Facultad de Ciencias y Tecnologías Químicas
Universidad de Castilla- La Mancha

# XIV YOUNG SCIENCE SYMPOSIUM

Del 8 al 10 de Julio de 2020

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# Bienvenida del Decano

Os damos la bienvenida a esta nueva edición del 14th Young Science Symposium, que organiza la Facultad de Ciencias y Tecnologías Químicas de la Universidad de Castilla – La Mancha junto con sus jóvenes investigadores. En esta ocasión en formato virtual.

Es el XIV Simposio, que nació con este formato en la edición del año 2013. Previamente, y desde el año 2007, se había desarrollado como jornadas de sesiones cortas a lo largo de los meses de abril a junio. En todos los casos, ha sido siempre un compromiso de la Facultad por la investigación y hacia sus jóvenes investigadores. Promover y divulgar la investigación en los campos científicos y tecnológicos que tiene que ver con la Química, la Ingeniería Química, y la Ciencia y Tecnología de los Alimentos, que son las enseñanzas tradicionales de nuestro Centro.

El objetivo es doble: divulgar y dar a conocer lo que nuestros jóvenes investigadores hacen, e introducirlos en la organización de pequeños eventos científicos, que a escala reducida sirvan de ejemplo de cómo se organizan los congresos a los que después ellos asisten, a nivel nacional e internacional, presentando los resultados de sus trabajos científicos. Constituye también una actividad formativa para los estudiantes de nuestros programas de doctorado, después de los cambios que éstos han experimentado a partir del año 2011. En esta edición se sigue contando con la participación de jóvenes investigadores de otros campus (Toledo y Albacete) de la Universidad de Castilla-La Mancha, cuyos temas de investigación están estrechamente relacionados con los campos científicos y tecnológicos que se desarrollan en este Centro, hecho que fomenta la interdisciplinaridad y el debate entre nuestros jóvenes investigadores. Es buena prueba de la magnífica aceptación del Simposio, que va creciendo de forma muy clara durante los últimos años. A modo de incentivar a aquellos jóvenes doctorandos y nuevos doctores que participan como ponentes en estas Jornadas, en esta convocatoria 2020, se concederá el V PREMIO CIENCIA JOVEN. Asimismo, se otorgará el III PREMIO CIENCIA JOVEN al mejor póster, ya que este año contamos también con contribuciones en este formato.

Desde la Facultad, que aporta la infraestructura y los recursos necesarios, hemos de felicitar al grupo de jóvenes que han estado detrás de la organización de esta edición, por su dedicación y entusiasmo. Agradecer a todos los participantes su respuesta y favorable acogida. A los investigadores invitados. A los patrocinadores; incluso bajo las condiciones especiales de este año. Al Rector, y a la Vicerrectora de Investigación y Política Científica de la UCLM, por su apoyo a estos Simposios. Y, así, con estos respaldos, el convencimiento del éxito de esta nueva edición del Simposio Ciencia Joven.

**Ángel Ríos Castro**

*Decano de la Facultad*

## Comité Científico:

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**Ana Raquel de la Osa Puebla,** *'Chemical Engineering'*

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**Antonio Manuel Rodríguez García,** *'JIQ-RSEQ - EYCN EuChemS'*

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**Cristina Montes Correal,** *'Analytical Chemistry'*

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**Alberto José Huertas Alonso,** *'Organic Chemistry'*

**María del Prado Caballero Espinosa,** *'Inorganic Chemistry'*

**Luis Fernando León Fernández,** *'Chemical Engineering'*

Estamos encantados de retomar durante los días 8, 9 y 10 de Julio la iniciativa del **"Young Science Symposium"**, siendo este año su **decimocuarta edición**.

Con este fin, un grupo de **jóvenes doctorandos** pertenecientes a la **Facultad de Ciencias y Tecnologías Químicas de Ciudad Real**, **Facultad de Ambientales de Toledo** y **Facultad de Medicina de Albacete** presentarán una serie de comunicaciones sobre el trabajo que están desarrollando en sus respectivas áreas. También contaremos con algunos investigadores invitados externos que nos darán una visión más general sobre la investigación en España, tanto en instituciones públicas como en la empresa privada.

Con el fin de incentivar y despertar el espíritu investigador entre los alumnos de la facultad, nuestra futura "cantera", la asistencia a las jornadas podrá ser convalidada por **un Crédito de Libre Configuración**. Creemos que esta iniciativa puede ser muy interesante para acercar a los alumnos a la verdadera actividad de los laboratorios de investigación de la facultad.

Por otro lado, a modo de incentivar a aquellos jóvenes doctorandos y nuevos doctores que participan como ponentes en estas jornadas, en esta convocatoria 2020, se concederá el **"V PRIZE OF YOUNG SCIENCE FACULTY OF SCIENCES AND TECHNOLOGY"**. Asimismo, se otorgará el **"III PREMIO CIENCIA JOVEN"** al mejor póster, ya que este año contamos también con contribuciones en este formato.

Por todo ello, os agradecemos vuestra participación y esperamos que la experiencia os sea de provecho, aunque este año tenga que ser en esta versión online debido a la situación de alerta sanitaria de este año.

No olvidéis visitar nuestra página web donde podréis encontrar las últimas novedades de estas jornadas, instantáneas de las presentaciones, así como el Libro de abstracts en formato electrónico:

http://www.uclm.es/CR/FQuimicas/

# Programme:

## Wednesday, 8th July 2020.

**9:00- Opening ceremony chaired by the Rector Magnificus of the UCLM and the Dean of the Faculty of Chemical Sciences and Technology.**

**9:30- Invited Lecturer: Prof. Rafael Van Grieken (Rey Juan Carlos University).** ***"La carrera académica: luces y sombras de un complejo camino".***

**10:30- 1st session**

- *"Plasmonic nanosensing based on AuNPs for UV-Vis/ Colorimetric assessment of global aminoacids amount in food supplements".* **Manuel Bartolomé**. *Analytical Chemistry.*
- *"Nanostructured electrodes prepared by magnetron sputtering for water electrolysis".* **Ester López.** *Chemical Engineering*.
- *"Ring-Opening Polymerization of Ciclohexene Oxide Catalyzed by Scorpionate Aluminium Complexes".* **Miguel Ángel Gaona**. *Inorganic Chemistry.*
- *"Benzoazoles as two-photon absorbing chromophores".* **Beatriz Donoso**. *Organic Chemistry*.

**11:30- Break.**

**12:00- Invited Lecturer: Dr. Teresa Garde-Cerdan (Institute of GrapeVine and Wine Science). "Foliar application of biostimulants in the vineyard: A tool to improve grape and wine quality and attenuate the effects of climate change".**

**13:00-14:30-Poster session I.**

**16:00- 2nd session**

- *"Grape seed extract supplementation modulates hepatic lipid metabolism in healthy rats and could be effective for preventing the progression of overweight-related metabolic disorders".* **Eduardo Guisantes**. *Food Sciences and Technology.*
- *"Kinetics on Supercritical Epoxidation of Vegetable Oils".* **Juan Catalá.** *Chemical Engineering.*
- *"Speciation of a polyphenolic pigment in free solution and encapsulated into a nanocarrier system. Quantification of this bioactive in micellar medium in nutraceuticals".* **Esther Pinilla.** *Analytical Chemistry.*
- *"Effects of central leptin on metabolic reprogramming of adipose tissue. Role of PPARβ/δ and FGF21".* **Lorena Mazuecos**. *Biochemistry.*
- *"Substituted Alkynylbenzenes and Bithiophenes-Based Crystalline Structures: X-Ray Study and Optical Waveguide Behaviour".* **Carlos Tardío.** *Organic Chemistry.*

## Thursday 9th of July 2020

**9:00- Invited Lecturer: Prof. Juan Ignacio Cirac (Director and Scientific Member Max Planck Institut of Quantum Optics). "Ordenadores cuánticos: Cómo, cuándo y para qué".**

**10:00- 3rd session**

- *"Analytical control of gold and platinum nanoparticles in clinical and toxicological matrices".* **Sergio Fernández**. *Analytical Chemistry.*
- *"Flavan-3-ol composition of chocolate panned raisins".* **Carolina Olivati.** *Food Sciences and Technology.*
- *"Microwave-responsive Graphene-based hydrogels for drug delivery applications".* **Jorge Leganés.** *Organic Chemistry.*
- *"Characterization and reactivity with* $NO_2$ *of diesel soots generated under different combustion conditions".* **María Inmaculada Aranda**. *Physical Chemistry.*
- *"Development of organocatalysts for the synthesis of oxazolidones for potential antibiotics"***. María del Prado Caballero**. *Inorganic Chemistry.*

**11:15- Break.**

**11:45- Presentation of the Territorial Section of the Royal Spanish Society of Chemistry (STCLM-RSEQ)**

**12:00- 4th session.**

- *"Glycolysis process for polyurethane composites recyclin".* **Jesús del Amo.** *Chemical Engineering.*
- *"Push-pull azaheterocyclic chromophores: Synthesis, photophysical properties and use for white light emission".* **Rodrigo Plaza.** *Organic Chemistry.*
- *"Why and how do we study the interstellar chemistry?".* **Sergio Blázquez.** *Physical Chemistry.*
- *"Influence of ultrasounds on wine microbiota".* **Raquel Muñoz.** *Food Sciences and Technology.*

**13:00-14:30- Poster session II.**

**16:00- Invited Lecturer: David Medina Cruz (Ph.D. Northeastern University at Boston, USA, The Green Chemistry Lab). "Green Nanobiotechnology and Biofactories: from a laboratory curiosity to a market product helping society".**

**17:00- 5th session**

- *"Resistance to oxidation of* $SO_2$ *free red wines elaborated with natural antioxidants".* **Lourdes Marchante.** *Food Sciences and Technology.*
- *"Acrylamide magnetic hydrogels: different methods for the introduction of magnetic nanoparticles".* **Jesús Herrera.** *Organic Chemistry.*

- *"Emerging contaminants treatment in a real waters matrix by AOP processes".* **Jihane Ben**. *Analytical Chemistry.*
- *"Comparative study about formation of Secondary Organic Aerosol from ozonolysis of organic compounds".* **Alba Escalona.** *Physical Chemistry.*
- *"Development of a combined electro-scrubbing process for the treatment of volatile organic compounds".* **Florymar Escalona.** *Chemical Engineering.*

## Friday 10th of July 2020

**9:00- Invited Lecturer: Dr. Jesús Martínez de la Fuente (Aragón Materials Science** Institute). "Hybrid Nanoparticles for Therapy and Diagnosis: New Biotechnological Challenges for Hyperthermia".

10:00- 6th session

- *"Effect of oenological by-products as natural antioxidants and flavourings in meat products".* **Marina Alarcón**. *Food Sciences and Technology.*
- *"A screening method for the assessment of the release of silver nanoparticles from food containers".* **Ana Isabel Corps**. *Analytical Chemistry.*
- *"Effect of COVID-19- lockdown on the air quality of different cities".* **María Gabriela Viteri.** *Physical Chemistry.*
- *"Electrochemical technologies for the elimination of antibiotics in hospital urine".* **Ángela Moratalla**. *Chemical Engineering.*
- *"Graphene-based sulfonate hydrogels: Soft scaffolds for cell culture".* **Josué Muñoz**. *Organic Chemistry*.
- *"Antitumoral effects of resveratrol through adenosinergic system on cervical cancer cell line".* **Sonia Muñoz**. *Biochemistry.*

**12:00- 7th session**

- *"Bimetallic Zinc Catalysts for Ring-Opening Copolymerization Processes".* **Felipe de la Cruz.** *Inorganic Chemistry.*
- *"Tumor lockdown: how to beat cancer through confinement measures".* **Miriam Nuncia**. *Biochemistry*.
- *"Accurate quantification of polycyclic aromatic compounds adsorbed on soot samples".* **Sonia Lara.** *Physical Chemistry.*
- *"Determination of biological markers oxidative stress by capillary liquid chromatography mass spectrometry".* **Yassine Benmassaoud**. *Analytical Chemistry.*
- *"Applying statistics criteria in rational selection of probiotic Lactobacillus strains to be used as biocontrol agents".* **Sara Rodríguez**. *Food Sciences and Technology.*

- *"$CO_2$ reduction by simulated sunlight and $TiO_2$-based catalysts".* **Verónica Rodríguez**. *Chemical Engineering.*

**13:30- Colloquium, Prize winners and Closing Ceremony chaired by the Vice-Chancellor of Research and Scientific Policy of UCLM, the Dean of the Faculty of Chemical Sciences and Technology and Ricardo Jurado (AGROVIN, sponsor of the V Prize of Young Science) who presents "Application of ultrasound in the production of quality wines".**

# INVITED SPEAKERS

## CI-1

# La carrera académica: luces y sombras de un complejo camino

Rafael Van Grieken*

*Rey Juan Carlos University, Madrid, Spain.*

The intention of this talk is to try to summarize the implications of choosing a path, the academic path. It is a long-distance race through a path of which the entire route is not known, but rather a series of rules that determine it, but which are never well explained. The academic career, which includes both the university and the research centres, is above all the result of intertwined intellectual challenges and normally satisfies those who follow it, even from its beginnings in the doctoral stage. However, in addition to satisfaction, it also entails negative aspects that should be avoided, or at least cushioned, following certain guidelines that will be developed throughout the talk. It is a complex path, not at all simple, that requires balances that are not easy to achieve such as competing by collaborating, moving away without losing your academic links or publishing scientific results without losing sight of the fact that this is not the only learning process in research.

This is not a standard scientific talk but rather the compendium of multiple reflections that come from numerous meetings and debates that constitute the baggage of a university professor's experience. A professor who has also had the opportunity to manage important areas in his university (vice-rectorate for research), the management of the state agency for university quality (ANECA), and the political management of education (at all levels) and research in the Community of Madrid. From this privileged position, the lights and shadows of the complex academic path will be analysed and, I hope, discussed.

## CI-2

# FOLIAR APPLICATION OF BIOSTIMULANTS IN THE VINEYARD: A TOOL TO IMPROVE GRAPE QUALITY AND ATTENUATE THE EFFECTS OF CLIMATE CHANGE

*T. Garde-Cerdán, P. Rubio-Bretón, S. Marín-San Román, E. Baroja, E.P. Pérez-Álvarez*

*Grupo VIENAP, Instituto de Ciencias de la Vid y del Vino (CSIC, Gobierno de La Rioja, Universidad de La Rioja), Ctra. de Burgos Km. 6, 26007 Logroño, La Rioja.*

*Agricultura y Ciencia y Tecnología de Alimentos*

## 1. Introduction

Traditionally, the vineyard fertilization has been carried out directly on the soil, in order to be absorbed by the plant roots. Currently, due to the contamination problems caused by its excessive use, new fertilization techniques more precise and effective are sought. One of these techniques is the foliar fertilization, which provides a quick and efficient assimilation by the plant of the products applied [1, 2], which allows reducing costs and contributing to sustainable agriculture. Ammonium and amino acids are the main nitrogen sources for yeasts, and their concentration can decisively influence the wine aroma, since the formation of volatile fermentative compounds can be affected by the content of grape nitrogen compounds [3, 4]. Among the amino acids, proline is metabolized by yeast only when there are no other nitrogen sources and in presence of oxygen in the medium [5, 6]. On the other hand, phenylalanine is precursor of 2-phenylethanol, a compound with rose notes [7], and of several phenolic compounds, including *trans*-resveratrol, a compound very important in the wine healthy properties [8]. Otherwise, fertilizers that include amino acids in their composition have appeared on the market, whose effectiveness has not been tested, and may represent an economic expense without justification. Therefore, the first objective of this study was to investigate the use of foliar application in the vineyard of different nitrogen sources as a tool to improve the grape quality.

Moreover, for some time now, an imbalance has been observed between technological maturity, defined fundamentally by sugar content and acidity, and phenolic maturity, responsible for the taste properties and color of the berries. This fact is due to several factors, among which climate change stands out, whose main effects are the increase in temperature and the decrease in rainfall. The conditions favor the imbalance between technological and phenolic maturity, so that the color of the grapes and wines is impaired [9]. An alternative to mitigate the effects of climate change may be the use of elicitors, substances that trigger plant defense mechanisms when are applied exogenously. In viticulture, one of the elicitors most widely used is methyl jasmonate (MeJ), whose application in the vineyard has improved the composition of grapes and wines [10]. For this reason, the second objective of this study was to evaluate the effect of the foliar application of MeJ on the grape composition, thus helping to find tools to fight the consequences that the current weather situation has on grape maturation.

## 2. Experimental Section

In the case of nitrogen sources, foliar applications were made with five nitrogen products at 1 kg N/ha: proline, phenylalanine, urea and two commercial fertilizers, one without amino acids in its formulation and the other with amino acids. The elicitor used was methyl jasmonate (MeJ), using a 10 mM aqueous solution. The treatments were carried out, in triplicate, in two phenological moments of the plants, at veraison and one week later. For each treatment, 200 mL/plant were applied, using Tween 80 as surfactant. Control plants were treated only with an aqueous Tween 80 solution.

The grapes of all the treatments were harvested at the optimum moment of technological maturity. The musts were obtained after destemming and crushing the grapes, and aliquots of each sample were frozen for subsequent determination of amino acids by HPLC [11], stilbenes by HPLC [12], and volatile compounds by GC-MS [13]. It should be noted that for the determination of phenolic compounds in the MeJ trial, prior to the harvest, 50 berries were sampled from each treatment and repetition, which were frozen for their subsequent analysis by HPLC [14]. The results were statistically analyzed using analysis of variance (ANOVA) with the SPSS statistical program version 21.0 (Chicago, USA). The differences between the measures were compared using the Duncan test with a significance level of 0.05.

## 3. Results

Regarding the use of nitrogen sources as biostimulants in the vineyard, the results obtained have shown that the foliar application of proline did not affect the must composition. However, phenylalanine and urea treatments and commercial fertilizers improved the amino acid composition of the samples. Regarding the content of stilbenes, resveratrol and piceid, only the foliar application of urea favored significantly their synthesis by the plant. The treatment carried out with phenylalanine stimulated the synthesis in the vine of 2-phenylethanol and 2-phenylethanal, thus improving the must aromatic composition. For all these reasons, it could be said that the applications of phenylalanine and urea were the most promising nitrogen treatments in order to improve the health properties and quality of the grapes and wines.

With regard to the use of methyl jasmonate as elicitor in the vineyard, this study shows for the first time the effect of its foliar application on the composition of grapes of the Tempranillo variety. Foliar application of MeJ led to an increase in the concentration of anthocyanins and stilbenes, as well as an increase in the synthesis of some amino acids, including phenylalanine. However, it did not affect the content of primary aromas in the grapes. This study has shown that plants can react to the foliar application of MeJ by accumulating mainly phenolic compounds. Therefore, the application of MeJ can be a simple and interesting viticultural tool to increase the concentration of phenolic compounds in grapes, helping to mitigate the effects of climate change on grape maturation.

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## CI-3

## Quantum computers: How, when and what for

*Juan Ignacio Cirac Sasturain*

*Max Planck Institute of Quantum Optics Hans-Kopfermann-Str. 1 85748 Garching, Germany*

*Physics*

The discovery of quantum physics led to one of the greatest scientific and technological revolutions that humanity has experienced. Lasers, semiconductors, or nuclear power plants are a few examples of the applications of this theory. In recent years we have experienced a second "quantum revolution", where the phenomena most extraordinary in quantum physics, which until recently had not been confirmed, can be exploited to give rise to new technologies. In particular, new ways of processing information that promise to make a quantum leap in the ability to calculation of the supercomputers. In fact, today there is a great deal of effort to build these quantum computers. However, this requires several technological challenges. In this talk I'll explain what the power of quantum computers is based on, what will be useful and I will review the current situation and prospects for to move from small prototypes to large installations.

## CI-4

# Green Nanobiotechnology and Biofactories: from a laboratory curiosity to a market product helping society

*David Medina Cruz*

*Northeastern University at Boston, USA, The Green Chemistry Lab*

*Chemical Engineering Department*

Antimicrobial resistance to antibiotics (AMR) and chemotherapy-resistant cancer are two of the main concerns that the healthcare system should face nowadays. Current chemotherapy drugs and antibiotic treatments are becoming ineffective or have plenty of drawbacks related to misuse and overuse. Therefore, new alternatives are needed, and nanotechnology is rising as a powerful solution over time. Nevertheless, how nanomaterials are created has plenty of influence in their features and applications. Traditional synthesis of nanostructures, taking knowledge from both physics and chemistry, is subjected to several disadvantages, such as the production of toxic-by-products and harsh conditions, as well as biocompatibility issues once they are applied in biomedical settings. Therefore, green nanotechnology is presented as a suitable answer, allowing the generation of nanostructures in a cost-effective and environmentally friendly approach employing living organisms, such as bacteria or fungi, and biomolecules produced from natural sources, such as plants.

The instauration of the Green Chemistry Lab at Northeastern University supposed an effort in the aim to develop and implement green-nanotechnological approaches to solve some of the problems in the healthcare system. As such, different projects have been proposed. For instance, bacteria-mediated synthesis of nanomaterials, in which pathogenic bacteria are employed as biofactories for the production of selective antimicrobial agents. Alternatively, the human-cells mediated synthesis of nanostructures, which offers a dual effect: the generation of valuable noble-metal nanomaterials and the biological cross-linking of extracellular matrixes for reinforcement of biomedical devices and agents. Other approaches include the production of metal/metalloid-based nanostructures employing natural extracts of Aloe Vera, pepper, or exotic fruits.

Each one of these approaches can confer different biomedical applications to the produced nanomaterials, from the fight of antimicrobial resistance to antibiotics to the reduction in the size of tumors or the trapping/sensing of different viruses, such as Ebola or COVID-19. As such, a few of the methods have been patented and licensed for the registration to date of two biotechnology companies: SynCell Biotech and Nanolyx Inc, whose goal is to bring these green nanotechnology-based platforms to society.

## CI-5

# Hybrid Nanoparticles for Therapy and Diagnosis: New Biotechnological Challenges for Hyperthermia

*Jesús Martínez de la Fuente*

*Aragón Materials Science Institute, ICMA, CSIC-University of Zaragoza & CIBER-BBN*

The use of nanoscale structures has been used by Nature for billions of years. Two properties that make them very interesting for use in biomedical applications are (i) they have a typical size similar to biological components and (ii) the possibility of manipulating and designing materials with almost à la carte properties controlling the size, composition and shape. The research of our group is mainly focused on the use of magnetic nanoparticles and their ability to generate heat applying an alternating magnetic field. For that, different iron oxides nanoparticles has been prepared by different methodologies and with different size and organic or inorganic coatings. The potential of using the generated heat by applying alternating magnetic fields in the microenvironment of magnetic nanoparticles to activate multienzymatic industrial bioprocesses has been explored. This feature will be also used for the treatment of cancer by magnetic hyperthermia or targeted enzyme therapy. Our research is divided into two distinct but clearly related fields through a common nexus: the type of material used to generate heat by applying an alternating magnetic field (magnetic nanoparticles).

1) **Magnetic Hyperthermia for Biocatalysis**: We report here how the properties of magnetic nanoparticles (NPs) and of thermophilic enzymes can be combined to obtain NP-enzyme systems capable to be activated in a wireless fashion. Conjugation of α-amylase and L-aspartate oxidase to the surface of magnetic nanoparticles and using different strategies obtaining different orientations of the enzymes respect to NP surface. This allowed us to create effective biocatalysts with different activities (ranging from 17 to 87 % of the initial activity). Furthermore, spectroscopic studies showed that the conjugation of the enzyme to the NP modifies its 3D structure and that different conjugation strategies lead to different stretching of the protein. Results clearly demonstrate that the application of an AMF activates the nano-systems, without a significant increasing in the reaction media temperature. Furthermore, we successfully reused the nano-systems for at least three consecutive cycles of AMF activation with the loss of only the 40% of the initial activity.

2) **Magnetic Hyperthermia and 3D cell culture models**: Understanding the mechanisms involved in the cellular damage generated by magnetic hyperthermia is crucial for its successful application. In order to evaluate the treatment efficacy, two different 3D cell culture models were prepared using a collagen matrix, which is one of the major components of the tumour extracellular matrix. A strong effect of the hyperthermia treatment was observed on the location of the particles within the 3D cell culture for one of the models. The treatment facilitated the migration of the particles from the outer areas of the 3D structure, achieving a faster homogeneous distribution

throughout the whole structure and providing access to the particles to the inner cells. Moreover, although in both models’ cells were exposed to the same amount of nanoparticles, as a consequence of the 3D model generation, the cell death mechanism activated by the magnetic hyperthermia treatment was different in both models.

# ORAL SPEAKERS

**(appearance in alphabetic order)**

## O-1

# EFFECT OF OENOLOGICAL BY-PRODUCTS AS NATURAL ANTIOXIDANTS AND FLAVOURINGS IN MEAT PRODUCTS

M. Alarcón*, M. S. Pérez-Coello, M. C. Díaz-Maroto, M. E. Alañón, A. Soriano

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*Food Technology Area*

In the last years, a growing interest of ready-to-eat meat products has resulted as to their easiness to be cooked and consumed. However, oxidative phenomena are one of the major causes of their quality deterioration. This fact causes the necessity to use antioxidants in meat products. New trends aimed at the employ of natural sources and agri-food by-products revalorization as natural preservatives have emerged in order to replace the traditional additives. Therefore, this work has been performed to study the antioxidant capacity and sensorial contribution of several oenological by-products added to different meat products.

Different types of meat products were prepared: pork and venison burgers, a cooked pork product and pork cured sausages (*salchichón*). In all experiments, two control samples were tested: C (without antioxidants) and AC (with 400 ppm sodium ascorbate). Likewise, oenological by-products were used in different concentrations (0.05-5%): oak wood, stems, vine-shoots, inactive dry yeasts and wine lees. Total phenolic content (TPC) and antioxidant capacity was evaluated, as well as the lipid and protein oxidation development. The effect in sensory characteristics of natural antioxidants was also checked.

The addition of oenological by-products increased considerably TPC and similar trends were observed in antioxidant capacity. In consequence, a protector effect of lipid and protein oxidation was showed, being C and AC the most oxidized samples. Among all experiments carried out, oak, stems and vine-shoots were the most effective. On the other hand, natural antioxidants modified color, enhanced the texture, and provided new odor and taste attributes to meat products.

Therefore, the oenological by-products studied could be a relevant option as natural preservatives providing interesting new flavors to meat products.

## O-2

# CHARACTERIZATION AND REACTIVITY WITH $NO_2$ OF DIESEL SOOTS GENERATED UNDER DIFFERENT COMBUSTION CONDITIONS

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*Physical Chemistry*

Soot is one of the pollutants generated in the incomplete combustion of fuels. This pollutant cause changes in the climate that affect the environment globally and has negative effects on the human health. Two samples of diesel soot generated in different operation conditions (mode E and mode G), have been characterized using Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS). In addition, the reactivity of these two different samples with $NO_2$ have been studied using a Knudsen flow reactor and mass spectrometer as detection system. For the analysis with DRIFTS the samples were diluted with KBr and the IR spectra obtained have been compared with the IR spectrum of a standard reference material (SRM) Diesel NIST 1650b. Both samples (mode E and G) show similar bands in the IR spectra and they are also similar to the IR bands of the reference diesel NIST 1650b, what means that all the samples have the same functional groups in their structure. The heterogeneous reactivity of the diesel soot with $NO_2$ have been studied determining the initial uptake ($\Upsilon_0$), which corresponds to the initial probability of the collision between a probe gas and the soot sample leads to disappearance of the reactant[1]. The $\Upsilon_0$ obtained have been compared between them and with the $\Upsilon_0$ available in bibliography.

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## O-3

# PLASMONIC NANOSENSING BASED ON AuNPs FOR UV-Vis/ COLORIMETRIC ASSESMENT OF GLOBAL AMINOACIDS AMOUNT IN FOOD SUPLEMENTS

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*Química Analítica*

A novel, simple, rapid and highly sensitive colorimetric sensor based on citrate stabilised gold nanoparticles (AuNPs) for the assessment of aminoacids global content has been developed in this work. Firstly, AuNPs from five different sizes were synthesized by Turkevich-Frens method and later characterized by UV-Vis, DLS, SEM, and Z potential techniques to select the most suitable AuNPs size for sensor performance. Chemical and physical parameters affecting the sensing system such pH, AuNPs concentration, incubation time and maximum ionic strength were also evaluated. Aminoacids could induce AuNPs aggregation at low pH (2.6) owing to the interaction between amino and carboxylic groups located at aminoacids and citrate AuNPs surface and naked AuNPs respectively. This aggregation causes a change in colour solution from red to blue as well as a shift in absorption band from 518 nm to 650 nm, whose absorbance ratio will be the analytical signal related to the aa amount, just monitoring by UV-Vis spectroscopy. The proposed sensor shows excellent analytical performance characteristic in terms of linear range (480 nM to 1.8 µM), LOD (160 nM) and precision (repeatability and reproducibility). Selectivity of the sensor was evaluated by an extensive interference study (six of them in different ratios). Finally, this sensing methodology was successfully applied to different commercial nutritional supplements with very good agreement between our results and those obtained by the official Kjeldahl method and with the declared values by the manufacturer too.

## O-4

# EMERGING CONTAMINANTS TREATMENTS IN REAL WATERS MATRIX BY ADVANCED OXIDATIVE PROCESSES (AOP's)

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*Analytical chemistry*

The discharge of pharmaceutical products in the environment has become a major concern. Conventional treatments are not efficient to achieve complete removal of these pollutants and it becomes necessary the development of new more-efficient treatment technologies. Advanced Oxidation Processes (AOPs), characterized by the production and use of strong oxidizing agents such as hydroxyl radical (OH•), have been efficiently applied in the recent years. In this work, it has been evaluated the simultaneous degradation by photochemical, electrochemical and photoelectrochemical processes of five pharmaceutical compounds, sulfadiazine (SDZ), naproxen (NAP), diclofenac (DIC), ketoprofen (KET) and ibuprofen (IBUP) from three different water matrixes (sewage, pore water and surface water) were studied employing photochemical, electrochemical and photoelectrochemical processes. Results show different removal rates for each drug, which can be explained in terms of their chemical structure. All processes were able to reach 100% of removal in the three water matrixes studied. During the photochemical treatment the SDZ, KET and DIC were the most photoactive substances and very low degradation percentages were obtained for other compounds. Electrochemical technology was able to achieve better removal rates and, in turn, coupling light irradiation to electrochemical process improved significantly the efficiencies reached. In comparing the degradation of the different drugs, a synergism was confirmed in the coupling of technologies for NAP and IBUP and antagonism for the other compounds. Regarding to the matrix effect, it was observed that the more complex in the matrix, the worse are the degradation results (surface water > pore water > sewage). This has been explained in terms of the presence of interferents, which compete with target compounds.

## O-5

# Determination of biological markers oxidative stress by capillary liquid chromatography mass spectrometry

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*Química Analitica*

Oxidation stress is an important factor in the pathogenesis of several diseases, including cardiovascular, neurodegenerative and certain forms of cancers. Various methods have been used to measure the extent and the nature of oxidative stress, ranging from oxidation of DNA to proteins, lipids, and free amino acids.

The present work describes an HPLC-MS method involving the separation of Tyrosine from 3-NItrotyrosine, 3-Chlorotyrosine, 8-hydroxy-2'-deoguanosine and 4-Hydroxynonenal by HPLC and MS quantification under positive-ion chemical ionization conditions. Determination of the biomarkers oxidative stress amounts was allowed in a concentration range of 5–500 $\mu g \cdot L^{-1}$. The limit of detection, the limit of quantification and the standard deviations achieved were lower than 2 $\mu g.L^{-1}$, 6 $\mu g.L^{-1}$ and 4.52%, respectively. The described method was applied for the determination of Oxidative biomarkers in human serum and plasma samples.

## O-6

# WHY AND HOW DO WE STUDY THE INTERSTELLAR CHEMISTRY?

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*Physical Chemistry*

In 1953, Stanley L. Miller and Harold Urey (1) conducted an experiment where, from simple molecules such as water, methane, ammonia and hydrogen, they managed to obtain prebiotic molecules as amino acids. This experiment that simulated the first moments of the primitive Earth may be a possible explanation for the presence of life on the planet. These simple molecules, in addition to many others, have been found in molecular clouds in the interstellar medium (ISM) and other bodies such as comets. Knowing the chemistry of these molecules in such extremely cold environments ($T = 10 - 100$ K) is fundamental to understand how more complex molecules can be generated, just as it happened in the Miller and Urey's experiment, and give rise to fundamental compounds in life as we know it. In our laboratory, the gas-phase kinetics of the reactions of OH radicals (ubiquitous in the ISM) with simple molecules present in molecular clouds, such as methanol, ethanol, formaldehyde, methyl formate, acetone or acetaldehyde is carried out by the pulsed CRESU system (French acronym for *Reaction Kinetics in a Uniform Supersonic Flow*). To emulate the typical temperatures of ISM the gas mixture is expanded through a Laval nozzle from a high to a low-pressure region. Currently, our CRESU apparatus reaches temperatures between 11.7 and 177.5 K, avoiding the gas condensation onto the reactor walls. This technique, together with the *Pulsed Laser Photolysis* (PLP) and *Laser Induced Fluorescence* (LIF) techniques, allows us to determine the rate constant of these reactions in the gas phase at ultra-low temperature (2-6), thus contributing to the improvement of astrophysical models to have a greater and better knowledge of the evolution of the cosmos.

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## O-7

# Development of organocatalys for the synthesis of oxazolidinones for potential antibiotics

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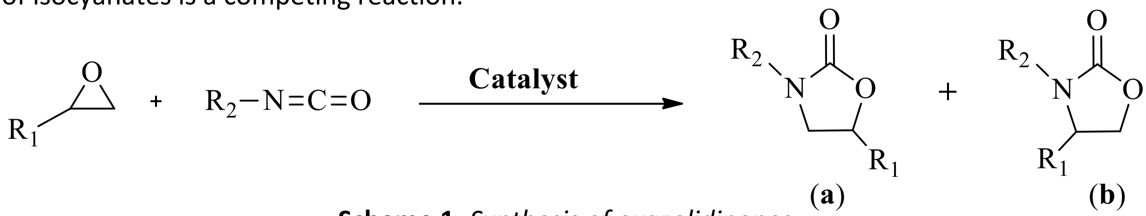
*Química Inorgánica*

Metal-free organocatalysts based on imidazolium salts showed high efficiency catalyzing the cycloaddition reactions to prepare oxazolidinones (Scheme 1). These synthetic drugs are active against a large spectrum of Gram-positive bacteria, includig *Staphylococcus*, *Streptococcus* and *Enterococcus* which have developed resistance to antibiotics. *Linezolid* was the first oxazolidinone for the treatment of infections caused by this germ.[1]

Imizazolium salts performed as bifuntional catalysts[2] and exhibited improved activity for preparation of *N*-heterocycle compunds. Oxazolidinones were prepared in the optimal reaction conditions (3 mol % of organocatalyst at 90 °C in chlorobenzene for 24 hours) aromatic and aliphatic epoxides were reacted with isocyanates to prepare the corresponding oxazolidin-2-ones. The activity of catalysts has an important role in the synthesis since the cyclotrimerization of isocyanates is a competing reaction.[3]



**Scheme 1.** *Synthesis of oxazolidinones.*

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## O-8

# KINETICS ON SUPERCRITICAL EPOXIDATION OF VEGETABLE OILS

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*Chemical Engineering*

The great importance of the use of epoxidized vegetable oils is due to their suitability as an alternative bio-based precursor for many industrially relevant products.[1]

The most widespread way of carrying out the epoxidation of the double bonds present in vegetable oils involves the use of percarboxylic acids, that come from organic acids. Side products generation reactions are the main issue in conducting the selectivity of the process to the formation of epoxides.

Environmental concerns related to the use of mineral acids and organic solvents, in addition to the need to simplify and improve the purification processes of the products obtained, leads to a growing interest in the outlining of an alternative route to the traditional epoxidation process.

