melt-blown technology. The entire filtration time was divided into 5 periods. After each period of time the tested filter was dried and stratified into layers differing in fiber diameter and porosity. The retention capacity of each layer was calculated gravimetrically. Porosity of the initial fibrous structure and the structure loaded with the deposit collected on the fibers was determined using a scanning electron microscopy in 2-dimentional space. Experimental data presents the time developing values of filtration efficiency and pressure drop for each type of the tested filters. Results show qualitative difference of filters behavior depending on their initial structure. Deposits are distributed inside the cartridge with different manner. When bacteria are present in the water stream, the biofouling due to their intrinsic colonization significantly changes the filter loading. The introduction of nanocomposite Ag and ZnO particle on the filter fiber significantly reduces this effect, according of our measurements.

## Biography

Leon Gradon is a tenured Professor of Chemical Engineering at Warsaw University of Technology with 45 years of academic and research experience. He is the author and co-author of 19 monographs and chapters, 4 academic books, over 220 per-reviewed papers in scientific international journals and 65 patents. Several applications of his inventions at technical scale are ultrasonic nebulizer, pneumatic nebulizer, bag filters, disposable respirators, dry powder inhaler and diesel engine filters. He is an Editorial Board Member in four international journals: *Chemical and Process Engineering Journal, International Journal Occupational Safety and Ergonomics, Journal of Aerosol in Medicine, KONA Powder and Particles* and *Advanced Powder Technology*.

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# Impact of sludge properties on solid-liquid separation of activated sludge

**Morten Lykkegaard Christensen**
Aalborg University, Denmark

Solid-liquid separation of activated sludge is important both during the biological treatment of wastewater and for dewatering of excess sludge. The separation of solid from the treated wastewater can be done by using clarifiers (conventional plants) or membranes (MBR). Further, excess sludge is usually mechanical dewatered before further handling. Solid-liquid separation is a costly part of wastewater treatment. The separation process depends on the composition and the properties of the sludge. Sludge contains sludge flocs, filaments, single cells, dissolved extracellular polymeric substances (EPS) and ions. The best separation is obtained for sludge that contains strong, compact flocs without single cells and EPS. Di and trivalent ions improve the floc strength and improve the separation whereas monovalent ions (e.g. from road salt, sea water intrusion and industry) impairs the separation. High pH e.g. due to the inlet flow impairs the separation process due to floc disintegration. In membrane operation, single cells and dissolved EPS clogs the membrane whereas strong sludge flocs courses the membrane and thereby reduce membrane fouling. In filtration dewatering small cells and EPS, blinds the cake and thereby lowers the dewaterability. Thus, in all separation processes single cells and dissolved EPS should be avoid e.g. anaerobic storage and high shear levels should be avoid as this erode the flocs and results in more single cells. Sludge can be aerated during storage or nitrate added to avoid anaerobic condition. Further, pumping and mixing should be gently and pipes with sharp bends should be avoided.

## Biography

Morten Lykkegaard Christensen has completed his PhD from Aalborg University, Denmark. He is currently an Associate Professor at Aalborg University and Head of the Separation Science Group. He has published more than 40 papers in reputed journals.

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# Nanofiltration for the separation of polyvalent and monovalent ions in high saline solutions

**R Ibanez, Ortiz P, Urtiaga A** and **Ortiz I**
Universidad de Cantabria, Spain

This work, as part of a global membrane process for the recovery of alkali and acids from reverse osmosis (RO) desalination brines, focuses on the nanofiltration (NF) separation of polyvalent and monovalent anions, more specifically sulfate and chloride. This pretreatment stage plays a key role in the whole recovery process. Working with model brines simulating the concentration of RO concentrates, 0.2-1.2 M chloride concentration and 0.1 M sulfate concentration, the experimental performance and modeling of the NF separation is reported. The study has been carried out with the NF270 (Dow Filmtec) membrane. The effect of operating pressure (500-2000 kPa), ionic strength (0.4-1.3 M) and chloride initial concentration (0.2-1.2 M) on the membrane separation capacity has been investigated. Additionally, the Donnan steric pore model (DSPM) together with experimentally determined parameters, effective pore radius (rp), thickness of the membrane effective layer (δ) and effective membrane charge density (Xd), was proved accurate enough to satisfactorily describe the experimental results providing the tools for process design and optimization. Finally, In this work, we provide for the first time the analysis of partitioning effects and transport mechanism in the NF separation of sulfate and chloride anions in concentrations that simulate those found in RO desalination brines.

## Biography

R Ibanez is an Associate Professor of Chemical Engineering at the Chemical and Biomolecular Engineering Department of the University of Cantabria, Spain. She coordinates the research group IPS "Sustainable Processes Engineering" focusing her work in the research and development of advanced separation technologies and their sustainable applications. She has been involved in more than 30 national and international research projects and has been Coordinator of about 10 research projects. She has published more than 60 papers in reputed journals and participates regularly in international conferences.

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# Identification of inhibitors of the Fe-2OG dependent oxygenases by capillary electrophoresis with laser-induced fluorescence

**S M Krylova**
York University, Canada

2-oxoglutarate and Fe (II)-dependent dioxygenases catalyze the removal of N-alkyl groups from damaged DNA in eukaryotes and bacteria. Silencing these demethylating enzymes may be beneficial for the enhancement of chemotherapeutic treatments and reduction of their cytotoxic effects. Therefore, a direct and efficient quantitative analysis using biologically-relevant substrates is needed for detection of demethylase activity of *E. coli* and human Fe-2OG dioxygenases and its application for high throughput screening of potential inhibitors. Previously reported techniques utilize coupled enzyme reactions, detect co-products, require complex processing or use radioactive substances. We developed a direct and rapid method based on capillary electrophoresis with laser-induced detection allowing real-time detection of both the substrate and the product separated with high efficiency using no post-enzymatic processing and without the use of radioactive substances. Here we report the CE-based activity assay of two members of the Fe-2OG dioxygenase superfamily of enzymes-hABH2 and hABH3, and demonstrate that the activity can be selectively inhibited by small molecules or short DNA aptamers. The inhibition selectivity of hABH2 over hABH3 enzymes can be advantageously used for qualitative and quantitative assay of these enzymes mixtures. The simple and specific differential analysis can be potentially employed to distinguish hABH2 and hABH3 enzymes expressed by the same types of cells *in vivo*. The minimal processing, short analysis time, low cost and availability of automation makes this assay useful for developing therapies targeting Fe-2OG dioxygenases.

## Biography

S M Krylova has obtained her PhD from the Russian Academy of Sciences, Russia. She has over 10 years of research leadership experience in the area of Medical Diagnostics and Drug Development in biotechnology and pharmaceutical companies in Canada. She has been a contract Faculty Member at York University in Toronto since 2008. She is also leading research projects in the area of Bioanalytical Chemistry as a Senior Research Associate in the Centre for Research on Biomolecular Interactions at York University.

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# Separation of heath compounds in Goji (*Lycium barbarum*) aqueous extracts by membrane technology

**Conidi Carmela**[1] and **Alfredo Cassano**[2]
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[2]ITM-CNR, Italy

In recent years, the recovery of antioxidant compounds from natural sources is a focus of great interest due to their potential use as natural ingredients in food, pharmaceutical and cosmetic formulations or as substitutes of synthetic products in the food industry. Several conventional extraction techniques have been reported for the recovery of target compounds from raw materials, such as solvent extraction, ultrasound-assisted extraction, pressurized-liquid extraction, supercritical fluid extraction and resin-based extraction. These extraction methods are characterized by some drawbacks, including the degradation of the target compounds due to high temperatures and long extraction times (as in solvent extractions) and health related risks. Membrane operations are recognized as powerful tools for the purification and concentration of various solutions (e.g., juices, extracts and whey) and the separation of valuable compounds from different food matrix. This study was aimed at developing a sustainable process for the purification of natural antioxidants from Goji berries and leaves. This process is based on the combination of an aqueous extraction with membrane operations in order to avoid the use of organic solvent or adsorbents. The aqueous extraction was studied in order to obtain the maximum yield of phenolic compounds. At this purpose, different parameters such as the extraction time and temperature, the pH and the solid/liquid ratio, were optimized. Aqueous extracts were processed through membrane operations, such as ultrafiltration (UF) and nanofiltration (NF), in order to evaluate the overall bioactivity of fractionated extracts in comparison with that of the unprocessed extracts.

## Biography

Conidi Carmela has completed her PhD from University of Calabria and the Institute on Membrane Technology, ITM-CNR. She has completed her Post-doctoral studies from Instituto de Ingenieria de Alimentos para el Desarrollo, Universitat Politècnica de València, Spain. She is a Post-doctoral Researcher at the Institute of Membrane Technology of CNR, where she is involved in different research activities devoted to the purification and concentration of antioxidant compounds in products and byproducts of food processing. She has published more than 30 papers in international journals.

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## Emerging separation technologies

**Session Chair**
**Leon Gradon**
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**Session Co-chair**
**Sandrine Ricote**
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### Session Introduction

**Title:** **Understanding and predicting industrial membranes performances in separation technology**
**Helene Marie,** Dow Benelux BV, Netherland

**Title:** **Influence of coal power plant exhaust gas on the structure and performance of ceramic nanostructured gas separation membranes**
**Kai Wilkner,** Forschungszentrum Julich, Germany

**Title:** **A simple, accurate, time-saving and green method for the determination of 15 sulfonamides and metabolites in serum samples by ultra-high performance supercritical fluid chromatography**
**Feng Zhang,** Chinese Academy of Inspection & Quarantine, China

**Title:** **Protonic ceramic membranes under asymmetric steam atmosphere**
**Sandrine Ricote ,** Colorado School of Mines, USA

**Title:** **Separation science that's built for biopharma**
**Robert van Ling,** Thermo Fisher Scientific, Netherlands

**Title:** **Removal of divalent heavy-metal ions from aqueous solutions by adsorption process with titanium dioxide nanowires**
**Snežana Maletic,** University of Novi Sad, Republic of Serbia

**Title:** **Development of a liquid-liquid extraction method of resveratrol from cell culture media using solubility parameters**
**Mohamad Houssam Al balkhi,** Universite de Picardie Jules Verne, France

**Title:** **Highly selective sieving in porous graphene-like carbon nitride for helium/light isotopes separation**
**Qu Yuanyuan,** Shandong University, China

**Title:** **2D crystal-based membranes for photocatalysis and separation**
**Yuyoung Shin,** University of Manchester, UK

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# Understanding and predicting industrial membranes performances in separation technology

**Helene Marie**
Dow Benelux BV, Netherland

Nanofiltration (NF) membranes cover a range of salt and neutral molecule selectivities that lay between reverse osmosis and ultrafiltration membranes. This unique performance enabled a breakthrough in industry during the past decades. Their high water permeability along with good rejection makes them usable as a replacement for different treatment processes at a reasonable energy cost. However, there are some hindrances in the design of new NF processes and improvement of existing ones since NF applications are mostly associated with complex transport phenomena of which modeling is difficult. The understanding of transport phenomena through NF membranes by use of modeling and prediction may help to implement NF membrane technology more broadly and will enlarge the NF market beyond water filtration. Considering that NF is an industrial scale method for purification and concentration of oligosaccharides mixtures, we decided to study some industrial membranes performances against various saccharides. We studied independently each component of the solute/membrane/solvent system and their pair-interaction to achieve a deep theoretical knowledge. We then collected rejection data and analyzed membrane performances while varying parameters, such as temperature and concentration. Thanks to the characterization of the membranes and their performance, we were able to parameterize a model based on the extended Nernst-Planck equation for rejection results. Finally, the model was assessed against mixture separations. Going forward, the outcome of this study will enable more rigorous membrane selection for targeted industrial applications and to predict its performance.

## Biography

Helene Marie has completed her PhD from Compiegne University of Technology, France and CEA Leti. She is a Lead Engineer at Dow Benelux B.V. in Corporate R&D and works closely with the Dow Water & Process Solution Business Unit.

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# Influence of coal power plant exhaust gas on the structure and performance of ceramic nanostructured gas separation membranes

**K Wilkner, J Eiberger, M Bram, M E Ivanova** and **W A Meulenberg**
Forschungszentrum Julich, Germany

Carbon capture and storage or utilization is a key technology to decrease $CO_2$ emissions from conventional power plants, until cost efficient energy supply from renewable sources is possible. Membrane-based systems to capture $CO_2$ from flue gas streams are considered a promising alternative to conventional absorption technology. In the present work the effect of coal power plant exhaust gas on amino-modified mesoporous ceramic membranes was investigated. Testing membranes in direct contact with exhaust gas represents a new approach, as testing under simulated flue gas conditions has already been undertaken. Flue gas exposure experiments were carried out at a lignite-fueled power plant and a hard-coal-fueled power plant. Most experiments were conducted using a test rig designed to bring planar membrane samples in direct contact with unconditioned flue gas in the exhaust gas channel. Another test rig was designed to test membrane modules with pre-treated flue gas. The tested membranes had an asymmetric structure consisting of a macroporous α-$Al_2O_3$ support coated with a mesoporous ɣ-$Al_2O_3$ or 8YSZ interlayer. The microporous functional top layer was made of amino-functionalized silica. The tests revealed different degradation mechanisms such as gypsum/fly ash deposition on the membrane surface, pore blocking by water condensation, chemical reactions and phase transformation. A detailed analysis was carried out by XRD, XPS and SEM to evaluate their impact on the membrane in order to assess membrane stability under real conditions. The suitability of these membranes for this application is critically discussed and an improved mode of membrane operation is proposed.

## Biography

K Wilkner holds a Diploma in Physical Engineering from the University of Applied Science Aachen. Since 2011, he has been working at the Institute of Energy and Climate Research: Materials synthesis and processing (IEK-1) in Forschungszentrum Julich. Since 2012, one of his responsibilities is to test membranes in direct contact with flue gas of lignite and hard-coal-fueled power plants.

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# A simple, accurate, time-saving and green method for the determination of 15 sulfonamides and metabolites in serum samples by ultra-high performance supercritical fluid chromatography

**Feng Zhang**
Chinese Academy of Inspection & Quarantine, China

Sulfonamides are a large group of synthetic antibiotics which has anti-bacterial properties. They have a good antibacterial effect on both Gram-positive and Gram-negative bacteria. Sulfonamides are commonly prescribed in human and veterinary medicine against many kinds of infections. In order to investigate the pharmacodynamics and pharmacokinetics of different sulfonamides, establishing a detection method of multiple sulfonamides and their metabolites in serum is necessary. An analytical method based on ultra-high performance supercritical fluid chromatography (UHPSFC) with photo-diode array detection (PDA) has been developed to quantify 15 sulfonamides and their $N_4$-acetylation metabolites in serum. Under the optimized gradient elution conditions, it took only 7 min to separate all 15 sulfonamides and the critical pairs of each parent drug and metabolite were completely separated. Variables affecting the UHPSFC were optimized to get a better separation. The performance of the developed method was evaluated. The UHPSFC method allowed the baseline separation and determination of 15 sulfonamides and metabolites with a limit of detection ranging from 0.15 to 0.35 g/mL. Recoveries between 90.1 and 102.2% were obtained with satisfactory precision since relative standard deviations were always below 3%. Hence, the proposed method is simple, accurate, time-saving and green; it is applicable to a variety of sulfonamides detection in serum samples.

## Biography

Feng Zhang has completed his PhD from Dalian Institute of Chemical Physics, Chinese Academy of Sciences in the year 2005. He has done his Post-doctoral research from Max Planck Institute of Biochemistry under the funding of Max-Planck Society. He has worked as a Senior Expert of Food Safety at the Chinese Academy of Inspection and Quarantine. In 2014, he was assigned as the Director of the Institute of Food Safety and focusing on Chromatography/Mass Spectrometry Techniques applied to food, tobacco, proteomics, metabolomics and pharmaceutical research. He has published more than 40 papers in reputed journals, 4 books and has authorized 3 patents.

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# Protonic ceramic membranes under asymmetric steam atmosphere

**Sandrine Ricote**[1], **Anthony Manerbino**[2] and **W Grover Coors**[3]
[1]Colorado School of Mines, USA
[2]Solid State Energy, USA
[3]Protonetics, USA

A comprehensive analysis of proton transport in protonic ceramic membrane devices is presented. Thin, dense membranes of BaZr0.8Ce0.1Y0.1O3-d, BZCY81, may now be fabricated with relative ease at commercial scale. These devices have potential for supporting the emerging hydrogen economy and reducing dependence of fossil fuels. With protonic ceramic electrolytes it is possible to galvanically transport pure hydrogen from one side of a membrane to the other, making it possible to fabricate electrochemical devices and systems that were previously impractical or impossible. $H_2$ can be produced from natural gas by steam reforming, whereby hydrogen may be extracted from a reacting stream of methane and steam in a protonic membrane reformer, PMR; Liquid hydrocarbons, such as ethylene and benzene, may be produced from dry methane in a catalytic membrane reactor by methane dehydroaromatization, MDA, with hydrogen extracted from the feed gas; Ammonia can be synthesized by pumping hydrogen through the membrane to react with nitrogen in a process called solid-state ammonia synthesis, SSAS; and $H_2$ can be produced from water vapor by steam electrolysis in a protonic ceramic electrolysis cell (PCEC). In order for these devices to become commercially viable, a clear understanding of their operation in various use environments is necessary. All the devices listed above consume electric power to pump hydrogen across the membrane, which must be supplied by an external power source. The power consumed is the product of the applied voltage and the current consumed by the galvanic device, so it is important that the device have low resistance and high faradaic efficiency with respect to proton transport and that power is not wasted by parasitic losses. The proton current depends on the effective resistance, which depends on electrode performance, faradaic efficiency and bulk materials properties. Well-designed electrodes can, in principle, be developed with low effective resistance, but in the final analysis it is the conductivity of the electrolyte membrane that limits the performance of these devices. The proton conductivity of BZCY81 is only a few millisiemens per centimeter in reducing atmosphere. More importantly, conductivity in BZCY is a strong function of water vapor pressure, making the electrolyte a mixed proton/steam conductor. This is an unusual characteristic that is unique to protonic ceramic electrolytes. The impact depends on the application. For example, MDA requires nominally dry methane on the feed side, while PMR requires moist atmosphere with steam-to-carbon ratio greater than unity. SSAS, on the other hand requires dry atmosphere on the permeate side, where nitrogen reacts with hydrogen to produce anhydrous ammonia. Steam electrolysis is carried out under moist oxidizing conditions on the feed side. In all cases, the desired permeate is hydrogen, as dry as possible to avoid the need for separation of hydrogen from steam, but as a practical matter, some steam will be present either due to steam permeation or added to the sweep gas intentionally. The transport properties of the membrane in each of these devices depend strongly on $pH_2O$ and $pH_2$ on the feed and permeate sides. Knowledge of the electrolyte conductivity as a function of $pH_2O$ and $pH_2$ on each side of the membrane is essential for designing cost-effective galvanic systems since this determines the protonic flux density.

## Biography

Sandrine Ricote has obtained her PhD on Ceramic Proton Conductors at the University of Burgundy, France. She has worked four years in the Department of Energy Conversion and Storage at the Danish Technical University as a Post-doc and then as a Scientist. In 2012, she moved to the Department of Mechanical Engineering at Colorado School of Mines. She is currently a Research Associate Professor with a main focus on ceramic proton conductors.

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# Separation science that's built for biopharma

**Robert Van Ling**
Thermo Fisher Scientific, Netherlands

Biopharmaceuticals now dominate the leading positions in both sales and revenue tables over traditional small molecule drugs and drug pipelines continue to lead by biopharmaceuticals. In addition, as some of the earlier biopharmaceuticals begin to come off patent, we are seeing tremendous growth and attention in biosimilars. Both biopharmaceuticals and biosimilars however present new challenges compared to small molecules due to their complexity and variability; and as such manufacturers need advanced analytical tools to be able to effectively characterize these biopharmaceuticals to ensure efficacy and safety of the product. In this seminar, I will overview the latest advances made by Thermo Fisher Scientific with respect to UHPLC instrumentation and column chemistries for complete biopharmaceutical separation and characterization. The Thermo Scientific™ Vanquish™ Flex UHPLC system is truly built for biopharma offering precise and reproducible separations, coupled with flexibility to execute all major biopharmaceutical applications. Our extensive portfolio of column chemistries combines with the Vanquish Flex to offer a complete solution for biopharmaceutical analysis. Examples of characterizing monoclonal antibodies to determine amino acid sequence, charge variants, aggregates, purity and glycan profiles will be presented.