In this context, arises the possibility of obtaining the oxygen transfer agent from an unconventional source, such as carbon dioxide. $CO_2$ can be not only one of the reagents for epoxidation, but also the reaction media if it is used under supercritical conditions, as an effective solvent. In addition, supercritical $CO_2$ ($scCO_2$), can be removed after the process has been completed simply by means of controlled depressurization. Previous work by the research group itself confirmed the feasibility of this reaction as well as the positive effect on the overall process performance of various enhancer compounds.

The background obtained by the group considered mild operating conditions and optimized the amount of the reagents and enhancer compounds. The study of the epoxidation kinetics in supercritical media would allow an in-depth understanding of the mechanism involved, how the use of $CO_2$ affects the occurrence of the oxirane ring opening phenomena and the subsequent formation of side products, as well as the evolution of the whole system over time, and its overall behavior when it is developed at different temperatures.

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## O-9

# A screening method for the assessment of the release of silver nanoparticles from food containers

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*Analytical Chemistry*

Silver nanoparticles (AgNPs) are used in packaged products to enhance the quality and shelf-life due its antimicrobial and bactericidal properties. Silver ions detached from the surface of the nanoparticles are responsible for these properties. However, AgNPs could migrate into food too and generate health risks and/or toxicological effects to the consumers.[1] The common approach to tackle this issue is to monitor AgNPs in food simulants that have been in contact with the packaging material according to the European Food Safety Authority (EFSA).[2] For this reason, there is a need for analytical methods that evaluate the migration of AgNPs in these simulants.

The analytical techniques currently available provide structural and quantitative information on AgNPs but they are time-consuming, require expensive equipment and generate more information than it is required. Qualitative analytical methods are good alternatives because they classify samples as positive or negative in a rapid, simple, and cost-effective way, which is also most valuable for routine laboratories. Nevertheless, to our knowledge, screening methods for AgNPs have not been used so far. Therefore, the aim of this work is to apply an instrumental screening method of AgNPs for the assessment of their migration from plastic containers into food simulants.

The analytical method is based on the chemiluminescence of the luminol/$AgNO_3$ system in the presence of AgNPs. Total silver migration and AgNPs released were investigated from plastic vessels containing nanosilver, into different food simulants, namely water and 3 % acetic acid at different temperatures and contact times. A nanosilver free plastic food container was used as control. Probability of detection (POD) curves were constructed at three cut-off values next to the limit of detection (1 µg/L) for each food simulant and performance parameters such as false negative and positive rates as well as unreliability region (UR), specificity, sensitivity, and reliability rates were calculated for validation. Silver migration occurred in both food simulants and conditions but, according to the screening method, not as AgNPs (0 % positive). All the samples were spiked with AgNPs around the UR and a reliability rate of 0.89 was obtained. The results of screening were confirmed by asymmetric flow field flow fractionation coupled to inductively coupled plasma mass spectrometry (AF4-ICP-MS).

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## O-10

# BIMETALLIC ZINC CATALYSTS FOR RING-OPENING COPOLYMERIZATION PROCESSES

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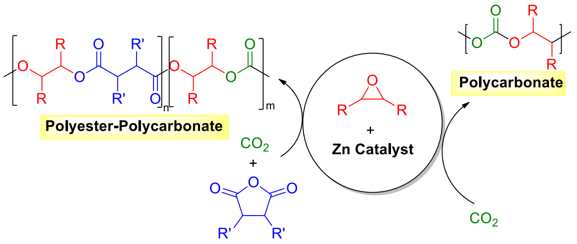
*Inorganic Chemistry*

The transformation of carbon dioxide into high-value added organic chemicals and polymers has received much attention in recent years as carbon dioxide is seen as an alternative carbon feedstock for a sustainable chemical industry.[1] One of the most relevant processes is the reaction of carbon dioxide with epoxides to afford polycarbonates, which have found many applications. In this context, the terpolymerization between $CO_2$, an epoxide and cyclic anhydrides has also showed the potential to fine-tune the properties of the resulting polymeric material. For instance, these terpolymers have shown improved degradability and enhanced thermal and/or mechanical properties.[2] In this contribution, we report the synthesis of novel bimetallic zinc acetate complexes supported by heteroscorpionate ligands for the ring-opening copolymerization of cyclohexene oxide and $CO_2$ and the terpolymerization of cyclohexene oxide, phthalic anhydride and $CO_2$, obtaining polymeric materials with a broad range of thermal properties (Scheme 1).[3]



**Scheme 1**

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## O-11

# GLYCOLYSIS PROCESS FOR POLYURETHANE COMPOSITES RECYCLING

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*Chemical Engineering*

Polyurethanes (PUs) are generally thermoset polymers resulting from the addition between a polyol and an isocyanate. The characteristic functional group obtained by the nucleophilic addition of the oxygen of the polyalcohol to the carbon of the cyano group is the urethane. Based on their applications, PUs can be classified into foams (flexible or rigid) and CASEs (Coatings, Adhesives, Sealants, Elastomers). The global tendency in the polyurethane market is the development of advanced products or composites with one or more improved properties, incorporating different additives or fillers for this purpose, therefore, it becomes essential to find solutions for the recycling of this waste at the end of its useful life, considering the presence and destination of their fillers.

PUs are the sixth group of polymers most used in the world and generate a large volume of waste, being its management an environmental challenge due to its low biodegradability. Traditionally, PUs waste has been deposited in landfills but it necessary to find new and more environmentally friendly alternatives.

Physical recycling processes are successfully used in thermoplastic polymers, but they are useless for most PUs due to their thermostable nature. Therefore, chemical recycling processes are of special interest, making possible to obtain chemical products from the PUs foams waste, such as polyether polyol for the synthesis of new PUs foams or even the fillers and additives in the case of PUs composites waste.

Glycolysis is the most widely used chemical recycling process for PUs waste and it consists of a transesterification reaction, in which the ester group bonded to the carbonyl carbon of the urethane is exchanged for the hydroxyl group of the glycol. This reaction results in the formation of a polyol and an unstable carbamate at the reaction temperatures, which promotes the formation of secondary amines and carbon dioxide. In addition, with an excess of glycol in the reaction, a biphasic product is obtained, where the upper phase is formed mainly by the recovered polyol and the bottom phase by the excess of glycolysis agent and by-products of the reaction; achieving, this way, a recovered polyol of high purity. Besides, our research has demonstrated that the polyurethane foams synthesized from the recovered polyol meet the conditions and specifications for which are designed and that some organic and inorganic fillers can also be recovered and reused.

## O-12

# BENZOAZOLES AS TWO-PHOTON ABSORBING CHROMOPHORES

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*Organic Chemistry*

Two-photon absorption (TPA) spectroscopy [1] has emerged as an attractive tool to further study the basic properties of materials and matter–light interactions. In recent years, interest in materials that exhibit TPA has widely increased due to the development of applications such as 3D microfabrication [2], optical data storage [3], and two-photon induced fluorescence (TPIF) imaging [4].

TPA properties are closely related to the molecular architecture, which makes it essential to identify structure–property relationships. The best results are obtained with centrosymmetric quadrupolar π-conjugated dyes with terminal donor or acceptor substitution, and with symmetrical designs displaying donor groups in the periphery of the molecule, that is, D-π-D or D- π - A- π -D patterns. In this sense, a series of donor–π–acceptor– π –donor (D- π -A- π - D) benzoazole dyes with 2*H*-benzo[*d*][1,2,3]triazole (BTZ) or benzo[*c*][1,2,5]tiadiazol (BTD) cores have been prepared.

The two photon absorption properties of the different compounds synthesized were studied to establish the relationship between structure and properties. These results provided cross sections of up to 1500 GM, with a predominance of $S_2 \leftarrow S_0$ transitions and a high charge-transfer character. Time-dependent DFT calculations supported the experimental results.

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## O-13

# DEVELOPMENT OF A COMBINED ELECTRO-SCRUBBING PROCESS FOR THE TREATMENT OF VOLATILE ORGANIC COMPOUNDS

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*Ingeniería Electroquímica y Ambiental*

Volatile organic compounds (VOCs) are characterized by their high activity in atmospheric photochemical reactions, so they can cause serious environmental problems such as the greenhouse effect, photochemical smog and stratospheric ozone depletion. Human health is also affected because VOCs can be easily absorbed through the skin and mucous membrane, damaging the body and the metabolic system, and in some cases can even be carcinogenic. Therefore, the control of air quality and the elimination of VOCs is a topic of interest today.

Thus, the objective of this research is to study the elimination of toluene as a representative of VOCs, using "electro-scrubbing": an emerging technology for the treatment of gaseous currents, which consists of the combination of an absorption column with an electrochemical oxidation process mediated, whose purpose is to retain gaseous pollutants and then degrade them using acid solutions of metal pairs as a catalyst.

For this purpose, a defined volume of pure toluene is bubbled with air in order to generate currents charged with the contaminant that circulates through an absorption column in countercurrent to a washing solution. The activation of the metal catalyst and the efficiency of the contact between the phases are carefully analysed parameters. The exhaust gas stream is captured in a hexane trap that allows the quantification of undegraded toluene. The removal efficiency and possible reaction intermediates are analyzed by mass-coupled gas chromatography (GC-MS) and HPLC.

## O-14

# COMPARATIVE STUDY ABOUT FORMATION OF SECONDARY ORGANIC AEROSOL FROM OZONOLOYSIS OF ORGANIC COMPOUNDS

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*Physical Chemistry*

Assessing the effect of anthropogenic aerosol on climate change is a huge challenge since it is considered, at present time, one of the main uncertainty sources for the understanding of the Earth's climate[1]. The ozonolysis of terpenes has been reported as a potential source of new particles under natural conditions[2], and likewise, some organics compounds could be a source of SOA (Secondary Organic Aerosol) by fast reactions of some alkenes with ozone. Comparative SOA formation from ozonolysis of 2,3-dihydrofurane, 2,5-dihydrofurane, styrene, indene and 2,5-dimethylfurane has been studied in this work.

The reactions have been carried out in a Teflon chamber filled with synthetic air mixtures, under atmospheric pressure and room temperature. The particle formation was followed by SMPS (Scanning Mobility Particle Sizer) and CPC (Condensation Particle Counter).

In order to evaluate SOA formation, it has been checked the conditions that lead to the formation and growth of new particles. The main parameters for their characterization are nucleation, their temporal profile and influence of different amounts of reagents. Various series of experiments were carried out changing initial concentrations of both organic compound and $O_3$. It was also checked the influence of the presence or absence of $SO_2$, and the influence of the relative humidity. Results were analyzed taking into account the different organic compounds structures.

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## O-15

# NOVEL APPROACH FOR THE ANALYTICAL CONTROL OF GOLD AND PLATINUM NANOPARTICLES IN CLINICAL AND TOXICOLOGICAL MATRICES

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*Analytical Chemistry*

The use of metallic nanoparticles as gold (AuNPs) and platinum (PtNPs) are rapidly growing due to their physicochemical and biological properties which makes them useful in biomedicine. However, the information about their toxicological profile is scarce and it is mandatory to create new methodologies for their characterization and quantification in complex clinical and toxicological matrices. One of the most powerful techniques proposed to solve this concern is single particle inductively coupled plasma mass spectrometry (SP-ICP-MS). It allows to obtain simultaneously information about the particle concentration (number and mass-based), size and its distribution at clinically relevant concentrations with minimal or even without sample preparation [1]. Nevertheless, its applicability for these types of matrices should be demonstrated. The aim of this work has been to develop novel strategies based on SP-ICP-MS for the analytical control of AuNPs and PtNPs in biofluids and culture media. Working under optimum conditions it is possible to obtain a correct NPs characterization in 60 seconds of analysis at ng $L^{-1}$ level of AuNPs (40, 60, and 80 nm) in Dulbecco's Modified Eagle Medium cell culture medium and PtNPs (50, and 70 nm) in human urine and blood serum samples, both individually and in mixed solutions. The size values are in accordance with transmission electron microscopy measurements. Also, these strategies have allowed to follow NPs transformations over time. The results of this study will be very useful to achieve an adequate interpretation and management of the interactions between NPs and biological systems in future (pre)clinical trials.

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## O-16

# RING-OPENING POLYMERIZATION OF CICLOHEXENE OXIDE CATALYZED BY SCORPIONATE ALUMINUM COMPLEXES

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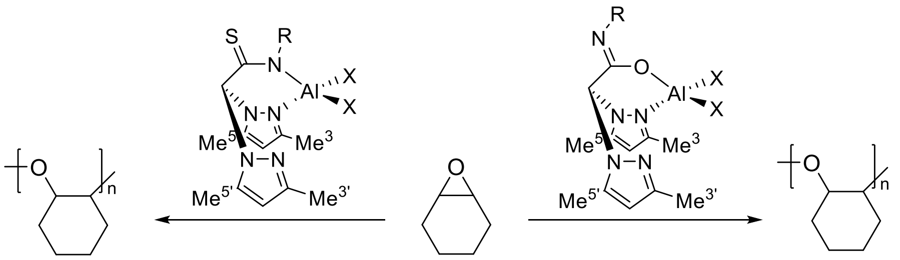
*Organometallic Chemistry*

Aluminum is the most abundant metal in the Earth's crust and its complexes can be used as Lewis acid catalysts or as co-catalysts for a range of chemical transformations.[1] In particular, they have found application as homogeneous catalysts in polymerization processes. Recently, aluminum catalysts have received significant attention since these can be used as efficient catalysts for Ring-Opening Polymerization (ROP) processes of cyclic esters, propylene oxide and cyclohexene oxide, amongst others.[2]

In this context, our research group has significantly contributed to the development of novel aluminum complexes as catalysts for a considerable range of catalytic processes, such as the cyclic carbonates formation or ROP of cyclic esters.[3] Therefore, we decided to investigate the use of scorpionate aluminum complexes as catalysts for the synthesis of poly(cyclohexene oxide) in order to prepare new polyether materials which can find important application in industry (Scheme 1).



**Scheme 1.** Ring-opening polymerization of CHO catalyzed by aluminum complexes.

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## O-17

# Grape seed extract supplementation modulates hepatic lipid metabolism in healthy Wistar rats and could be effective for preventing the progression of overweigh-related metabolic disorders

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*Food Science and Technology*

Several epidemiological studies have related diets characterised by high plant consumption with a lower incidence of overweigh-related metabolic disorders. In this context, flavanols constitute one of the most common families of non-essential nutrients present in edible plants with wide bioactive potential to improve the health status, nevertheless the mechanisms of actions are not completely understood [1]. Thus, we aimed to evaluate the effects of a long-term dietary dose of a grape seed extract (GSE) in the hepatic lipid metabolism of young-mature Wistar rats.

Firstly, the characterization of the GSE showed a high content on polyphenols, mainly flavanols with different degree of polymerization. Secondly, we observed that GSE increase serum lipid levels and overexpression of hepatic genes related with fatty acid uptake, glycogen synthesis and VLDL secretion. Concomitantly, GSE stimulate oxidative metabolism and mitochondrial biogenesis in the liver, deducted from the overexpression of Acox-1 and PGC-1α, as well as, the downregulation of Plin-5.

Our preliminary results indicate that GSE consumption could be beneficial in preventing fat liver accumulation by promoting the secretion of lipoprotein particles and stimulating the liver oxidative metabolism. However, more studies are needed to check these hypotheses.

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## O-18

# ACRYLAMIDE HYDROGELS: DIFFERENT WAYS TO INTRODUCE MAGNETIC NANOPARTICLES

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*Organic Chemistry*

Hydrogels have been extensively studied by the scientific community due to its excellent properties, what make them useful for such a big number of applications. Hydrogels are three-dimensional networks, physically or chemically crosslinked, that can absorb water though not soluble in it.[1] The properties of these soft systems can be tuned by changing its composition or by introducing nanoparticles. In this study, different kinds of magnetic nanoparticles are introduced within acrylamide hydrogels, like $CoH_2$NPs, $CoFe_2O_4$NPs and $Fe_3O_4$NPs, in order to study its magnetic properties. Moreover, Superparamagnetic Iron Oxide Nanoparticles (SPIONs) are introduced into acrylamide hydrogels by two sorts of strategies, which are *blending method* and *in situ co-precipitation*.[2] Advantages and disadvantages of both methods will be discussed.

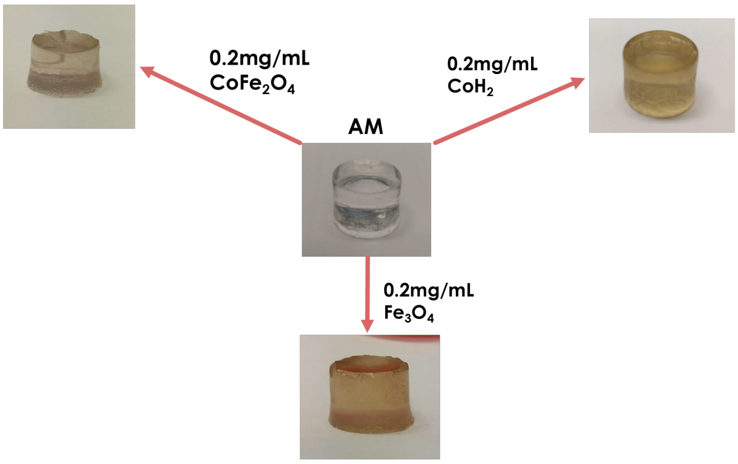

**Figure 1.** Different magnetic hydrogels synthesized by blending method.

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## O-19

# Accurate quantification of polycyclic aromatic compounds adsorbed on soot samples

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*Química-Física*

The incomplete combustion processes contribute to a large extent generating atmospheric pollutants in gas phase and particles in suspension. The main source of particulate matter is traffic.[1] Their emissions are constituted by soot that is mainly composed of black carbon and SOF (soluble organic fraction) where organic compounds such as Polycyclic Aromatic Compounds (PACs) are adsorbed. They are considered as toxic persistent substances.[2] Soot has been classified as the second largest contributor to global warming after $CO_2$.[3] Therefore, knowing the chemical composition of soot is important in order to develop new (bio) fuels and engines that generate minimal environmental impact and cause less risk to human health.

In this study, three real soot samples, two diesel and one biodiesel have been generated by a EURO 3 engine in different engine conditions (mode E and G).[4,5] A previously developed and validate methodology consisting of a microwave extraction (MAE) and GC-MS/MS analysis to identify and quantify the PACs present on the SOFs has been applied to analyse the collected soot samples. Among the PACs, polycyclic aromatic hydrocarbons (PAHs) and their derivatives nitro-PAHs (NPAH) and oxygenated-PAHs (OPAH) have been analysed. The results show that using the mixture of extraction solvents pyridine-acetic acid, the amount of PACs extracted was higher than the amount found using acetone-toluene. The total amount of PACs extracted was 50, 28 and 20 ng $mg^{-1}$ for diesel-mode E, biodiesel-mode G and diesel-mode G, respectively. NPAH have not been identified in any sample and the amounts of OPAHs were lower than those of the PAHs. A second extraction of the biodiesel soot sample with pyridine-acetic acid greatly improved the concentration of PACs in the SOF (46 ng $mg^{-1}$). In addition, the total amount of PACs extracted from the real soot samples was much lower than that extracted from diesel particulate matter standard reference material NIST 1650b (692 ng/mg). The results obtained confirm the differences in the composition of soot samples that could be attributed to the conditions of temperature at which the soot is produced, to the formation of PACs that takes place during combustion (very unknown process) and/or to the properties of the fuels.

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## O-20

# MAGNETIC-RESPONSIVE HYDROPHOBIC HYDROGELS FOR DRUG DELIVERY APPLICATIONS

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*Química Orgánica*

Hydrogels, 3D water-swollen crosslinked polymer networks, have stood out for the past two decades as particularly promising biomaterials for controlled drug delivery applications. These smart materials may respond to specific stimulations, such as pH, temperature, light, electric or magnetic fields.[1] Magnetic-responsive hydrogels acquire magnetic sensitivity upon incorporation of magnetic nanoparticles (MNPs) into the polymer network, for which favorable polymer matrix-NP interactions must occur. Additionally, to fulfil their role as drug reservoirs, hydrogels must also be mechanically robust in order to withstand fracture during cargo release and must be compatible with commercial marketed drugs, the majority of which are hydrophobic in nature.

Based on these premises, we herein fabricated a multi-functional nanocomposite hydrogel for magnetic-stimulated drug delivery. By introduction of diaminotriazines (DATs) in the hydrogel backbone, a hydrophobic, physically-crosslinked and magnetic responsive hydrogel was fabricated (Fig. ) The key multi-functionality of the hydrogel laid on the DAT skeleton: strong H-bonding interactions between DAT hydrophobic moieties allowed formation of a stable physically-crosslinked network, as well as a suitable hosting and release environment for hydrophobic compounds.[2] Additionally, DAT centers served as efficient anchoring sites for MNP formation, which conferred the hydrogel with magnetic-responsiveness.

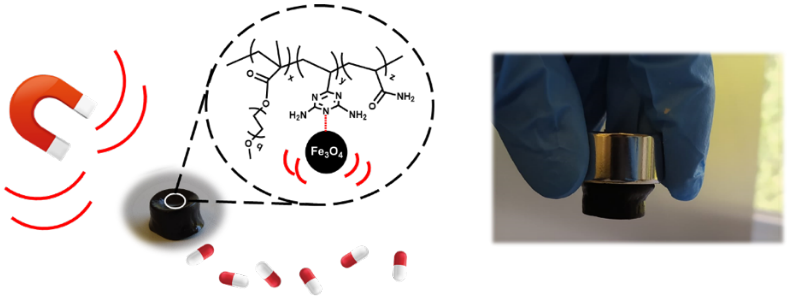

Fig. Magnetic-responsive diaminotriazine hydrogels

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## O-21

# NANOSTRUCTURED ELECTRODES PREPARED BY MAGNETRON SPUTTERING FOR WATER ELECTROLYSIS

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*Chemical Engineering*

Water electrolysis to obtain hydrogen in combination with intermittent renewable energy resources constitute a sustainable alternative to fossil fuels (see Figure 1). Anion Exchange Membrane water electrolysis (AEMWE) solves the problems associated with other traditional alkaline and proton exchange membrane water electrolysis techniques. Among the most promising non-platinum materials, nickel- and Ni-alloy-based layers have demonstrated high electrocatalytic activity and stability when they act either as electrodes in alkaline electrolyzers. Commonly, the catalyst load is incorporated onto the support using traditional techniques with little capacity to control de catalyst microstructure and composition. To solve these problems, we propose the use of Magnetron Sputtering (MS) technique to prepare the active electrode catalyst films for AEMWE. In this study, the influence of different fabrication parameters of nickel electrodes has been studied in a three-electrode glass cell and in a complete electrolysis cell obtaining a great stability and efficiency in relation to the amount of catalyst. In addition, a thorough structural and chemical characterization of these porous thin film electrodes by SEM, XPS, XAS and XRD, before and after being used, has been carried out.

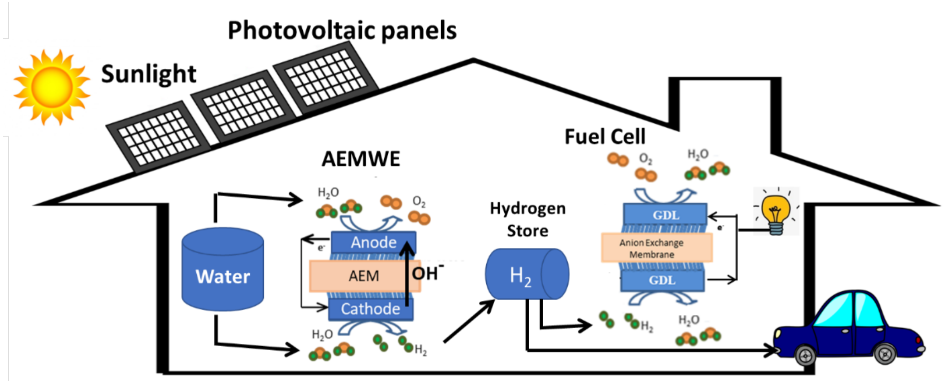

*Figure 1. Coupling system of photovoltaic panels for domestic electrical energy self-sufficiency.*

## O-22

# RESISTANCE TO OXIDATION OF $SO_2$ FREE RED WINES ELABORATED WITH NATURAL ANTIOXIDANTS

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*Food Science and Technology*

As a result of the harmful effects that $SO_2$ can have on health, the oenological sector is looking for natural antioxidants that can replace or reduce the dose of $SO_2$ used in winemaking. Chitosan, inactive dry yeast and different winery by-products have been studied as alternatives to the use of $SO_2$. However, the resistance to oxidation of the red wines fermented with these products is unknown. Electron paramagnetic resonance (EPR) is presented as a technique that allows the direct detection of free radicals in complex systems and allows determination of the level of oxidation in wines [1]. In this sense, the present study aimed to evaluate the influence of chitosan, inactive dry yeast and two extracts from winery by-products (stem and shoot) on the formation of 1-hydroxyethyl radical in red wines.

Six types of red wines from grapes *Vitis vinifera* L. Cv. Cabernet Sauvignon were elaborated in triplicate: control wine without $SO_2$, wine with $SO_2$, wine with chitosan, wine with inactive dry yeast, wine with stem extract and wine with shoot extract. The resistance to oxidation of the wines was evaluated by EPR using POBN as spin trap. The results showed that all wines produced 1-hydroxyethyl radical induced by air bubbling and exposure to visible light during 6 min. The lowest concentration of the 1-hydroxyethyl radical was found in wines elaborated with $SO_2$, chitosan and inactive dry yeast. While the wines with stem and shoot extracts showed lower resistance to oxidation. Chitosan and inactive dry yeast could be considered as potential natural alternatives for reducing $SO_2$ in red winemaking because they allowed the elaboration of red wines capable of controlling the formation of 1-hydroxyethyl radical.

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## O-23

# “EFFECTS OF CENTRAL LEPTIN ON METABOLIC REPROGRAMMING OF ADIPOSE TISSUE. ROLE OF PPARβ/δ AND FGF21”

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*Ciencias de la Salud*

The adipose tissue is a dynamic organ that serves as a nutritional sensor of the energy stored. The flexibility of the adipocytes signaling an excess or demand of energy has the origin in an extremely well-coordinated control of transcription that allow the adipocyte to shifts rapidly in response to changes in nutritional status and nutrients intake. These molecular processes are commonly known as metabolic/transcriptional reprogramming, indicative of physiological adaptations of an organism. Leptin is one of the most important adipokines identified signaling at central level to indicate excess of energy. Thus, metabolic disorders are generally accompanied by impairing in leptin signaling due to a phenomenon referred as leptin resistance. However, the molecular mechanisms or initial events that lead to the development of these pathological states are not well defined and require further investigation. In this research work we have studied potential central leptin effects on visceral white adipose tissue metabolic reprogramming dependent on PPARβ/δ activity, a transcription factor involved in lipid homeostasis. The study was performed in young male Wistar rats of 3-months-old. Central leptin treatment (0,2 µg/day, 7 days) was administered by intracerebroventricular surgery and mini-osmotic pumps were placed in the interscapular subcutaneous space. Inhibition of PPARβ/δ activity was performed by “in vivo” intraperitoneal pharmacological administration of the selective antagonist GSK0660. The results obtained in this experimental work show that PPARβ/δ activity is required for the anorexigenic effects of central leptin and its lipolytic actions exerted, through the Central Nervous System (CNS), on white adipose tissue. Moreover, the pharmacological inhibition “in vivo” of this nuclear receptor induced lipid accumulation, increased adiposity and partially downregulated the browning capacity in epididymal white adipose tissue, impairing the oxidative and functional state of the adipocytes. Furthermore, central leptin treatment during 7 days decreased the circulating levels of the fibroblast growth factor 21 (FGF21), and at the same time that increased its endogenous adipose tissue expression, in parallel with the increased expression of key thermogenic markers, which are also related to the lipolysis process. Unexpectedly, GSK0660 treatment dramatically inhibited the endogenous FGF21 expression in epididymal white adipose tissue, suggesting a potential interaction between the transcription factor PPARβ/δ and FGF21 expression not previously described.

## O-24

# ELECTROCHEMICAL TECHNOLOGIES FOR THE ELIMINATION OF ANTIBIOTICS IN HOSPITAL URINE

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*Electrochemistry*

In last decade, the daily use of drugs has resulted in the continuous discharge of these drugs into the aquatic environment. The presence of pharmaceutical compounds in waste effluents has become a serious environmental problem on a global scale, which has led to an increased interest in detecting their presence in seas and rivers. The origin of the problem is that not all drugs are eliminated by conventional treatments carried out in the wastewater treatment plants. As a result, the presence of these drugs in aquatic systems poses a potential risk, and even partially unknown, for health and for the environment. Furthermore, this situation gives rise to an increase in bacterial resistance, which causes the drugs to be every less and less effective.

In this context, electrochemical advanced oxidation processes (EAOPs) are an interesting alternative to conventional technologies due to its capacity to degrade complex molecules. The objective of the present work was to achieve the elimination of different pharmaceuticals such as antibiotics using a microfluidic flow-through cell.

### *Acknowledgments*

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## O-25

# GRAPHENE-BASED SULFONATE HYDROGELS: SOFT SCAFFOLDS FOR CELL CULTURE

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*Química Orgánica*

Heparin is an interesting molecule which plays an important role in a lot of biological processes. Their sulfonate and carboxylate functional groups allow the interaction with growth factors and proteins.[1] However, its high anticoagulant effect may often lead to hemorrhagic complications.

Hydrogels have attracted much attention in the field of tissue engineering in recent times, having numerous applications, especially as scaffolds for cell cultures.[2] We have also shown that the introduction of graphene in the hydrogel structure improves their biocompatibility supporting the growth of cultured brain cells and, allowing neuronal adhesion.[3]

In this work, we have carried out the preparation of novel graphene hybrid hydrogels based on sodium 4-vinylbenzenesulfonate (VBS), as a monomer mimicking heparin structure. Different hydrogels were characterized and compared in order to study their capacity to support living neurons.

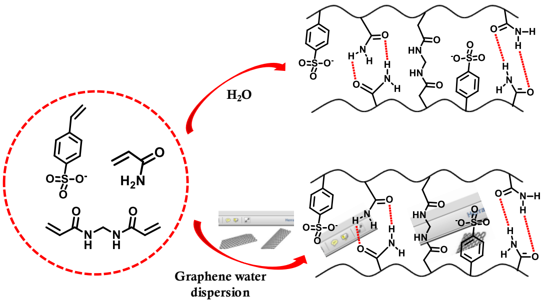

**Figure 1.** VBS based hydrogels in the presence and absence of graphene.

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## O-26

# INFLUENCE OF ULTRASOUNDS ON WINE MICROBIOTA

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*Tecnología de Alimentos*

The characteristic microbiota of a winemaking process involves numerous genera and species of yeast and bacteria. Yeasts, specifically *S.cerevisiae*, are responsible for carrying out the alcoholic fermentation. It has a great capacity to grow in the must due to its tolerance of high sugar content and low nitrogen concentration. On the other hand, other non-*Saccharomyces* yeasts may or may not be present. *Dekkera anomala* is one of the most problematic yeasts in winemaking.

Moreover, lactic acid bacteria transform glucose into lactic acid as they have an aerotolerant anaerobic metabolism. The number of lactic acid bacteria during alcoholic fermentation is normally very low, however, their population increases very significantly in wines where malolactic fermentation takes place to decrease again in the finished product. The aim of this work was to study how different ultrasound treatments affect the vitality and viability of some of the characteristic microorganisms of wine: *Lactobacillus plantarum* and two different species of yeast, *Saccharomyces cerevisiae* and *Dekkera anomala.*

Six different treatments were applied to fresh crops with a population of $10^6$ cfu/mL, varying the conditions of exposure time, power and the use or not of electric pulses. To study the effect on viability, plate counts were carried out, while the variation in vitality was quantified by obtaining the kinetic parameters of the microorganisms after each treatment. The mediums used were MRS and YPD for bacteria and yeasts respectively. In each case, in parallel, a positive control without application of ultrasound was used.

The results showed that on some occasions the treatments had an effect on vitality or even increased it slightly; in others, the microbial metabolism was attenuated, which is mainly reflected in an increase in the latency phase and loss of viability. Finally, only one of the applications characterized by using the highest power and not using pulses, was able to destroy all the microorganisms.

## O-27

# Antitumoral effects of resveratrol through adenosinergic system on cervical cancer cell line

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*Biochemistry*

Cancer is a multifactorial disease that affects millions of people worldwide. Despite the efforts made, an effective treatment against this disease has not been found yet. Natural compounds, such as polyphenols, have emerged as preventive agents against cancer. Currently, we are focused on resveratrol, a phytoalexin mainly present in peanuts, grapes and red wine, which has shown a promising effect in inhibiting proliferation and cancer progression in several tumoral models. However, its molecular mechanisms remain unclear. Adenosine receptors ($A_1$, $A_{2A}$, $A_{2B}$ and $A_3$) have emerged as a promising target in cancer due to its involvement in various stages of tumorigenesis, such as proliferation, angiogenesis and metastasis. Therefore, the main aim of this study was to determine antitumoral effects of resveratrol through adenosinergic system on human HeLa cells, a cancer cell line derived from epithelioma cervix. Cell viability by XTT method, adenosine receptors quantification by Western-blotting and gene expression by real time PCR were assayed. Results herein showed a significant decrease on HeLa cell viability in a time- and concentration-dependent manner after treatment with resveratrol. Accordingly, there was a reduction in the number of treated cells. In addition, the treatment with this polyphenol caused an increase in $A_1$ and $A_{2A}$ gene expression and a decrease in $A_{2B}$ protein level. Furthermore, 5'-Nucleotidase activity was determined and it was observed that resveratrol significantly reduced this activity in plasma membrane. As resveratrol has been shown to be a non-selective adenosine receptors agonist, these results suggest that a possible mechanism underlying antitumoral effect of resveratrol could be through adenosine receptor binding.

## O-28

# Tumor lockdown: how to beat cancer through confinement measures

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Despite progress in the treatment of breast cancer, a high percentage remains incurable and the disease is considered the leading associated cause of death in women worldwide. In this regard, several factors are involved in the initiation and progression of breast tumors. Tumor cells depend on external signals to grow. These signals come from the surrounding microenvironment, in which adipose tissue is the majority component. In addition, this tumor microenvironment (TM) provides a niche for tumor cells that may be responsible for the often poor response to treatment that certain breast tumors have. Triple negative breast tumors (TNBC) are characterized by the absence of hormone receptors on their membrane, so they lack specific targets that can be used for their treatment. Chemotherapy is the most widely used option, but the development of resistance is a frequent problem. These therapeutic limitations make TNBC the molecular subtype with the worst prognosis.

In the present study, we evaluated the role of the adipose niche, in particular of adipose stem cells (ASC), which is the population with the greatest plasticity of adipose tissue, and then, the may have a more important influence on the initiation and progression of the tumor, as well as on the response to chemotherapeutic drugs in the most aggressive and worst prognosis breast cancer subtype (TNBC).

To this end, we have generated an *ex vivo* niche tumor model from ASCs obtained from TNBC patients to recreate the patient's pathophysiological conditions in the laboratory as close as to reality as possible. Exposure to a conditioned medium prepared from these cells ($ASC^{TNBC}$-CM) promotes tumorigenicity and invasiveness in breast cancer. Moreover, after exposure to $ASC^{TNBC}$-CM, the inhibitory effect of three chemotherapeutic agents, cisplatin, doxorubicin and docetaxel, is reduced, increasing cell viability, the occurrence of metastasis during treatment and recurrence after it.

The *ex vivo* tumor niche model used in this work provides relevant information for the understanding of TNBC tumors. In view of the role played by the adipose niche in the chemoresistance of TNBC, it is necessary to develop new therapies based not only on attacking the tumor population, but also the cells surrounding it. Thus, therapies that target both cancer cells and their TM can be more effective than those that target only tumor cells, since the lockdown of communication between the two may reverse the resistance to chemotherapy.