## Biography

Robert Van Ling has started working at LC Packings, focusing on Nano and Capillary LC. Following the successful introductions of UltiMate, the world's first Nano LC system, LC Packings was acquired by Dionex Corp., in which he has held several roles in conveying the use of Nano LC-MS in Proteomics, for both ESI and MALDI applications and later moving more towards separation of intact proteins and Protein Therapeutics. Since 2012, he has been working as a Member of the Thermo Fisher Scientific family. Currently, he is supporting wide portfolio of chromatography consumables and specifically sample preparation and characterization of large molecules and biologics.

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# Removal of divalent heavy-metal ions from aqueous solutions by adsorption process with titanium dioxide nanowires

**Snezana Maletic[1], Jelena Trickovic[1], Marijana Kragulj Isakovski[1], Srđan Roncevic[1], Jasmina Agaba[1], Akos Kukovecz[2,3]** and **Bozo Dalmacija[1]**
[1]University of Novi Sad, Republic of Serbia
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The key objective of this work was to investigate kinetics and adsorption capacities of divalent metal ions ($Cu^{2+}$, $Pb^{2+}$, $Cd^{2+}$) from water on $TiO_2$ nanowires at pH 3 and 7. Brunauer-Emmett-Teller (BET) analysis showed that the surface area of the TiO2 nanowires was 115,9 $m^2g^{-1}$. The point of zero charge (pHpzc) was 4.8. Adsorption experiments were performed using the conventional batch technique at room temperature (25±2 °C). The background solution was 0.01 M $CaCl_2$ in deionized water. Initial concentrations of heavy metal ions were in the range 0.05-5 mg $L^{-1}$. The amount of adsorbent corresponded to a sample/solution ratio that resulted in 20-80% uptake of given metal ion. The samples were agitated on horizontal shaker for 30 h. The time to reach adsorption equilibrium was obtained from a kinetic study performed over 72 h. The adsorption kinetics of divalent metal ions on $TiO_2$ nanowires was investigated using pseudo-first order, pseudo-second order and intraparticle diffusion models. Adsorption of metal ions was controlled by chemisorption which was supported by the suitability of the pseudo-second order model. Intraparticle diffusion model data showed that diffusion cannot be considered as the limiting step of adsorption. The equilibrium adsorption data were modeled using Freundlich and Langmuir adsorption isotherms which both showed good agreement with experimental data. The highest removal efficiency of heavy metals was observed at pH=7, except in the case of Pb which has been removed better at pH=3. Adsorption affinities increased in the following order: PbpH3>CdpH7>PbpH7>CdpH3>CupH7>CupH3.

## Biography

Snezana Maletic is an Associate Professor and the Chair of Chemical Technology and Environmental Protection, University of Novi Sad, Faculty of Sciences. She has completed her PhD in Chemistry from University of Novi Sad, Faculty of Sciences, Department of Chemistry, Biochemistry and Environmental Protection in 2010 and BSc in Chemistry from University of Novi Sad Faculty of Sciences, Department of Chemistry in 2003. Her research interests include environmental protection, chemical technology, remediation of contaminated water, soil and sediment, bioavailability of inorganic/organic pollutants in sediments/soils investigation and adsorption of inorganic pollutants on nanomaterials. She is Head of the laboratory for the analysis of environmental samples accredited according to ISO 17025 protocols.

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# Development of a liquid-liquid extraction method of resveratrol from cell culture media using solubility parameters

**Mohamad Houssam Al Balkhi**[1, 2], **Mohammad Amin Mohammad**[2, 3], **Leo-Paul Tisserant**[1, 4] and **Michele Boitel-Contia**[1]
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[2]University of Damascus, Syria
[3]University of Bradford, UK
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The extraction of bioactive compounds, produced by plant cell cultures, directly from their culture medium, which contains other by-products, is a great challenge. Resveratrol extraction from its grapevine cell cultures is considered here as an example to improve the extraction processes from plant cell cultures using solubility parameters. Successive liquid-liquid extraction (LLE) processes were exploited to extract resveratrol from the culture medium with an extraction ratio approaching 100%, high selectivity and minimum amounts of solvents. The calculations of partition coefficients as a function of solubility parameters demonstrated that benzyl benzoate is the most suitable intermediate solvent to extract resveratrol from its aqueous medium at a benzyl benzoate/medium ratio of 1:100 v/v. The calculations also illustrated the high ability of methanol and ethanol to extract resveratrol from benzyl benzoate. The physicochemical properties of benzyl benzoate and processing conditions were exploited to separate it from aqueous media and organic solvents. The agitation method, component ratios and extraction time were studied to maximize the extraction yield. Under the best studied conditions, the recovery of resveratrol from different culture media approached approximately 100% with a selectivity of approximately 92%. Ultimately, the improved extraction processes of resveratrol are markedly efficient, selective, rapid and economical.

## Biography

Mohamad Houssam Al Balkhi has obtained his PhD in 2009 from the University of Picardie Jules Verne, France. He has worked as an Assistant Professor at the University of Damascus, Syria for many years. Actually, he is a Post-doctoral Fellow at the University of Picardie Jules Verne working on Plant Biotechnology and the development of extraction methods of active compounds. He has published many papers in reputed journals.

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# Highly selective sieving in porous graphene-like carbon nitride for helium/light isotopes separation

**Yuanyuan Qu**
Shandong University, China

An efficient membrane for helium separation from natural gas is quite crucial for cryogenic industries. However, most experimentally available membranes fail in separating helium from small molecules in natural gas, such as $H_2$, as well as in $^3He/^4He$ isotopes separation. Using first-principles calculations, we theoretically demonstrated that the already-synthesized graphitic carbon nitride (g-$C_3N_4$) has high efficiency in helium separation from the gas molecules ($H_2$, $N_2$, CO and $CH_4$) in natural gas and the noble gas molecules (Ne and Ar). The selectivity of He over $H_2$ molecule at room temperature is calculated to be as high as $10^7$. More interestingly, the g-$C_3N_4$ membrane can also serve as a quantum sieving membrane for $^3He/^4He$ separation with a predicted transmission ratio of 18 at 49 K, thus offers a combined means of both He and $^3He$ isotope separation. Furthermore, for another experimentally available porous graphene-like carbon nitride ($C_2N$-*h*2D), we theoretically demonstrated that highly efficient light isotopes separation, such as $^3He/^4He$, can be reached via quantum sieving effect. Under moderate tensile strain, the quantum sieving of the $C_2N$-*h*2D membrane can be effectively tuned in a continuous way, leading to a temperature window with high $^3He/^4He$ selectivity and permeance acceptable for efficient isotopes harvest in industrial application. This mechanism also holds for separation of other light isotopes, such as $H_2/D_2$, $H_2/T_2$. Such tunable quantum sieving opens a promising avenue for light isotopes separation for industrial application.

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## 2D crystal-based membranes for photocatalysis and separation

**Yuyoung Shin, Kai Ge Zhou, Eric Prestat, Sarah J Haigh** and **Cinzia Casiraghi**
University of Manchester, UK

The isolation of graphene has unveiled a wide range of novel 2-Dimensional (2D) materials with outstanding properties. Liquid-phase exfoliation (LPE) is a simple technique for production of 2D-crystal dispersions, which can be used to form coatings and membranes. 2D crystal-based membranes have already shown interesting properties, such as selective permeation of water, opening the possibility of using these membranes for gas or liquid separation. In this talk, we give two examples of 2D crystal-based membranes. The first membrane is obtained by LPE of graphitic carbon nitride (g-$C_3N_4$), which has been shown to be an efficient photo-catalyst for many reactions under visible light. Photo-degradation studies show that the membranes are very efficient in the degradation of several dyes. This is attributed to the membrane structure: As the catalyst is a porous laminate, the reactant can flow through the pores of the membrane and because the space between the g-$C_3N_4$ nanosheets is comparable to the size of the dyes, the probability of the reactants to be close to the catalyst is enhanced, making the reaction very efficient. The second type of 2D-crystal membrane is prepared by mixing LPE graphene with a polymer of intrinsic microporosity (PIM-1). Graphene is expected to improve membrane permeability, control over diffusion selectivity and to reduce the polymer ageing. Here we show characterization of those membranes by Raman spectroscopy and transmission electron microscopy and we show preliminary results on $CO_2$ permeability.

### Biography

Yuyoung Shin has obtained her BSc in Chemistry from University of Sussex in 2010 and her MPhil in Chemistry from University of Cambridge in 2012, working with Dr Mark Miller on thermodynamics of charged nano-droplets. She has completed her PhD under the guidance of Prof. Cinzia Casiraghi at University of Manchester, working on synthesis and characterization of graphene-based membranes. During her studies, she contributed to a number of publications and recently had two publications on synthesis and characterization of graphene-based membranes.

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2nd International Conference and Expo on

# Separation Techniques

September 26-28, 2016 Valencia, Spain

## Scientific Tracks & Abstracts

## (Day 2)

Day 2 September 27, 2016

# Novel separation techniques in chemistry

**Session Chair**
**Rafael Lucena Rodriguez**
University of Cordoba, Spain

**Session Co-chair**
**Tso-Fu Mark Chang**
Tokyo Institute of Technology, Japan

## Session Introduction

**Title:** **High strength au film fabricated by advanced electrochemical technique in supercritical $CO_2$ emulsified electrolyte for mems accelerometers**
**Chun-Yi Chen,** Tokyo Institute of Technology, Japan

**Title:** **Next generation clarification for processing high density cell culture fluids supplemented with a flocculating agent**
**Sladjana Tomic-Skrbic,** Merck KGaA, Germany

**Title:** **Separation of chiral nanotubes with an opposite handedness by oligopeptide adsorption: A molecular dynamics study**
**Giuseppina Raffaini,** Politecnico di Milano, Italy

**Title:** **Integrating extraction and stirring in microextraction techniques**
**Rafael Lucena Rodriguez,** University of Cordoba, Spain

**Title:** **Nanoscale wiring by cu electrodeposition in supercritical $CO_2$ emulsified electrolyte with continuous-flow reaction system**
**masato Sone,** Tokyo Institute of Technology, Japan

**Title:** **Arsenic speciation in edible marine algae: presence of water and lipid-soluble arsenic compounds**
**Sara Garcia-Salgado,** Universidad Politecnica de Madrid, Spain

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# High strength Au film fabricated by advanced electrochemical technique in supercritical $CO_2$ emulsified electrolyte for MEMS accelerometers

**Chun Yi Chen**[1, 2], **Masaharu Yoshiba**[1], **Haochun Tang**[1], **Tso Fu Mark Chang**[1, 2], **Daisuke Yamane**[1, 2], **Katsuyuki Machida**[1, 2, 3], **Kazuya Masu**[1, 2] and **Masato Sone**[1, 2]
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[2]Japan Science and Technology Agency, Japan
[3]NTT Advanced Technology Corporation, Japan

In recent years, Au has become promising material used as the movable structures and proof mass in micro-electrical-mechanical system (MEMS) accelerometers, because the density of Au is about 10 times higher than that of silicon and the sensitivity of the MEMS accelerometer can be improved with a reduction in the density. However, Au is known to be a soft material and the mechanical strength becomes a concern in miniaturization of the MEMS device. One of the approaches is to decrease the grain size of the Au material to increase the mechanical properties according to the Hall-Petch relation. Pulse plating has been reported to be effective to fabricate metal films with finer grain, higher uniformity and lower porosity. On the other hand, application of supercritical carbon dioxide (Sc-$CO_2$) in electroplating of metal film has also received recent research interests. Physical and transport properties such as density, viscosity, diffusivity and reactant solubility in Sc-$CO_2$ can be adjusted through control of pressure and temperature. Sc-$CO_2$ is often used as a substitute or additive to control overall physical and transport properties of the reaction medium. Surface tension and viscosity of Sc-$CO_2$ are much lower than those of aqueous solution. In this work, grain refinement, surface smoothening and compressive strength enhancement of Au films were achieved by an electroplating using Sc-$CO_2$ emulsified electrolyte.

## Biography

Chun Yi Chen has completed her PhD from Tokyo Institute of Technology, Japan. She was a Research Assistant Professor for next generation battery project in Waseda University from 2012 to 2015. She is currently a Research Assistant Professor of Precision and Intelligence Laboratory in Tokyo Institute of Technology, focusing on advanced electrochemical technique for biomedical materials and devices. She has published many papers in reputed journals and received 6 awards from the international conferences.

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# Next generation clarification for processing high density cell culture fluids supplemented with a flocculating agent

**Sladjana Tomic Skrbic**
Merck KGaA, Germany

The first step in the purification of a therapeutic product is clarification that typically combines multiple separation technologies such as centrifugation, tangential flow microfiltration and depth filtration. Recent advances in mammalian expression systems have led not only to very high cell density cultures and increased protein titers, but also for low viability feed streams with high proportion of solid fraction that made those techniques less attractive. Complexity and issues at scale-up limits the use of the centrifugation, while clarification of such high density cell harvests solely by depth filtration can be costly due to high filter areas required. In this study, we present a complete clarification solution that combines cell harvest pretreatment with a polycationic flocculating agent (pDADMAC), followed by depth filtration using enhanced depth filters that were specifically developed for filtration of flocculated or precipitated feed streams. Multiple antibody feed streams have been treated with pDADMAC and filtered using Clarisolve depth filters resulting in improved removal of cells and cell debris, efficient reduction of DNA and high process yield. Overall, this innovative clarification approach leads to reduced filter area, enhanced impurity removal and process simplification of high density cell harvests that can be readily incorporated into current clarification platforms.

## Biography

Sladjana Tomic Skrbic has studied Biotechnology at Beuth University of Applied Sciences, Berlin, Germany and completed her PhD at Max-Planck-Institute of Biochemistry, Germany and Heidelberg University, Germany. She was a Post-doc at the Martin Luther University Halle-Wittenberg, Germany. Presently she is a Senior Process Development Scientist at Merck KGaA in Darmstadt, Germany, where she provides high quality scientific and technical support to Merck customers developing or manufacturing biopharmaceuticals. She also publishes and presents at conferences on bioprocess technologies.

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# Separation of chiral nanotubes with an opposite handedness by oligopeptide adsorption: A molecular dynamics study

**Giuseppina Raffaini**
Politecnico di Milano, Italy

The separation of enantiomeric chiral nanotubes that can form non-covalent complexes with an unlike stability upon adsorption of chiral molecules is a process of potential interest in different fields and applications. Using fully atomistic molecular dynamics simulations we can study the adsorption and denaturation of an oligopeptide taken from human serum albumin formed by 16 chiral amino acids having a helical structure in the native state on both the inner and the outer surface of the chiral (10, 20) and (20, 10) single-walled carbon nanotubes having an opposite handedness, and of the armchair (16, 16) nanotube with a similar diameter for comparison. In the final adsorbed state, the oligopeptide loses in all cases its native helical conformation, assuming elongated geometries that maximize its contact with the surface through all the 16 amino acids. We find that the complexes formed by the two chiral nanotubes and the chosen oligopeptide have a strongly unlike stability both when adsorption takes place on the outer convex surface of the nanotube, and on the inner concave surface. Thus, the molecular simulations indicate that separation of chiral enantiomeric carbon nanotubes for instance by chromatographic methods can indeed be carried out using oligopeptides of a sufficient length. Moreover, membranes formed by aligned chiral single-walled carbon nanotubes of a given handedness might also act as chromatographic chiral selectors for appropriate racemic mixtures, with also possible application in the field of proteomics. The favorable protein–nanotube interaction would yield significantly different retention times.

## Biography

Giuseppina Raffaini has received her Bachelor's degree in Chemistry and the Post-graduate Diploma at Advanced School in Polymer Science G. Natta; the inter-university Master's in Biomaterials in 2005 and PhD in Materials Engineering from Politecnico di Milano. In 2008, she became an Assistant Professor and Associate Professor in 2014 at the Politecnico di Milano. Her research interests are molecular dynamics simulations of protein adsorption on biomaterials, inclusion complexes and self-assembling of modified cyclodextrins. She is the co-author of 40 original peer-reviewed ISI papers (H-index Scopus=17), 2 invited reviews and 5 contributions to books.

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## Integrating extraction and stirring in microextraction techniques

**Rafael Lucena Rodriguez**
University of Cordoba, Spain

The performance of a given microextraction technique depends on both thermodynamic and kinetic factors. In fact, thermodynamics defines the total amount of analyte that can be isolated while kinetics describes the time required to achieve the mass transference equilibrium. Therefore both aspects, which in some cases may be opposite forces, have to be studied in depth and they have being the focus of an intensive research in the last decades. Agitation is usually found in the scientific literature as a kinetic variable of paramount importance in microextraction techniques since it facilitates the diffusion and therefore the transference, of the analytes from the bulk sample to the solid/liquid phase that acts as extractant. Sample agitation is common to the majority of the microextraction techniques although it can be performed in two different ways. In the simplest approach, the agitation is done by an external element (typically a magnetic bar). Although this strategy has been successfully applied in many well established techniques like solid phase microextraction (SPME) may have two shortcomings, namely the higher turbulence is produced far from the sample/extractant interface and the magnetic bar, especially if it is coated with a polymer, may co-extract a part of the analyte. These shortcomings may be overcome if a second approach, where the agitation element and the extractant phases are integrated in the same device, is applied. This simple idea, which was firstly proposed by Prof. Sandra in the so-called stir bar sorptive extraction, has been exploited in different formats in the last years. This communication tries to give a general overview of the contributions of our research group in this context. The main microextraction techniques developed in our laboratories, including the so-called stir membrane extraction, will be discussed in depth presenting the main research lines for the next years.

### Biography

Rafael Lucena Rodriguez is a Professor at the Analytical Chemistry Department of the University of Cordoba since 2010. He has coauthored 80 scientific articles and several chapters mainly on microextraction techniques. He has been Guest Editor in one special issue of *Analytical and Bioanalytical Chemistry* journal. He is the Editor of Microextraction Tech blog. His main research interest comprises different areas, especially the development of new microextraction techniques as well as the evaluation of ionic liquids and nanoparticles in this context. Currently he is also working on bio-recognition.

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# Nanoscale wiring by Cu electrodeposition in supercritical $CO_2$ emulsified electrolyte with continuous-flow reaction system

**Masato Sone**[1,2], **Tso Fu Mark Chang**[1,2], **Tetsuya Shimizu**[1] and **Nao Shinoda**[1]
[1]Tokyo Institute of Technology, Japan
[2]Japan Science and Technology Agency, Japan

Copper wiring into nanoscale holes with high aspect ratio by electrodeposition is an important problem for 3-D integration in integrated circuit technology toward miniaturization of electronic devices. However, void and pinhole found in Cu wiring for the integration can cause trouble for miniature device. Cu electroplating method without void and pinhole is needed. We have proposed novel electroplating methods with supercritical carbon dioxide (sc-$CO_2$) emulsion (EP-SCE). The electrochemical reaction is carried out in an emulsion of sc-$CO_2$ in electrolyte with surfactants. Sc-$CO_2$ has low viscosity and compatibility of hydrogen. Thus, this method is applicable in fine Cu wiring. The aim of this report is to examine Cu electrodeposition by using sc-$CO_2$ emulsified electrolyte into nano-scale Cu wiring on the viewpoints of dissolution of Cu seed layer, gap-filling capability into nano-scale holes and contamination in the plated Cu. Moreover a continuous-flow reaction system is proposed and examined for filling of Cu into holes with 60 nm in diameter and aspect ratio of 2 and 5 by EP-SCE on a round-type large-area hole test element group with diameter of 300 mm, which has an integrated structure of Cu seed layer on TiN barrier layer sputtered on Si substrates.