## O-29

# FLAVAN-3-OL COMPOSITION OF CHOCOLATE PANNED RAISINS

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*Food Sciences and Technology*

Raisins and chocolate are foods rich in phenolic compounds with great acceptance by consumers. However, since they are foods with different compositions, the combination of these two foods in a single product produces a complex matrix. Regarding the phenolic compounds, flavan-3-ols are the principal phenolic compounds in the chocolate and are an important class in grape phenolic composition too. Thus, the aim of this study was to evaluate the influence of this combination (chocolate + raisin) on the flavan-3-ols profile in the chocolate panned raisins (dragée) produced. For that, high performance liquid chromatography (HPLC) coupled to different types of detectors was applied. In general, it was noticed that the concentration of flavan-3-ols in the dragées produced was significantly high due to the contribution of chocolate. For the flavan-3-ol monomers group, five monomers ((+) – catechin (C), (-) – epicatechin (EC), (-) – epigallocatechin (EGC), (-) – gallocatechin (GC) and (-) - epicatechin 3-gallate (ECG)) were detected in the dragées produced and in the raisins used as it's center. EC was the major monomer with approximated value of 117 mg/kg of dried weight product (DW) in the dragées, an amount significantly ($\alpha = 0.05$) higher than those found for the raisins (3,46 mg/kg of product – DW). The results showed that the chocolate used to cover the raisins provided a significant increase in all monomers when compared to the raisin used as the dragée center, except for ECG, which was present in higher concentration in the raisins (0,87 mg/kg of product – DW) than in the dragées (0,17 mg/kg of product – DW). In the dragées evaluated, the EC alone accounted for about 23% of the total flavan-3-ols found, demonstrating to be the main phenolic compound in these products. For the dimers group, was detected the presence of procyanidins PB1, PB2 and PB4 in all samples evaluated, being the PB4 the one with the highest concentration in the dragée samples (approximately 247 mg/kg of product – DW) , followed by PB2, while PB1 remained in concentrations statistically equal ($\alpha = 0.05$) to the raisin used as dragée center.

## O-30

# SPECIATION OF A POLYPHENOLIC PIGMENT IN FREE SOLUTION AND ENCAPSULATED INTO A NANOSCARRIER SYSTEM. QUANTIFICATION OF THIS BIOACTIVE IN A MICELLAR MEDIUM IN NUTRACEUTICALS

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*Química Analítica*

Nowadays, the use of bioactive lipophilic molecules in food industry offers unresolved issues such as their poor aqueous solubility, reduced bioavailability and pH sensitivity. Curcumin is one of these compounds that belongs to the family of polyphenols which offers beneficial properties for human health. Those mentioned problems have been tried to be solved using nanotechnology for developing nanostructured organic compounds.[1] Several techniques for nanoencapsulation of bioactive compounds have been developed, which has raised serious concerns due to the lack of toxicological information about these nanostructured systems. Due to this fact, it is necessary to develop analytical procedures to monitor and discriminate between these two different environments of analytes.

Here, nanocurcumin was prepared by encapsulating this bioactive into water soluble nanomicelles to solve a problem of food industry and a smart application based on carbon material was developed, being able to distinguish between nanoparticulated and free forms of curcumin. Using well-designed and characterized graphene quantum dots (GQDs), it was demonstrated a novel application for the speciation of curcumin in both environments without the need of an analytical separation technique or complex instrumentation. In particular, the determination of free curcumin was based on a gradual quenching on GQDs photoluminescent emission as well as a red shifting on its maximum wavelength by increasing its concentration. In contrast, nanocurcumin only produced a quenching effect on the GQDs intensity remaining constant its maximum wavelength. These findings form the basis of a new method for the recognition of nano-sized curcumin and free curcumin species in complex matrix. Its applicability was tested in enriched beverage and nutraceutical samples containing either free curcumin or nanocurcumin. Results reveal an excellent selectivity of the ensuing GQDs as fluorescence probe for discriminating between micellar formulations of curcumin and free curcumin in food.

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## O-31

# PUSH-PULL AZAHETEROCYCLIC CHROMOPHORES: SYNTHESIS, PHOTOPHYSICAL PROPERTIES, AND USE FOR WHITE LIGHT EMISSION.

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Conjugated push–pull chromophores based on azaheterocyclic fragments have been investigated in recent years.[1] Indeed, the photophysical properties of these materials can be easily tuned by environmental stimuli such as polarity, pH, and the presence of metal cations.[2] Six-membered nitrogen heterocycles such as pyridine, quinoline, and (benzo)diazines act as moderate-to-strong electron-withdrawing groups. When protons or metal cations are added, the photophysical properties are modified due to the interaction with the electron lone pair of the nitrogen atoms of the heterocycle, which leads to an increase in the electron-withdrawing character and enhances the intramolecular charge transfer to the chromophore.[3] This kind of phenomenon has been used to obtain sensors and a variety of optical switches.[4]

In this communication, we will describe a new series of conjugated push-pull pyrimidines and (iso)quinolines. They exhibited emission solvatochromism in a variety of non-polar solvents, and tunable halochromism. Some of the compounds remained moderately luminescent after protonation and these had a orange-red emissive form, which was used to obtain white light emission, both in solution and in thin film, by the controlled protonation of the initially violet-blue-green emitting materials. This methodology has potential applications in white OLED fabrication with two forms of a single emitter in equilibrium.

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## O-32

# $CO_2$ PHOTOCATALYTIC REDUCTION WITH CNT/$TiO_2$/Cu BASED NANOCOMPOSITES PREPARED BY SUPERCRITICAL $CO_2$

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*Chemical Engineering*

From all the possible solutions to reduce atmospheric $CO_2$ concentration, our group has gained some experience in the $CO_2$ photoreduction with simulated sunlight and $TiO_2$-based catalysts.

In previous works we reported that the $TiO_2$-based catalysts (nanoparticles, nanofibers) synthesized in supercritical conditions improved the catalysts properties and $CO_2$ reduction yields regarding those catalysts prepared by traditional synthesis.[1,2] However, there was still room for improvement. In this context, the present work reports novelties found when synthesizing $TiO_2$ composites modified by the addition of carbon nanotubes (CNT). Particularly, the CNT/$TiO_2$ proportion and Cu load for different CNT/$TiO_2$ and CNT/$TiO_2$/Cu nanocomposites have been assessed.[3] In addition, two types of supercritical synthesis were implemented and it could be observed that the catalysts synthesized in one step exhibit better photocatalytic activity than those obtained with the two steps synthesis.

The best results were obtained when the $TiO_2$ nanoparticles were supported on to carbon nanotubes with a proportion of CNT/$TiO_2$ (50%-50%). The excellent optoelectronic qualities of carbon nanotubes combined with $TiO_2$ increased the generation of products in the $CO_2$ photoreduction. Specifically, they were 15 (methane) and 4 (carbon monoxide) times higher than with commercial $TiO_2$, P25. However, the effect of copper doping was overshadowed by the effect of CNT support.

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## O-33

# Applying statistics criteria in rational selection of probiotic Lactobacillus strains to be used as biocontrol agents

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In recent years, there has been an increasing interest in alternatives to the use of additives to ensure food safety. The use of probiotics has been concentrated on human health, but their use as biocontrol agents is gaining importance nowadays. Ninety-eight *Lactobacillus* (*Lb*.) strains belonging to thirteen species isolated from different fermented food and drinks were screened to select those with the best probiotic and antimicrobial properties. Firstly, a screening based on growth kinetics under gastrointestinal tract (GIT) conditions was carried out. Twenty-eight strains were able to grow under simulated GIT conditions and these strains were assayed for autoaggregation, coaggregation and hydrophobicity. 7.1% were strongly autoaggregating strains after 4 hours of incubation. Differences between strains were observed in both aggregation activities. Strains belonging to *Lb. plantarum* and *Lb. paraplantarum* species showed the highest coaggregation percentages with the two pathogens tested: *Salmonella choleraesuis* and *Staphylococcus aureus*. Regarding hydrophobicity, there was correlation between the results obtained for the two solvents (xylene and toluene) although better results were obtained with toluene. Twenty strains, selected by principal component analysis (PCA), were identified as potential probiotics and were selected for further study forming biofilms and adhering to Caco-2/TC7 cells. The percentage of cells that form biofilms was around 53-75%. Adhesion to Caco-2/TC7 cells was around 51-97%, and Lb29 strain belonging to *Lb. paracasei* had the greatest percentage of adhesion (97%). Furthermore, antimicrobial activity of all ninety-eight strains was also carried out. 51% of strains showed antimicrobial activity against the four species indicators used: *Salmonella choleraesuis*, *Staphylococcus aureus*, *Bacillus cereus* and *Listeria monocytogenes*. The results showed that three strains, *Lb. paracasei* Lb38, *Lb. plantarum* Lb93 and *Lb. brevis* Lb99, showed both the best probiotic profile and antimicrobial properties. Therefore, these strains have displayed great potential to be considered both as potential probiotics and biocontrol agents in the food industry.

## O-34

# SUBSTITUTED ALKYNYLBENZENES AND BITHIOPHENES-BASED CRYSTALLINE STRUCTURES: X-RAY STUDY AND OPTICAL WAVEGUIDE BEHAVIOUR

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In last years, organic micro-nano/crystalline structures have received great attention as optical waveguides due to their ability to confine and propagate the light. For this reason, they are potential components of photonic circuits.[1]

Photonic properties are highly dependent on the structure of the crystal, which is formed thought non-covalent intermolecular interactions between the molecules. In this way, these interactions play an important role in the photonic behaviour.[2]

With the aim to establishing a structure-property relationship, fibrillar crystalline structures have been obtained by slow diffusion, through self-assembly of substituted alkynylbenzenes and bithiophenes. All compounds were synthesized by C-C coupling reactions under microwave radiation.

X-Ray studies of crystals have shown the crucial role of non-covalent interactions in the supramolecular structure. By fluorescence microscopy, it has been demonstrated that these crystalline structures show optical waveguide behaviour, being potential structures to be integrated as components in photonic circuits.

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## O-35

# EFFECT OF COVID-19 LOCKDOWN ON THE AIR QUALITY OF DIFFERENT CITIES

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COVID-19 health crisis has forced an unprecedented confinement of population and drastic drop of industrial activities and transport in many countries. The benefits in air quality are evident for citizens and from remote sensing. Nevertheless, the atmospheric surface concentrations of pollutants are strongly dependent on meteorology and show high variability. Thus, the simple comparison of given days before and during the crisis may result misleading. Thus, in this work we statistically assess the changes of pollutants considering a wide database (2013-2020) from surface air quality monitoring stations.

This work reports the short-term effects on air quality of such unprecedented paralysis of industry and transport in different size cities in Spain, one of the countries most affected by the virus and with the hardest confinement measures. Data series from ground-air quality monitoring networks have been analysed to assess changes in atmospheric pollutants during March-April 2020 due to this sudden paralysis of activity. The results show significant concentration drops of primary pollutants, including NOx, CO, SO2, BTX, NMHC and NH3. On the other hand, particulate matter changes were small due to the existence of other natural sources and no clear effect was observed for ozone concentrations. The results and conclusions for these cities may serve as behaviour model for other continental sites and help understand future crisis.

# PRESENTATIONS FLASH & POSTERS

**(appearance in alphabetic order)**

## F-1

# SUPERCRITICAL FLUID EXTRACTION OF BIOACTIVE COMPOUNDS

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*Ingeniería química*

In recent years, the increase in the demand of nutraceutical and pharmaceutical products of natural origin has led to the search of sources of bioactive products. In this sense, the agro-food sector of Castilla-La Mancha offers the possibility to obtain these products from low-value materials or residues, allowing both to satisfy the market need and encourage economic promotion. Special interest has been paid to compounds with antioxidant and anti-inflammatory potential for the treatment of skin-related diseases such as atopic dermatitis. Several studies have shown the feasibility of extracting these compounds from essential oils of plants, residues of the wine industry or even purple garlic.

Bioactive compounds are usually extracted with organic solvents. However, these methods have certain limitations such as high energy costs, high temperatures and low selectivity. For this reason, alternative methods are being studied, like supercritical fluid extraction (SFE). This technology is environmentally friendly and offers the versatility needed to treat different raw materials. Carbon dioxide ($CO_2$) is the most widely used supercritical fluid, as it is inert, non-toxic and allows extraction at lower temperatures and pressures.

Lavender essential oil has been extensively studied for its antioxidant and anti-inflammatory potential, which is attributed to one of its predominant compounds, linalool. In this sense, the present work focuses on the supercritical extraction of this compound for its application in drugs and nutraceuticals. In order to obtain linalool, the equilibrium system of lavender essential oil with $CO_2$ has to be studied, through the analysis of the influence of pressure and temperature, as well as the optimal conditions for supercritical extraction and isolation of this substance, which will be quantified by liquid and gas chromatography.

## F-2

# REMOVAL OF EMERGING POLLUTANT DIBUTYLHYDROXYTOLUENE FROM WATER WITH CNT/$TIO_2$ CATALYSTS IN A VISIBLE LED PHOTOREACTOR

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*Chemical Engineering*

Heterogeneous photocatalysis based on $TiO_2$ has been used in several studies as an effective Advanced Oxidation Process (AOP) for the removal of refractory pollutants in water streams. Further, since this technology allows using solar energy, it would be an economically and environmentally sustainable solution. However, the main drawback of using $TiO_2$ as solar photocatalyst is that its high band-gap energy (~ 3.2 eV) allows working efficiently only with ultraviolet (UV) light. In this work, in order to reduce the $TiO_2$ band gap so that it could absorb visible radiation, carbon nanotubes (CNT) are used to synthesize CNT/$TiO_2$ composites in supercritical $CO_2$ medium (Hemalatha et al., 2015). CNTs have been chosen to modify $TiO_2$ because they can also improve the photooxidation efficiency by other vias such as increasing pollutant adsorption to the catalyst surface, and accepting the photogenerated electrons in $TiO_2$ particles, thus hindering their recombination with holes.

The objective of this study has been to explore the visible light assisted photodegradation of BHT (2,6-Di-tert-butyl-hydroxytoluene), a synthetic phenolic antioxidant (E-321) widely used as antioxidant and/or UV-filter, employing different CNT/$TiO_2$ composites synthesized in supercritical $CO_2$. First, the synthesis process of the catalytic material is analyzed and the effect of CNT percentage on the physico-chemical properties of CNT/$TiO_2$ composites established. Next, we investigate the photocatalytic removal of BHT and the effect of process parameters, such as pH or composite CNT load, on pollutant degradation is determined. The roles played in the photocatalytic reaction by different reactive oxidative species such as $h^+$, $OH^•$, $^1O_2$, *and* $O_2^{\cdot-}$, have also been evaluated using appropriate scavengers.

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## F-3

# CHEMICAL CHARACTERIZATION OF LA MANCHA CHELVA WINES: INFLUENCE OF WINEMAKING TECHNIQUE

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The present research work had a two principal aims one is a study of the volatile composition of Chelva wines from Castilla-La Mancha and on the other research the effect that different winemaking techniques, wine treatment with glycosidic enzymes and pre-fermentative maceration of the must, have on the volatile compounds of the aroma of La Mancha Chelva wines. Volatile compounds were isolated by solid phase extraction and them analyzed by gas chromatography coupled to mass spectrometry. The aroma of control wines was characterized by higher concentrations of $C_6$ and benzenic compounds. The pre-fermentative maceration of the must with the solid parts of the grape and the treatment of the wine with glycosidic enzymes increased the concentration of the main varietal compounds of Chelva wines. The compounds responsible for aromatic differentiation of wines based on the processing technique used were mainly isoeugenol, β-damascenone, (Z)-3-hexene-1-ol, linalool and ethyl hexanoate. According to the results it may be concluded that all studied winemaking techniques provides a viable alternative to traditional winemaking methods for improve and enhancing the aroma profile of La Mancha Chelva wines, increasing the offer to the consumer, which favors the differentiation of our wines on the national and international market.

## F-4

# Gas-phase reactivity of acetaldehyde with OH at ultra-low temperatures (11.7-177.5 K)

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*Physical Chemistry*

Acetaldehyde ($CH_3CHO$) has been observed in the cold sources of the interstellar medium (ISM). As hydroxyl (OH) radical is ubiquitous in many interstellar environments, it is a key intermediate in many reactive processes in the ISM. Hence, the gas-phase kinetics of the OH + $CH_3CHO$ reaction is important to be known at the ultra-low temperatures of the ISM (T~10-100 K). This reaction has been studied due to its importance in atmospheric (down to 200K) and combustion chemistry (up to 1400 K). To date, the only determination of the rate coefficient, *k*(T), below 200 K for the title reaction was carried out by Vöhringer-Martínez et al.[2] at 60, 77, 106 and 118 K. These authors observed an increase of *k*(T) with respect to that at room temperature of ~5 times at 60 K and ~2 at T>100 K. In this work, the temperature dependence of *k*(T) for the OH+$CH_3CHO$ reaction has been determined in a extended range between 11.7 K and 177.5 K. It is essential to include the kinetic data at ultralow temperature (close to 10 K) to include in a pure gas-phase model that predicts the abundances of existing and/or undetected species (e.g. $CH_3CO$ radicals, main product at IS temperatures) in the ISM, as will be presented. To study this reaction the CRESU (French acronym for *Reaction Kinetics in an Uniform Supersonic Flow*) system allows us to achieve ultra-low temperatures, by cooling down the gases below the freezing point without their condensation, based on a supersonic expansion [3]. This technique was coupled to PLP-LIF (Pulsed Laser Photolysis/Laser Induced Fluorescence) to perform the kinetic study. The potential pressure dependence of *k*(T) has been measured at several temperatures, as well.

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## F-5

# MECHANOCHEMICAL PREPARATION OF PIEZOELECTRIC NANOMATERIALS: BN, $MoS_2$ AND $WS_2$ 2D MATERIALS AND THEIR GLYCINE-COCRYSTALS.

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*Química Orgánica*

Piezoelectric materials have a unique property that converts mechanical energy into electrical energy or vice versa.[1] Barium titanate is the first piezoelectric ceramic ever discovered, but the ceramic lead zirconate titanate, also known as PZT, is the most used material for piezoelectric harvesting.[2] Different nanomaterials in the dichalcogenides family have been theorised to have piezoelectric behaviour. Some of them have been tested, such as boron nitride and molybdenum disulphide, and confirmed experimentally. Still, the procedures to obtain these nanomaterials, with the right quality to observe the piezoelectric behaviour, are enormously expensive, halting its possible applications.

Here, we show different 2D-layered nanomaterials through their mechanochemical exfoliation with glycine such as boron nitride (BN), molybdenum disulphide ($MoS_2$), Tungsten disulphide (WS2) and graphen (FLG) of 2D nanomaterials (FLG, BN, MoS2 and WS2). We have also successfully synthesised the cocrystals for these nanomaterials,2 which makes it possible to enhance their piezoelectric responses.[3]

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## F-6

# SUSTAINABLE ORGANIC CHEMISTRY: SYNTHESIS AND EXTRACTION OF HIGH VALUE-ADDED CHEMICALS FROM TABLE OLIVE WASTEWATERS

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Spain is the world-leader table olive producer, and the Spanish-style green olives comprise almost 50% of total table olive production. During table olive processing, high amounts of lye are employed to remove olive bitterness, mainly due to a secoiridoid glucoside called oleuropein,[1] to make them edible. Also, high amounts of water are necessary to remove lye completely.

The physicochemical composition of these wastewaters is characterised by an enormous amount of organic matter (mainly phenolic compounds), high Chemical Oxygen Demand (COD) and pH,[2] which give them a considerable phytotoxicity and, together with the huge amount of wastewater produced by table olive industry, pose a threat to the environment. For this reason, a wide range of studies have merged nowadays aimed to valorise this residue.[3]

In this study, we present for one hand the extraction of tyrosol (TY) and hydroxytyrosol (HT) from table olive processing wastewaters and their quantification by Nuclear Magnetic Resonance (NMR). For the other hand, the synthesis of high added-value chemicals, namely 5-Hydroxymethylfurfural (5-HMF) and levulinic acid (LA) from the sugar-rich aqueous phase after extraction, using environmentally friendly conditions, such as microwave radiation, homogeneous catalysis or water as solvent.

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## F-7

# TRACEABILITY AND QUALITY ASSESEMENT OF WINE CORK STOPPERS USING VOLATILE FRACTION ANALYSED BY SPME-GC-MS

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*Area of Food Technology*

Nowadays, natural cork stoppers are still the chosen closures for bottling quality wines, representing a high cost for wineries and one of the most important factors that influence wine quality. The certification of its quality and its traceability is a great challenge for the cork industry, being the physical-chemical characterization a very useful tool when establishing these parameters. The volatile fraction of cork is made up of a high number of compounds that, even when in a small concentration, can influence the final aroma of the wine. On the other hand, these compounds come from the cork sheets used to make the stoppers and, therefore, can be good indicators of their origin. In this work, a total of 140 volatile compounds (terpenes, esters, acids, aldehydes and hydrocarbons) from natural cork stoppers of different qualities and origins have been analyzed, using a fast and sensitive technique such as SPME-GC-MS. Based on these compounds, it has been possible to establish differences between stoppers of different origins and qualities, showing that the volatile composition of corks can be very useful for the differentiation and classification of cork stoppers, as well as to establish their traceability.

## F-8

# "Teamwork to beat breast cancer"

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*Bioquímica*

In 2019, the number of new cancer cases diagnosed in Spain reached 277,234, with breast cancer being the second most prevalent. Some types of breast cancer are incurable diseases with limited therapeutic options. Among the approved therapies, are chemotherapies that modify the genetic material. One of the main problems of these treatments is the development of resistances. Targeting DNA repair mechanisms, like the inhibition of the Chk1 kinase, has become one interesting therapeutic option because then cells cannot repair the DNA modification. In this study, we explored synergistic interactions of Chk1 inhibitors (rabusertib and SAR020106) with those clinical treatments for breast cancer. In addition, we evaluated the role of these agents to overcome platinum resistance. Using a panel of breast cancer cell lines, we identified a synergistic interaction between Chk1 inhibitors and platinum compounds. This association increased apoptosis through a caspase dependent mechanism. Rabusertib demonstrated activity in combination with cisplatin in one breast cancer platinum-resistant cell line, inducing an increase in cell death and, therefore, overcoming the resistance to this compound. Combination of Chk1 inhibitor with cisplatin and gemcitabine resulted in more activity than single or double combinations. In conclusion, in this study we identify novel therapeutic options for the clinical development of Chk1 inhibitors and confirm that the inhibition of this kinase can overcome acquired resistance to cisplatin.

## F-9

# CARBON-BASED NANODOTS AS EFFECTIVE ELECTROCHEMICAL SENSING TOOLS TOWARDS THE SIMULTANEOUS DETECTION OF BIOACTIVE COMPOUNDS IN COMPLEX MATRICES

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*Química Analítica*

A comparative study about the electroanalytical performance of three types of carbon-based nanodots (CND, CQD and GQD) has been accomplished. After the structural (TEM, FTIR, Raman and fluorescence) and electrochemical characterization (using specific redox probes, outer and inner sphere systems), the electroanalytical capabilities of these carbogenic nanodots as suitable sensing tools towards the simultaneous detection of several bioactives like vitamins (ascorbic acid or Vit C, pyridoxine or Vit B2, riboflavin or Vit B6) and amino acids (cysteine and tyrosine) were also evaluated and discussed attending to the main interactions responsible for improvement in peak currents and potentials. Finally, GQD-based electrodes, selected as the best choice, were submitted to an exhaustive electroanalytical performance characteristics evaluation. The results in terms of reproducibility and repeatability evidenced the good precision of the electrodes considering a potential manufacturing process (batch-to-batch evaluation). Detection limits (LOD) for riboflavin, ascorbic acid and tyrosine were found to be of 0.83 µM, 0.1 mM and 0.12 mM respectively, whereas their corresponding quantification limits (LOQ) were 2.50 µM, 0.30 mM and 0.35 mM, respectively. GQD-Nf was satisfactory applied for the simultaneous determination of three diverse analytes in commercial sports supplements whose their discrimination is usually difficult for their closer oxidation potentials. The DPV signals of Mincartil, Vitax and L-Tyrosine BioTech samples showed RSD values (n=5) for riboflavin (Mincartil) of 4.2 (current) and 3.1% (peak potential), for ascorbic acid (Vitax) of 1.6 (current) and 1.1% (peak potential), and for tyrosine (L-Tyrosine Biotech) of 4.6 (current) and 0.7 % (peak potential), respectively. The success of this simple drop-casting procedure was also proved by affording the simultaneous detection of three diverse bioanalytes in complex commercial matrices and with lower detection limits in comparison to other reported proposals from similar nature.

## F-10

# GAS-PHASE KINETICS OF OH($H_2O$) AND $H_2O_2$($H_2O$) COMPLEXES AT ULTRALOW TEMPERATURE: FORMATION AND OH REACTIVITY.

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*Physical Chemistry*

Water vapor, $H_2O$, is one of the most abundant species in the Earth's atmosphere ($T$~220-298 K, in the troposphere), but it can also be found in the atmosphere of many objects in the solar system at low temperature conditions ($T$≤150 K), such as Jupiter's and Saturn's moons. One of the most relevant processes involving $H_2O$ in our atmosphere is the formation of the main diurnal oxidant, the hydroxyl (OH) radicals, which act as a major sink for many species. In addition, $H_2O$ can form hydrogen-bonded complexes with many atmospheric species and consequently influences the radiation balance on Earth and affects the atmospheric chemistry. Water complexes have received much attention in past decades in atmospheric chemistry, because of their impact on the radiative balance and their reactivity towards atmospheric radicals. As complexation is favored at low temperatures, the CRESU (French acronym of *Cinétique de Réaction en Ecoulement Supersonique Uniforme*)[1] technique is an excellent tool to investigate the formation of water complexes and their reactivity in the gas-phase. We studied and reported for the first time, the formation and OH-reactivity rate coefficients of OH($H_2O$) and $H_2O_2$($H_2O$) complexes at ultra-low temperature. Photolysis of gaseous $H_2O_2$ was used as a source of OH radicals in a pulsed CRESU apparatus and the OH loss was monitored by laser induced fluorescence as a function of $[H_2O]_0$ in excess with respect to $[OH]_0$. A kinetic model that interprets the experimental observations provides the rate coefficients for the formation of OH($H_2O$) and $H_2O_2$($H_2O$) complexes and for the OH-reaction of $H_2O_2$($H_2O$) and $(H_2O)_2$ at 21.7 K.

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## F-11

# PROTACS: BREAKING THE BARRIERS OF BREAST CANCER

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*Bioquímica*

Breast cancer (BC) it is the most common tumor in women worldwide. In Spain in 2018, more than 32,000 patients were diagnosed with this disease, representing 28.7% of all cancer cases in women. It is not just one disease, people affected do not always have the same prognosis or the same treatments. This is because BC is classified according to the presence of molecules located on the surface of the tumor cell that serve as a pharmacological target. Triple negative breast cancer (TNBC) is the most aggressive subtype. It lacks these targets; therefore, novel therapies are needed. At present, TNBC is only treated with chemotherapy, which has two drawbacks: inespecificity and drug resistance.

PROTACs (proteolysis targeting chimera) are a new generation of targeted therapies drugs. A PROTAC is composed of three parts; an E3 ligand that binds to an E3 ligase, a ligand that binds to the protein of interest, and a linker connecting the two ligands. E3 ligase can marker the protein by ubiquitination, targeting it for destruction by the proteasome. In our study, we explore the antitumoral activity of two PROTACs, MZ1 and ARV-825, against BRD4 protein. Both PROTACs efficiently and maintained deleted the levels of BRD4. We evaluated the antiproliferation effect using MTT assay, colony-forming assay, and three-dimensional cultures. They showed a profound antiproliferative effect in using 2D and 3D cultures. To explore the molecular mechanism of drugs we used flow cytometry. The analysis of protein expression was determined by western blot. A profound effect on apoptosis was observed in these experiments. *In vivo* studies included mice that were injected with MDAMB-231 JQ1 (BRD4 inhibitor, no PROTAC) resistant cells. Administration of MZ1 was able to recue tumor growth in nude mice of MDA-MB-231 JQ1 resistant cells, by reducing the expression of BRD4. We describe the profound activity of MZ1 and ARV-825 in TNBC cell lines and in an *in vivo* resistant model. This data provides options for further clinical development of these agents in TNBC.

## F-12

# Autonomous self-healing hydrogels and their mechanism

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*Organic Chemistry*

The idea of making a device able to repair itself when it gets damage is very attractive. This self-healing ability is desired for engineers and chemists who have been working thoroughly to obtain these auto-reparable products. This kind of materials is no longer unattainable but is currently a real fact, as already some commercial coating agents and mobile protectors with self-healing ability exist and other devices are focused of intensive research.

Here, we present a hydrogel, which is a 3D polymeric material able to absorb large amounts of water without being dissolved by the aqueous medium. Our hydrogel shows the ability to self-heal at a particular amount of water and keeps the water inside the hydrogel at ambient conditions. In order to improve the self-healing behaviour of our hydrogel, the first step is to understand the mechanism. Therefore, different experiments were performed: a) computational studies and thermal analysis were performed to understand the role of a specific amount of water inside the polymeric network and b) when the water was replaced by DMSO, the hydrogel lost the self-healing ability. The analysis of these studies allowed us to conclude that self-healing is due to the formation of hydrogen bonds between water molecules inside the network. One water molecule acts as a bridge between the solvation cells of the two ammonium groups (Fig. 1). The amount of water needs to be precise as otherwise a high water content also leads to the loss of the self-healing feature.

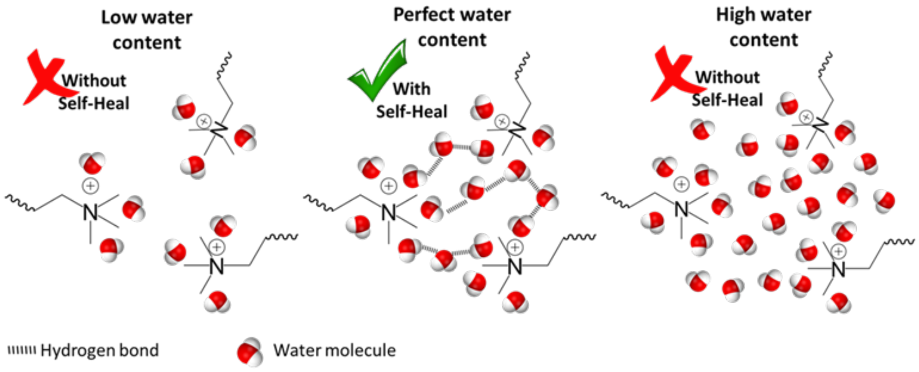

**Figure 1:** Proposed hydrogel self-healing mechanism

## F-13

# $CO_X$-FREE HYDROGEN PRODUCTION FROM AMMONIA DECOMPOSITION REACTION

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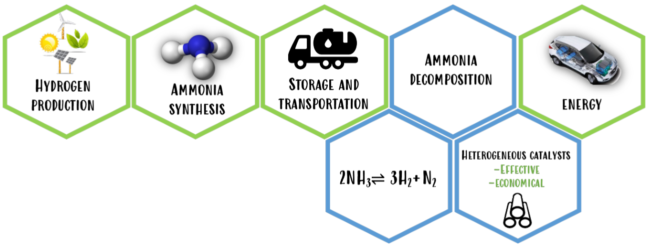
*Chemical Engineering*

Alternative energies are urgently needed due to the decrease in the reservoirs in fossil-fuels and environmental damage caused by fossil-fuels combustion. Hydrogen ($H_2$) is widely recognized as an important "energy vector" because it can be used as an environmentally clean energy carrier for portable applications using fuel cells and internal combustion engines, releasing only non-toxic by-product such as water. The security of the storage and distribution techniques for hydrogen is a problem that is currently slowing the utilization of the existing hydrogen fuel cell technologies. Thereby, alternative hydrogen storage technologies are required.

One of these alternatives can be storage hydrogen chemically in hydrogen-rich molecules (called "hydrogen carriers") such as methanol, methane, metal amine salts, ammonia and related compounds or hydrides. Among them, ammonia ($NH_3$) with high $H_2$ content (17.6 wt%), suitable physical properties and well established technologies of production and distribution, has been considered as an excellent hydrogen carrier[1]. In addition, the ammonia decomposition reaction occurs at high temperatures and only produces nitrogen ($N_2$) and hydrogen, therefore, $NH_3$ is a carbon-free hydrogen carrier due to its decomposition only yields $N_2$ and $H_2$.

The aim of this project is to explore different novel catalysts to find an effective and economical process to produce hydrogen from ammonia decomposition. In this sense, high activity (close to 100% conversion), high stability of catalyst and both low load of noble metal and low reaction temperature (<500°C) are required.



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## F-14

# Safety assessment of strains of *Lactobacillus* isolated in different fermented foods: biogenic amines production.

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*Food Sciences and Technology*

Biogenic amines (BA) are low molecular weight substances, formed mainly by decarboxylation of amino acids present in food by some microorganisms. They are toxic compounds that can cause adverse effects on consumers. Since the accumulation of BA presents a toxicological risk to the health of the consumer, is very important to control their occurrence and production in food. Therefore, is essential to evaluate the safety of the strains to be used for food production. The aim of this work was to analyse the BA production by strains of *Lactobacillus* spp. isolated from several food ecosystems. This bacterial group has been used for many years such a safe microorganism to produce fermented foods, but in previous studies it has been demonstrated that certain strains of lactobacilli could produce BA. The results of the analyses showed that 58% of the strains were BA non-producers. In addition, it was possible to verify how the decarboxylation of amino acids capacity is strain dependent.

## F-15

# DEGRADATION OF PESTICIDES BY A COUPLED OXIDATION ADVANCED PROCESS

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*Chemical engineering*

The need to increase the quality and quantity of agricultural products requires the control of living organisms that produce damage to crops; the most common way to deal with this problem is the use of pesticides. However; the lack of adequate human training on the correct application has led to an excessive use of them and thus the pollution of soil, groundwater and surface water. The degradation of pesticides listed as persistent organic pollutants (POPs) has been studied through various treatment technologies. For example, biological treatments reported that the biodegradation is significantly affected by specific concentrations of pesticide, pH, temperature and long treatment time. Furthermore, in many cases, the last degradation product is more toxic that the objective pesticide. Attractive alternative technologies as Advanced Oxidation Processes (AOP's) for the degradation of (POPs) have emerged as promising methods for wastewater treatment. Because they have been reported to be relatively simple, easy to control, and result in a high oxidation degree and compatibility with the environment.

This work evaluates the electro-oxidation-ozone coupled treatment as a viable process for the degradation of pesticides such as Endosulfan and Clopyralid. These technologies increase the formation of radical oxidizing agents as $\cdot OH$, $O_3$, and $H_2O_2$. These radicals achieved high efficiency in the treatment by improving removal rates, reducing treatment time, energy consumption and operating costs.

The results of the electrofenton-electrooxidation-ozonation ($EF/EO/O_3$) for the endosulfan degradation show: Electrooxidation with Boron doped diamond (BDD) electrodes process achieved 49% of endosulfan degradation efficiency in 2 h, Ozonation process removed 62% in 2h and electrofenton allows a 72% of endosulfan degradation efficiency. The integrated process $EO/O_3$ improved the removal with 89% of removal efficiency in 2 h, but the $EF/EO/O_3$ increases the removal to 92% and decreases the treatment time to only 1h, optimizing the process with a lower energy consumption.

## F-16

# HAS CENTRAL LEPTIN THE ABILITY OF CONTROLLING THERMOREGULATION?

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*Bioquímica*

Leptin at central level seems to play an important role in the protection of peripheral tissues. In heart, it protects against damage produced as a result of diseases like obesity, which degenerates into diabetes, hypertension or heart failure. Our group has recently demonstrated that some actions of central leptin in the heart are produced through the nuclear receptor PPARβ/δ. Herein we study the role of the heart in the thermogenic effect of central leptin.

To this end, we analysed the effects of intracerebroventricular leptin infusion 7 days (0.2μg/day) on cardiac metabolism in 3-month-old Wistar rats with normal leptin sensitivity; and examine the effects of pharmacological inhibition of PPARβ/δ with the specific antagonist GSK0660 in rats infused with leptin. We focus on the study of the mRNA levels of FGF21 and its receptors in the heart, and those of several genes involved in cardiac lipid metabolism, thermogenesis and immune response such as LPL, ATGL, UCP-1, FAS, TBX15, HIF-1α, HIF-2α, CCL5, CD64, CD32 and CD3g.

Our results indicate that central leptin decreased serum levels of FGF21. Nevertheless, the mRNA levels of FGF21 increased in the heart of leptin treated rats. Moreover, the gene expression of the specific receptors FGFr1 and FGFr2 were markedly increased by central leptin. Central leptin treatment induced the expression of LPL, ATGL, UCP-1, FAS, TBX15, HIF-1α and HIF-2α in the heart in a PPARβ/δ-dependent manner, indicating that central leptin treatment is involved in the regulation of hypoxia, immune response and thermogenesis in the heart independently of their effects on food intake. These results support a model in which overexpression of PPARβ/δ by central leptin protects the heart against cell damage.

## F-17

# PM2.5 AND MICROORGANISMS ANNUAL VARIABILITY MEASUREMENTS ON A SUBURBAN ATMOSPHERE IN THE CENTRAL IBERIAN PENINSULA.

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*Microbiología*

The aim of this study was to assess outdoor air quality in the Technological Campus of Toledo during the summer of 2018. For this reason, during this period the concentration of $PM_{2.5}$ particles was determined, as well as the diversity and the total microbial load, and associated to those particles. Due to the fact that summer sampling completed one year of study, the final analysis of the results obtained was carried out.

Sampling was taken for 5 consecutive weeks with two different types of samplers: 1) a $PM_{2.5}$ particle collector by filtration and 2) an air collector by impact on solid surfaces. The determination of the particles was carried out by gravimetry. For the growth of microorganisms, three culture media were used: Tryptone Soya Agar (TSA) and R2A, selective for bacteria, and Rose Bengal agar (RB), selective for fungi. After counting the grown colonies, a representative number of colonies were isolated and purified. Then, the genotyping of the isolates was developed using RAPD-PCR technique and the main genotypes were identified by sequencing of 16S rRNA fragment.

After analyzing the annual data, it was revealed that the concentrations of $PM_{2.5}$ obtained were within the limits established by Spanish and European legislation. In the samples taken by the MAS-100 Eco, the fungal concentration exceeded the bacterial concentration in all seasonal periods. In addition, in these counts, there were significant differences between the two specific media used for bacteria. Regarding the meteorological parameters, only temperature correlated positively with $PM_{2.5}$ concentration and total fungal and bacterial counts in the air.

The identification of the main genotypes revealed a high genetic diversity in winter and summer period. However, Gram (+) bacterial genera such as *Staphylococcus* and *Bacillus* predominated in all seasons.

## F-18

# ROLE OF IONIC STRENGTH AND ORGANIC MATTER AS CRITICAL FACTORS FOR THE ENVIRONMENTAL BEHAVIOUR OF METALLIC NANOPARTICLES

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*Analytical Chemistry*

Metallic nanoparticles (NPs) are being extensively used in many technological fields and daily products. Among them, platinum nanoparticles (PtNPs) are used in automotive catalytic converters due to their unique properties. However, chemical reactions and mechanical abrasion at the catalyst surface can lead to their emission [1]. Once these NPs are released into aquatic environments, their persistence and bioavailability are dependent both on their own physiochemical properties and water characteristics, such as the organic matter content or the ionic strength, which are expected to have a strong influence on their fate [2]. Little is known about the stability and transformations that these NPs can suffer, and adequate analytical methods are key to bring some knowledge on these questions. The development of novel and powerful analytical techniques able to provide reliable information about these topics is needed, which is indeed a challenging task for analytical chemistry nowadays.

For this purpose, a methodology based on asymmetric flow field flow fractionation hyphenated to inductively coupled plasma mass spectrometry (AF4-ICP-MS), previously optimized, was applied to study the influence of water characteristics onto PtNPs' behaviour, since it can provide reliable data about particle size range, concentration and composition. The effect of ionic strength (using different synthetic waters as test media with a broad range of ionic strength values) was studied over time (0 h and 48 h of exposure). Relevant changes in fractionation profiles were observed when increasing the ionic strength, which could be a sign of aggregation. Also, the presence of organic matter (using humic acid as a model in different concentrations, 2 and 5 mg $L^{-1}$) over that system was tested. It was found that its presence may improve PtNPs stability over time.

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## F-19

# STRATEGIES IN THE DESIGN OF ELASTIC ELECTRODES FOR CONTINUOUS SYSTEMS IN SOFT ROBOTICS

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*Organic Chemistry*

Hydrogels are biocompatible soft materials that resemble biological tissues more than any other material. However, the use of these systems in soft robotics has been limited to aqueous environments. In recent literature, hydrogels have relied on external water to swell or shrink in response to stimuli and, therefore, to actuate macroscopically. In recent years, advances in materials science inspired by biological structures are enabling the manufacture of different types of functional, light, and safe robotic devices.[1,2] Recently in our group, we found a way to make electroactive hydrogels capable of actuating when a low electric field is applied, even outside water. The problem with the actuation is that the electrodes we use tend to break and separate from the hydrogels. (**Figure 1**)

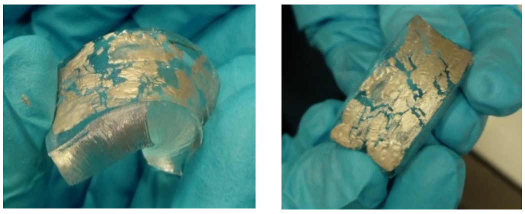

**Figure 1.** Silver enamel coated Hydrogel

In this work, we have identified different strategies to solve the degradation of the electrodes, such as applying stretchable and resistant gels filled with conductive fillers (conductive polymers and metallic nanoparticles). These materials have been synthesized to act as flexible electrodes and/or as an interface between different electrodes and the hydrogel actuator.

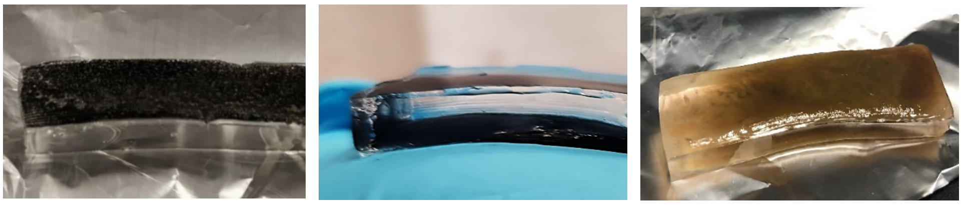

**Figure 2.** Conductive gels ensembled on the hydrogel actuator

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## P-1

# $CO_2$ FIXATIONTECHNOLOGIES: FROM BIOLOGY TO ELECTROCHEMISTRY

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*Ingeniería Química*

When the terrestrial plants first appeared in the ancient geologic time, the atmospheric $CO_2$ concentration was several-fold higher than today. This might be due to that the relative abundance of land plants was mainly responsible for the substantial reduction in $CO_2$ concentration by photosynthetic assimilation. In biology, there are different $CO_2$ fixation pathways that can efficiently work, and each of these has evolved a mechanism to fixate $CO_2$ [1]. Recycling of $CO_2$ into fuels and chemicals is a potential approach to reduce $CO_2$ emission and fossil-fuel consumption, and there is a feasible way for biological production of fuels and chemicals from $CO_2$ under normal conditions through biosynthetic pathways and bioengineering. From this point of view, there are mechanisms for the electrochemical reduction of $CO_2$, that can reduce this compound into more reduced chemical species using electrical energy most frequently to produce urea, salicylic acid, methanol and even carboxylic acids [2]. In this work, we aim to use NaOH produced from an electrochemical cell (cathodic area, stainless steel electrode) designed to generate $Cl^-$ from a NaCl solution (anodic area, boron doped diamond electrode), and fixing a flow of $CO_2$ into a adsorption glass column to produce carbonates and bicarbonates as secondary products, that can be commercialized and used for the desorption of $CO_2$ from the environment.

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## P-2

# HYDROGEN-CHLORINE PEM REVERSIBLE FUEL CELL ENERGY STORAGE SYSTEM

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*Electrochemistry*

Energy has been excessively dependent on fossil fuels and until relatively recently, it has not been aware of an important side effect of this source: climate change. Over thousands of years, the concentration of carbon species in the atmosphere slowly decreased, as a consequence of various processes that led to the formation of large accumulations of hydrocarbons. Furthermore, there is no perfect energy source, but probably solar and wind energy have the least impact on the planet, since in principle they seem to be the ones with which natural cycles are least disturbed. However, electricity generation is not easily adapted to demand. For this reason, energy storage alternatives are sought. The aim of this work is to develop a PEM reversible fuel cell capable of producing and storing energy, based on electrochemical technology, with the capacity to simultaneously fix carbon dioxide in the form of carbonates. In a first stage, based on the chlor-alkaline process, the electrolytic cell transforms sodium chloride into chlorine gas ($Cl_2$), hydrogen ($H_2$) and sodium hydroxide (NaOH). These two gas reagents are introduced, again into the cell, with the goal of producing electrical energy (fuel cell). In the second stage, one of the by-products obtained during the electrolysis mode (NaOH), it is used in a washing process, which after a crystallization stage is transformed into sodium carbonate, a compound that can be stored and buried. Thus, on the one hand, a green energy storage system is achieved, and on the other hand, a by-product is valued, reducing the carbon footprint.

## P-3

# GOOD FAT, BAD FAT: HOW NICHE AFFECTS BREAST CANCER DEVELOPMENT

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*Bioquímica*

**Introduction**. Most cancer studies are based solely on the study of tumor cells. However, here, we study how the microenvironment surrounding them affects the pathogenesis of the disease.

**Objective**. To study how tumor-associated fibroblasts (TAF) affect tumor cell gene expression in the context of the tumor microenvironment.

**Material and methods**. We compared the profile of gene deregulation induced in response to normal fibroblasts (NAF) vs CAF using the GSO public database **GSE70884.** Venn diagrams have been constructed to identify common genes. The functional analysis has been carried out using the Enrichr platform. The association with forecast has been developed using the KM Plotter tool.

**Results.** We showed how the difference in gene expression induced by the factors secreted by CAF is greater than that of NAF. In addition, the number of overexpressed genes shared between different cell lines is greater when they are exposed to the environment conditioned by CAF. The functions of genes overexpressed in response to CAF are mostly related to the immune system. Finally, the combined analysis of genes overexpressed by CAF with poor disease progression (HR≥1) has a higher prognostic value.

**Conclusions.** CAF-induced deregulation of gene expression is more intense and homogeneous than NAF-induced. Besides the fact that the microenvironment influences the tumor cells, it is involved in the tumor's immune response and in the worse evolution of the tumor.

## P-4

# PERSPETIVES AND CHALLENGES AT HIGH TEMPERATURE FOR GREEN HYDROGEN PRODUCTION BY THE HYBRID SULFUR CYCLE

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*Chemical Engineering*

The future of the energy sector clearly relies on the development of the renewable energies. However, there are two issues that must be addressed regarding these energies in order to make them perfectly suitable for the current energy system distribution. Those issues are that they are because they are dependent on the weather and they are also intermittent. The solution is found on the storage technologies. One of the cleanest ways to storage the electricity produced from renewable energies is to transform them in chemical energy in the form of Hydrogen.

The hybrid sulfur cycle has emerged as a very promising technology for this purpose. In this process, electric energy is used to produce hydrogen in an electrolyzer where the equations showed below occur. What makes this process more appealing than the production of hydrogen directly from water is that it is needed around five times less energy with the hybrid sulfur cycle.

$SO_2\,(g) + 2\,H_2O\,(g) \rightarrow H_2SO_4\,(aq) + 2\,H^+ + 2\,e^-$ **Anodic reaction**
$2\,H^+ + 2\,e^- \rightarrow H_2\,(g)$ **Cathodic reaction**

Up to now, this process has only been studied at low temperatures below 100ºC. Nevertheless, in this work temperatures in the range of 100ºC to 170ºC will be studied as a higher thermal efficiency of the process has been stated by different researchers. By working at high temperatures, the kinetics of the reaction are boosted, and the resistance of the materials decreases with temperature. Making the electrolysis step more efficient. Results show that by increasing temperature, less potential needs to be applied in order to start the reaction but other parasitic reactions (which produce oxidants in the anode which degrades the membrane and sulfur in the cathode) are also boosted resulting in some problems that must be addressed in further studies.

## P-5

# THE AROMA OF LA MANCHA TRUJILLO MELONS: VOLATILE AND SENSORY CHARACTERIZATION

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The main objective of this research was the detailed study of the aroma melons of the variety Trujillo grown in the region of Castilla-La Mancha. The melons were harvested for three consecutive years. The aroma free volatile compounds of the melons were isolated by Solid Phase Extraction (SPE) and then analyzed by Gas Chromatography coupled to Mass Spectrometry (GC-MS). The sensorial profile of the melons was evaluated by expert tasters using the attributes that had previously been selected as those that the best described the sensorial characteristics of the melons. Sixty-two free volatile compounds were identified and quantified in the Trujillo melons of the Castilla-La Mancha region in this period. Aldehydes, alcohols, acids and benzene compounds were the major constituents of aroma of La Mancha Trujillo melons. Sensory profile of melons aroma was characterized by aromas of jam/marmalade, cucumber, fresh fruit, sweet and ripe fruit, while the taste profile of the melons presented basic flavors such as sweet and acid and aromas retro-olfactory way like honey, jam, cucumber, fresh fruit, ripe fruit and green. This study is the first characterization of the aroma of the Trujillo melons from the La Mancha region and the data suggest that these melons have a complex aromatic profile and can be a viable alternative to expand the traditional market.

## P-6

# BIOELECTROCHEMICAL TECHNOLOGY FOR THE TREATMENT OF RECALCITRANT POLLUTANTS

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*Chemical Engineering - Bioelectrochemistry*

Conventional Bioelectrochemical Systems (BESs) consist in biological anodes and abiotic cathodes, in which bacteria attached to the electrode is able to (bio)catalyze the anodic reaction. Commonly, BESs working as Microbial Fuel Cells (MFCs) have been used to produce electrical energy from organic compounds in wastewater, using oxygen as oxidant at the cathode. Applications in BESs have been shooting up in recent decades, as well as improving the materials used in their design.

In this study, several hazardous and recalcitrant pollutants such as chlorophenols (CPs), used in the formulation of herbicides, pesticides, preservatives and disinfectants, have been treated through an electro-reductive hydrogenation process at the cathode of our BESs, seizing the catalytic activity of the electroactive biofilm for the anodic reaction, thus enhancing the overall energetic balance of the electrochemical cell. The bio-assisted electro-reductive treatment of the CPs allowed the total dechlorination of the molecule, dramatically increasing de biodegradability and reducing the toxicity of the original wastewater.

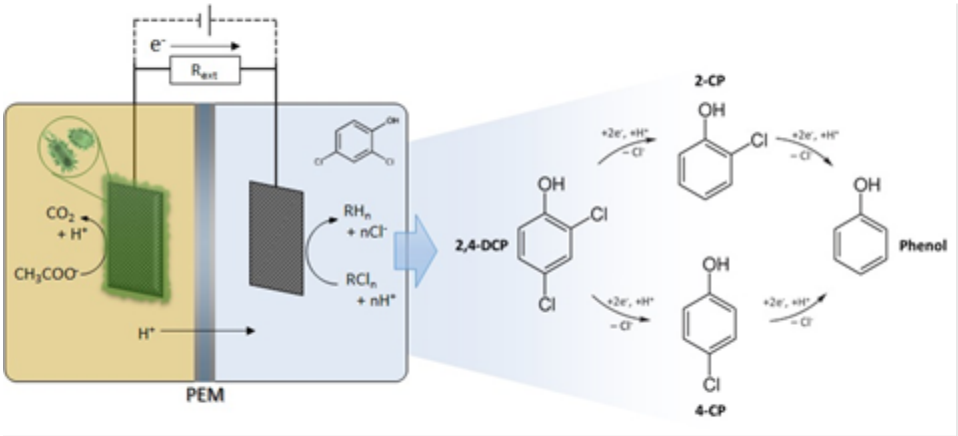

Figure 1. Scheme of the bio-assisted electro-reductive dechlorination process.

## P-7

# TARGETING APOPTOSIS IN BREAST CANCER TO AVOID RESISTANCE TO CHEMOTHERAPY

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Despite advances in cancer research, breast cancer remains one of the leading causes of death among women. In particular, triple negative breast cancer (TNBC) is the most aggressive subtype with the worst prognosis, as it lacks specific treatments. In many cases, it is resistant to the chemotherapy drugs used for its treatment. For this reason, many research groups are focused on finding alternative therapies for this type of tumor. In this line, the main objective of this work has been to evaluate drugs that can reverse acquired resistance to chemotherapy drugs. To this end, we have generated a cisplatin-resistant model from a TNBC cell line, MDA-MB-231, obtaining the MDA-MB-231R line. This line showed an alteration in the expression of proteins of the BCL-2 family proteins, involved in apoptosis processes, so we decided to evaluate the action of the inhibitor, Obatoclax, on this resistant model. Our results show that Obatoclax can inhibit cell proliferation and the capacity of invasion and migration, while increasing apoptosis in the resistant model, showing no statistically significant differences with the parental lines. BET protein inhibitors (BETi) have shown an effect on the population of cancer stem cells in TNBC, which are largely responsible for these tumors. Thus, after evaluating the effect of Obatoclax individually, its combination with BETi, JQ1, was assessed. The combination of both drugs showed a potent synergistic effect on the TNBC cell line with acquired resistance to chemotherapy, so it could be an alternative to reverse the intrinsic resistance. For this reason, we also decided to evaluate the action of a new generation compound that acts on BET proteins, PROTAC ARV-825. This compound is capable of inhibiting cell proliferation at low doses and shows an effect on proteins of the BCL-2 family. In addition, it was shown to have a greater effect on resistant TNBC cells. In summary, the use of a combined BCL-2 and BET protein inhibition strategy could be a good therapeutic alternative to treat tumors with acquired resistance in TNBC.

## P-8

# ORGANIC LASER: AMPLIFIED SPONTANEOUS EMISSION (ASE) OF BENZOTHIADIAZOLE-BASED DERIVATIVES

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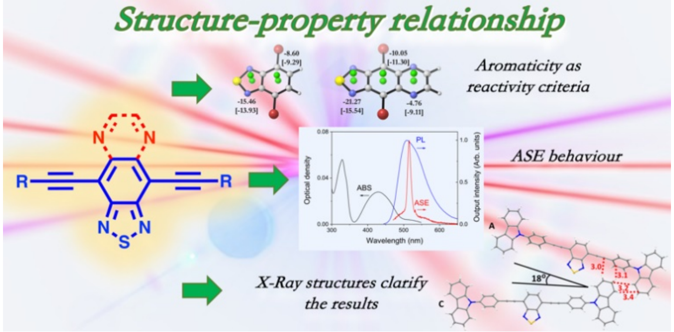
*Organic Chemistry*

In recent years, interest in searching for new organic materials with optimal properties for use as an active medium in thin film organic lasers (TFOLs) has promoted extensive research.[1] TFOLs have great potential for using in a wide range of applications such as optical communications, spectroscopy, biosensing and chemical sensing.[2]

The potential of a material for laser applications can be easily evaluated characterising its amplified spontaneous emission (ASE) properties when deposited as a thin film in a waveguide configuration.

Our group have designed, synthesized and evaluated the ASE properties of a serie of benzothiadiazole derivatives.[3] The synthetic strategy entailed the use of Sonogashira and Stille reactions based on the different aromatic nature of the central core. We observed that BTD derivatives showed narrowing of the photoluminescence spectra, thus revealing their potential laser applications. X-ray diffraction analysis was carried out to understand the observed results and to establish structure–property relationships.



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## P-9

# BRS VIOLETA GRAPE: ANTHOCYANINS PROFILE AFTER THE PROCESSES OF MAKING A GUMMY

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The development of products based on Brazilian grapes is important to satisfy the macro trends of the market and develop the valorization of national fruits. The BRS Violeta grape is a teinturier cultivar and shows itself as an interesting source for the development of a natural coloring in foods. In addition, several studies have been looking for the development of gummies fortified or enriched with natural alternatives. Therefore, this work aimed to use the grape juice of BRS Violeta grape for the preparation of a gummy containing gelatin and honey. For this, the grape juice was obtained by the steam juice extraction method, in an "steam juicer"; then the gummy was made with the grape juice (62%), gelatin (12%), honey (25%), agar (0.7%) and natural grape flavor (0.3%). So, the anthocyanins were extracted from grape, juice and gummy. Finally, the anthocyanins profile of grape, juice and gummy were determined using high-performance liquid chromatography with diode array detector coupled to mass spectrometry with electrospray ionization chamber and ion trap analyzer (HPLC-DAD-ESI-MS$^n$). The anthocyanin profiles were composed of monoglycosilated (3-glc) and diglycosilated (3,5-glc) anthocyanins. A total of 26 anthocyanins were detected in grape and juice and 18 in the gummy. All samples showed the presence of non-acylated and coumarylated anthocyanins in both 3-glc and 3,5-glc forms. But the acetylated 3-glc and 3,5-glc anthocyanins were reported just for the grape and juice. In the gummy, these anthocyanins were degraded. The grape presented a total of 80.9% of 3,5-glc anthocyanins, while the juice and the gummy showed 84.9 and 93.15%, respectively. It´s demonstrates that after the processing of obtaining juice and gummy there was an increase in 3,5-glc anthocyanins concentration as well as a reduction of 3-glc anthocyanins concentration. Therefore, during obtaining the gum candy, there was mainly the degradation of 3-glc and acetylated anthocyanins, thus increasing the proportion of 3,5-glc anthocyanins.

## P-10

# ELECTRO-REFORMING PROCESSES: AN OPPORTUNITY TO BOOST BIOETHANOL

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*Chemical engineering*

In the last decade, bioethanol has postulated as one of the most promising substitutes to traditional fossil fuels in terms of energy consumption. However, the commercial price and global demand of this compound crossed a recession period from 2010 mainly in Europe, situation which remains nowadays. This is due to different causes, such as competition between international markets (US exports), oversizing of production plants and government regulations (blending gasoline normative). Therefore, the overall production of this compound far exceeds the current market needs, being available as a platform molecule for the production of value-added chemicals. In this context, the electro-reforming systems constitute one of the most promising alternatives within the ethanol valorization processes. Through this technology, ethanol molecule is electro-oxidized at the anodic side of a proton exchange membrane (PEM) cell, leading to the formation of electrons, protons and organic compounds, while reduction takes place in the cathodic compartment, generating high purity hydrogen (important energy vector). Available literature have generally focused on the hydrogen production, being works which deal with the organic compounds co-production scarce [1]. For that, reason this research work is focused on maximizing and optimizing the organic production of industrial interest (acetaldehyde, acetic acid and ethyl acetate among others) apart from hydrogen, in order to increase the overall profitability of the process. Thus, the ethanol electro-reforming process was carried out in a PEM cell of 5 $cm^2$ (active area) at mild operational conditions (1 atm and 80 ºC) over PtRu/C anodic catalysts. The initial ethanol concentration and the Pt:Ru ratio were optimized (4 M and 2:1 mass ratio) to achieve the highest electro-catalytic activity. To that aim, linear sweep voltammetry essays (0-1.4 V) and chronopotentiometry tests (0.2-0.8 A) were conducted, analysing the product distribution (hydrogen and organic compounds).

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## P-11

# HYDROGELS BASED ON CHITOSAN: BIOMEDICAL APPLICATIONS AND MAGNETIC PROPERTIES

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*Organic Chemistry*

Chitosan (CS) is a polysaccharide obtained from alkaline hydrolysis of chitin, one of the most abundant natural amino polysaccharide extracted from the exoskeleton of crustaceans and insects, from fungal cell walls, etc. Among the several types of biomaterials studied and developed in the past few years for biomedical applications, the chitosan-based hydrogels have attracted much attention because of its advantages like low cost, biocompatibility and versatility.

Despite the aforementioned advantages, CS-based hydrogels have weak mechanical properties being not suitable for biomedical applications [1]. This drawback can be avoided by combining chitosan with other monomers (such as acrylamide) [2] or nanomaterials (graphene [3], magnetic nanoparticles [4]…) in order to tune the hydrogels properties and to develop smart materials with stimuli-responsive properties.

In this work, we highlight chitosan-based hydrogels as suitable scaffolds for biomedical applications. To enhance their mechanical strength, acrylamide (AM) has been added. By incorporating graphene (G) into the hydrogels, we are able to modify their biological properties and their structure. Furthermore, we achieve a magnetic-responsive hydrogel generating magnetic nanoparticles in the matrix by the *in-situ* precipitation method.

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## P-12

# Controlling Cell Plasticity to Overcome Cancer Resistance

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*Bioquímica. Oncología Traslacional*

**Objective:** Triple negative breast cancer (TNBC), is the most heterogeneous breast cancer subtype and the one with the worst prognosis. TNBC lacks specific therapeutic targets, what difficult their clinical management and restrict their treatment to radio- and chemotherapy. More importantly, TNBC usually exhibits resistance to chemotherapy, mainly due to the elevated proportion of cancer stem cells (CSCs). BET proteins are epigenetic players known to control TNBC stemness and then, BET inhibitors (BETi), such as JQ1, might be promising strategies to target CSCs and revert chemoresistance in these tumors.

**Materials and methods:** TNBC cells models with acquired resistance to chemotherapeutic agents were generated by pulsed exposure to increasing doses of the drugs (IC30 to IC70 for 6 months). qPCR studies were performed to assess the expression of a panel of stemness markers in these models in comparison to parental cell lines and to evaluate the impact of JQ1. Proliferation and tumor progression assays upon treatment were completed on the resistant models. Flow cytometry studies were also performed to evaluate cell cycle and apoptosis of resistant cells upon treatment with JQ1.

**Results:** TNBC cells with acquired resistance to chemotherapy are enriched in stemness markers. Despite their aberrant overexpression, JQ1 efficiently decreased the expression of these markers in the resistant models. Moreover, these drugs slowed down cell proliferation and hampered colony formation and invasion capabilities in chemo-resistant cells. Evaluation of cell cycle confirmed that these BET-targeting compounds led to a marked arrest in G0/G1 unravelling apoptosis in these models.

**Conclusions:** BETi efficiently target CSC markers in TNBC, what might constitute a good strategy to revert resistance in TNBC.

## P-13

# D-A NAPHTHALENDIIMIDE DERIVATIVES WITH POTENTIAL APPLICATION AS ORGANIC FIELD-EFFECT WAVEGUIDES (OFEW)

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*Organic Chemistry*

In our research group we have an extensive experience in heterocycles which have shown interesting properties with application as new materials in photonic devices (optical waveguides) [1] (Figure 1a) and electronic devices (organic field effect transistors, OFETs) (Figure 1b) [2]. Currently, electronics is reaching its limit and the new emergent technology in the last years is the photonics. In that sense, on a nanometric scale, a new and very recent formula arises to combine the advantages of photonics and electronics. These new devices are called organic field effect waveguides (OFEW) (Figure 1c) [3].

For this reason, it is necessary to find organic compounds that can show the necessary requirements for both applications. In this sense, naphthalenediimide derivatives are very interesting due to their planar structure, their acceptor character, their self-assembling capacity and the possibility of combining with donor groups of different nature through simple chemical modifications, to obtain D-A systems that may have interesting optoelectronic properties.

In this work, three different Donor-Acceptor (D-A) compounds based on a naphthalendiimide core branched with different alkynyl donor groups (triphenylamine, phenylcarbazole and benzothiophene) were synthesized, totally characterized and tested in OFETs and optical waveguides, with the aim in mind of designing an OFEW.

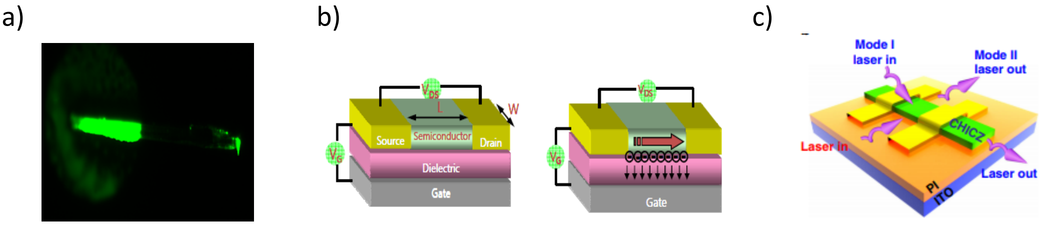

**Figure 1.** Scheme of: a) Optical waveguide, b) Organic Field-Effect Transistor (OFET), c) Organic Field-Effect Waveguide (OFEW)

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# CÓMO FUE LA EDICIÓN DEL AÑO PASADO...

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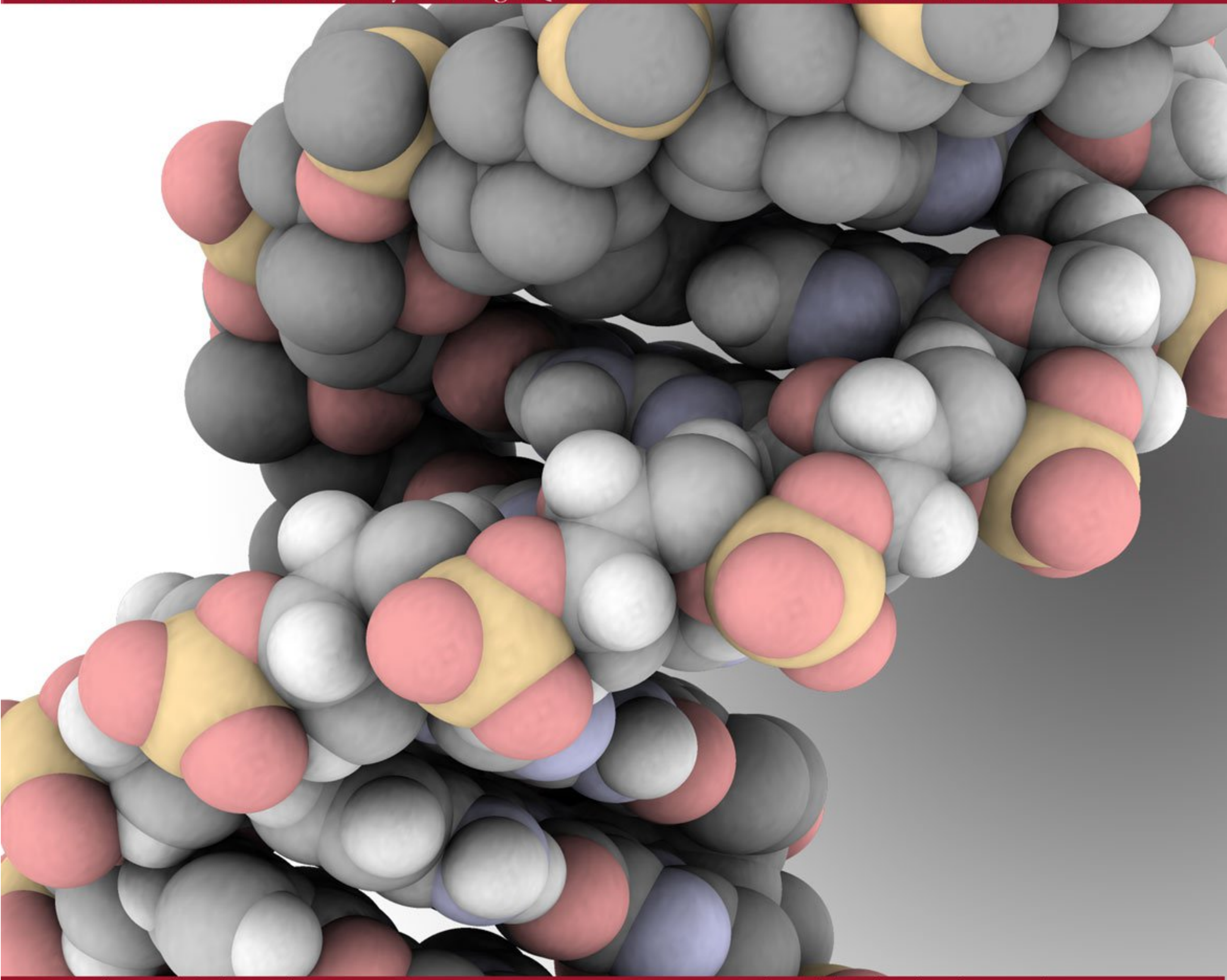

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# ÍNDICE

# PRESENTACIÓN

El número de Junio es un monográfico dedicado al XIII Simposio de Ciencia Joven, donde se recogen todas las comunicaciones orales y posters que han participado en el evento, además de las conferencias invitadas. Este número también recoge información relativa a los premios otorgados en el simposio.

El comité editorial.

# LOS QUÍMICOS NOVELES DE LA UCLM COMPARTEN CON SUS COMPAÑEROS SUS LÍNEAS DE TRABAJO DE INVESTIGACIÓN

*El rector de la Universidad de Castilla-La Mancha inaugura el XIII Simposio Ciencia Joven que se celebra en Ciudad Real*

La Facultad de Ciencias y Tecnologías Químicas de la Universidad de Castilla-La Mancha (UCLM) en Ciudad Real acoge el Simposio Ciencia Joven desde el 5 al 7 Junio, durante el que los investigadores noveles presentan sus líneas de trabajo a sus colegas del Campus, así como a otros llegados de fuera. El encuentro, que cumple su decimotercera edición, ha sido inaugurado por el rector de la UCLM, Miguel Ángel Collado.

Los jóvenes investigadores de la Facultad de Ciencias y Tecnologías Químicas de la universidad de Castilla-La Mancha (UCLM) en el Campus de Ciudad Real, así como otros llegados de Albacete y Toledo, presentan a la comunidad académica sus líneas y resultados de investigación con motivo de la celebración del decimotercer Simposio Ciencia Joven, una iniciativa organizada por ellos mismos que a su vez les supone una oportunidad para acercar a los estudiantes a la tarea investigadora.

El encuentro ha sido inaugurado esta mañana por el rector de la Universidad de Castilla-La Mancha (UCLM), Miguel Ángel Collado, quien ha felicitado a la Facultad de Químicas y a sus organizadores por esta iniciativa, muestra de la “vitalidad” del centro, y por su “entusiasmo, trabajo y compromiso con la investigación pese a las dificultades”.

Para el rector, este encuentro refleja que en la Universidad de Castilla-La Mancha hay gente joven “con talento, capacidad e inteligencia” que aseguran el futuro de la investigación. Frente a ello está el problema de los recursos económicos y materiales, de ahí que el rector haya pedido a los nuevos gobiernos surgidos de las urnas que “asuman con mayor entusiasmo la investigación” y apuesten por “la financiación, la regularidad y consolidación de convocatorias y la divulgación de la ciencia” a fin de que la sociedad valore la tarea investigadora. “La ciencia contribuye a seguir creciendo y a vivir mejor”, ha dicho.

# LOS QUÍMICOS NOVELES DE LA UCLM COMPARTEN CON SUS COMPAÑEROS SUS LÍNEAS DE TRABAJO DE INVESTIGACIÓN

Junto al rector de la UCLM, el decano de la Facultad de Ciencias y Tecnologías Químicas, Ángel Ríos, se ha referido a la celebración del simposio de Ciencia Joven como una excelente oportunidad para los investigadores noveles, ya que el mismo les ayudará a conocer cómo se prepara un congreso científico, les servirá de apoyo en su tarea formativa investigadora y les permitirá conocer el trabajo que realizan otros compañeros.