## Biography

Masato Sone has completed his Doctor degree of Engineering at Tokyo Institute of Technology. He has worked as a Researcher in Nippon Oil Company from 1996 to 2000. He was an Assistant Professor and then a Research Associate Professor at Tokyo University of Agriculture & Technology from 2000 to 2005, and became an Associate Professor at Tokyo Institute of Technology in 2005 and working till date. He has published more than 127 papers in scientific journals and 18 books. His majorities are microelectronics, surface finishing, chemical engineering, liquid crystal and polymer science. His recent research topic has been "Novel Nano Wiring Process Using Supercritical Carbon Dioxide for Integrated Circuit Technology".

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# Arsenic speciation in edible marine algae: Presence of water and lipid-soluble arsenic compounds

**Sara Garcia Salgado[1], Georg Raber[2], Kevin A Francesconi[2] and M Angeles Quijano[1]**
[1]Universidad Politecnica de Madrid, Spain
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Nowadays, there is considerable interest in arsenic speciation in food products due to the different toxicity exhibited by the different arsenic compounds. This is accentuated in the case of marine algae, because they contribute substantial amounts of arsenic to the human diet and their consumption is increasing due to their properties as food additives, nutritional values and suggested medical applications. Organic arsenic compounds are abundant in marine ecosystems. Although most of the arsenic compounds identified so far have been water-soluble species, the early work on arsenic marine chemistry focused on lipid-soluble compounds. In 1988, an arsenolipid was first rigorously characterized and identified as an arsenosugar-containing phospholipid in algae. Subsequently, several arsenic-containing fatty acids and hydrocarbons have been discovered in different fish products, which origin was presumed to be algae. In this work, we report the water and lipid-soluble arsenic compounds found in 9 commercially available edible marine algae from Japan and Spain. The extraction of water-soluble arsenic species was performed by microwave-assisted extraction, using deionized water as extracting agent, and they were determined by HPLC-(UV)-HG-AFS. Lipid-soluble arsenicals were extracted by mechanical shaking with a (2:1; v/v) chloroform/methanol mixture, purified by SPE on home-made silica columns and determined by online HPLC-ICPMS/ESMS analysis. 6 water-soluble arsenic species, comprising DMA, As(V) and 4 arsenosugars (glycerol, phosphate, sulfonate and sulfate sugars), as well as 14 lipid-soluble arsenic species (3 arsenic-containing hydrocarbons and 11 arsenosugar-containing phospholipids), were found in the water and chloroform extracts, respectively, of the edible marine algae analyzed.

## Biography

Sara Garcia Salgado has completed her PhD in 2013 from Technical University of Madrid, Spain. She is an Assistant Professor in this University and Deputy Director for External Relations at School of Civil Engineering. She has published 10 papers in reputed journals and published two books.

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**Notes:**

Day 2 September 27, 2016

# Separation Processes in Chemical Engineering

**Session Chair**
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**Session Co-chair**
**Adolfo Iulianelli**
CNR-ITM, Italy

## Session Introduction

**Title: Fabrication of Pt metallization on silk via supercritical carbon oxide-assisted electroless plating for wearable medical devices**
**Wan-Ting Chiu,** Tokyo Institute of Technology, Japan

**Title: Pervaporative removal of polyaromatic hydrocarbons from model diesel composition using a fabricated polyimide membrane and process optimization**
**Debarati Mitra,** University of Calcutta, India

**Title: Pd-alloy supported membranes: hydrogen separation/purification and membrane reactor applications**
**Adolfo Iulianelli,** CNR-ITM, Italy

**Title: Effect of pressure on crystal structure of metal oxides formed in supercritical $CO_2$ emulsified solution**
**Tso-Fu Mark Chang,** Tokyo Institute of Technology, Japan

**Title: Fabrication of electrospun nanofiber mat for tetracycline adsorption**
**Liu Qing,** Chinese Academy of Sciences, China

**Title: Separation of cerium from other rare earth elements by solvent extraction**
**Carlos Antonio de Morais ,** Nuclear Technology Development Center, Brazil

**Title: Analysis of the transport of the individual gas mixture components in polymers of intrinsic microporosity and PIM-based mixed matrix membranes**
**Johannes Carolus Jansen,** CNR-ITM, Italy

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# Fabrication of Pt metallization on silk via supercritical carbon oxide-assisted electroless plating for wearable medical devices

**Wan Ting Chiu**[1, 2], **Yuma Tahara**[3], **Chun Yi Chen**[1, 2], **Tso Fu Mark Chang**[1, 2], **Tomoko Hashimoto**[3], **Hiromichi Kurosu**[3] and **Masato Sone**[1, 2]
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[2]Japan Science and Technology Agency, Japan
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As the medical technology advances, the requirements of the next-generation healthcare devices are urgently demanded. Implantable and wearable medical devices are the latest applications over the decades. Nickel, copper and aluminum are widely used in the aforementioned devices because of the simple process and low cost, however, adverse reactions such as allergies and Alzheimer's disease might take place due to the releasing of metal ion. A biocompatible material thus becomes the most urgent demand. Platinum is considered to be the most promising material owing to its irreplaceable biocompatibility. Moreover, silk is a common material used in clothes. The combination of Pt and silk is considered to be a promising candidate for the medical devices. Electroless plating can put these composite materials into practice and further achieve homogeneous metallized-surface due to the low deposition rate. Typical electroless plating consists of pretreatment to clean and roughen the surface, catalyzation to embed the catalysts as a nucleation site into the substrate, and the plating step for the metallization. In spite of the dominance of Pt, electroless plating of Pt remains less studied due to the hardship of controlling the deposition of platinum by electroless technique via the traditional catalyzation. An up-to-date technique of supercritical carbon dioxide ($sc\text{-}CO_2$) assisted catalyzation is practiced in this study to overcome the instinct difficulty of Pt plating. With the help of the exceptional self-diffusivity, low surface tension, and affinity to non-polar materials of $sc\text{-}CO_2$, the catalyst can be inlaid to the substrate while the substrate structure remains undamaged.

## Biography

Wan Ting Chiu is currently a Doctoral student majoring in Materials Science and Engineering in Tokyo Institute of Technology. She has received her MS from Chemical Engineering Department at National Tsing Hua University in 2014. She has worked as a Research Assistant in Institute of Physics, Academic Sinica. She has a strong background in phase diagram and phase equilibria. Her current research topic is metallization of flexible texture for medical wearable devices.

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# Pervaporative removal of polyaromatic hydrocarbons from model diesel composition using a fabricated polyimide membrane and process optimization

**Debarati Mitra, Monalisha Samanta, Sania Yasmin** and **Sayan Roychowdhury**
University of Calcutta, India

Diesel combustion in transportation and other industrial activities release toxic air pollutants like polyaromatic hydrocarbons (PAHs) that can cause serious health effects. According to the worldwide fuel charter, PAH in diesel has to be limited to 2% m/m (max.). Removal of PAHs from diesel is conventionally achieved via hydroprocessing which is hazardous, expensive and gives low conversions. Membrane pervaporation, a unique combination of permeation and evaporation which is comparatively a simple and inexpensive method can be applied for separating PAHs from diesel efficiently. An aromatic polyimide membrane was fabricated and successfully used for the said purpose. The efficiency of the process was evaluated in terms of permeation flux of PAHs. The effect of different physicochemical parameters on the permeation flux was investigated and the process was optimized using response surface methodology with a view to maximize the flux of PAHs.

## Biography

Debarati Mitra has completed her MTech degree from the University of Calcutta and subsequently completed her PhD in Chemical Engineering from Jadavpur University. She is presently working as an Assistant Professor in the Department of Chemical Technology, University of Calcutta. She has published 18 papers in reputed international journals and has been engaged in research in the fields of membrane separation processes, biotechnology and biopolymers.

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# Pd-alloy supported membranes: Hydrogen separation/purification and membrane reactor applications

**Adolfo Iulianelli, Giuseppe Bagnato** and **Angelo Basile**
CNR-ITM, Italy

The main industrial process for producing hydrogen is represented by the natural gas reforming, which consists of a multi-step process in which the steam methane reforming takes place in harsh conditions (800-1000 °C and 15-20 bar), followed by two water gas shift reactors and other separation/purification stages for producing high purity hydrogen. However, the natural gas composition of the feeding stream can vary widely from source to source, but each of them contains some traces of $H_2S$. This can damage dramatically the permeation characteristics of the Pd-based membrane and, consequently, affect the overall performance of the membrane reactor. In the last years, many authors studied the preparation of composite membrane based on Pd alloyed with other metals, such as Au, Ag, Pt or Cu, responsible of higher resistance to sulfur contamination. In this study, a composite membrane constituted of a thin layer of Pd-Au supported on porous supports has been fabricated for their utilization in hydrogen separation field. For comparison, a supported pure Pd-membrane has been also studied in order to compare the performance under long-time operation of the different membranes and for evaluating the $H_2S$ effects on both systems. They have been also used in membrane reactors to carry out steam methane reforming reaction, by comparing the experimental results with an equivalent conventional reactor.

## Biography

Adolfo Iulianelli has completed his Degree in Chemical Engineering and PhD in Chemical and Material Engineering. Presently, he works at the Institute on Membrane Technology of the Italian National Research Council. He has published more than 50 articles in international scientific ISI journals, more than 20 chapters in international books, author of one patent and a book and of more than 50 papers in proceedings of national and international conferences. He is a Reviewer of more than 20 scientific ISI journals, Associate Editor of *International Journal of Membrane Science & Technology*, Editor of *Journal of Membrane Science and Technology*, *Advances in Chemical Engineering and Process Technology, Journal of Fuels and Scientific World* as well as serving as Guest Editor for *International Journal of Hydrogen Energy*.