En el simposio, en el que colabora la Real Sociedad Española de Química, se han inscrito un total de 120 investigadores del Campus de Albacete, Ciudad Real y Toledo y se han admitido 35 comunicaciones orales y 28 contribuciones en formato póster en los distintos ámbitos que abarca la Facultad: Química Inorgánica, Ingeniería Química, Química Orgánica, Química Analítica, Tecnología de los Alimentos, Química Física, Matemáticas y Bioquímica. Entre otras líneas de investigación, durante el simposio, según ha explicado en representación de la organización la investigadora Virginia Moreno, se expondrán cuestiones como los nanomateriales en alimentos y medioambiente, fluidos supercríticos sostenibles con el medioambiente, el grafeno o las propiedades del vino, entre otras.

Junto a los ponentes investigadores noveles, el simposio cuenta con la intervención de cinco ponentes externos invitados del Instituto IMDEA Nanoscience, y de las universidades de Valencia y Murcia.

**Gabinete Comunicación UCLM. Ciudad Real, 5 de junio de 2019**

# INTERFACING MOLECULES AND NANOMATERIALS: FROM CARBON NANOTUBE ROTAXANES TO SINGLE-MOLECULE EXPERIMENTS

## EMILIO M. PÉREZ

We provided a global perspective of the work we have carried out in the last five years in the search for novel strategies to interface molecules (0D), single-walled carbon nanotubes (SWNTs, 1D) and bidimensional materials (2D).

In particular, we presented the synthesis of mechanically interlocked derivatives of SWNTs (MINTs), which are rotaxane-type materials, the first example of, a 0D-1D mixed dimensional heterostructure interfaced through the mechanical bond (see Figure).[1] We also showed that the effect of the mechanical bond on the properties of SWNTs is unique, and clearly different from the noninterlocked, supramolecular compounds.[2] We also showed that encapsulation of organic molecules into SWNTs results in modulation of their electronic properties.[3]

In the flatland area, we reported a method to functionalize graphene covalently with exquisite (>97%) atomic selectivity and yield (92%).[4] We described improved methods for the production of 2D materials through liquid-phase exfoliation (LPE),[5] and report simple strategies to construct functioning electronic devices from the resulting nanoflakes.[6] Finally, we described a “click” reaction protocol for the covalent modification of TMDCs.[7]

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# RETOS DEL CONTROL DE NANO MATERIALES EN ALIMENTOS

## YOLANDA PICÓ

El control de los nanomateriales en los alimentos sigue siendo un reto difícil de abordar pero que debe instaurarse para garantizar la seguridad de los consumidores y fijar algunos aspectos relacionados (por ejemplo, el etiquetado o el control). El mayor reto para la química analítica es que son un nuevo tipo de analitos, cuya caracterización involucran tanto información química (composición, masa y concentración numérica) como física (tamaño, forma y agregación). Además existe una gran variedad de nanomateriales (basados en óxidos inorgánicos, en carbono, moléculas orgánicas) que complica todavía más la determinación de estas sustancias. Por todo ello, en esta charla se analiza la situación actual, evaluando y comparando los métodos analíticos implicados y estudios desarrollados hasta ahora para la identificación y cuantificación de nanomateriales en los alimentos. El papel de cada una de las técnicas empleadas (microscopia electrónica, Raman, fraccionamiento en campo de flujo, plasma de acoplamiento inductivo (partícula única, etc.) se analizará en profundidad. Se valoraran el rango de aplicaciones y las ventajas y limitaciones de cada técnica, así como las perspectivas de futuro haciendo hincapié los principales desafíos específicos que quedan dentro de este tema. Por último, también contemplamos perspectivas futuras que plantea el control de nanomateriales en alimentos.

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# GREEN CHEMISTRY OR 'DREAM' CHEMISTRY: CLEAN BIOCATALYTIC PROCESSES OF INDUSTRIAL INTEREST

## PEDRO LOZANO

The development of sustainable chemical processes turns around two main axes: the selectivity of catalytic transformations, and the easy and clean separation of pure products. The use of enzymes in ionic liquids (ILs) was reported great advantages (*i.e.* improved activity and enantioselectivity, enhanced stability, etc.),[1] and its combination with supercritical technologies was resulted in synergetic and outstanding platforms for developing (multi)catalytic green chemical processes under flow conditions able to directly provide pure products.[2] This has been demonstrated by several examples of flow synthetic processes in IL/$scCO_2$ biphasic systems at 100 bar and 40-100ºC (*e.g.* DKR of *rac*-1-phenylethanol,[3] biodiesel,[4] monoglycerides,[5] etc. Straightforward protocols for carrying out biotransformations, product recovering and biocatalysts/ILs reusing without any loss in the overall greenness character of the process, have been developed in recent years.

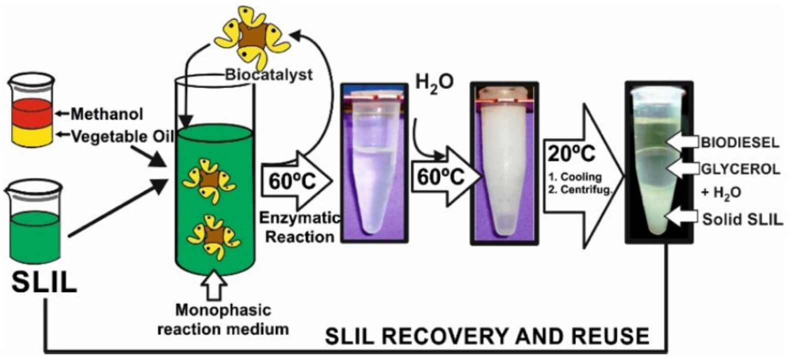

**Figure 1.** Schema of the protocol for the biocatalytic synthesis of biodiesel in SLIL, and product separation with full recovery and reuse of the SLILs

# GREEN CHEMISTRY OR 'DREAM' CHEMISTRY: CLEAN BIOCATALYTIC PROCESSES OF INDUSTRIAL INTEREST

## PEDRO LOZANO

Sponge-Like Ionic liquids (SLILs) are hydrophobic ILs based on cations with long alkyl side-chains, *e.g.* octadecyltrimethylammoniumbis(trifluoromethylsulfonyl)imide, etc.), which behaves as sponge-like systems by switching from liquid to solid phase with temperature.[6] Based on this new property, the SLILs have been applied for developing straightforward and clean approaches for biocatalytic synthesis and extraction of nearly pure compounds of added value (*e.g.* terpene esters,[7] biodiesel,[8] etc.). As can be seen in Figure 1, the SLILs are able for dissolving (soaking) hydrophobic compounds at temperatures compatible with enzyme catalysis (*e.g.* vegetable oil and methanol mixtures are monophasic in $[C_{18}mim][NTf_2]$ at 50°C[7]).

By cooling reaction mixtures based on these SLILs under room temperature, they are solid phases that can be fractionated by centrifugation, allowing the release (wringing out) of reaction products from the SLIL net, and resulting in two phases: a solid SLIL phase and a liquid phase containing synthetic products (flavour esters, biodiesel, etc.). This approach for product separation can be improved by using centrifugal filters, demonstrating the excellent suitability of this straightforward and green approach for carrying out synthesis and separation of pure products with full recovery and reuse of the reaction system (SLIL and biocatalyst).[6]

**Acknowledgements.** Work supported by the MINECO CTQ-2015-67927-R, and Fundación SENECACARM 20790/PI/18 grants.

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## INVESTIGACIÓN EN ENERGÍA: PROGRESO VS CIRCULARIDAD

## DAVID SERRANO GRANADOS

A pesar de los avances que se han producido en los últimos años en el desarrollo e implementación a escala comercial de las energías renovables, todavía en la actualidad más del 80 % de la energía primaria consumida a nivel mundial procede de combustibles fósiles (carbón, petróleo y gas natural). Como consecuencia de las emisiones de gases de efecto invernadero así generadas, la concentración de $CO_2$ en la atmósfera no deja de crecer año tras año, habiendo superado ya la barrera de las 400 ppm, provocando una aceleración de los efectos del cambio climático.

IMDEA Energía es un instituto de investigación creado por la Comunidad de Madrid con la misión de contribuir a un sistema energético sostenible y de mínimo impacto ambiental, basado en tecnologías energéticas limpias y renovables. Entre las temáticas de investigación que desarrolla y estudia, cabe destacar las siguientes: producción de combustibles sostenibles, energía solar de concentración, sistemas y dispositivos de almacenamiento de energía, redes eléctricas inteligentes, eficiencia energética, valorización de las emisiones de $CO_2$ y análisis integral de los sistemas energéticos.

El progreso de la investigación en el ámbito de la energía durante las últimas décadas no siempre ha progresado de forma lineal, al estar fuertemente condicionado por factores externos de tipo político y económico. Ello ha llevado a una cierta recurrencia y circularidad en las temáticas de moda que se abordan de forma prioritarias. En este sentido, conceptos, como los de biocombustibles de tercera generación, los vehículos eléctricos, las pilas de combustible, el hidrógeno como vector energético o la valorización de residuos, que en el momento presente son de gran actualidad iniciaron en realidad su desarrollo hace ya bastante tiempo.

# EVALUATION OF THE ATMOSPHERIC IMPACT OF THE USE OF FUELS AND BIOFUELS

## MARÍA INMACULADA ARANDA

The combustion of fossil fuels and biofuels generates Volatile Organic Compounds (VOCs), nitrogen oxides ($NO_x$), CO, $CO_2$ and particulate matter (PM). These pollutants are causing changes in the climate that affect the environment globally and have an impact in the human health. In addition, fossil fuels are a non-renewable energy source. For those reasons new fuels are being developed from biomass waste (biofuels), which could replace the fossils fuels being used in the same motors, without any modification. In general, the use of biofuels represents lower emissions of compounds such as $CO_2$ and PM[1], however, in most studies has been observed that biodiesel blends increase $NO_x$ emissions or are similar to diesel fuel[2]. Moreover, some studies have shown that the addition of oxygenated compounds in fuels reduces the formation process of soot[3]. Thus, the use of oxygenated compounds (alcohols, ethers, hydroxyethers…) as additives in fuels is being investigated. In this sense, our research group evaluates the atmospheric impact of the use of these additives in a massive way in new fuels, from the point of view of the chemistry and atmospheric reactivity. Additionally, our group evaluates the atmospheric impact of the gas phase emissions generated in the combustion of these fuels and biofuels, through the kinetic study, evaluation of the Global Warming Potential (GWP), ozone generation capacity and determination of the reaction products. Finally, respect to the PM (soot) emitted, our group carried out studies of physico-chemical characterization and reactivity of these particles with different gases.

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# ELECTROCHEMICAL DETECTION OF RHODAMINE B BY MOLECULARLY IMPRINTED POLYMER MODIFIED SCREEN PRINTED CARBON ELECTRODE AND EXTRACTION BY MAGNETIC MULTIWALLED CARBON NANOTUBE POLY(STYRENE-CO-DIVEINYLBENEZNE)

## YASSINE BENMASSAOUD

Due to its potential health risks on human bodies, the use of Rhodamine B, as well as several other dyes, is banned from food industry. thus, several analytical methods were developed for its determination. furthermore, molecularly imprinted polymers are synthetic receptors for a targeted molecule allowing a better discrimination in a complex matrix.

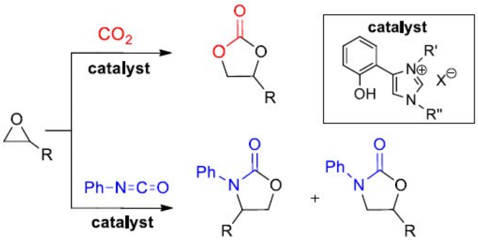
In this study, a molecular imprinted polymer was prepared and used as a screen-printed carbon electrode modifying film for the rapid electrochemical detection of Rhodamine B. A magnetic solid phase extraction was also carried out using magnetic multiwalled carbon nanotubes poly(styrene-co-divinylbenzene) composite (MMWCNTs-PS-DVB) as sorbent. The MIPs synthesis was optimized by varying the activated $TiO_2$ and MWCNTs amounts. SPCE electrode was then modified using an optimum volume of $TiO_2$-MWCNTs-MIP and compared to a non-modified SPCE, a $TiO_2$-SPCE and a MWCNTs-SPCE by cyclic voltammetry. The magnetic solid phase extraction and detection conditions were optimized as well. the developed method provided a quantification linear range from 5-100 µg $L^{-1}$. The limit of detection, limit of quantification and relative standard deviation were respectively 1.44 µg $L^{-1}$, 4.81 µg $L^{-1}$ and 6.59 %. Finally, the present method was applied in real food samples analysis.

# SYNTHESIS OF HETEROCYCLE COMPOUNDS CATALYZED BY IMIDAZOLIUM SALTS

## MARÍA DEL PRADO CABALLERO

Metal-free organocatalysts based on imidazolium salts showed high efficiency catalyzing the cycloaddition reactions to prepare heterocycle compounds such as cyclic carbonates and oxazolidinones (Scheme 1).[1,2] Imidazolium salts performed as bifuntional catalysts and exhibited improved activity for the fixation of $CO_2$. Mechanistic studies revealed that the hydroxyl group promoted the activation of the epoxide ring by nucleophilic attack of the iodide.

Oxazolidinones are an important class of heterocycle compounds which are commonly used as precursors to antibacterial medicines. Using the optimal reaction conditions (2 mol% of 5-hidroxyphenylimidazol at 80 °C in toluene for 24 hours) aromatic and aliphatic epoxides were reacted with phenylisocyanate to prepare the corresponding oxazolidinones. The activity[3] of catalysts has an important role in the synthesis since the cyclotrimerization of isocyanates is a competing reaction.



**Scheme 1.** Synthesis of cyclic carbonates and oxazolidinones.

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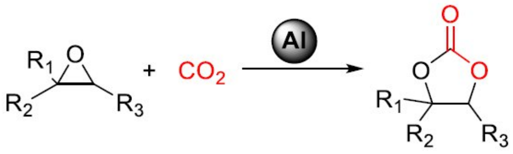
# SYNTHESIS OF BIO-DERIVED CYCLIC CARBONATES FROM RENEWABLE RESOURCES

FELIPE DE LA CRUZ

The development of bio-based high-value added chemical products to replace petrolbased molecules is one of the main objectives for the scientific community and industry. In this context, a range of new platform molecules derived from waste biomass have been developed and used as monomers for the production of polymeric materials, as solvents and as chemical intermediates.[1] One of the most abundant form of carbon on Earth is carbon dioxide ($CO_2$) and it is a potential sustainable feedstock for the chemicals industry.[2] The most important transformation of carbon dioxide is its catalytic coupling with epoxides to prepare cyclic carbonates.[3] This reaction has been widely studied over the last decade with the development of a wide range of highly efficient metal- and organic catalysts for this reaction.[3]

In this contribution we report the aluminum catalyzed coupling reaction of carbon dioxide with bio-derived epoxides under mild reaction conditions to afford highly-substituted natural-derived cyclic carbonates with excellent diastereoselectivity, obtaining in some cases one diastereoisomer as the major product.



Highly diastereoselective synthesis;
Excellent yields; Earth-abundant catalyst

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# SCREENING AND PRELIMINARY BIOCHEMICAL AND BIOLOGICAL STUDIES OF [RuCl(p-CYMENE)(N,N-BIS(DIPHENYLPHOSPHINO)-ISOPROPYLAMINE)][BF4] IN BREAST CANCER MODELS

## ELENA DOMÍNGUEZ

Breast cancer is the second leading cause of cancer death worldwide. Despite progress in drug discovery, identification of the correct population is the limiting factor to develop new compounds in the clinical setting. Therefore, the aim of this study is to evaluate the effects of a new metallodrug, [RuCl(p-cymene)(N,N-bis(diphenylphosphino)-isopropylamine)][BF4] (pnpRu- 14) as a lead pnp-Ru compound by screening and preliminary biochemical and biological studies in different breast cancer subtypes. The results show that complex pnpRu-14 is much more effective in promoting in vitro cytotoxic effect on HER2+ and RH+/HER2- breast cancer than the reference metallodrugs cisplatin, carboplatin or RAPTA-C. It is important to highlight that pnpRu-14 shows an impressive cytotoxicity against BT474 cells. Caspase-dependent apoptosis is the mechanism of action for these compounds. In addition, treatment of SKBR3, BT474, T47D and MCF7 cancer cells with pnpRu-14 caused an accumulation of cells in the G0/G1 phase cells. The Human serum albumin (HSA), DNA and H1 histones binding properties of the lead compound are reported. Pharmacokinetic and biodistribution studies show a quick absorption of pnpRu-14 in serum with no significant accumulation in any of the tested organs. This work provides evidence to support the preclinical and clinical development of pnpRu-14 in breast cancer.

# 2*H*-BENZO[*d*][1,2,3]TRIAZOLE DERIVATIVES WITH APPLICATION IN ORGANIC ELECTRONICS

## BEATRIZ DONOSO

In the last decades, organic electronics has attracted great attention due to the development of organic semiconductor materials. These materials have practical applications in the latest generation devices organic field effect transistors (OFETs)[1], organic light emitting diodes (OLEDs), organic photovoltaic devices (OPVs), as well as optical waveguides[2] which are essential elements for their implementation in photonic circuits. In our research group, we have studied the preparation and self-assembly properties of different 2*H*-benzo[*d*][1,2,3]triazole derivatives with the aim to determine their behavior as organic waveguides and semiconductors in OFETs (**Figure 1**). As a green approach, we have used computational calculations to avoid unnecessary synthesis. We have calculated the energy and topology of frontier molecular orbitals, and the photophysical properties of a set of derivatives. The selected compounds have been synthesized using cross-coupling reactions. Currently, we are studying the behavior of these compounds as semiconductors in OFETs and optical waveguides.

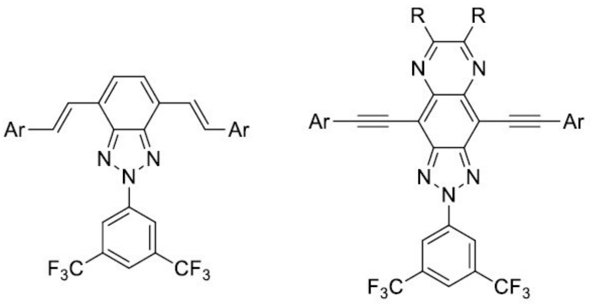

**Figure 1.** 2*H*-benzo[*d*][1,2,3]triazole derivatives.

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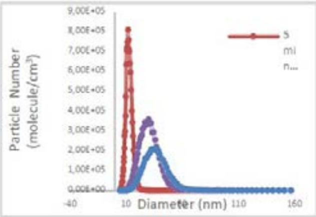
# FORMATION OF SECONDARY ORGANIC AEROSOL (SOA)

## ALBA ESCALONA

Atmospheric aerosols are considered as one on the main uncertainty sources in the current understanding of the Earth's climate[1]. Previous studies showed that some compound aromatic emitted to the atmosphere give a high SOA (Secondary Organic Aerosol) formation potencial[2,3]. Reaction of the alkenes with ozone proceeds through the formation of a Criegee intermediate (CI). Very recently, it has been found that stabilized CI (sCI) can undergo reactions with $SO_2$ several orders of magnitude faster than assumed so far[4] producing $SO_3$, which contributes efficiently to the formation of ground level sulfuric acid[5,6].

We study the ozonolysis reaction of different volatile organic compound following the conditions that lead to the formation and growth of new particles. The effect of water vapor and $SO_2$ concentrations during the process are also studied and discussed. The reactions have been carried out in a Teflon chamber filled with synthetic air mixtures at atmospheric pressure and room temperature and following the particle formation by SMPS (Scanning Mobility Particle Sizer) and CPC (Condensation Particle Counter).



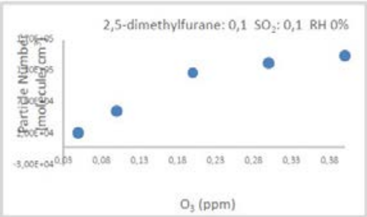


**Figure 1.** Distribution of particle size at different reaction times. Figure 2. Effect of [$O_3$] on the production of SOA

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# YEASTS WITH PROBIOTIC POTENTIAL

## PILAR FERNÁNDEZ-PACHECO

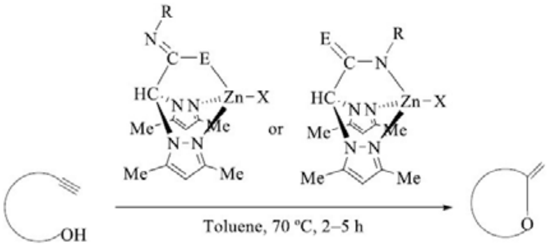
The intestinal microbiota is the most populated community of the human body in which trillions of microorganisms live in symbiosis, contributing to the normal functioning of the organism through its interactions with the host. However, a dysbiosis can cause multiple alterations. One of the most widely used current approaches in the modulation of intestinal microbiota balance is based on the administration of probiotics. Currently, a wide range of probiotic products are marketed, almost all of them are bacteria, but there is also a probiotic yeast (*Saccharomyces boulardii*). For this reason and due to the increased of researching in this field, the aim of this work was to study the behavior of potential probiotic yeasts already selected by previous works under in vitro and *in vivo* conditions. The study was carried out with 5 strains belonging to *Saccharomyces cerevisiae* (2), *Hanseniaspora osmophila* (2) and *kluyveromyces thermotolerans* (1), isolated from food ecosystems and selected due to their good probiotic characteristics *in vitro*. All of them underwent gastrointestinal conditions in studies in vitro through the use of Simgi® equipment. It allows the dynamic emulation of the different physiological variables (peristaltic movements, pH, gastrointestinal fluids, etc.) getting effects very similar to those occurred in humans. The results showed that two strains (*S. cerevisiae* and *H. osmophila*) were the most resistant to the process showing a minimal loss of viability. Both were used for experiments *in vivo* with mice and contributed to the recolonisation of gut microbiota process after the use of antibiotics. On the other hand, they showed a transitory adhesion capacity to the mucosa since their concentration decreased drastically after their administration was stopped, besides, they did not show ability to invade tissues by hyphae formation, as show the pathogenic yeasts.

These strains could be used to produce probiotic products (foods and drugs).

# REACTIVITY OF ZINC COMPLEXES TOWARDS ALKYNES: CATALYTIC INTRANOLECULAR HYDROALKOXYLATION OF ALKYNYL ALCOHOLS

## MIGUEL ÁNGEL GAONA

Alkyne is a very interesting functional group, as it can be used as a building block to many useful compounds through the generation of new C-C, C-H or C-X bonds.[1] There transformations are usually catalyzed by transition metals, establishing efficient and selective procedures under a variety of conditions. One of these reactions is the intramolecular hydroalkoxylation of alkynyl alcohols for the synthesis of oxygen-containing heterocycles, with several applications in the pharmaceutical and the fine chemicals industries.[2] However, these catalysts usually involve expensive and/or toxic metals, making them less attractive. In this scenario, zinc-based have grown in interest, due to its abundance and low toxicity.[3] Herein, we describe the synthesis of a series of heteroscorpionate alkyl and amido zinc complexes, their reactivity towards a variety of alkyne compounds and their catalytic activity towards the intramolecular hydroalkoxylation of alkynyl alcohols.



X = Et, $NSiMe_3$
E = S, R = Adam, Naph, Ph
E = O, R = Adam, Ph, (*S*)-MePhCH

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# ANOTHER APPLICATION OF YEASTS: BIODETOXIFICATION OF POLLUTANTS

## BEATRIZ GARCÍA-BÉJAR

Over the past years, biodetoxification has become a new elimination alternative of compounds such as microbial toxins, chemical pollutant and waste industrial products. Detoxification techniques used in the present are catalogued by the elimination mechanisms carried out: chemical, physical or microbial. Microbial detoxification methods could be a useful and a specific action tool that provide a new way for reducing the presence of pollutants from industrial waste, as heavy metals, or dangerous biotoxins such as mycotoxins. This capability has been traditionally studied in bacteria and microalgae, being the fungi clade barely investigated.

In this study, 213 yeast strains from 20 different species, isolated from Castilla – La Mancha region, were incubated in separated minimal contaminated media supplied with 2 important pollutants for the industry, Aflatoxin $B_1$ and Zinc. Incubation process was carried out during 5 days at 30ºC. At the end of the process, aliquots were selected and analysed by HPLC and voltammetric techniques respectively. Additionally, strains with the best activity were subjected to complementary assays: biocontrol capability against mycotoxigenic moulds and heavy metal tolerance.

The results show that detoxification capability is toxin and strain dependant. Also, it has been noticed that is not directly related with cell growth, which could indicate that there are mechanisms of bioaccumulation or bioadpsorption involved. In general, yeasts were able to remove more percentage of Zinc than Aflatoxin $B_1$, higher than 50% of this heavy metal. Regarding the biocontrol activity, almost 50% of the strains presented an inhibition of the mycotoxigenic moulds' mycelium. On the other hand, most of the strains grew at 100 ppm concentration of Zinc. Promising strains will be further studied in order to know more about the mechanisms involved in biodetoxification process.

# EFFECIENT BILIRUBIN REMOVAL BY BSA FUNCTIONALIZED POLIMERIC PARTICLES

## MARÍA DEL PRADO GARRIDO

Hyperbilirubinemia is an illness caused by a hepatic failure, inducing a dramatic increase of bilirubin concentration in blood. In addition, the majority of patients with hyperbilirubinemia are also affected by kidney failure. Existing systems for the treatment of these patients are complex, expensive and not selective towards bilirubin. Hence, the development of a new material for efficient bilirubin removal is justified.

The main objective of this research was the syntesis of P(St-co-MMA) particles on whose surface PEGMA was incorporated at the identity point of a suspension polymerization reaction, making the particles more compatible with blood. After that, in order to improve the selectivity towards bilirubin, the biofunctionalization of the particles took place by the immobilization of Bovine Serum Albumin (BSA) in its surface.

First of all, polymeric particles with a proper particle diameter to avoid the coagulation of blood during its passing through the particles bed were synthesized. After that, the immobilization of BSA in the particles took place. Finally, some in vitro and in vivo tests were carried out, obtaining bilirubin removals of 83% and 72%, respectively, in a short period of time.

Considering these results, it can be stated that the new synthesized particulate material is suitable for an efficient and fast bilirubin removal completely compatible with a conventional hemodialysis system.

### ACKNOWLEDGEMENTS

Authors gratefully acknowledge the fellowship for PhD studies (FPU17/00750) from the Spanish Ministry of Science, Innovation and Universities and grant from the Regional Government of Castilla-La Mancha (SBPLY/17/180501/000269).

# FLEXIBLE, MULTIFUNCTIONAL NANORIBBON ARRAYS OF PALLADIUM NANOPARTICLES FOR TRANSPARENT CONDUCTION AND HYDROGEN DETECTION

## ELENA HERNÁNDEZ

A multifunctional material system consisting of planar arrays of nanoribbons was made with Pd nanoparticles (NPs) obliquely deposited on polyethylene terephthalate (PET) substrates. The PET substrates were first irradiated with a linearly polarized 266 nm laser to obtain a laserinduced periodic surface structure (LIPSS)[1]. As-prepared specimens were comprised of large area, high coherence nanoripple pattern with a mean spacing 200 nm and average amplitude 46 nm. Separately, in a gas phase aggregation vacuum equipment, the Pd NPs (diameter 5 nm) were deposited on the LIPSS at an incidence angle 60-65° directed as to become partially shadowed by the ripple ridges. This resulted in the formation of an array of NP ribbons (thickness ~ 20 nm) on the ripple ridges, the mean ribbon width depending on the deposition incidence angle. After the NPs deposition the surface pattern preserved the spacing and the analysis of AFM and SEM images confirmed the shadow effect and the Pd NPs deposition. These planar arrays were studied as candidate for both flexible, transparent conductors and hydrogen sensors.

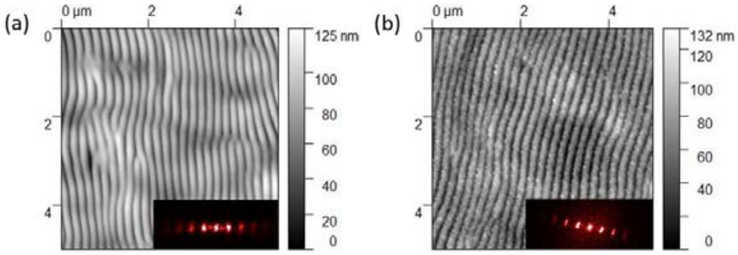

Figure 1: AFM height images (5 x 5 µm$^2$ size) of (a) PET substrate with LIPSS and (b) Pd nanoparticles on PET substrate with LIPSS. The inset images correspond to the Fast Fourier Transform of each height image.

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# HYDROGELS WITH IRON AND COBALT MAGNETIC NANOPARTICLES

## JESÚS HERRERA

Hydrogels are physically or chemically crosslinked hydrophilic 3D polymer networks, which absorb and retain large amounts of water, though not soluble in it, with maintenance of shape[1]. Our research group has a large experience synthesizing hydrogel (Figure 1). It is well known that hydrogels can store drugs inside and, depending on the structure and composition, they can deliver the drug in response of a certain stimulus[2]. In this communication, we show the synthesis of hydrogels functionalized with Co and Fe Nanoparticles and also with Few Layer Graphene (FLG) and Graphene Oxide (GO). These hydrogels will be applied to the design of scaffolds for 3D cell cultures. The presence of nanoparticles makes them sensitive to a magnetic stimulus, in order to deliver, for example, grown factors. Furthermore, graphene is expected to contribute to the mechanical, electrical and thermal properties, and at the same time, cellular adhesion can be increased.

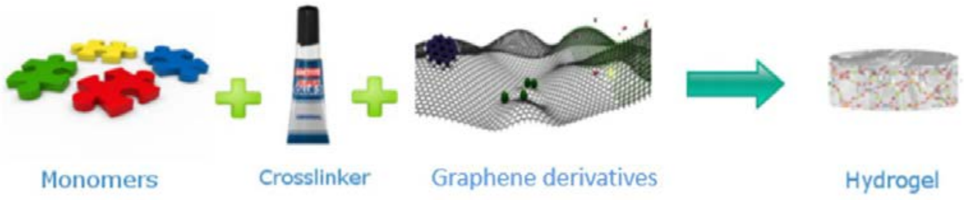

**Figure 1.** General scope of hydrogels synthesis.

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# APPLICATION OF THE GC-MS/MS AND LASER-BASED MASS SPECTROMETRY TECHNIQUES FOR THE DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS (PAH) AND THEIR DERIVATIVES (OPAH AND NPAH) IN REFERENCE AND REAL SOOT SAMPLES

SONIA LARA

The incomplete combustion processes contribute to a great extent generating atmospheric pollutants in gas phase and particles in suspension. The main source of particulate matter is traffic (May et al, 2014). Their emissions are constituted by soot that is mainly composed of black carbon and SOF (soluble organic fraction) where organic compounds such as Polycyclic Aromatic Compounds (PACs) are adsorbed. They are considered as toxic persistent substances (WHO, 2010). Soot has been classified as the second largest contributor to global warming after $CO_2$ (IPCC, 2013). Therefore, to know the chemical composition of soot is important in order to evaluate its impact on human health and climate change. In this study, an analytical methodology (microwave assistant extraction, MAE and GC-MS/MS) has been developed and validated for the analysis of PACs (PAH, OPAH, NPAH) from soot particles generated from fuels and biofuels combustion. For that, three soot samples have been investigated Printex-U, a standard reference material (SRM NIST1650b) and a real diesel soot sample. The results showed that the SRM and real sample present differences regarding the adsorption of the heaviest and NPAH since the recoveries of internal standards were 50-136% and 1-41%, respectively. In addition, total PACs retained in the diesel soot was markedly poorer (9 ng/mg- 27 ng/mg) than the extracted from SRM (699 ng/mg). Laser-based mass spectrometry techniques (L2MS and HRLMS) were also applied to the soot samples in order to obtain more information about the composition of SOF. The results obtained confirmed that diesel soot had a low content of SOF since the PACs were strongly retained, particularly the heavy ones. These differences in the composition of soot samples can be attributed to the conditions in which the diesel soot was produced.

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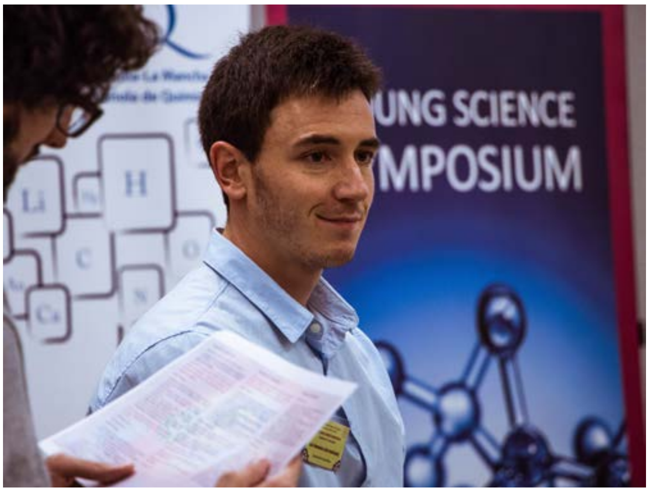
# CAPILLARY ELECTOPHORESIS METHOD FOR THE DISCRIMINATION BETWEEN BIOELECTROCHEMICAL SYSTEMS FOR THE REDUCTIVE TREATMENT OF HAZARDOUS POLLUTANTS

## LUIS FERNANDO LEÓN



Conventional Bioelectrochemical Systems (BESs) consist in biological anodes and abiotic cathodes, in which bacteria attached to the electrode is able to catalyze the anodic reaction. Commonly, BESs working as Microbial Fuel Cells (MFCs) have been used to produce electrical energy from organic compounds in wastewater, using oxygen as oxidant at the cathode. Applications in BESs have been shooting up in recent decades, as well as improving the materials used in their design.

In this study, several hazardous compounds from different wastewaters have been treated through a reductive process at the cathode of our BESs, seizing the catalytic activity of the biofilm at the anode to reduce the energy input for the electrolysis.

# FUNCTIONALIZATION OF POLYMERS IN SUPERCRITICAL CARBON DIOXIDE

## SONIA LÓPEZ

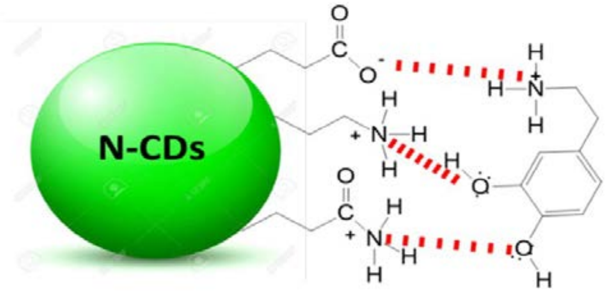
The growing demand for "intelligent" polymers has led to the continuous search for simple and efficient methods for their generation. Polymer-drug conjugates are finding increasing use as novel anticancer agents. Polymeric systems conjugated with a drug result in the controlled release of drugs as this occurs when a carrier material, specifically a polymer, is combined with an active principle so that it is released from the system in a pre-designed way. The release of drugs achieves more effective therapies as it eliminates the administration of both overdoses or doses below the minimum effective dose. In this context, click chemistry emerges as a simple and extremely powerful methodology due to its ability to easily and effectively interconnect different substructures. This has resulted in a wide range of applications in biomedical sciences, organic synthesis and materials science. Click chemistry has the advantage of being a highly reliable methodology, clean, with excellent performance and compatible with a large number of functional groups. One of the most well-known reactions is copper(I)-catalyzed alkyne azide cycloaddition (CuAAC). Terminal alkyne group reacts with an azido group to form a thermally and hydrolytically stable triazole ring, where N,N-dimethylformamide (DMF) or tetrahydrofuran (THF) are the most common solvents used to achieve the conjugation of chemical product. This work aims to find an option that avoids the use of toxic solvents such as THF or DMF, and to use a solvent that can protect medicines from degradation and is additionally environmentally sustainable, using supercritical technology. In recently, considerable attentions have been focused on using supercritical carbon dioxide ($scCO_2$) as a reaction medium for organic reactions due to their attractive physical and toxicological properties. This research focuses on the conjugation of a polymer, polyethylene glycol (PEG), with an active ingredient, coumarin, by means of click chemistry, carrying it out for the first time using supercritical technology without the use of a ligand being necessary for the reaction to be carried out satisfactorily. In order to carry out the reactions corresponding to click chemistry based on Cu catalysis, it is necessary that the polymers previously incorporate azide or alkyne groups on which to carry out the functionalization.

# NANOMOLAR DETECTION OF DOPAMINE IN HUMAN FLUIDS BY N-DOPED CARBON NANOPARTICLES

## MARWA LAULEB

Nitrogen doped carbon nanoparticles, obtained by an easiest hydrothermal and eco-friendly process, was applied as fluorescence sensor for dopamine in urine and serum human fluids at nanomolar concentration range. The HR-TEM images demonstrated well dispersed nanoparticles with 19 nm as mean size, and the ATR and XPS analysis demonstrated the surface composition based in carboxylate and amide/amine functionals groups. The obtained nanoparticles were excitation wavelength dependent, with an optimal emission at 438 nm, which is the basis of the quantification of dopamine when quenches the signal. A linear trend between 0-652 nM was the range of application with a detection limit at 4 nM, acceptable accuracy (>80%) and precision (RSD<10%), showing a highest selectivity with related analytes and acceptable accuracy.

**Keywords:** N-doped carbon dots, dopamine, quenching, human fluids.

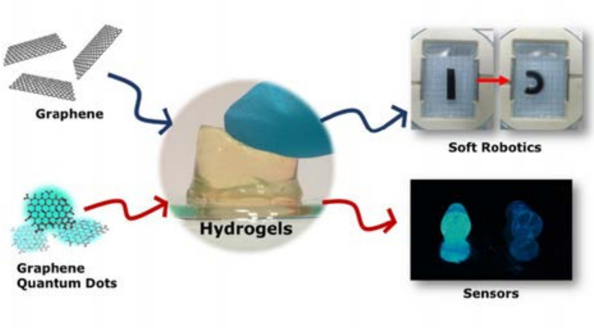


**Scheme 1**. Schematic representation of the interaction between N-CDs and DA.

# SMART MATERIALS FOR SENSING AND SOFT ROBOTICS APPLICATIONS

## ANA MARTÍN

Hydrogels, three-dimensional polymeric networks, have been extensively studied as suitable materials for a wide range of applications, from agriculture to biological uses. In recent times, composite hydrogels have been developed as the combination between hydrogels and fillers, improving the properties of the final material. Specially, graphene derivative-based hydrogels have recently opened a new field in science and technology because of their unique properties [1]. In this work, graphene (G) and graphene quantum dots (GQDs) were used to prepare composite hydrogels in order to understand their role in the hydrogel structure, regarding their sizes and chemical surfaces, in comparison with GO [2]. These nanomaterial-based composite hydrogels have been thoroughly characterised and applied in several applications, that range from sensing to soft robotics. For instance, a GQD-based hydrogel can be used as sensor for polyaromatic compounds in water [3], because of their fluorescent properties. In its case, Gbased hydrogels can be used as a soft actuator or as fingertips, modulating the precision of gripping in robotics hands [4].



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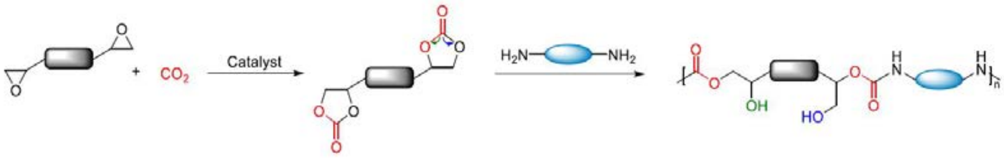
# FIXATION OF $CO_2$ INTO CYCLIC CARBONATES FOR THE GENERATION OF POLYURETHANES FREE OF ISOCYANATES (NIPU)s

## MARC MARTÍNEZ DE SARASA

In the past few years, a compelling growth of interest has emerged within the plastic industry for the production of polyurethane materials using greener intermediates and processes to substitute the classical route first reported by Otto Bayer during the late 1940s.[1-4] One of these processes is the polyaddition between di-cyclic carbonates and diamines.[5] These polymers combine numerous properties (light weight, excellent strength, energy absorbing performance, comfort features etc.) required in different fields such as medical, automotive and industrial.[6]

In this work, we report the synthesis of a wide range of new polyurethanes free of isocyanates from $CO_2$, bis-epoxides and diamines derivates with different thermal properties for their specific use within industry (Scheme 1).



Scheme 1

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# ELECTROCHEMICAL PROPERTIES AND SENSING CAPACITIES OF DIFFERENT CARBON DOTS FAMILIES USING SCREEN PRINTED ELECTRODES

## CRISTINA MONTES

The novelty of this work lays in the comparison of the electrochemical properties of three new carbon-based nanodots species with different structures thus involving diverse crystallinity and quantum confinement degree: carbon nanodots (CNDs), carbon quantum dots (CQDs) and graphene carbon dots (GQDs), here employed as potential sensing tools on screen printed electrodes. The three carbon-based nanodots species were synthesized by different routes, topdown and bottom-up [1, 2] and characterized by TEM, FTIR, Raman, UV-Vis and fluorescence. Measured sizes by TEM were similar between them being the smaller diameter found for CQDs (around 3 nm) while the highest one was found for GQDs (9 nm). All structures were passivated with different functional groups of oxygenated species. Since the aim of this work is to elucidate the potential effect of the carbogenic core structure (crystalline or amorphous) on their electrocatalytic properties, cyclic voltammetry assays were carried out using different specific redox probes such as: potassium hexacyanoferrate(III), hexaamine-ruthenium(III) chloride and dopamine hydrochloride, everyone with specific surface active degree. Better electrocatalytic properties were obtained for the CND, CQD and GQD modified electrodes compared to the bare one. However, CND and GQD electrodes showed better electrochemical features than CQD electrode in terms of reversibility and electronic transfer rate. The electroanalytical capabilities of these carbogenic nanodots as sensing tools were also considered for the detection of bioactives analytes like different vitamins such as ascorbic acid (Vit C), tocopherol isomers (Vit E) and several kind of aminoacids (lysine, arginine, histidine, cysteine, methionine, tyrosine, etc) present in biological samples like food products and biological fluids.

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# BLUE AGGREGATION-INDUCED EMISSION BY HYDROGELATION PROCESS

## JOSUE MUÑOZ

A novel stable physical hydrogel was prepared by radical polymerization of a monomer system including acrylate monomers and a vinyltriazine derivative. For the first time, a phenyltriazine compound is used as monomer in the construction of a hydrogel. The strong interactions between the triazine molecules[1] are the origin of the blue fluorescence that the gel exhibits when being excited under UV-light (Figure 1). Restricted Intramolecular Rotation (RIR) caused by polymer formation and Restricted Intramolecular Motion (RIM) engendered by H-bonds operate as Aggregation-Induced Emission (AIE)-mechanisms.[2] Fluorescence response with respect to the pH change has been studied. The idea of combining soft materials and AIE properties expands the applications of these materials, being able to be used as sensor for ions or molecules capable of interacting with the different functional groups of the polymer network. As well as, the possibility of incorporating other substances into the hydrogel may also allow the development of light-emitting soft materials.

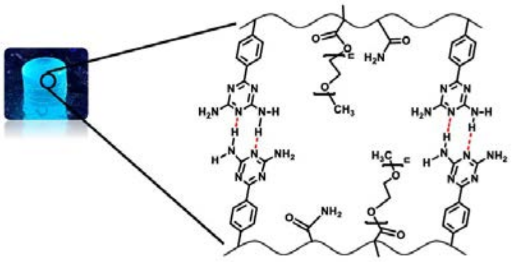

**Figure 1.** Digital image and chemical hydrogel structure

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# ADENOSINE RECEPTORS, RESVERATROL AND CANCER

## SONIA MUÑOZ

Extracellular adenosine is one of the major constituents of the tumor microenvironment and plays a crucial role in apoptosis, angiogenesis and metastasis in cancer. The effects of this purine are triggered through four G-protein coupled receptors: $A_1$, $A_{2A}$, $A_{2B}$ and $A_3$. $A_1$ and $A_3$ receptors inhibit adenylyl cyclase activity through Gi/o protein whereas $A_{2A}$ and $A_{2B}$ receptors activate this enzymatic system through Gs protein. Current efforts are focused on resveratrol (RSV), a diet polyphenolic phytoalexin found in many plant species such as grapes, peanuts and berries and also in red wine. This molecule has shown promising effects in inhibiting proliferation and cancer progression in several tumoral models. However, its molecular mechanisms are poorly understood. Recently, our group has found that RSV acts as a non-selective adenosine receptor agonist. This study provides strong evidence that adenosine receptors are a primary target for RSV. Adenosine receptor have been involved in cancer. Therefore, the aim of the present work was to study the antitumoral effect of RSV and the possible mechanism involving adenosine receptors. To this end, two tumoral cell lines were used, rat C6 glioma and human HeLa epithelioma cervix cells which is one of the most aggressive cancer in woman. Cell viability assays shown that RSV inhibited proliferation in a time and concentration dependent manner in both tumoral cell lines. RSV was also able to modulate these receptors and their corresponding transduction pathway. All together these results suggest that antitumoral effect of RSV could be through adenosine receptors activation.

# INTERSTELLAR DARK CLOUDS CHEMISTRY: KINETIC BEHAVIOUR OF NEUTRAL-NEUTRAL REACTIONS AT VERY LOW TEMPERATURES

## ANTONIO JESÚS OCAÑA

In the field of astrochemistry, complex organic molecules (COMs), are defined as molecules which are formed by C, H, O and/or N atoms and have 6 or more of atoms[1] Understanding how the COMs which are present in interstellar dark clouds are formed and destroyed is crucial in order to model the abundances of them. Usually, the rate coefficient (k) values used in modelling are extrapolations from temperature dependences reported at T > 200 K but it is known that these reactions are much faster than estimated by Arrhenius equations obtained at T > 200 K[2].

The aim of this work is to provide new kinetic data for the gas phase reactions of OH radicals with COMs at dark clouds temperatures (10-100K), using the continuous and pulsed[3] CRESU (French acronym that stands for Reaction Kinetics in Uniform Supersonic Flow) technique. This technique is based on the isentropic gas expansion through a Laval nozzle from a high-pressure region to a low-pressure region to cool down the gas to get uniform jets in temperature and total gas density over several tens of cm. The impact of the measured rate coefficients will be discussed at the conference.

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# EVALUATION OF CHEMICAL VOLATILE PROFILE OF MANGO BY-PRODUCTS BY HS-SPME-GC-MS AS POTENTIAL SOURCE OF FLAVOURING COMPOUNDS

## RODRIGO OLIVER

Mango (*Mangifera indica* L.) is an appreciated tropical fruit for its unique characteristics of aroma and flavor. During its processing stages, large quantities of bio-wastes are produced which represent from 25 to 60 % of the fruit approximately. Several studies have reported the value-added of mango wastes especially as a natural source of functional and nutraceutical ingredients derived from the vast variety of bioactive compounds existing in those discarded fractions (Jahurul et al., 2015). However, other different potential applications of mango byproducts in the food industry have already not been addressed until now.

Therefore, due to the appreciated sensory characteristics of mango, it is thought that revalorization of mango wastes as a source of natural flavourings may be considered an alternative for exploiting those by-products. For that scope, the chemical composition of outstanding mango by-products (skin, seed) in comparison with mango pulp were analysed by HS-SPME-GC-MS with the aim to characterize their volatile composition and evaluate the flavouring potential of mango by-products.

Results showed that mango peel exhibited large quantities of volatile compounds (monoterpens, sesquiterpens, alcohols, aldehydes,…) in higher concentrations than in mango pulp. Furthermore, the odor activity values (OAVs) indicated that mango peel was also the part of mango with the highest values in herbaceous green and floral features. Therefore, mango peel by-product is proposed as a natural source of volatile compounds for prospective applications in the food industry.

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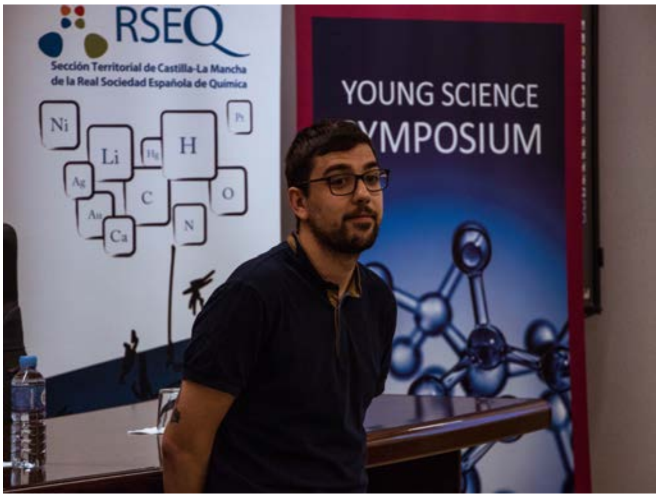
## GRAPHENE BASED MATERIALS APPLICATIONS

### ANTONIO PATÓN



Since Geim and Novoselov won the Nobel Prize in physics for the isolation of one perfect atomic layer of graphene in 2010, the interest of researchers towards graphene field has awakened. In the search for the best way to synthesize graphene, a large number of new carbon nanomaterials have been discovered. One of the most important materials discovered around graphene is graphene oxide (GO), which can be defined as few functionalized layers of graphene with molecules of carbon, oxygen and hydrogen attached to the structure [1]. This material and its derivatives are widely used in a wide range of applications, from electronic devices and medical treatments to adsorber media.

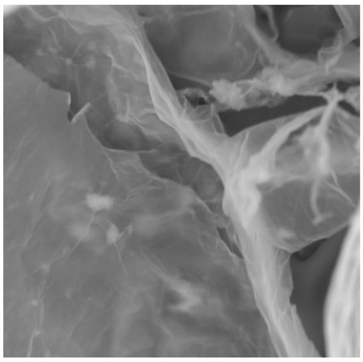


**Figure 1:** SEM Image of graphene oxide

The development of this applications is one of the new research area for this new material. Two of the most important applications for graphene oxide are the adsorption of chemical molecules and the electrochemical applications. On one side, the adsorption capacity of this new materials provides a reusable resource for harmful molecules as dyes, heavy metals or organic compounds. On the other side, the electrochemical applications based on the doping of heteroatoms as catalyst for oxygen reduction reaction (ORR) provide new green free-metal catalyst for this purpose.

# NEW ELECTROFENTON REACTORS FOR WASTEWATER TREATMENT

## JOSÉ FERNANDO PÉREZ

ElectroFenton has been extensively studied on laboratory scale for the abatement of hardly biodegradable organic pollutants in wastewater. However, most of the works are carried out on a laboratory scale. The objective of the present work is the design and construction of electroFenton reactors with potential to be scale up and applied on an industrial scale. Figure 1 shows schematically the final prototype which integrates synergistically different technological solutions:

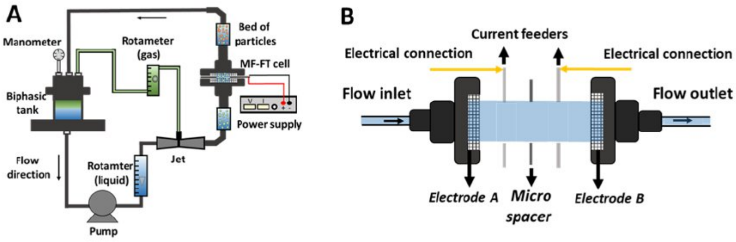


**Figure 1.** A) Schematic setup of the system; B) A micro-fluidic flow-through cell

In particular, the reactor incorporates an efficient pressurized-jet aerator to supply large oxygen flow rates at moderate pressure, a microfluidic flow-through reactor to simultaneously minimize ohmic resistance and maximize mass-transfer, equipped with state-of-the-art electrodes and a fluidized bed of particles to promote Fenton reaction. Results show an outstanding performance of the microfluidic flow-through cell in comparison to a commercial flow-by, a synergistic combination of anodic oxidation and electroFenton in this reactor[1] or the lowest specific energy consumption ever reported under similar conditions for the production of hydrogen peroxide. The communication summarizes the development and current application of this reactor.

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## PHENOLIC COMPOUNDS PROFILE OF DIFFERENT BERRY PARTS FROM NOVEL *VITIS VINIFERA* L. RED GRAPE GENOTYPES AND TEMPRANILLO USING HPLC-DAD-ESI-MS/MS: A VARIETAL DIFFERENTIATION TOOL

### JOSÉ PÉREZ

Phenolic compounds are bioactive secondary metabolites found in plants and fruits with potential beneficial effects on human health. In addition, these compounds are regarded as antioxidants and antimicrobials in food. Grape phenolic composition is a determinant factor for red wine quality, since it is related to important sensory attributes such as color, bitterness and astringency, and also to aging aptitude. Moribel and Tinto Fragoso are two novel red grape genotypes (*Vitis vinifera* L.) recently identified in Castilla-La Mancha, which are not previously registered in any national or international data base. They have been studied genetically but until now, no deep studies are still available about their phenolic composition. The aim of this work was to perform a comprehensive study of the phenolic profile of Moribel and Tinto Fragoso red grape genotypes (*V. vinifera* L.) for the first time and compare them to that of Tempranillo using HPLC-DAD-ESI-MS/MS over two consecutive vintages (2016 and 2017).

More than 50 phenolic compounds were identified in different berry parts from these grapes, of which, as far as we know, some anthocyanin and flavonol dihexosides were reported for the first time in *V. vinifera* grapes. According to phenolic composition, these novel red grape cultivars have interesting oenological properties. Tinto Fragoso may be a potential source of anthocyanins, which are responsible for the bluish-red and purple color of young red wines, and Moribel genotype was characterized by having a phenolic profile similar to Tempranillo cultivar, which may be due to a genetic relationship. Application of Principal Component Analysis (PCA) to experimental data showed a good separation of the novel grape genotypes and Tempranillo according to the phenolic profile of skins and seeds, mainly based on the proportion of trisubstituted anthocyanin derivatives, flavonols and flavan-3-ols, being a useful tool to differentiate these grape genotypes.

## SIMPLE AND SENSITIVE METHOD FOR DETERMINING GOLD NANOPARTICLES AND FOOD DYES IN DIFFERENT SAMPLES USING SURFACE-ENHANCED RAMAN SPECTROSCOPY

ESTHER PINILLA

Metallic nanoparticles, especially, gold nanoparticles (AuNPs) are used in several applications such as photoelectrochemical materials, optical sensors, diagnostic tests with biomedical imaging or catalysts showing unique physicochemical and optical properties due to the oscillation of localized surface plasmons (LSP). Despite their benefits, there are serious concerns for their possible environmental and biological impact. For these reasons, it is necessary to develop analytical methodologies controlling these particles.

SERS (Surface-Enhanced Raman Spectroscopy) has emerged thanks to the incorporation of noble metal nanoparticles to common Raman spectroscopy, providing so an enhanced signal due to the resonant excitation of LSP on metal nanoparticles surface that generates a magnification of electromagnetic field.1

In this report, on the one hand SERS signal has been indirectly used to determine AuNPs, incorporating on the substrate different organic molecules that belong to the family of synthetic food dyes with good ability to be adsorbed on metallic gold surfaces and that could be suitable candidates as AuNPs-sensor. Erythrosine B showed to be the best candidate producing a good SERS activity in presence of this nanomaterial. The proposed analytical method has been thoroughly validated for determining AuNPs in environmental, cosmetic and biological samples considering the principles of analytical nanometrology.[2] The obtained results were satisfactory with a lineal range between 1-12 ng $L^{-1}$, detection limit of 1 ng $L^{-1}$ and precision (%RSD) of 4.2 for 8 replicates of 6.8 ng $L^{-1}$ AuNPs solution. In the opposite way, this method was also used for determining erythrosine in food samples. This last method was also validated with a lineal range between 5-150 μg $L^{-1}$ and a detection limit of 4.7 μg $L^{-1}$ as figures of merit.

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# VALORIZATION OF ETHANOL INTO VALUE ADDED COMPOUNDS BY ELECTROREFORMING ON A PEM CELL

## ALBERTO RODRÍGUEZ

In recent years bioethanol has become one of the most promising alternatives to traditional fossils fuels. However, the overproduction on a global scale (biodiesel crisis) and also on a national scale (surplus in wine production and industrial waste of alcohol production) have required the search of different strategies for the valorization of this compound. In this research work, the electrochemical reforming process is proposed as an alternative, which presents some advantages (less deactivation and selectivity limitations) compared to conventional catalytic processes. Using a polymer membrane electrochemical reactor (PEM cell), in the anodic compartment the oxidation reaction of ethanol takes place, producing protons and organic compounds of industrial interest. Reduction reaction takes place in the cathodic compartment, generating high purity hydrogen. It is well known that no special attention is given to the organic compounds in the available literature [1]. For that reason, the aim of this research work is to maximize the production of valuable liquids obtained in the anodic compartment, derived from the ethanol oxidation, in order to improve the valorization of this compound. Some of these products are acetaldehyde, acetic acid and ethyl acetate. Acetaldehyde is an important intermediate in organic synthesis, used as a raw material for the production of acetic acid, acetic anhydride, ethyl acetate, pyridine, medicines or plastics. Industrially, it is obtained from the direct catalytic oxidation of ethylene through Wacker process, which is very intensive in terms of energy, so the electrocatalytic alternative is presented as a more efficient option. Under this scenario, ethanol electro-reforming process was carried out at 80 ºC, 1 atm and 1.15 ml/min of flow rate for cathodic and anodic chamber. In order to verify the viability of the system, different experiments were carried out: linear voltametries, chronopotenciometries at various levels of intensity, stabilities essays to estimate the total consume and impedance spectroscopy essays. Commercial Pt/C was used for the cathode and PtRu/C (synthetized through the modified polyol method) for the anode.

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## LEPTIN. "FROM 1969 TO THE PRESENT DAY"

### BLANCA RUBIO

The development of cardiovascular diseases (CVD) is the main cause of death and disability of the elderly world population, being some of the major risk factors obesity or Diabetes Mellitus (DM). In search of preventive strategies, and after its discovery as a regulator of body fat reserves (Halaas JL et al., 1995), leptin began to be studied as a point of control of energy metabolism. This hormone is produced mainly by the white adipose tissue, but the receptors for the hormone are distributed in a great variety of tissues, being very important its discovery in the hypothalamic nuclei related to the appetite control, metabolism and thermoregulation.

Our group tries to respond to the effects caused by the use of central leptin in peripheral tissues; however, and unlike other studies, our measures are based on the activation of the sympathetic nervous system as an intermediary of the effects of leptin. In this case we have shown that central leptin causes alterations in the heart metabolism, such as favoring the metabolic fuel change, reducing lipotoxicity or generating a specific pattern of cardiac atrophy (Mora et al., 2018). Similarly, white adipose tissue is able to guide a browning model by increasing the metabolic rate, favoring oxidative metabolism and thermogenesis.

Our current studies focus on the use of nuclear PPAR receptors as intermediates of the action of central leptin, based on the use of the specific PPARβ / δ antagonist.

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# ALTERNATIVE PHOTOSENSITIZERS TO ENHANCE THE SENSITIVITY OF NMR SPECTROSCOPY HYPERPOLARIZATION METHODS ON MICROCOILS

## MARGARITA RUIZ DE CASTAÑEDA

Nuclear Magnetic Resonance (NMR) Spectroscopy is one of the most powerful and versatile analytical techniques available today used in chemistry, physics, biology and medicine. NMR is widely employed to probe the local structure and dynamic properties of atoms. However, NMR suffers from an intrinsically low sensitivity, precluding the application of NMR spectroscopy to mass- and volume-limited samples.

Here, we present the use of hyperpolarization methodology to enhance the NMR sensitivity. These experiments are initiated with the excitation of a photosensitizer by an external laser source.

Furthermore, we have combined the use of microcoils with the hyperpolarization methodology to provide a breakthrough in both mass and concentration sensitivity for the study of low solubility samples. In previous works, our group have been able of detecting subpicomole quantities of sample and a sample concentration in the µM range for a fluorinated compound.[1]

The most common photosensitizers are flavins. However, flavins are prone to photoreduction during the hyperpolarization experiments as the process is not perfectly cyclic and they form aggregates. Thus, we are focusing on the development of alternative photosensitizers as triazines derivatives and organometallic compounds of ruthenium.

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# SYNTHESIS OF FURFURAL BY DEHYDRATION OF PENTOSES. TOWARDS FULL BIOMASS VALORISATION

## MANUEL SALGADO

Furfural (FF), or furan-2-carbaldehyde, is a green key compound which has been recognized as a top-value bio-based chemical by United States Department of Energy [1]. Therefore, it is useful to be employed in a wide range of applications, from the chemical industry to the agrochemical sector [2], as well as in the production of bio-fuels and solvents. FF production is generally carried out by the initial dehydration of pentoses, such as xylose. This process usually involves acidic conditions, for instance, with inorganic mineral acids, heterogeneous catalysts or sulfonic acids. Moreover, the mechanism for FF production has been also suggested by many researches [3]. Particularly, FF can be obtained from $C_5$-sugars found in hemicelluloses, one of the three major components in lignocellulosic biomass, including cellulose and lignin [2]. Nowadays, due to the diminishing of fossil fuel reserves, together with the detrimental augmentation of greenhouse effects, the worldwide scientific community has focused on novel routes employing sustainable C-sources for the synthesis of the top platform chemicals [4]. Regarding to the top, the main goal of this work is the optimization of some variables (temperature, time, solvent, catalyst…) in order to achieve the best yield of FF from xylose, as well as the influence of these variables over the valorization of some lignocellulosic wastes, always keeping the Green Chemistry Principles.

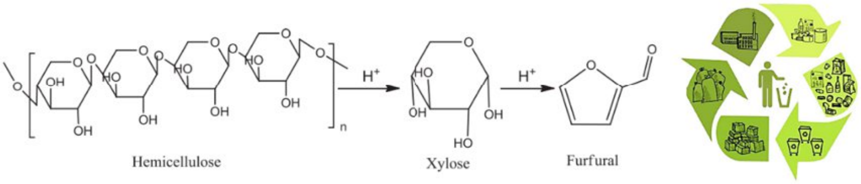

**Figure 1.** General chemical path from biomass to furfural

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# STUDY BY AF4-ICP-MS OF THE PtNPs' BEHAVIOUR IN NATURAL AND SYNTHETIC FRESHWATERS

## ARMANDO SÁNCHEZ

Platinum nanoparticles (PtNPs) present exceptional catalytic reactivity which makes them useful for automotive catalysts. However, mechanical abrasion and chemical reactions at the catalyst surface may cause PtNPs' emission through exhaust gasses of vehicles, leading to dispersion and accumulation of PtNPs all over environmental compartments [1]. The study of these NPs in relevant environmental samples is necessary to know the potential associated risks. This is indeed a challenging task for analytical chemistry, and the development of novel and powerful analytical techniques is necessary [2]. New analytical strategies based on the coupling of hydrodynamic separation techniques with elemental specific detectors have been recently proposed. One of the most promising combination is asymmetric flow field flow fractionation hyphenated to inductively coupled plasma mass spectrometry (AF4-ICP-MS). This system can give simultaneous information about particle size range, concentration and composition, but there are nearly not applications for PtNPs, especially in environmental samples. In this work a methodology based on AF4-ICP-MS has been developed for the study of PtNPs in water samples. Once the optimization of the separation and acquisition conditions in AF4-ICP-MS is done, PtNPs (5, 30 and 50 nm) were separated in one single fractogram run. It was also possible to detect transformations of these PtNPs in natural and synthetic freshwaters samples under the influence of relevant environmental factors such organic matter, pH, ionic strength or incubation time.

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# RESVERATROL IS A NON-SELECTIVE ADENOSINE RECEPTOR AGONIST: POTENTIAL ROLE IN NEURODEGENERATION

## ALEJANDRO SÁNCHEZ

Aged population is increasing fast in the last decades. Hence, neurodegeneration is one of the greatest global challenges for health research in the next future due to the major risk factor is the age. Currently, it is described that around 50 million of people live with any kind of dementia. By 2050, the incidence of neurodegeneration-associated diseases is estimated to be triplicated, which supposes an over cost for health services. Resveratrol (RSV) is a natural polyphenolic compound produced by plants under stressful conditions that has shown multiple beneficial properties for human health, including neurodegeneration. Unfortunately, the precise molecular mechanisms by which this phytochemical exhibits its biological functions remain still unclear. Therefore, the aim of this work was to investigate whether RSV was able to induce modulation on adenosine-mediated signaling. Our results strongly indicated, by biochemical and informatics approaches, that RSV acts as a non-selective adenosine receptor agonist, which affects to adenosine-mediated signaling in rat glioma C6 cell line. On the other side, long-term RSV supplementation in diet reversed the age-related effect on adenosinergic system in total brain from SAMP8 mice, an animal model of Alzheimer's disease. Accordingly, plasma membrane-expressed $A_1$ receptor was found to be increased whereas no changes on plasma membrane-expressed $A_{2A}$ receptors were detected in RSVtreated mice as compared to their age-matched controls. Moreover, an increase of $A_1$ receptormediated signaling was observed, but a desensitization of $A_{2A}$ receptor-mediated signaling was induced by RSV. On the other side, adenosine production/degradation carried out by the enzymes 5'-Nucleotidase and Adenosine Deaminase, respectively, were both found to be significantly reduced in RSV-treated mice, suggesting an alteration on adenosine level. In conclusion, RSV acts as a non-selective agonist, exerting a tissue-dependent biological effect. Taking all of this into account, adenosine receptors should be considered as a primary target for resveratrol and, although further experimentation is required, new therapeutic strategies involving resveratrol and adenosine-mediated signaling should be aimed in the future for neurodegeneration-associated diseases.

# ALKYNYL 1*H*-BENZO[*d*]IMIDAZOLE DERIVATIVES: APPLICATIONS IN ORGANIC ELECTRONICS

## CARLOS TARDÍO

In the last years, self-assembly of π-conjugated organic molecules has undergone a big growth due to the improvement of the optoelectronic properties offered by these structures with respect to isolated molecules. The shape, morphology and photophysical properties play a fundamental role in the applicability of the structures.[1] Multifunctionality is a desirable objective in these new structures. In this context, we have synthesised a series D-A-D of alkynyl compounds using 2*H*-benzo[*d*]triazole and benzo[c][1,2,5]thiadiazole as the acceptor cores. Self-assembly of these derivatives has generated structures with a high applicability in organic electronics as waveguides[3] and OFETs.[2] With the aim to define the structure-property relationship, in this report we have changed the acceptor moiety to 1*H*-benzo[*d*]imidazole (Figure 1). The structures resulting from self-assemble were studied by SEM and PL microscopy. The main objective is to study the applicability of these compounds as organic semiconductors in OFETs, or in the field of nanophononics as optical waveguides.

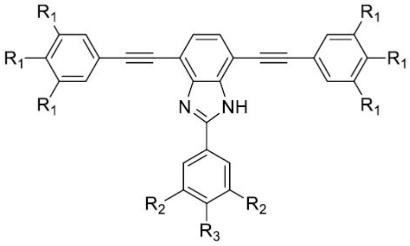

**Figure 1.** Structure of alkynyl 1*H*-benzo[*d*]imidazole derivatives

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# ELECTROCHEMICALLY-ASSISTED DEWATERING FOR THE REMOVAL OF OXYFLUORFEN FROM A COAGULATION/FLOCCULATION SLUDGE

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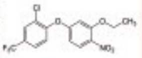
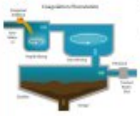
## Introduction

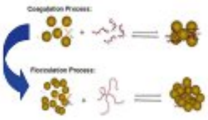
Use of **herbicides** in developed countries is an essential part of **agricultural production**, it is aimed to improve indirectly crop yield by killing or inhibiting the growth of weeds that compete with the desirable main plants for nutrition.



**Oxyfluorfen** is one of the most commonly used herbicides. It is a hazardous organochlorinated diphenyl-ether compound that has been used to control certain annual broadleaf and grassy weeds.

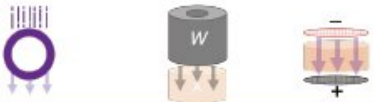


The use of these chemical compounds is associated with **undesirable impacts** in the environment, and many **technologies have been developed and tested** to remove this kind of compounds from synthetic and real water effluents.

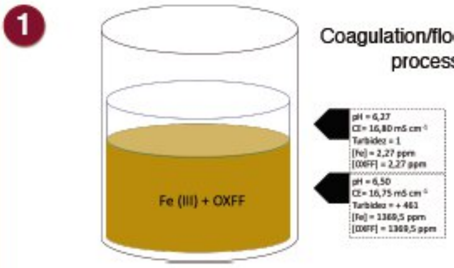


Nowadays, one of the priorities in the field is to **reduce the waste amounts** of sludges from the different treatments including electrochemical processes.

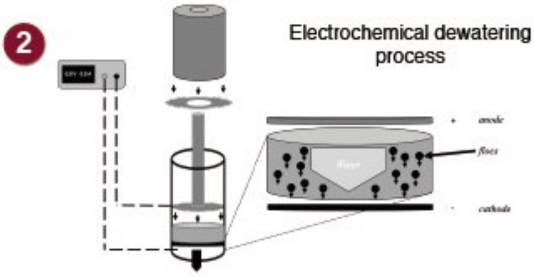
This work explores the **effect of electrochemical dewatering**, to **reduce and oxydize pollutants such as oxyfluorfen from sludges** procedent from a coagulation/flocculation process.



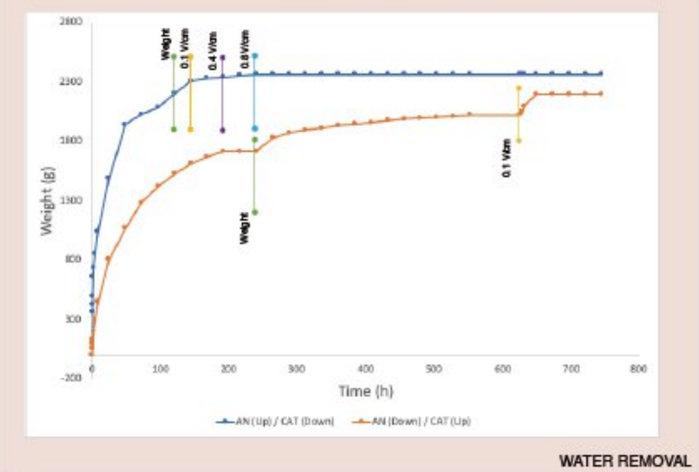
## Materials and methods



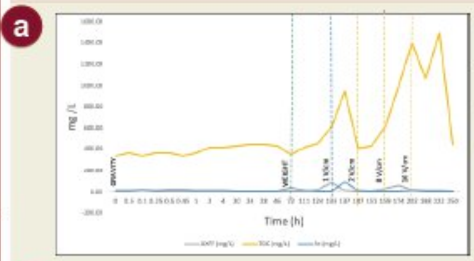
Coagulation/flocculation process



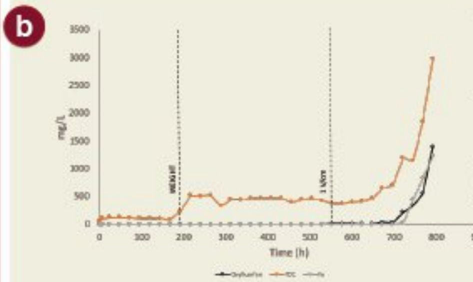
Electrochemical dewatering process



WATER REMOVAL



Anode (Up) and Cathode (Down) experiments show that there more energy needed to complete the dewatering of the sludge, but there is almost no oxyfluorfen liberation. TOC is liberated to the liquid sample when electric field is increased.