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# Effect of pressure on crystal structure of metal oxides formed in supercritical $CO_2$ emulsified solution

**Tso Fu Mark Chang[1, 2], Wei Hao Lin[1, 3], Chun Yi Chen[1, 2], Yung Jung Hsu[3] and Masato Sone[1, 2]**
[1]Tokyo Institute of Technology, Japan
[2]Japan Science and Technology Agency, Japan
[3]National Chiao Tung University, Taiwan

Metal oxides such as $TiO_2$ and ZnO are widely applied as biosensors, biomaterials and as the support in a drug delivery system because of the high biocompatibility. The synthetic approaches include hydrothermal methods, cathodic deposition, anodic oxidation and sol-gel method. Among them, cathodic deposition offers a low-cost yet effective process for production of metal oxides with controllable morphology. However, the as-deposited $TiO_2$ and $SnO_2$ are usually amorphous. An additional heat treatment process, for example, annealing at 400°C for 1 hr, is needed to obtain crystalline $TiO_2$. The need of the post-heat hinders the applicability of the products. Therefore, it is practically significant if crystalline metal oxides can be obtained directly from the cathodic deposition without the additional heat treatment, or lower the temperature needed in the heat treatment. On the other hand, the effect of pressure on the crystallinity of metal oxides deposited from the solution phase is rarely investigated. In a previous study, grain size of the $TiO_2$ cathodically deposited with a supercritical $CO_2$ (sc-$CO_2$) emulsified electrolyte (SCEM) was found to be increased with an increase in the pressure. In this later study, crystal structure of the $TiO_2$, ZnO, and $SnO_2$ were found to be affected by the applied pressure used during the deposition. The as-deposit $TiO_2$ and $SnO_2$ were found to be composed of nano-crystallines when the SCEM was applied. In this presentation, the effect of pressure on crystal structure of metal oxides deposited using the SCEM will be reported.

## Biography

Tso Fu Mark Chang has received his BASc in Chemical Engineering from the University of Toronto (2004), MS in Chemical Engineering from National Tsing Hua University (2007) and PhD in Materials Science and Engineering from Tokyo Institute of Technology (2012). He is currently an Assistant Professor of Precision and Intelligence Laboratory at Tokyo Institute of Technology. His research interests include pressure and solvent effects on reactions in supercritical $CO_2$ and characterization of the materials fabricated in supercritical $CO_2$. He has published more than 50 papers in reputed journals.

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2nd International Conference and Expo on
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# Fabrication of electrospun nanofiber mat for tetracycline adsorption and other membrane separation processes

**Liu Qing**
Chinese Academy of Sciences, China

Electrospinning is a novel technique to produce nanofibers with a diameter of 100 nm to >1. In electrospinning, when a high voltage is supplied to a liquid droplet, a charged liquid jet is formed. The bending instability between the jets cause thinning and elongation of the fibers, subsequently, resulting in formation of uniform nanofibers, which have high specific area, porosity and interconnecting pore structures, thus, are suitable for energy and environmental applications. In our work, a novel $Fe_3O_4$/polyacrylonitrile (PAN) composite nanofiber mat was prepared by a simple two-step process, electrospinning and solvothermal method. Surface characterization demonstrated formation of a uniform cubic phase $Fe_3O_4$ nanoparticles coating (about 20 nm in thickness) on the PAN nanofiber backbone. The coating doubled the specific surface area of NFs, from 8.4-17.8 $m^2g^{-1}$, as confirmed by nitrogen sorption isotherm analysis. To evaluate the feasibility of Fe3O4/PAN composite NFs as a potential adsorbent for antibiotic removal, batch adsorption experiments were conducted using tetracycline (TC) as the model antibiotic molecule. The results showed that $Fe_3O_4$/PAN composite NFs was effective in removing tetracycline with no impactful loss of Fe at pH regime of environmental interest (4 to 8). The maximum adsorption capacity calculated from Langmuir isotherm model was 257.07 mg $g^{-1}$ at pH 6. The composite NFs also exhibited good regenerability over repeated adsorption/desorption cycles. This highly effective and novel adsorbent can be easily modularized and separated, promising its huge potential in drinking and waste water treatment for antibiotic removal.

## Biography

Liu Qing is currently pursuing her PhD at Institute of Urban Environment, Chinese Academy of Sciences, China. She has received her Master in Chemical Engineering at National University of Singapore. Her current research focus is in solving environmental issues such as water contamination and recovery, air pollution, using membrane separation techniques, in particular, by electrospinning. She has published 5 papers in reputed journals.

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## Separation of cerium from other rare earth elements by solvent extraction

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This work presents an investigation of solvent extraction parameters to obtain high purity cerium from a mixture containing other rare earths elements, in alternative to the oxidation and selective precipitation or dissolution. The study was carried using a sample of a sulphuric liquor obtained from monazite leaching rich in light rare earth elements (La, Ce, Pr, Ne) provided by INB (Indústrias Nucleares do Brasil). Tests were realized in chloridric, nitric and sulfuric medium. For the experiments in nitric and chloridric medium, the rare earth elements from the liquor were precipitated in rare earths oxalate form, precipitated and then dissolved in the respective medium. The parameters investigated were: Type and concentration of oxidant agent, type and concentration of extractant, liquor acidity and volumetric ratio between organic and aqueous phases. Preference of the organic phase to extract cerium in its oxidase form (IV) above the other rare earths elements, allowing the obtainment of a high purity cerium solution was confirmed. Best results were achieved in nitric medium, P507® as extractant and a mixture of potassium persulfate and silver chloride as oxidant agents, yielding over 98% cerium extraction with over 99% of purity.

### Biography

Carlos Antonio de Morais has a BS degree in Chemistry from UFMG (graduated in 1990) and PhD in Metallurgical Engineering and Mining, also from UFMG in 2002. He is a CNPq researcher level 2 and Researcher in CDTN-Nuclear Technology Development Center, an agency belonging to the CNEN (National Nuclear Energy Commission) since 1990. He operates in the development, optimization and application of hydrometallurgical processes, using mainly leaching techniques, solvent extraction and chemical precipitation.

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# Effective adsorption and concentration of carnosine by nickel species within mesoporous silica

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Owing to the abundant existence, low-value food stuff, such as egg-laying hens, might be cheap resource of histidine-containing peptides, such as carnosine (Car), which have various functions with attractive application as food supplements. Herein, carboxyl group functionalized mesoporous silica of SBA-15 was synthesized to facilely incorporating NiO, ZnO and CoO for Car extraction. Among these, Ni/SBA-15 has the highest adsorption capability. The correlation between the most significant parameters such as adsorbent dose, pH, background salts, contact times, Car concentration and the elution was optimized, and the effects of these parameters on the adsorption efficiency of Car were investigated. Thanks to the adequate pore size and high Ni loading, the adsorption capacity of Car onto $Ni_{50}$/SBA-15 approached as high as 0.839 mmol (188.4) $g^{-1}$. The excellent adsorption characteristics of the current adsorbents toward Car were preserved in a wide pH window and could be hardly infected by the concentration of the background salts. The pseudo-second-order rate equation effectively described the uptake kinetics. The Langmuir model exhibited a better fit to adsorption isotherm than the Freundlich model. Therefore, nickel immobilized carboxyl functionalized SBA-15 is an efficient method for recovering histidine-containing peptides from muscle slurry of egg-laying hens and makes them favorable candidates as chromatographic column materials for HCPs analysis.

## Biography

Liming Zhao has completed his PhD from Jiangnan University (China). He is Professor and Director of a research center focusing on separation and purification technology (membrane and chromatography) at East China University of Science and Technology. He has published more than 50 papers in reputed journals and 2 monographs, and serving as an Editorial Board Member of repute.

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# Separation Techniques

September 26-28, 2016 Valencia, Spain

## Scientific Tracks & Abstracts

## (Day 3)

# Chromatography as a Separation Technique | Hyphenated Techniques | Role of Spectroscopy as Separation Techniques

**Session Chair**
**Hayrunnisa Nadaroglu**
Ataturk University, Turkey

**Session Co-chair**
**Snežana Maletic**
University of Novi Sad, Republic of Serbia

## Session Introduction

**Title:** **Anion exchange chromatography in lignocellulosics analysis**
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**Title:** **SFC-MS as a tool for flower absolute composition analysis**
**Santerre Cyrille ,** ISIPCA, France

**Title:** **Evaluation of solid-phase chromatography as a method for natural organic matter characterization**
**Aleksandra Tubic,** University of Novi Sad, Republic of Serbia

**Title:** **Robustness test of the chromatographic method for the quantification of chlorogenic acid in coffee brew**
**Jong-Sup Jeon,** Gyeonggi Province Institute of Health and Environment, Republic of Korea

**Title:** **Purification, characterization of lipase enzyme from lactobacillus brevis and immobilization onto magnetic florosil nps**
**Hayrunnisa Nadaroglu,** Ataturk University, Turkey

**Title:** **Ultrahigh pressure liquid chromatography as a separation technique for the simultaneous extraction and determination of four different groups of pharmaceuticals in compost**
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**Title:** **Simple HPLC–MS/MS method for simultaneous determination of aripiprazole and dehydroaripiprazole in human plasma using microelution solid phase extraction**
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**Title:** **Supercritical $CO_2$ in its multiple roles: solvent for fractionation of complex mixtures and blowing agent in polymer foaming**
**Martin Topiar,** University of Chemistry and Technology Prague, Czech Republic

**Title:** **Resolving terahertz spectral mixtures using the blind source separation approach – A method to study the dehydration kinetics**
**Lukasz Antoni Sterczewski,** Princeton University, USA

**Title:** **Numerical analysis on purification of methane through the hollow fiber membrane system**
**Yongtaek Lee,** Chungnam National University, Korea

**Title:** **Purification of phosphoric acid by liquid-liquid**
**Mariana Campos Assuncao,** PSL Research University, France

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## Anion exchange chromatography in lignocellulosics analysis

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Complex liquid samples such as lignocellulose hydrolyzates which contain both polar and nonpolar analytes as well as monomeric and oligomeric compounds are hard to analyze and require usually more than one analytical separation mechanism. In contrast, the reversed-phase mechanism anion exchange chromatography (AEC) allows for a separation of divergent analytes based on the acidity of the resulting anion. Thus, the aim of this study was to investigate the potential of AEC to analyze lignocellulosic biomass hydrolyzates completely using one analytical separation mechanism, as lignocellulose hydrolyzates contain a lot of potential anions with a variation in polarity and size. Therefore, the potential of generating anions from aldehydes, alcohols and phenols was investigated using an alkaline eluent. Additionally, the concentrations of aldehydes, alcohols and phenols derived from lignins were measured simultaneously with monosaccharides, oligosaccharides and uronic acids derived from cellulose, hemicellulose and pectin. Thus, parameters such as column temperature, eluent composition and chromatographic run time were examined. The final chromatographic method was set to a column temperature of 40°C, an eluent flow of 1 ml/min and an eluent consisting of sodium acetate and sodium hydroxide as well as ultrapure water. This method allows for a complete characterization of lignocellulose hydrolyzates with limit of detections in the range of 0.014 mg/L for 2,6-dimethoxyphenol and 21.9 mg/L for 4-methoxybenzyl alcohol. Finally, this method was used to characterize 17 hydrolyzates from lignocellulosic biomass simultaneously for cellulose, hemicellulose, lignin and pectin derived degradation products.