Anode (Down) and Cathode (Up) experiments show almost all water is extracted from the sample when applying 0.1 V/cm, but there is oxyfluorfen liberation that is correlated with TOC and Fe, and a decrease in pH values due to electrokinetic events.

OXYFLUORFEN REMOVAL

## Conclusions

1. The application of electric fields enhances the dehydration of polluted sludges and has influence on the movement of the pollutant species in the water flux.
2. The anode – cathode orientation has a relevant influence both in the species mobility and in pH, and also it has an influence in the dryness of the final cake.
3. It has been proven that the electrochemical dehydration of iron-coagulation sludges can reduce the amount of polluted wastes to a great extent.

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# POTENTIAL APPLICATIONS OF OENOLOGICAL BY-PRODUCTS AS ANTIOXIDANTS IN PROCESSED MEAT PRODUCTS

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## INTRODUCTION

One of the major causes of quality deterioration of meat products are the oxidative phenomena. Lipid oxidation is the main reaction produced which entails losses in nutritional and organoleptic quality reducing meat self-life. This fact causes the essential use of antioxidants in meat products [1]. In order to decrease the use of synthetic antioxidants, in recent years, research about antioxidants of natural origin has increased and, in particular, the by-products revalorization generated during the processing of food, show a wide interest in the food industry [2].

Study the antioxidant capacity of several oenological by-products in different meat products.

## MATERIAL & METHODS

## RESULTS

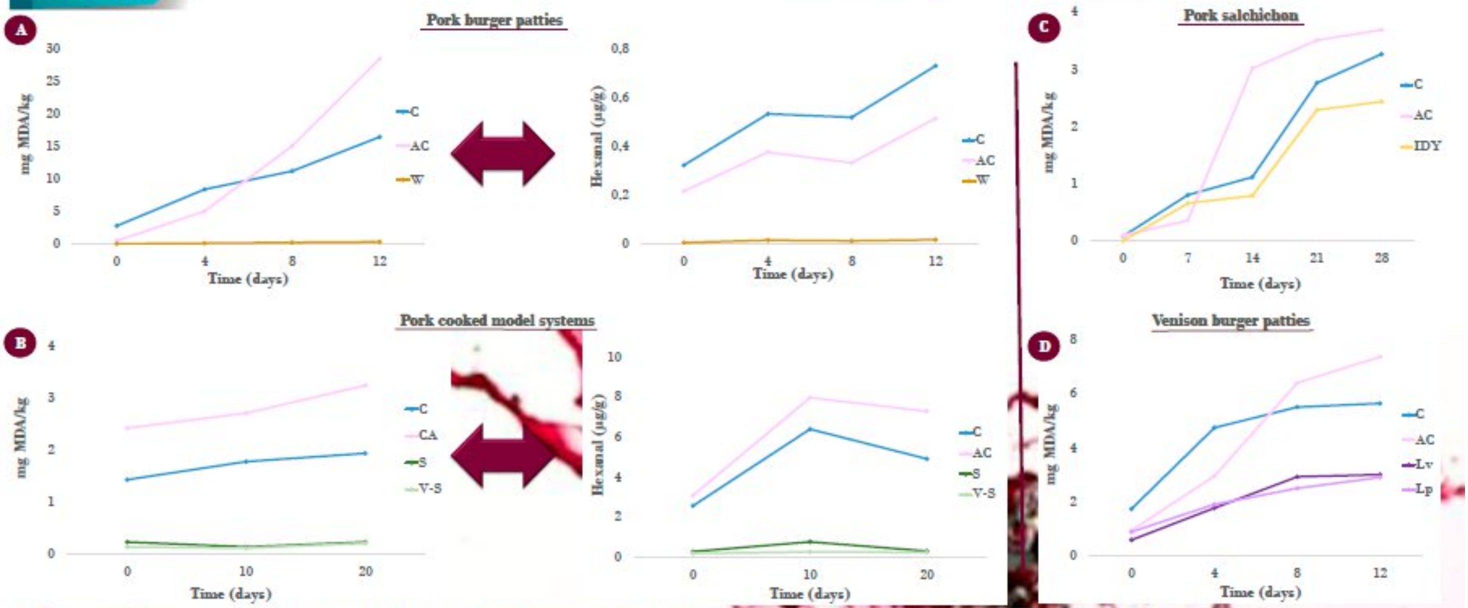

**Figure 1.** TBARs values (mg MDA/kg) and hexanal concentrations (µg/g) of different oenological by-products in processed meat products.

- A significant reduction of lipid oxidation (TBARs) was found in the different meat products using oenological by-products, especially oak wood extract.
- This effect was appreciated immediately after the natural antioxidant addition in samples.
- The inhibition of oxidation was reflected on the hexanal content which showed lower values compared to control samples.
- In all cases, natural antioxidants were more effective than sodium ascorbate. Likewise, AC was significantly higher than C samples, according to other authors [3].
- Selected concentrations of oenological by-products did not affect negatively the organoleptic characteristics of meat products.

## CONCLUSION

Oenological by-products could be employed as natural antioxidants against lipid oxidation in meat products.

## ACKNOWLEDGEMENTS

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# IONIC LIQUID AND MWCNTs FOR EXTRACTION OF CARBAMATE PESTICIDES FROM WATER SAMPLES PRIOR THEIR DETERMINATION BY CAPILLARY ELECTROPHORESIS

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## 1. Abstract

A rapid and simple method for the extraction of five N-methylcarbamate pesticides (carbaryl, carbofuran, promecarb, BDMC and methomyl) in water samples was developed. The procedure is based on temperature controlled ionic liquid dispersive liquid phase microextraction and MWCNTs combined with capillary electrophoresis. Some parameters that affected the extraction efficiency such as type and volume of extractant solvent, temperature, sorbent mass and type and volume of elution solvent were investigated in order to found the optimal extraction conditions.

## 2. Introduction

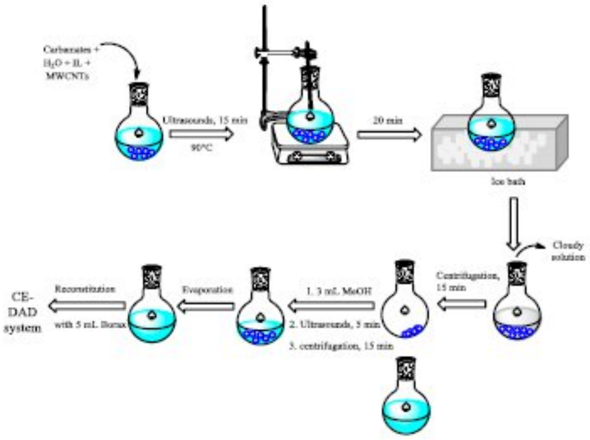
- ✓ Insecticide carbamates act as an inhibitor of acetylcholinesterase, the enzyme responsible for the hydrolysis of acetylcholine after transmission of information by this neurotransmitter. If this hydrolysis does not take place, the increase in acetylcholine concentration induces hyperactivity of nervous system resulting paralysis and death of the insect.
- ✓ The wide use of this carbamates causes the contamination of aquatic environments: rivers, lagoons, surface and underground water. Their trace-state determination, in various aqueous media, requires a preconcentration step followed by their analysis often conducted by HPLC-UV, HPLC LC-MS or CE-DAD.

## 3. Aims

- ✓ The development of a novel methodology to extract N-methylcarbamate pesticides with ionic liquid and MWCNTs from water samples.
- ✓ The separation and the quantification of the carbamates in water samples by CE-DAD.

## 4. Materials and Methods

### *Extraction procedure*



## 5. Results and Discussion

### Extraction and CE-DAD parameters

**Table 1.** Operating conditions for extraction and separation of NMCs by CE-DAD

| Parameter | Conditions |
|---|---|
| **Extraction procedure** | |
| Ionic liquid (IL) | 1-hexyl-3-methylimidazolium hexafluorophosphate |
| IL volume | 100µL |
| Sorbent material | MWCNTs |
| Sorbent mass | 10 mg |
| Elution solvent | DCM |
| Elution solvent volume | 3 mL |
| Sample volume | 10 mL |
| Buffer composition | 15mM borate solution |
| **CE method** | |
| Buffer pH | 11,47 |
| Separation voltage | 20 kV |
| Capillary dimension | 75 µm i.d. x 80 cm |
| Capillary temperature | 25°C |
| Injection time | 10 s |
| Injection pressure | 50 mbar |
| Wavelength | 209 nm |

### Detection of NMCs by CE-DAD

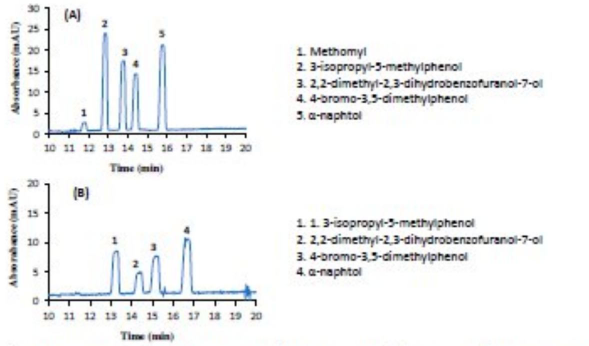


*Figure 1: Electropherogram of: (A) Standard solution 20 µg.mL-1 and (B) Extracted, spiked water sample.*

## 6. Conclusions

- ➢ In this work, the combination of IL and MWCNTs was used for the extraction of five N-methylcarbamate pesticides from water sample then determined by CE-DAD.
- ➢ The parameters that affected the extraction efficiency such as type and volume of extractant solvent, temperature, sorbent mass and type and volume of elution solvent were studied in order to found the optimal extraction conditions.

## Acknowledgements

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# BILIRUBIN METABOLISM IN HUMAN BODY AND ITS POSSIBLE CONTROL WITH SILICA MATERIALS

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## AIMS

The main objective of this project is the synthesis and study of silica nanoparticles as a biomaterial for treatment of critical hemodialysis patients with dangerously high serum bilirubin levels. For that goal, we have synthesised one type of amino-functionalized silica nanoparticles. The functionalization of the particles with an amine organic group is necessary for bilirubing binding and removal from patients' blood stream. A reduction in bilirubin levels was observed in PBS solutions, but further analysis techniques are needed to confirm that the decreasing levels are due to the removal capacity of amino-modified nanoparticles.

## SUMMARY

In critical patients with kidney failure, the development of simultaneous hepatic disease is especially dangerous, causing a rise in bilirubin serum levels that is not compatible with human life. Bilirubin builds up in skin, muscles and mucous membranes. It can even get into the brain and cause neurological damage (bilirubin encephalopathy).

In this study, we suggest the use of silica nanoparticles as a biomaterial for decreasing bilirubin levels in the blood of dialysed patients. The first step is the synthesis of nanosilica particles, functionalized with APTES as amine group donor for interaction with bilirubin carboxyl groups. Bilirubin adsorption assays are carried out in PBS solutions and human plasma to test the bilirubin removal capacity of the newly-synthesised biomaterial. If the results are promising, new sets of silica particles with other amino organic ligands will be produced and their bilirubin removal efficiencies will be compared.

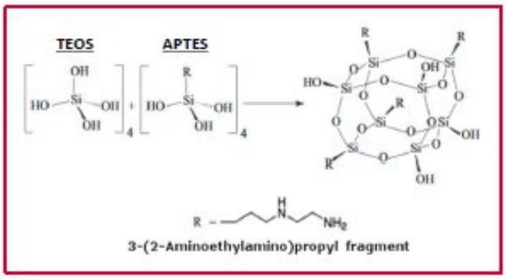

**Fig 1**. Diagram of the process of synthesis of silica nanoparticles modified with APTES.

## RESULTS

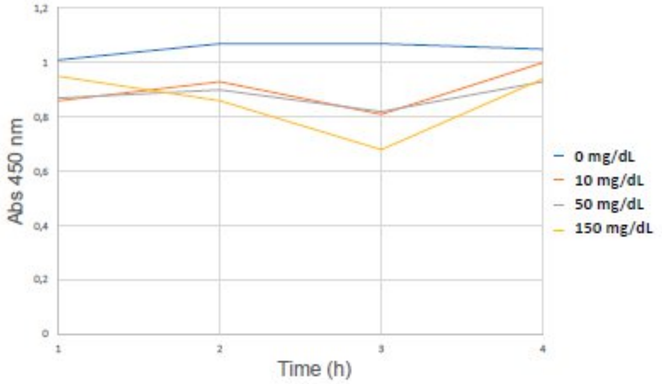

**Fig 2**. Bilirubin adsorption assays with different concentrations of amino-modified silica particles in bilirubin PBS solutions of 5 mg/dL. The reduction of bilirubin levels in the solutions over time were analysed by UV-visible spectroscopy, using the λmax of bilirubin (450 nm).

## CONCLUSIONS

- ➢ A sufficient amount of amino-modified nanosilica particles were successfully synthesised for bilirubin adsorption assays.
- ➢ A slight reduction of bilirubin is observed at 3 hours, especially when using 150 mg/dL of particles.
- ➢ It is necessary to recover the particles from the solutions and analyse their composition by techniques like FTIR or TGA to confirm that the reduction in bilirubin levels is due to its interaction with silica particles.

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# Assessment of Reversible Hydrogen-Chlorine PEM fuel cell

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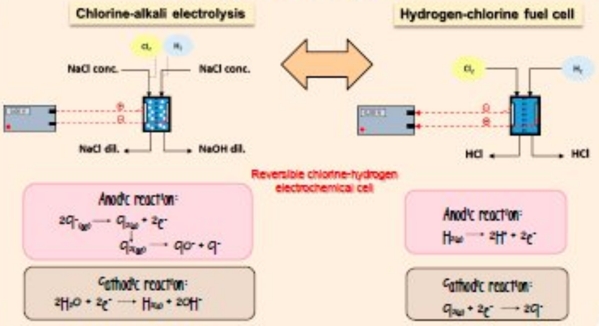
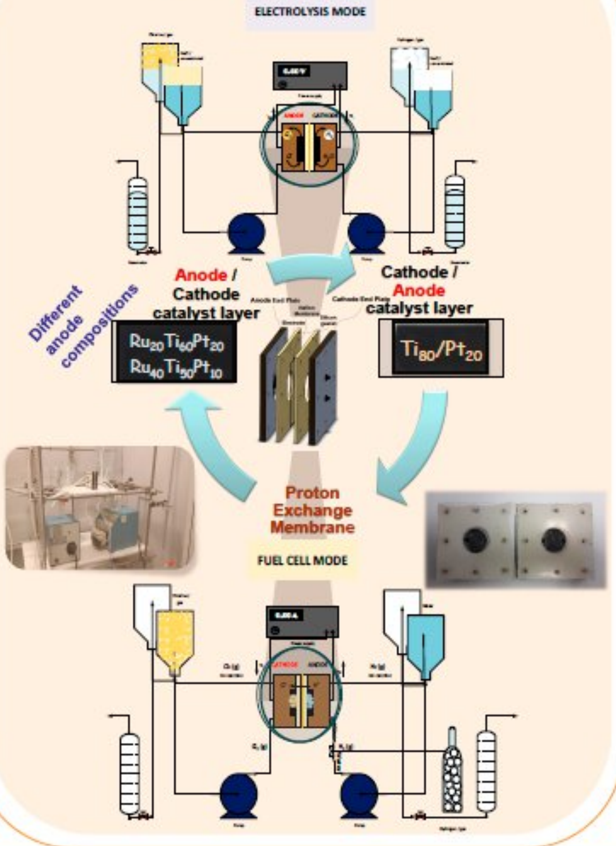
## INTRODUCTION



## OBJECTIVE

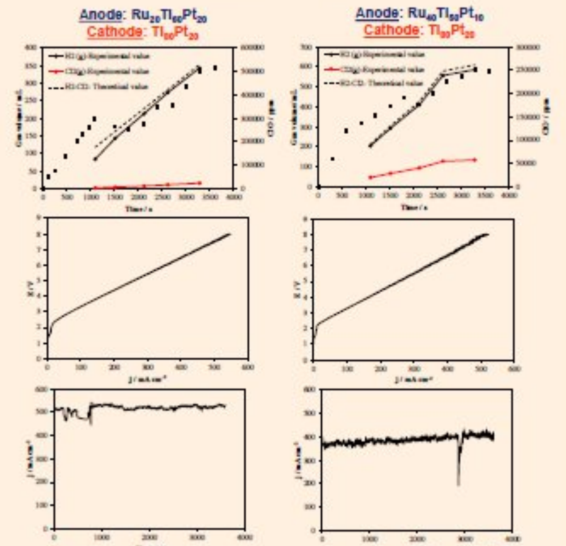
☐ Demonstrate the feasibility of reversible chlorine electrochemical cells operating in liquid phase

## EXPERIMENTAL



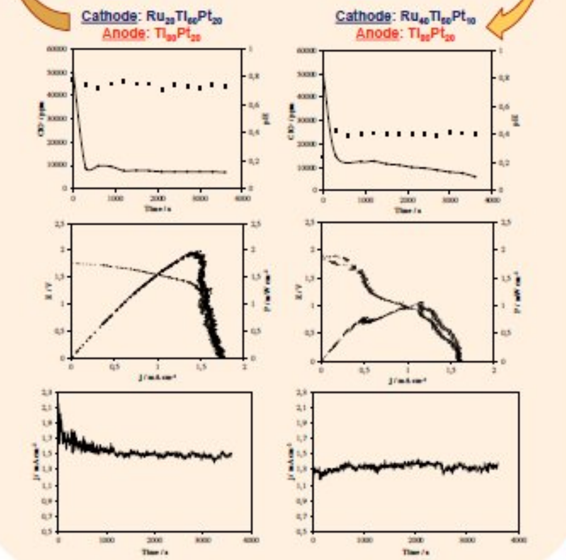
## RESULTS

### Electrolysis



REVERSIBILITY

### Fuel cell



## CONCLUSION

✓ A reversible chlorine-hydrogen PEM-Fuel cell is feasible operating with liquid phase. Further work is required to optimize the performance of the fuel cell mode

## ACKNOWLEDGEMENTS

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# OBTENTION OF HIGH VALUABLE COMPOUNDS FROM LOW-COST MATERIALS OF THE AGRO-INDUSTRIAL SECTOR OF CASTILLA-LA MANCHA THROUGH $scCO_2$

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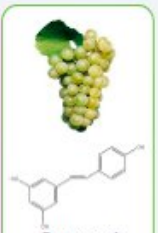
## INTRODUCTION

Nowadays, the increase in the demand of nutraceutical and pharmaceutical products of natural origin has led to the search of sources of bioactive compounds.

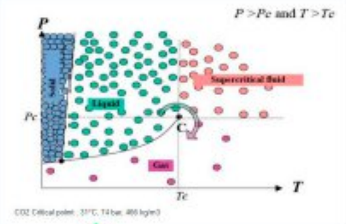
These compounds can be obtained from raw materials or waste from the agro-industrial sector of Castilla-La Mancha.


Polyphenols


Thiosulfinates


Resveratrol

ANTIOXIDANT AND ANTI-INFLAMMATORY POTENTIAL FOR TREATMENT OF SKIN DESEASES



SUPERCRITICAL FLUIDS EXTRACTION

$scCO_2$

- ✓ Tc=31℃, Pc=73 atm
- ✓ Easily separable
- ✓ Non-toxic
- ✓ Non-flammable
- ✓ Low cost

## AIM OF THE PROJECT

- ✓ Getting natural nutraceuticals and pharmaceuticals product through **a green technology.**
- ✓ Valorization of raw materials allowing the **economic** promotion of the agro-industrial sector.

## RESEARCH DEVELOPMENT

Evaluation of the solubility of raw materials in supercritical $CO_2$ ⇨ Extraction of valuable compounds ⇨ Modification of natural extracts ⇨ Determination of antioxidant and anti-iflammatory capacity ⇨ Economic review

## EXPERIMENTAL SETUP

SOLUBILITY TEST ⟶ **High pressure view cell**

**Figure 1.** Experimental setup

## MATERIALS AND METHODS

RAW MATERIALS

- Lavender essential oil
- Garlic oil
- Waste flows from wine

OPERATION CONDITIONS

- Pressure 100-300 bar
- Temperature 40-80 °C
- Contact time 6-72 h

## RESULTS

- ✓ After depressurization the solubility is calculated with the following equation:

$$Solubility = \left(\frac{kg\ lavender\ essential\ oil}{mCO_2\ in\ equilibrium}\right)$$

- ✓ The vapour phase was correlated using the semiempirical model of Chrastil:

$$\ln S = C_1 + \frac{C_2}{T} + C_3 \cdot \ln \rho$$

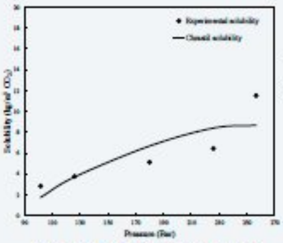


**Figure 2.** Influence of pressure in solubility of lavender essential oil in supercritical $CO_2$ (T=40 °C, contact time= 48 h)

**Table 1.** Optimised $C_1$, $C_2$ y $C_3$ values of carbon dioxide and essential lavender oil T=40 °C

| $C_1$ | $C_2$ | $C_3$ |
|---|---|---|
| -32,32 | -0,004 | 5,091 |

↑ **Pressure**
↑ **Solubility**

## CONCLUSIONS

- ☐ The use of $scCO_2$ avoids the handling of traditional organic solvents which are highly toxic and harmful to the environment.
- ☐ The samples obtained in liquid and vapor phases have to be analyzed by chromatography in order to determine the extracted compounds.
- ☐ An increase in pressure involves an increase in solubility of lavender oil in $scCO_2$.
- ☐ In Chrastil's equation, $C_2$ can be depreciated because, in this case, the experiments were carried out at isothermal conditions.

## ACKNOWLEDGEMENTS

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Castilla-La Mancha

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# GLYCOLYSIS PROCESS FOR POLYURETHANE WASTE RECYCLING

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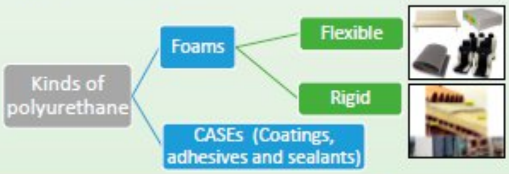
## INTRODUCTION

Polyurethanes (PUs) are generally thermostable polymers resulting from a series of additions between a multifunctional alcohol (polyol) and an isocyanate, resulting in a cross-linked structure. The characteristic functional group obtained by nucleophilic addition of the oxygen of the polyalcohol to the carbon of the cyano group is urethane.

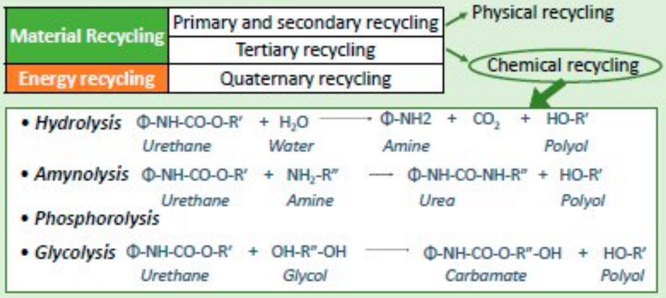
$$\underset{\textit{Polyol}}{n\ \text{HO-R-OH}} + \underset{\textit{Isocyanate}}{n\ \text{O=C=N-R}'\text{-N=C=O}} \rightarrow \underset{\textit{Urethane}}{[\text{-CO-NH-R}'\text{-NH-CO-O-R-O-}]_n}$$



PUs are the sixth group of polymers most used in the world and generate a large volume of waste, whose correct management is an environmental challenge, due to its low biodegradability. Traditionally, PUs waste has been deposited in landfills but due to its constant increase and the lower availability of landfills, it is necessary to find new and more environmentally friendly alternatives.

## POLYURETHANE WASTE RECYCLING

Physical recycling processes are successfully used in thermoplastic polymers, but they are useless for most PUs due to their thermostable nature, therefore, chemical recycling processes are of special interest. Besides, they make it possible to obtain chemical products from the PUs foams waste, such as polyether polyol, for the synthesis of new PUs foams.



| | |
|---|---|
| Material Recycling | Primary and secondary recycling |
| | Tertiary recycling |
| Energy recycling | Quaternary recycling |

- ***Hydrolysis*** $\underset{\textit{Urethane}}{\Phi\text{-NH-CO-O-R}'} + \underset{\textit{Water}}{H_2O} \longrightarrow \underset{\textit{Amine}}{\Phi\text{-NH2}} + CO_2 + \underset{\textit{Polyol}}{\text{HO-R}'}$
- ***Amynolysis*** $\underset{\textit{Urethane}}{\Phi\text{-NH-CO-O-R}'} + \underset{\textit{Amine}}{NH_2\text{-R}''} \longrightarrow \underset{\textit{Urea}}{\Phi\text{-NH-CO-NH-R}''} + \underset{\textit{Polyol}}{\text{HO-R}'}$
- ***Phosphorolysis***
- ***Glycolysis*** $\underset{\textit{Urethane}}{\Phi\text{-NH-CO-O-R}'} + \underset{\textit{Glycol}}{\text{OH-R}''\text{-OH}} \longrightarrow \underset{\textit{Carbamate}}{\Phi\text{-NH-CO-O-R}''\text{-OH}} + \underset{\textit{Polyol}}{\text{HO-R}'}$

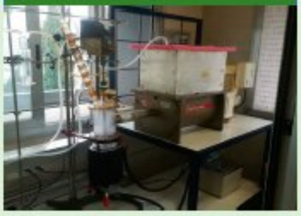
## GLYCOLYSIS PROCESS

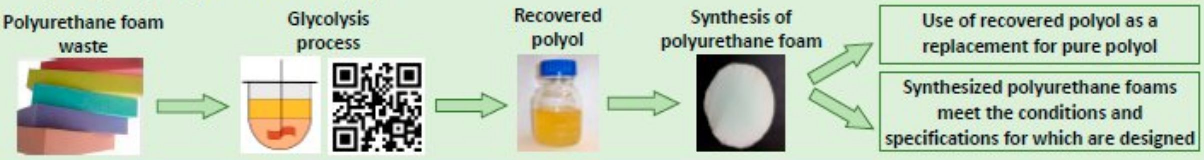
Glycolysis is the most widely used chemical recycling process for PUs and it is applied to elastomers, coatings, rigid foams, flexible foams and PU RIM (injection molding foams with reinforced reaction). In addition, with an excess of glycol in the reaction a biphasic product is obtained, where the upper phase is formed mainly by the recovered polyol and the bottom phase by the excess of glycolysis agent and by-products of the reaction; improving the quality of the recovered polyol.

### Reaction conditions

- Temperature = 193 °C
- N = 300 rpm
- Catalyst concentration in the glycolysis agent = 1.3 – 2.0 %
- Mass ratio of glycolysis agent to PU foam = 1.5:1

### Experimental unit





Use of recovered polyol as a replacement for pure polyol

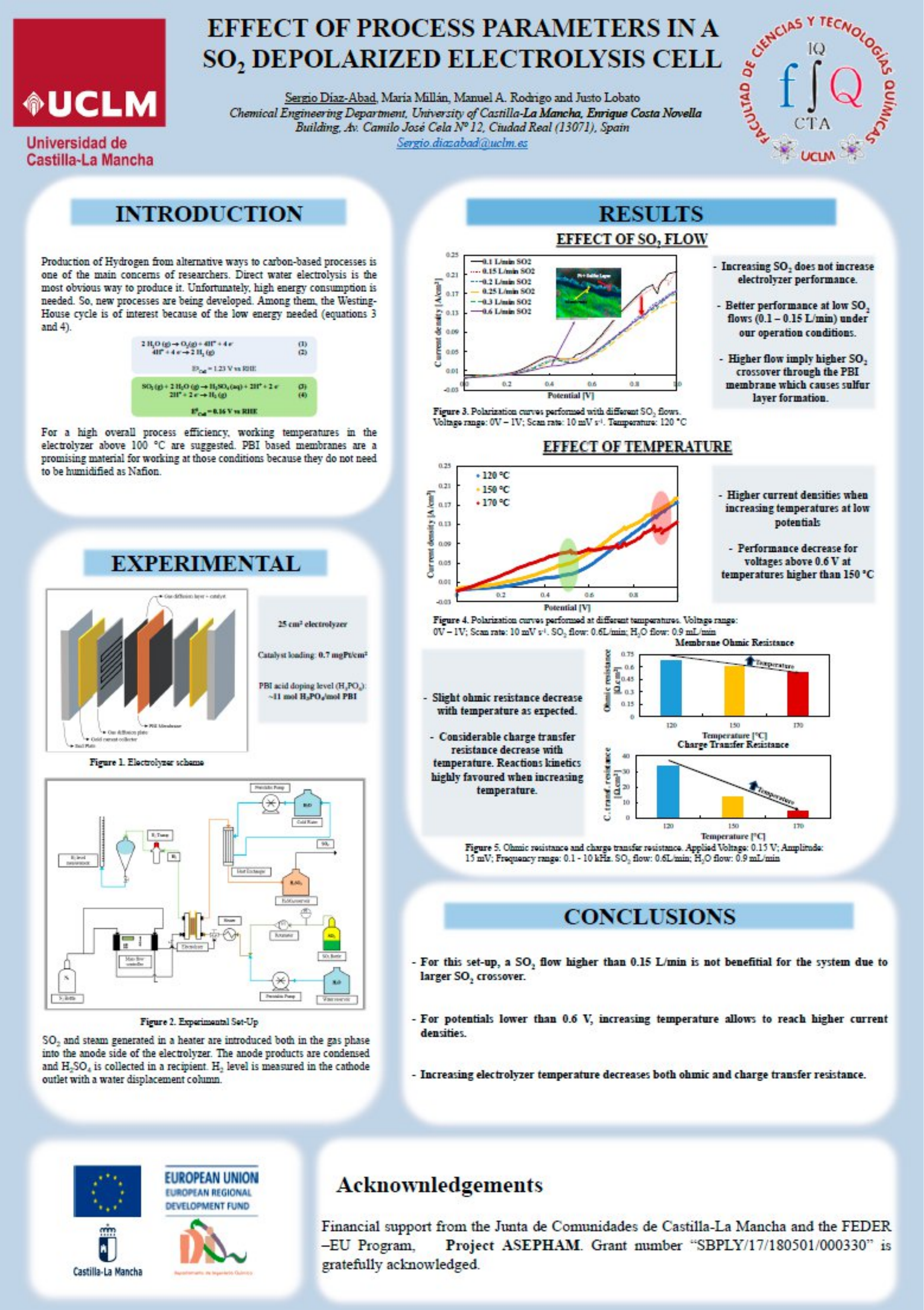
Synthesized polyurethane foams meet the conditions and specifications for which are designed

## CONCLUSIONS

One of the main advantages is the recovery of the raw materials (polyol) for the synthesis of new PUs foams, avoiding their deposit in landfills. In addition to the environmental benefits, it is worth highlighting the economic benefit. Previous studies have demonstrated that the glycolysis can be a profitable process, especially when it is carried out with crude glycerol as glycolysis agent since from two wastes, an added value product is obtained.

## ACKNOWLEDGMENT

Financial support from the European Union through project PolyUrethane Recycling towards a Smart Circular Economy (PUreSmart). Ref.: 814543.



# EFFECT OF PROCESS PARAMETERS IN A $SO_2$ DEPOLARIZED ELECTROLYSIS CELL

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## INTRODUCTION

Production of Hydrogen from alternative ways to carbon-based processes is one of the main concerns of researchers. Direct water electrolysis is the most obvious way to produce it. Unfortunately, high energy consumption is needed. So, new processes are being developed. Among them, the Westing-House cycle is of interest because of the low energy needed (equations 3 and 4).

$$2\,H_2O\,(g) \rightarrow O_2(g) + 4H^+ + 4\,e^- \quad (1)$$
$$4H^+ + 4\,e^- \rightarrow 2\,H_2\,(g) \quad (2)$$
$$E^0_{Cell} = 1.23\ V\ vs\ RHE$$

$$SO_2\,(g) + 2\,H_2O\,(g) \rightarrow H_2SO_4\,(aq) + 2H^+ + 2\,e^- \quad (3)$$
$$2H^+ + 2\,e^- \rightarrow H_2\,(g) \quad (4)$$
$$E^0_{Cell} = 0.16\ V\ vs\ RHE$$

For a high overall process efficiency, working temperatures in the electrolyzer above 100 °C are suggested. PBI based membranes are a promising material for working at those conditions because they do not need to be humidified as Nafion.

## EXPERIMENTAL

**Figure 1.** Electrolyzer scheme

**25 cm² electrolyzer**

Catalyst loading: **0.7 mgPt/cm²**

PBI acid doping level ($H_3PO_4$):
**~11 mol $H_3PO_4$/mol PBI**

**Figure 2.** Experimental Set-Up

$SO_2$ and steam generated in a heater are introduced both in the gas phase into the anode side of the electrolyzer. The anode products are condensed and $H_2SO_4$ is collected in a recipient. $H_2$ level is measured in the cathode outlet with a water displacement column.

## RESULTS

### EFFECT OF $SO_2$ FLOW

**Figure 3.** Polarization curves performed with different $SO_2$ flows. Voltage range: 0V – 1V; Scan rate: 10 mV s⁻¹. Temperature: 120 °C

- Increasing $SO_2$ does not increase electrolyzer performance.
- Better performance at low $SO_2$ flows (0.1 – 0.15 L/min) under our operation conditions.
- Higher flow imply higher $SO_2$ crossover through the PBI membrane which causes sulfur layer formation.

### EFFECT OF TEMPERATURE

**Figure 4.** Polarization curves performed at different temperatures. Voltage range: 0V – 1V; Scan rate: 10 mV s⁻¹. $SO_2$ flow: 0.6L/min; $H_2O$ flow: 0.9 mL/min

- Higher current densities when increasing temperatures at low potentials
- Performance decrease for voltages above 0.6 V at temperatures higher than 150 °C

- Slight ohmic resistance decrease with temperature as expected.
- Considerable charge transfer resistance decrease with temperature. Reactions kinetics highly favoured when increasing temperature.

**Figure 5.** Ohmic resistance and charge transfer resistance. Applied Voltage: 0.15 V; Amplitude: 15 mV; Frequency range: 0.1 - 10 kHz. $SO_2$ flow: 0.6L/min; $H_2O$ flow: 0.9 mL/min

## CONCLUSIONS

- **For this set-up, a $SO_2$ flow higher than 0.15 L/min is not benefitial for the system due to larger $SO_2$ crossover.**
- **For potentials lower than 0.6 V, increasing temperature allows to reach higher current densities.**
- **Increasing electrolyzer temperature decreases both ohmic and charge transfer resistance.**

## Acknowledgements

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# BIODIESEL PRODUCTION FROM WASTE OIL USING Fe/CaO AS BIFUNTCTIONAL CATALYST

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## ABSTRACT

The aim of this study was to analyze the catalytic performance of Fe/CaO as bifunctional catalyst in the transesterification reaction using frying oil with methanol. Quicklime was used as a source of calcium oxide and three chemical precursors of iron (III) were studied ($Fe_2O_3$, $FeCl_3$ and $Fe(NO_3)_3$*$9H_2O$). Heterogeneous catalysts Fe/CaO were synthesized using impregnation method; followed by calcination at 800°C. The catalysts were characterized by: X-ray diffraction (XRD), thermal gravimetric analysis (TGA), nitrogen adsorption/desorption (BET). The produced biodiesel has 91% of fatty acid methyl esters (FAMEs) content under optimum reaction parameters; atmospheric pressure, reaction temperature of 60 °C, 12:1 methanol:oil ratio, catalyst amount of 5wt%, 10wt% of Fe and 3 h of reaction.