### Biography

Nico Anders has been working in the field of analysis and renewables since 2009. He has obtained his PhD from the TU Braunschweig in the group of Prof. Dr. Vorlop in Technical Chemistry. Since 2013, he is working as a Junior Research Group Leader in the Aachener Verfahrenstechnik at the RWTH Aachen University. His research interests are analysis of lignocellulosic biomass, green analytical chemistry, conversion of lignocellulosic biomass and chromatographic separation.

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## SFC-MS as a tool for flower absolute composition analysis

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ISIPCA, France

Cosmetics and perfumes industries are very late for using supercritical fluids compared with pharmaceutical industry, for example. The protection and the environmental respect became a major subject of concern nowadays. Furthermore, with the increasing interest of the professionals for the natural raw materials with the complexity of their compositions, supercritical fluid chromatography (SFC) seems to be imperative to us as green chemistry tool. Hyphenation is very easy with mass spectrometry (MS) using various ionization sources (ESI, APPI, APCI). European legislation regulates 26 fragrance allergens at the moment with an exceeding stipulated cut-off level. In the near future, 32 other allergens will be newly added. In order to decrease analysis time and improve specificity, SFC coupled with diode array detector (DAD) has been tested. First step consists in stationary phase screening (Si, Hypercarb) using isocratic mode (95% $CO_2$/5% Ethanol as modifier). Hypercarb was selected as the most retentive stationary phase for this type of analysis. Then chromatographic resolution has been improved by checking the effect of column temperature, $CO_2$ back pressure, nature of modifier (methanol, ethanol, isopropanol) and gradient mode. First results indicate that at least 20 compounds can be efficiently separated in less than 12 minutes. Subsequently two methods have been developed: One, SFC-UV to quantify major components in essential oils (e.g. Eucalyptus and clove) and as second one based on SFC-MS to study flower absolutes composition. Indeed volatile compounds are already well known and described in the literature using gas chromatography (GC) coupled with MS but unvolatile fraction remains incompletely described.

### Biography

Santerre Cyrille has completed his Analytical Chemistry Engineering degree and Cosmetology Master's degree from the Conservatoire National des Arts et Metiers (CNAM) and University of Chatenay Malabry Paris XI, France. He is a Lab Manager and Member of a research team focusing on supercritical fluid chromatography (SFC) at ISIPCA and mass spectrometry at ICSN-CNRS (Gif sur Yvette-France).

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# Evaluation of solid-phase chromatography as a method for natural organic matter characterization

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University of Novi Sad, Serbia

Natural organic matter (NOM) is a ubiquitous constituent of surface and ground waters worldwide. It is well known that various inorganic and organic species interact with NOM in water matrices and these interactions can have a great impact on their mobility and behaviour, as well as the expression of toxic effects. NOM, if present in source water, can have a great negative influence on drinking water treatment, through disinfection by-products formation, requirements for increasing amounts of chemicals during treatment and deterioration of water quality in the distribution system. NOM is generally site specific, and the approximation of its influence is rather difficult to make based on simulations or previous knowledge gained from other sites. Thus, the characterization of NOM at the specific site of interest is of great help in understanding the processes in ambient waters as well as during treatment. There are various possibilities for NOM characterization, including solid-phase fractionation using XAD resins. The aim of this paper is to evaluate solid-phase chromatography as a method for NOM fractionation through a review of the results obtained by fractionation of 3 ground waters and 1 surface waters; which was repeated 2-5 times for each water type. The method is based on fractionation of the dissolved organic matter into four fractions: The humic acid, fulvic acid, hydrophilic acid and hydrophilic non-acid fractions; and measuring the DOC value after the fractionation. The fractionation method showed great recovery values for DOC (> 98%) compared to the DOC measured in the bulk water samples. The relative standard deviations (RSD) of the DOC values measured in the same fraction in each water sample range from 0.71-9.9%. An additional benefit of the method is that it gives satisfactory recovery results even after resin regeneration, with recoveries of 99%, 97% and 88%, for the resins used before and after the first and second regeneration cycles.

## Biography

Aleksandra Tubic is an Assistant Professor at the Chair of Chemical Technology and Environmental Protection, University of Novi Sad, Faculty of Sciences. She has completed PhD in Chemistry in 2010 and BSc in Chemistry from University of Novi Sad in 2003. Her research interests include environmental protection, chemical technology, ambient and drinking water treatment. She has also worked as Quality Manager of the laboratory for the analysis of environmental samples, accredited according to ISO 17025 protocols.

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# Robustness test of the chromatographic method for the quantification of chlorogenic acid in coffee brew

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Coffee has been for decades the most commercialized and widely consumed beverage in the world. Coffee beans contain a large variety of substances, which in many cases are biologically active such as caffeine and chlorogenic acids. Chlorogenic acids are water-soluble phenolic components of coffee and other plants formed by the esterification of certain trans-cinnamic acids, such as caffeic, ferulic and p-coumaric acids with quinic acid. The main subgroups of chlorogenic acid isomers in coffee are the caffeoylquinic acids, feruloylquinic acids, dicaffeoylquinic acids and in smaller amounts p-coumaroylquinic acids. In this study, we carried out robustness evaluation for method validation of quantification of chlorogenic acid in coffee brew using Youden's test. It was possible to determine the effect of each analytical parameter in the final analysis results. Seven analytical factors were selected and small variations were induced in the nominal values of the method. Then, 8 runs were performed aiming to determine the influence of each parameter in the final result. The 7 factors are: Concentration of $KH_2PO_4$ in mobile phase, column temperarture, flow rate, wavelength of detector, column supplier and initial mobile phase composition. Standard deviation of the differences Di (SDi) was calculated and that value was not larger than the standard deviation of the method carried out under within-laboratory reproducibility conditions. The experimental *t* values resulted below the 2-tailed t-critical value for all 7 factors. The tested procedure proved to be fairly robust since minor fluctuations in the operative parameter that can occur during the routine application of the method do not significantly affect its performance characteristics.

## Biography

Jong Sup Jeon has completed his PhD from College of Veterinary Medicine, Konkuk University, Republic of Korea. He is a DVM and Research Scientist for the Public Health in Gyeonggi Province, Institute of Health and Environment, Republic of Korea. He has published variable domestic and international papers (subjects: Antibiotics, hazardous materials in food and functional ingredients in cosmetics) in reputed journals and served as a co-worker in various field.

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# Purification and characterization of lipase enzyme from *Lactobacillus brevis* and immobilization onto magnetic florisil NPs

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Lipases (E.C.3.1.1.3; triglycerol acylhydrolases) are enzymes catalyzing reversible hydrolysis of animal and vegetable oils under normal conditions. Besides, they also catalyze reactions such as esterification and transesterification. In this study, a new lipase enzyme was isolated from *Lactobacillus brevis* and immobilized onto modified florisil with iron NPs and the usability of free and immobilized lipases as a detergent additive material was investigated. Lipase enzyme was purified using ammonium sulphate precipitation, DEAE-Sephadex ion-exchange chromatography and sephacryl S200 gel filtration chromatography techniques. Its molecular mass was determined to be 57 kDa by SDS-PAGE and gel filtration chromatography. Purified lipase was immobilized onto magnetic florisil NPs and determined immobilization conditions. Also immobilized lipase characterization was done using SEM, FTIR and XRD techniques. Immobilized lipase showed good thermo-stability and retained its activity at 80%, than free lipase enzyme at 60°C. The free and immobilized lipase enzymes were most stable in the alkaline pH. Also, immobilized lipase had more stability towards metal ions than free lipase enzyme. Washing performances of some detergents formulation were done and maximum percentage of olive oil was removed by the immobilized lipase than commercial detergents. The study on oil stain removal from cotton cloth indicated that oil removal was superior in the presence of immobilized lipase and immobilized lipase with detergent than the detergent alone.

## Biography

Hayrunnisa Nadaroglu has completed her PhD and MSc from Ataturk University, Graduate Institute of Sciences, Department of Biochemistry (Erzurum, Turkey) in Bioorganic Reactions using purified carbonic anhydrase isoenzymes. She is a Scientific Expert in process development on bioremediation of waste water, some nano-biotechnological applications onto environmental pollution and some industrial enzyme applications onto food technology (clarification of fruit juice, hydrolyzation of phytate etc.). She has published more than 120 papers in the field of biochemistry, food technology and nano-biotechnology in journals and conferences.

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# Ultrahigh pressure liquid chromatography as a separation technique for the simultaneous extraction and determination of four different groups of pharmaceuticals in compost

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Extracting and separating pharmaceuticals from complex environmental matrices such as compost is particularly challenging because of the complex nature of the samples, the different chemical characteristics of the compounds and the low detection limits required. In this study, ultrahigh pressure liquid chromatography (UHPLC) was used as a separation technique for the simultaneous extraction and determination of 4 different groups of pharmaceuticals in compost obtained from the thermophilic aerobic treatment of placenta. The pharmaceuticals were 2 non-steroidal anti-inflammatory drugs, ketorolac and naproxen, usually administered to humans; two fluoroquinolones- ofloxacin and ciprofloxacin (which are among the most commonly prescribed class of antibiotics in Mexico); 2 anti-cancer (antineoplastic or cytotoxic) chemotherapy drugs- ifosfamide and cyclophosphamide and 2 β-blockers-atenolol and propranolol, also called β-adrenergic blocking agents, which treat a variety of conditions, such as high blood pressure, glaucoma and migraines. The pharmaceuticals of each group were selected because they are commonly used in Mexico and environmental and health impacts have been reported. The clustering was based on the use of the drug and not on the similarity of the structure. The use of UHPLC allowed better detection and quantification of all pharmaceuticals; furthermore, shorter analysis time was required and lower costs were involved. Recovery values of the ultrasonic extraction for all compounds were on the range of 87% and 113%. The limits of detection and quantification for the eight pharmaceuticals were on the order of 0.66 ng $g^{-1}$ and 2 ng $g^{-1}$ respectively for all the pharmaceuticals analyzed. These values are lower than those values reported in the literature.

## Biography

Miguel Angel Lopez Zavala has completed his PhD and Post-doctoral studies at the Hokkaido University, Japan, in the field of Urban and Environmental Engineering. He is the Professor at the Tecnologico de Monterrey, Mexico. He is a Member and Evaluator of the National Research System of the National Council of Science and Technology. He was a Researcher of the Japan Science and Technology Agency at Hokkaido University, Japan. He is the author of more than 55 scientific papers published in international journals and proceedings of international conferences and congresses. He is an active Member of the International Water Association and a Treasurer of la IWA-Mexico from 2010 to 2011.

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# Simple HPLC-MS/MS method for simultaneous determination of aripiprazole and dehydroaripiprazole in human plasma using microelution solid phase extraction

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A selective and accurate high pressure liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) method has been developed and validated for simultaneous monitoring of Aripiprazole and its active metabolite Dehydroaripiprazole in human plasma using aripiprazole-d8 as the internal standard (IS). The analytes and IS were extracted from 200 µL of human plasma by solid-phase extraction using Oasis PRiME HLB 96-well µElution Plate, 3 mg sorbent per well (Waters, Madrid, Spain). Separations were carried out at 25°C in an ACE C18-PFP column (4.6 mm×100 mm and 3-µm particle size (SYMTA, Madrid, Spain) protected by a 0.2-µm on-line filter. The mobile phase consisted of a combination of 0.2% formic acid and 0.3% ammonia in MilliQ water pH=4.0 (solution A) and ACN (solution B) (65:35, v/v). The chromatogram was run under gradient conditions at a flow rate of 0.6 mL/min. Run time was 5 min followed by a re-equilibration time of 3 min, to give a total run time of 8 min. The volume injected into the chromatographic system was 5 µL. The analytes were detected using the mode multiple reaction monitoring in the positive ionization mode. The linearity of the method was established in the concentration range 0.15-110 ng/mL and 0.35-100 ng/mL for Aripiprazole and Dehydroaripiprazole, respectively. We validated the analytical method according to the recommendations of regulatory agencies through tests of precision, accuracy, recovery, matrix effect, stability, sensitivity and selectivity. The method was applied to 6 different bioequivalence studies of 10 mg aripiprazole formulation in 40 healthy Caucasian subjects.

## Biography

Wojnicz A is currently pursuing PhD from Autonomous University of Madrid, Spain. She is a Bioanalyst Scientist, working at Analytical and Pharmacokinetic Unit of Clinical Pharmacology Service of 'Hospital Universitario de la Princesa', Madrid, Spain. She has spent 3-months period at Department of Pharmacy & Pharmaceutical Science and Biochemistry of University of California San Diego (UCSD) to improve her knowledge with experts in mass spectrometry. She has published more than 7 papers in reputed journals.

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# Supercritical $CO_2$ in its multiple roles: Solvent for fractionation of complex mixtures and blowing agent in polymer foaming

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Carbon dioxide in its supercritical state (sc-$CO_2$) can find application in formation of microcellular polystyrene foams (PSF) with improved thermal properties in comparison with PSF on the market. A most important application of sc-$CO_2$ on industrial scale is supercritical fluid extraction (SFE) from plants. Both processes have been studied in our laboratory. Although the SFE is more selective than common extraction methods, $CO_2$ extracts still contain a mixture of chemical compounds. If a high concentrated isolate is required, suitable fractionation methods have to be involved into the process. Supercritical adsorption, a novel promising method combining SFE from plants with selective adsorption of extracted compounds, could be a solution when high purity isolates free of any traces of organic solvents are required. We used this method for fractionation of turmeric (*Curcuma longa* L.) isolate obtained by SFE. The major compounds analyzed by GC were turmerone (22.7 wt.%), ar-turmerone (13.2 wt.%) and curlone (13.8 wt%). Turmerones belong to widely studied substances with potential use in the treatment of neurodegenerative diseases. The efficiency of fractionation was studied in terms of adsorption conditions, adsorbent type and sorbent-to-feed ratio. The concentration of turmerones in isolate increased from the initial 49.7 wt.% up to 93.8 wt% with using a particular type of silica gel. Moreover, the concentration of β-sesquiphellandrene, a compound with a potential anticancer activity, in volatile fraction obtained from supercritical adsorption process was almost 16 times higher than in the initial sample. These are promising results for a more detailed subsequent research on this method.

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## Biography

Martin Topiar has completed his Master of Science in Synthesis and Production of Drugs from University of Chemistry and Technology Prague. Presently he is a PhD student at Institute of Chemical Process Fundamentals of the CAS, v.v.i. focusing on the SFE from plants with particular interest in a study of several types of fractionation techniques. He has published 3 papers in reputed journals and presented his work in many international conferences dealing with supercritical fluids.

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# Resolving terahertz spectral mixtures using the blind source separation approach: A method to study the dehydration kinetics

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Terahertz spectroscopy has gained popularity as a promising non-invasive investigation tool in recent decades. In studies of solid-state pharmaceuticals, its usefulness is enhanced, since it enables one to distinguish different polymorphic and pseudo-polymorphic forms. Terahertz absorption spectra are additive, meaning that the resulting spectrum of two or more compounds in the sample is an algebraic sum, in the linear region of the Beer's law. For this reason, the evolving-in-time linear mixture of unknown pure components can be resolved using the blind source separation approach, where both spectral sources and their concentrations are to be estimated. Such an evolution occurs naturally in temperature-induced (pseudo) polymorphic transitions, i.e. dehydration, where the terahertz spectrum is the hydrate and dehydrates's response with time-varying proportions. Conventionally, to estimate the kinetics of a reaction–an important physical-chemical parameter–the area of the unique spectral peak is integrated and normalized to evaluate the abundance, but a difficulty of strongly overlapping peaks unable its application. To overcome this problem, we show, that by employing the blind source separation procedure, we can resolve a complex, multi-compound spectral mixture with significant cross-bands, where the peak area method failed. We evaluate the performance of our approach in studies on dehydration of a well-known polycrystalline hydrate–α-D-glucose monohydrate. Seeing that the polymorphic transitions can completely change the properties of a pharmaceutical, our approach can find application in a drug development process, where their careful characterization is of utmost importance.

## Biography

Lukasz A Sterczewski has received his MSc degree in Electronics from Wroclaw University of Technology, Poland in 2014, working on terahertz time-domain spectroscopy. He is currently working towards a PhD degree from WrUT. In September 2015 he has joined PULSE (Princeton University Laser Sensing Laboratory) as a Visiting Student Research Collaborator. His research is focused on development on new spectroscopic techniques and advanced signal processing tools.

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# Numerical analysis on purification of methane through the hollow fiber membrane system

**Yongtaek Lee** and **Kyungwhan Cha**
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The polysulfone membrane was found to be applicable to the selective removal of $CO_2$ from a biogas, resulting in preparation of the relatively concentrated methane. The purified methane might be utilized as a vehicle fuel if the methane concentration is kept higher than the 95 mole %. Generally speaking, the concentration of methane in the biogas is around 20 mole %, which should be upgraded using a cascade operating system. Even if there are several conventional technologies, the membrane system is known to be not only a new technology, but also an economical method. The cascade membrane system may be constituted of either two membrane modules or three membrane modules, depending on the input biogas conditions and the degree of purification of methane. In this study, a theoretical analysis on the cascade membrane system is proposed and a proper numerical technique is applied to solve the obtained differential equations where the permeate flow rate and the concentration of the each component will be described in terms of the permeability and the partial pressure difference across the membrane. As the operating conditions are changed, the obtained flow rate and the concentration of methane as a product are found to be severely affected. As a design tool, this numerical analysis is strongly recommended since the proper operating condition will be suggested together with the suitable recycle flow rate for the target goal such as the recovery ratio and the purity of methane in the final product. The cascade schemes and the analysis results will be presented.

## Biography

Yongtaek Lee has completed his PhD from the State University of New York at Buffalo. He is currently the Head of the Department of Chemical Engineering, Chungnam National University. He has published more than 100 papers in reputed journals.

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# Purification of phosphoric acid by liquid-liquid

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Phosphoric acid is a weak oxyacid with many industrial applications depending on its degree of purification, including surface treatments or fertilizer production (merchant grade Phosphoric Acid, MPA), salt production for animal feeding or acidification of food and drinks for human consumption (Food grade Phosphoric Acid, FPA), and pharmaceutical industry (Pharmaceutical grade Phosphoric Acid, PPA). In order to produce these different grades, various purification technologies can be implemented including hydrometallurgical processes as it is the case in the Prayon's process based on the selective liquid-liquid extraction of phosphoric acid. The current solvent used by Prayon's process for purifying phosphoric acid is a mixture of 90 wt% di-iso-propylether (DIPE) and 10 wt% tri-n-butylphosphate (TBP). There is very few information about the physicochemistry involved in the purification of wet phosphoric acid by liquid-liquid extraction because wet phosphoric acid is a very complex medium (highly concentrated medium which can reach 14 M, high complexing power, only few data on metal speciation in phosphoric acid, etc.). Furthermore, liquid-liquid extraction of phosphoric acid involves very complex phenomena such as phase splitting and third phase formation which can be explained by the presence of supramolecular species in solution as well as coextraction of large amount of water. In the present paper, the physicochemistry involved in phosphoric acid and water extraction by new extraction solvents is presented with a focus on decrypting the role of the supramolecular organization. Inedit data on the transitions from triphasic systems towards biphasic systems are presented and a focus has been placed on the description of forces playing a role in these transitions.

## Biography

Mariana Campos Assuncao is currently a PhD student at Chimie ParisTech conducting a research project on the purification of phosporic acid in collaboration with Prayon. Before this experience, she has obtained a Master's degree in Nuclear Energy at Chimie ParisTech and an Engineering degree in Chemical Engineering at INSA de Rouen.

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