## 1. INTRODUCTION

Bifunctional heterogeneous catalysis is a novel trend that allows the transformation of waste oils into biodiesel in a one-step process [1]. It has been estimated that the use of this type of catalysts can reduce production costs derived from raw materials (refined oil) by up to 80%, additionally, eliminates separation processes with severe environmental effects and simplifies the process operations [2]. Essentially, a bifunctional catalyst is a material that contains in the same catalytic surface; acid sites to esterify free fatty acids and basic sites to transesterify triglycerides. In this work, a bifunctional catalyst of iron (III) supported in calcium oxide (CaO) was synthesized for the production of biodiesel from used frying oil with high content of free fatty acids.

## 2. EXPERIMENTAL

2.1 Characterization of used frying oil
2.2 Synthesis and characterization of Fe/CaO
Chemical precursors of iron (III): $Fe_2O_3$, $FeCl_3$ and $Fe(NO_3)_3$*$9H_2O$
2.3 Bifunctional heterogeneous catalytic evaluation
2.4 Optimization of reaction conditions
2.5 Characterization of biodiesel

## 3. RESULTS

### *3.1 Characterization of used frying oil*

| Acid value | Density at (15°C) | Kinematic viscosity at (40°C) |
|---|---|---|
| 1.53 mgKOH/g | 926.021 kg/m³ | 36.08 mm²/s |

### *3.2 Catalyst characterization*

- *X-Ray Diffraction (XRD)*

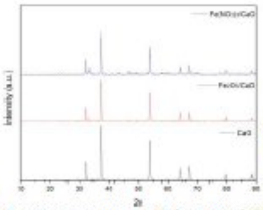


FIG 1. The XRD patterns of CaO, $Fe_2O_3$/CaO and $Fe(NO_3)_3$/CaO

- *Thermal Gravimetric Analysis (TGA)*

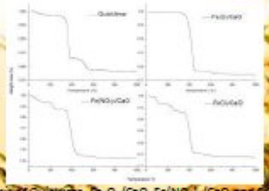


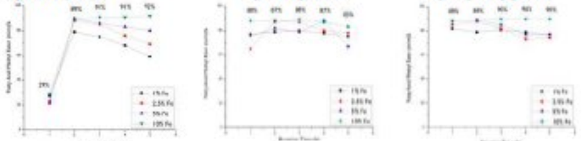
*FIG 2. TGA analysis of Quicklime, $Fe_2O_3$/CaO, $Fe(NO_3)_3$/CaO and $FeCl_3$/CaO catalysts*

- *Brunauer-Emmett-Teller method (BET)*

| Catalysts | Surface area (m²/g) | Pore volume (cc/g) |
|---|---|---|
| CaO | 7.995 | 0.0683 |
| $Fe_2O_3$/CaO | 12.150 | 0.0754 |
| $Fe(NO_3)_3$/CaO | 6.714 | 0.0267 |

### *3.3 Conversion of used frying oil*

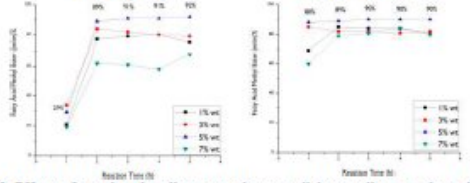
- *Effect of percentage of iron (III)*



*A) $Fe_2O_3$/CaO* *B) $FeCl_3$/CaO* *C) $Fe(NO_3)_3$/CaO*

*FIG 3. Influence of percentage of iron (III)*, reaction conditions: oil to methanol ratio 1:12, temperature 60°C

- *Effect of catalyst loading*



*A) $Fe_2O_3$/CaO* *B) $Fe(NO_3)_3$/CaO*

*FIG 4. Effect of* catalyst loading, reaction conditions: oil to methanol ratio 1:12, temperature 60°C, 10wt% Fe.

### *3.4 Biodiesel characterization*

| %FAMEs | Density at (15°C) | Kinematic viscosity at (40°C) |
|---|---|---|
| 91% | 879.28 kg/m³ | 4.6 mm²/s |

## 4. CONCLUSIONS

- $Fe_2O_3$/CaO catalyst has better performance with a high yield of methyl esters (91 % w/w).
- The best conditions were: 10 wt% of iron, alcohol: oil ratio 12:1, 5wt% of $Fe_2O_3$/CaO, reaction time= 3 hours and reaction temperature= 60 °C.

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[2] J. Gardy *et al.*, "A magnetically separable SO4/Fe-Al-TiO2solid acid catalyst for biodiesel production from waste cooking oil," *Appl. Catal. B Environ.*, vol. 234, no. 2010, pp. 268–278, 2018.

## Acknowledgments

The authors are grateful for the help provided with the catalyst characterization to Dr. Uvaldo Hernández Balderas from Centro Conjunto de Investigación en Química Sustentable UAEM-UNAM. The financial support of Universidad Autónoma del Estado de México through Project 4754/2019 CIB is also acknowledged. V.E.G. wishes to thank Consejo Nacional de Ciencia y Tecnología (CONACYT) for scholarship, No. 861011 to conduct postgraduate studies.

# STUDY OF DIFFERENT PRESERVATIVES FOR THE PLATINUM NANOPARTICLES STABILIZATION IN COMPLEX CLINICAL MATRICES

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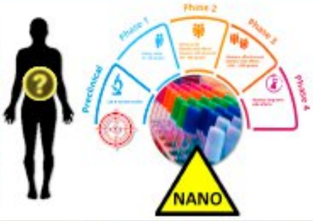
## INTRODUCTION

**Platinum nanoparticles (PtNPs)** are being applied in clinical and biomedical domains nowadays due to their physicochemical and antibacterial properties [1]. However, the use of PtNPs has been accompanied by a growing concern about their potential toxicity on human health. In order to have an adequate control of these applications it is necessary the study of PtNPs in **complex clinical matrices**. These studies are challenging for current analytical methods and there are still important open questions that starts even from the **stability** of metallic NPs in the presence of biofluids.



## OBJECTIVE

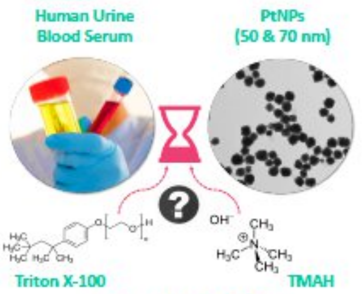
To evaluate the effect of several preservatives (tetramethylammonium (TMAH) and Triton X-100) to stabilize PtNPs in biofluids.

To test different concentrations of the most suitable preservative.

To achieve a correct characterization of various PtNPs sizes by Single Particle ICP-MS.

## MATERIAL & METHODS

### EXPERIMENTAL DESIGN



Triton X-100

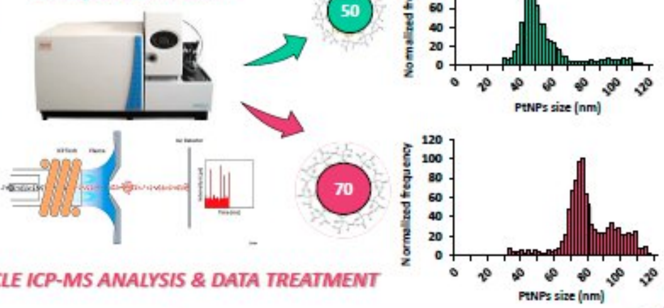
TMAH

Incubation at 4°C (24 h)

### SP-ICP-MS optimized method

| Instrument parameter | |
|---|---|
| RF-power | 1400 W |
| Nebulizer | Meinhard |
| Gas flows | |
| Plasma gas | 13 L min$^{-1}$ |
| Nebulizer gas | 0.9 L min$^{-1}$ |
| Auxiliary gas | 0.8 L min$^{-1}$ |
| Data acquisition | Time Resolved Analysis |
| Ion monitoring | $^{195}$Pt |
| Spray chamber | Scott type |
| Cones | Sampler & Skimmer - Ni |
| Sample flow rate | 0.8 mL min$^{-1}$ |
| Transport Efficiency | 2.0 % |
| Dwell Time | 5 ms |
| Acquisition Time | 60 s |

Quadrupole ICP-MS X-Series®

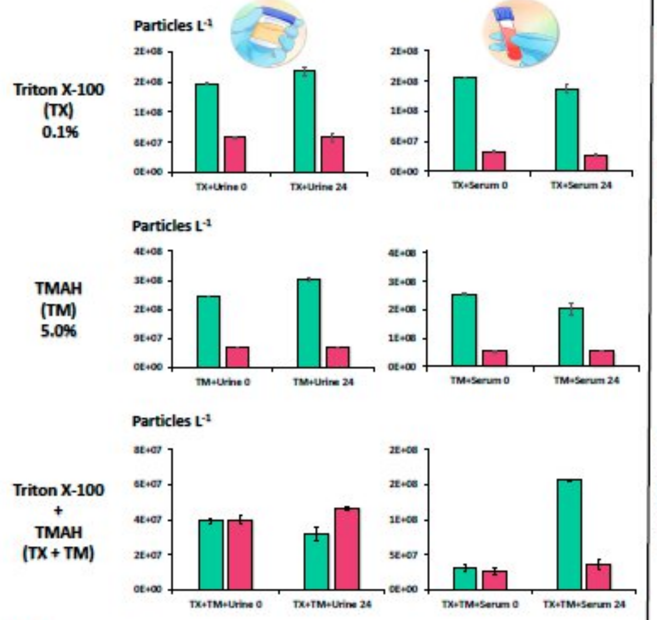


### SINGLE PARTICLE ICP-MS ANALYSIS & DATA TREATMENT

## RESULTS & DISCUSSION

### ① COMPARISON OF PRESERVATIVES

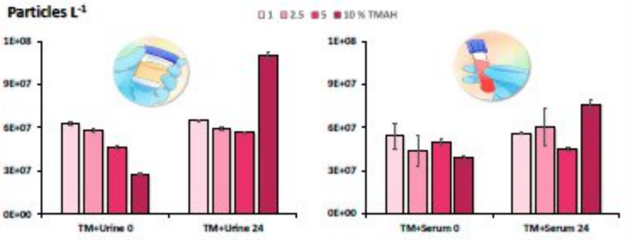
🟩 50 nm Nominal value (particles L$^{-1}$): 1.83E+08 🟥 70 nm Nominal value (particles L$^{-1}$): 6.92E+07



**TMAH** is the most suitable preservative to stabilize PtNPs in clinical matrices.

### STUDY OF TMAH CONCENTRATIONS ②

Nominal value (particles L$^{-1}$): 6.92E+07 🟥 70 nm

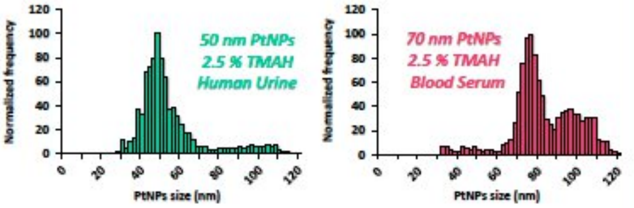


1.0 – 2.5 % TMAH

1.0 – 2.5 % TMAH

Results observed in particle mass concentration (ng L$^{-1}$) also confirm these TMAH intervals.

Particle size distribution (nm) at 24 h



24 h of incubation does not seem to induce a change in PtNPs sizes for both matrices.

## CONCLUSIONS

TMAH concentrations up to 2.5 % are acceptable to stabilize complex clinical matrices.
Further studies will be useful to assess toxicological effects over time.

## REFERENCES

[1] López-Serrano, A., Olivas, R. M., Landaluze, J. S., & Cámara, C. (2014). Nanoparticles: a global vision. Characterization, separation, and quantification methods. Potential environmental and health impact. Analytical Methods, 6(1), 38-56.

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Universidad de
Castilla-La Mancha

# POLYMERIC NANOPARTICLES FOR CONTROLLED DASATINIB DELIVERY IN BREAST CANCER THERAPY

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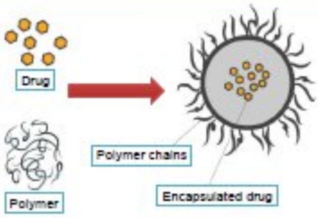
## 1. Introduction

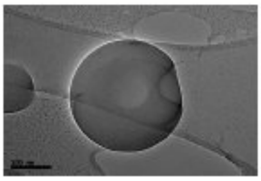
Dasatinib (DAS) is an inhibitor of several oncogenic kinases and the PDGFβ receptor. This mechanism of action makes it a great candidate for the treatment of breast cancer, but presents serious problems of solubility and primary metabolization by oral route.[1] In order to avoid these problems, in this work, we have carried out the encapsulation of DAS in poly(cyclohexene phthalate) (CHOPA) nanoparticles. Cytotoxicity and flow cytometry studies of the nanoparticles and the non-encapsulated drug in triple negative breast cancer cell lines were performed. The results showed a greater cytotoxic effect of the nanoparticles compared to the free drug.

## 2. Synthesis of Polymeric Nanoparticles

### Synthesis of CHOPA copolymer

### Synthesis of nanoparticles by nanoprecipitation and evaporation solvent method



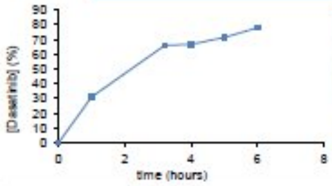


### DLS measurements and Z Potential

| Size | PDI | Z potential |
|---|---|---|
| 134.7 | 0.267 | 25.1 |
| 130.4 | 0.390 | 28.9 |
| 145.6 | 0.353 | 31.8 |
| 131.5 | 0.345 | 30.7 |

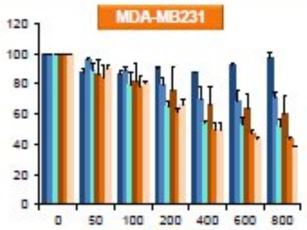
## 3. *In vitro* release profile at pH 7.4

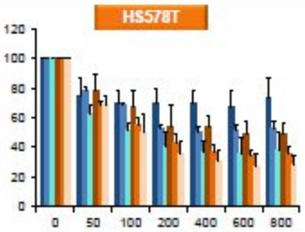
| Encapsulation Efficiency (%) | Loading Capacity (%) |
|---|---|
| 97.7 | 15.5 |

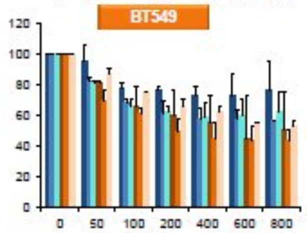


DAS release kinetics followed an exponential profile with a negligible burst and a sustained delivery over 6 hours for DAS-loaded CHOPA NPs at pH 7.4

## 4. Antiproliferative Studies *in Vitro*



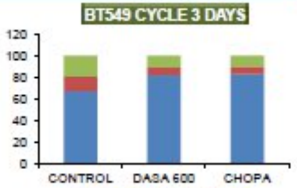


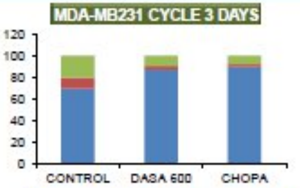


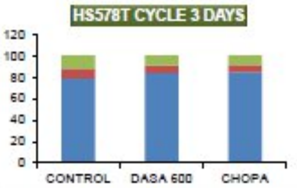
DAS-loaded CHO/PA NPs had a significant improved cytotoxic effect ($IC_{50}$ = 0.4 μM at 48 h and 0.2 μM at 72 h) in comparison with free DAS with an $IC_{50}$ > 0.8 μM at 48 h and 0.4 μM at 72 h

◆Dasatinib 24 hours ◆Dasatinib 48 hours ◆Dasatinib 72 hours
◆CHOPA 24 hours ◆CHOPA 48 hours ◆CHOPA 72 hours

## 5. Cell Cycle Arrest







Effect in cell cycle was carried out by treating cells with 600 nM of DAS and CHOPA. Cell cycle was analyzed after 24 hours of treatment by flow cytometry. No differences in the mechanism of action of the NPs were observed

## 6. Conclusions

A simple biodegradable and blood compatible copolymer has been developed as an alternative to FDA-approved polyesters for the generation of delivery nano-devices of DAS in breast cancer therapy. In vitro cytotoxicity in different breast tumor cell models demonstrated the superior efficacy of the NPs when comparable to free DAS.

## 7. References

1. (a) Zhang, J.; Yang, P. L.; Gray, N. S. *Nat. Rev. Cancer* **2009**, *9*, 28–39. (b) Yao, Q.; Choi, J.H. *ACS Appl. Mater. Interfaces* **2017**, *9*, 36642–36654.

# MECHANOCHEMISTRY IN THE SYNTHESIS OF NANOMATERIALS

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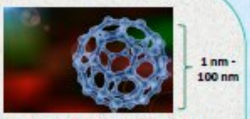
## INTRODUCTION

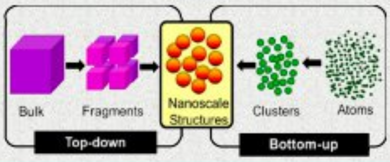
### Nanomaterials:

Engineered particles with improved physical, chemical and biological properties.



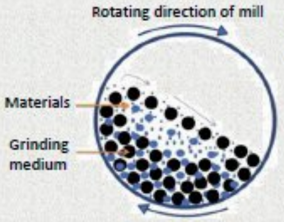
Nano-Zirconia ($ZrO_2$): Outstanding chemical stability, high strength and toughness, wear resistance, flexibility, good refractory properties and ionic conductivity.

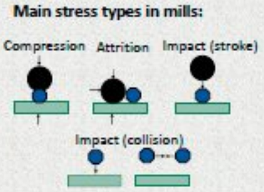
**A typical classification of nanomaterials synthesis methods:**



### Mechano-chemical process:

Chemical and physico-chemical changes of substances due to the influence of mechanical energy.

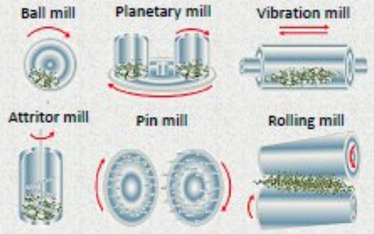




The less expensive, most simple and powerful way to produce nanoparticles at industrial scale.

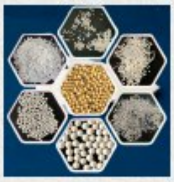
The properties of the nanomaterial obtained in a milling process and its subsequent heat treatment are function of several **parameters**:

➡ Type of the Mill



➡ Milling Media Material

Agate, Nylon, PTFE, PU, Zirconia, Stainless Steel



➡ **Ball to powder ratio**

*Weight ratio of the milling balls to the powder charge*

➡ **Milling time**

➡ Milling operating conditions (temperature, pressure and frequency)

➡ **Subsequent calcination temperature**

## RESULTS

**"The ball milling induced transformation of α-Fe2O3 powder in air and oxygen atmosphere"** by M.Zdujić et al.

Fig 1. XDR patterns of powders pre-milled for 7 h in air and subsequently milled in oxygen at different times, (○) α-$Fe_2O_3$; (●) $Fe_3O_4$; (□) $Fe_{1-x}O$ (BPMR=40).

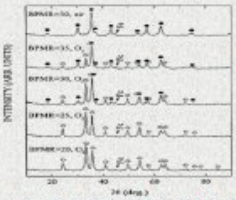


Fig 2. XDR patterns of powders milled for 5 h with various balls-to-powder mass ratio (BPMR), (○) α-$Fe_2O_3$; (●) $Fe_3O_4$.

**"Effect of ball milling time on the thermoelectric properties of p-type (Bi, Sb)2 Te3"** by J. H. Son et al.

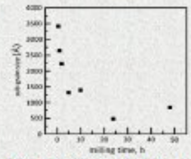


Fig 3. Estimated grain size of the p-type $(Bi,Sb)_2Te_3$ samples obtained in the milling process with Williamson-Hall equation.

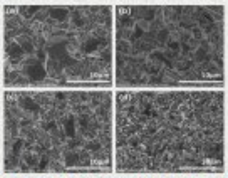
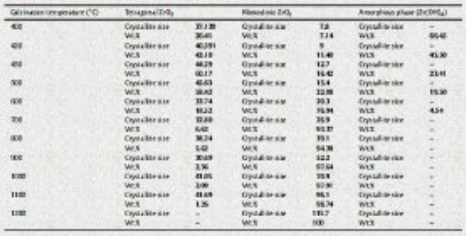


Fig 4. SEM images of fractured Surface of the bulk $(Bi,Sb)_2Te_3$ samples obtained from ball-milled powders from (a)0.5 h, (b) 5 h, (c) 10 h and (d) 48 h milling time.

**"Mechanochemical synthesis of zirconia nanoparticles: Formation mechanism and phase transformation"** by T. Dallali et al.

Table 1. Crystallite size and weight percent of the obtained zirconium hydroxide samples calcined at different temperatures for the corresponding phases.



| Calcination temperature (°C) | Tetragonal $ZrO_2$ | | Monoclinic $ZrO_2$ | | Amorphous phase ($Zr(OH)_4$) | |
|---|---|---|---|---|---|---|
| 400 | Crystallite size | [illegible] | Crystallite size | 7.8 | Crystallite size | – |
| | Wt.% | 36.45 | Wt.% | 7.14 | Wt.% | [illegible] |
| [illegible] | Crystallite size | [illegible] | Crystallite size | 9 | Crystallite size | – |
| | Wt.% | [illegible] | Wt.% | 11.48 | Wt.% | 45.30 |
| 450 | Crystallite size | 44.29 | Crystallite size | 12.7 | Crystallite size | – |
| | Wt.% | 60.17 | Wt.% | 16.42 | Wt.% | 23.41 |
| 500 | Crystallite size | [illegible] | Crystallite size | 15.4 | Crystallite size | – |
| | Wt.% | [illegible] | Wt.% | 22.08 | Wt.% | 19.30 |
| 600 | Crystallite size | 33.74 | Crystallite size | 30.3 | Crystallite size | – |
| | Wt.% | [illegible] | Wt.% | 76.94 | Wt.% | 4.54 |
| 700 | Crystallite size | 33.88 | Crystallite size | 35.9 | Crystallite size | – |
| | Wt.% | [illegible] | Wt.% | 93.37 | Wt.% | – |
| 800 | Crystallite size | 38.24 | Crystallite size | [illegible] | Crystallite size | – |
| | Wt.% | 5.62 | Wt.% | 94.38 | Wt.% | – |
| 900 | Crystallite size | [illegible] | Crystallite size | 32.2 | Crystallite size | – |
| | Wt.% | [illegible] | Wt.% | 97.64 | Wt.% | – |
| 1000 | Crystallite size | 41.05 | Crystallite size | [illegible] | Crystallite size | – |
| | Wt.% | [illegible] | Wt.% | [illegible] | Wt.% | – |
| 1100 | Crystallite size | [illegible] | Crystallite size | 98.1 | Crystallite size | – |
| | Wt.% | 1.26 | Wt.% | 98.74 | Wt.% | – |
| 1200 | Crystallite size | – | Crystallite size | [illegible] | Crystallite size | – |
| | Wt.% | – | Wt.% | 100 | Wt.% | – |

These results are providing new ideas to produce nanomaterials, which have not been extensively considered so far.

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13th YOUNG SCIENCE SYMPOSIUM 2019

# Gas-phase reactivity of acetone towards OH radicals at interstellar temperatures (11.7-64.4 K). Astrophysical Implications

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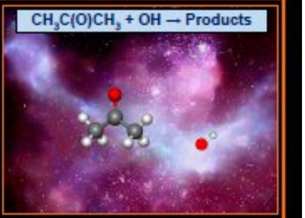
## 1. INTRODUCTION

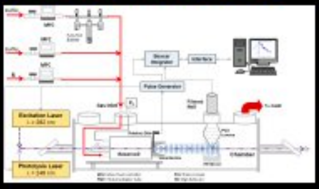
**Acetone ($CH_3C(O)CH_3$)** was detected for the first time in 1987 by Combes *et al.*[1] in the cold dense molecular cloud Sagittarius B2. More recently, its detection was confirmed by Snyder *et al.*[2] and acetone has been found in many other environments of the interstellar medium, such as Orion-KL hot core,[3] or Murchison meteorite.[4] **Hydroxyl radical (OH)** was detected for the first time in 1963 by Weinreb *et al.*[5] in Cassiopeia A, and later, in many other sources, such as Sagittarius B2.[6]

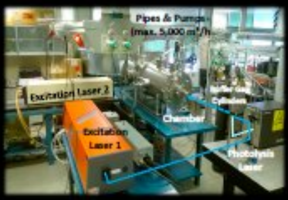
The purpose of our work is to determine **for the first time** the temperature dependence of the rate coefficient for the gas phase reactions of OH radicals with $CH_3C(O)CH_3$ at **very low temperatures**, between 11.7 and 64.4 K, and their potential pressure dependence at temperatures around 22, 50 and 64 K.

$CH_3C(O)CH_3 + OH \rightarrow$ Products



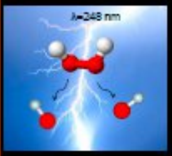
## 2. EXPERIMENTAL SET-UP



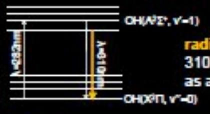


The **CRESU** (French acronym for *Cinétique de Réaction en Ecoulement Supersonique Uniforme* or *Reaction Kinetics in a Uniform Supersonic Flow*) technique was used to create a gas jet with a uniform temperature and gas density and the **pulsed laser photolysis/laser induced fluorescence** (PLP-LIF) technique was used to generate OH radicals and to monitor their temporal profile. The heart of the technique is the Laval Nozzle at the exit of which a uniform flow is formed.

## 3. THE PLP – LIF TECHNIQUE



For carrying out the kinetic experiment of the reaction, firstly the OH radicals have to be generated in situ. In this work, the **PLP** of **gaseous $H_2O_2$** at 248 nm was the **source of OH radicals.**



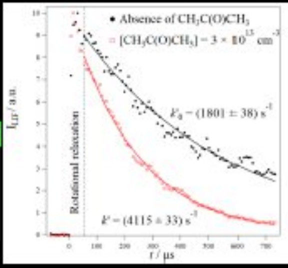
The **LIF from excited OH radicals** was detected at ca. 310 nm by a photomultiplier tube as a function of reaction time.

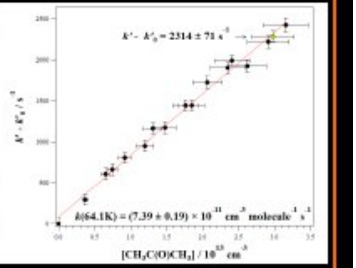
## 4. KINETICS OF THE $CH_3C(O)CH_3$ + OH REACTION

$$(I - I_{bck}) = (I_0 - I_{bck})e^{-k't}$$
$$\ln(I - I_{bck}) = \ln(I_0 - I_{bck}) - k't$$

$OH + CH_3C(O)CH_3 \rightarrow$ Prod. $k(T)$ — $k'$
$OH + H_2O_2 \rightarrow$ Prod. $k_{prec}$ — $k'_0$
$OH \rightarrow$ Other losses $k_{diff}$

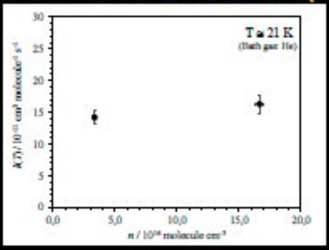
$$k' - k'_0 = k(T)\,[CH_3C(O)CH_3]$$

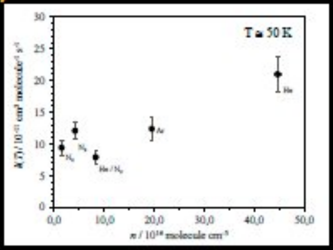


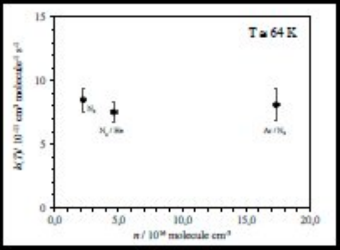


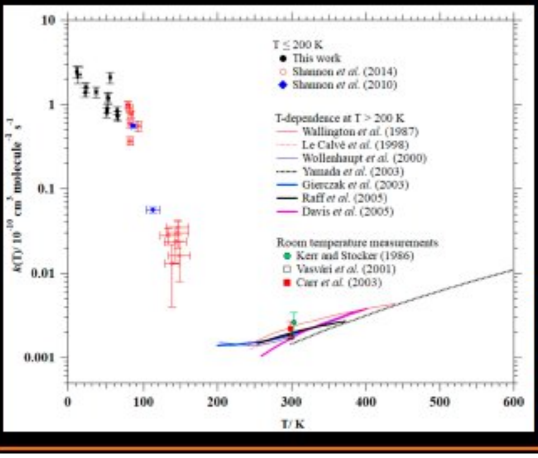
## 5. TEMPERATURE AND PRESSURE DEPENDENCE OF *k*(T)

| T(K) | *k*(T) ($10^{-11}$ cm³ molecule⁻¹ s⁻¹) |
|---|---|
| 11.7 ± 0.7 | 24.5 ± 1.7 |
| 13.0 ± 0.7 | 22.7 ± 2.3 |
| 21.1 ± 0.6 | 13.9 ± 0.5 |
| 21.7 ± 1.4 | 16.1 ± 0.6 |
| 36.2 ± 1.2 | 14.1 ± 0.7 |
| 50.5 ± 1.6 | 9.01 ± 0.25 |
| 51.6 ± 1.7 | 12.1 ± 0.4 |
| 49.9 ± 1.4 | 8.04 ± 0.33 |
| 52.1 ± 0.5 | 11.8 ± 0.8 |
| 54.2 ± 0.5 | 20.9 ± 1.3 |
| 64.2 ± 1.7 | 8.44 ± 0.21 |
| 64.1 ± 1.6 | 7.39 ± 0.19 |
| 64.4 ± 0.6 | 7.27 ± 0.40 |









## 6. CONCLUSIONS AND ASTROPHYSICAL IMPACT

The rate coefficients obtained in this work at temperatures around 22 K and 64 K reveal no pressure dependence between $3.37\times10^{16}$ and $16.7\times10^{16}$ cm⁻³, and between $2.24\times10^{16}$ and $17.4\times10^{16}$ cm⁻³ respectively, or bath gas dependence. At around 50 K, the rate coefficient at the largest density of $4.47\times10^{17}$ cm⁻³, is twice higher than the average values below that gas density, revealing a potential weak pressure dependence. This is somewhat surprising however because one would have expected finding a pressure dependence at 64 K as well since this has been observed by Shannon *et al.*[7] In the explored temperature range the experimental rate coefficient increases when decreasing temperature thus extending the trend observed by Shannon *et al.*[5] at temperatures higher than 79 K.

This new experimental data at ultra-low temperature for this reaction could be added to astrophysical databases, so it will help to improve the existing models to make them closer to the real reactivity in the ISM.

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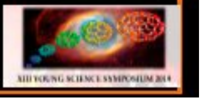
## ACKNOWLEDGMENTS

All authors from UCLM wish to thank ERC Project (NANOCOSMOS) for supporting this work.



# Mechanochemical synthesis of "Sweet Green Graphene"
# Part II

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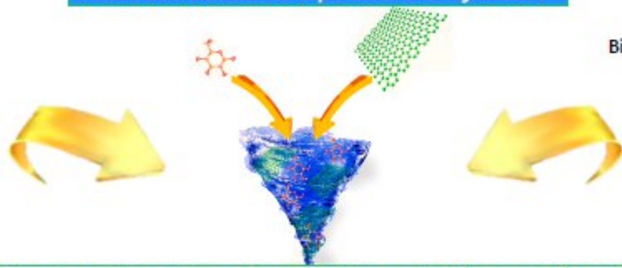
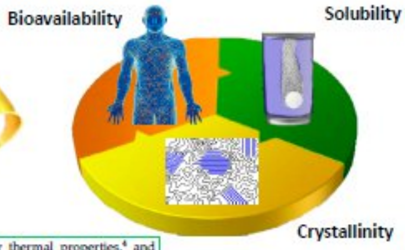
## INTRODUCTION

### Can we obtain Graphene Cocrystals?

### Graphene

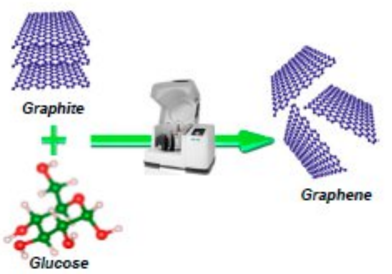


### Cocrystals

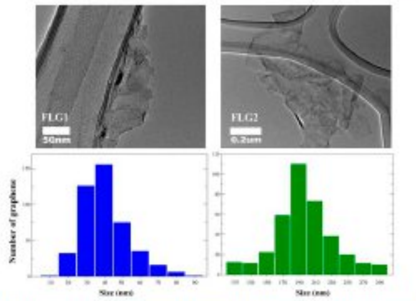


The use of cocrystals have solved problems in biology and chemistry,[1] including poor stability,[2] compressibility,[3] poor thermal properties,[4] and insufficient solubility.[5] Cocrystals are supramolecular structures of, at least, two neutral organic molecules via intermolecular interactions. These can be formed through mechanochemically treatments.[6] Mechanochemistry has also gained increasing importance in diverse fields of chemistry,[7] principally in the exfoliation and functionalization of nanomaterials like graphene.[8] We report the development of an environmentally friendly, cheap and simple approach for the synthesis of aqueous soluble few-layer graphene, useful for biological purposes, in the form of cocrystal with glucose.[9]

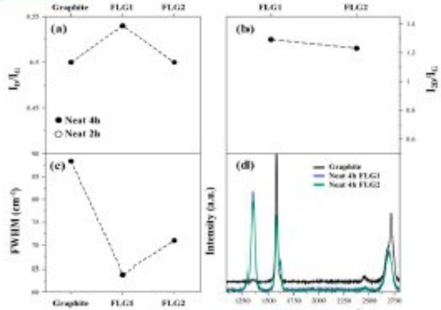
## SYNTHESIS OF GRAPHENE



**Neat 4 h** (75 mg of graphite and 4.5g of glucose and 4 h of ball milling)



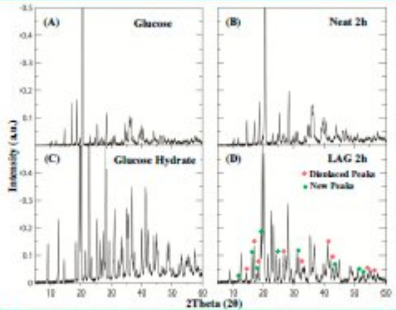
**Neat 4 h,** two different graphene batches, **FLG1** (50 nm) and **FLG2** (100-300 nm).



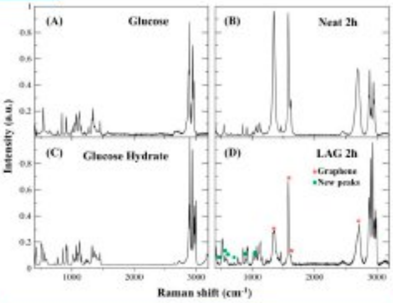
Raman of Neat 4h; (a) relation $I_D/I_G$ and $I_{2D}/I_G$ bands; (b) G and D band position; (c) FWHM of 2d band; and (d) Raman spectra for Graphite, FLG1 and FLG2.

## SYNTHESIS OF GRAPHENE COCRYSTAL

Mechanochemical synthesis for graphene cocrystal under **LAG** (liquid assisted gridding) conditions.



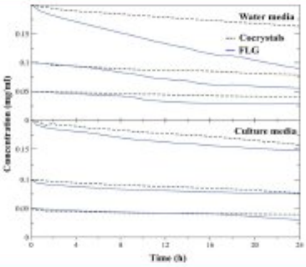
Powder X-Ray diffraction (PXRD) for glucose, glucose hydrate and Neat/LAG sample.



Raman for glucose, glucose hydrate and Neat /LAG conditions.

## CONCLUSION

We successfully applied a mechanochemical treatment with carbohydrates to exfoliate graphite in a simple and green approach. The addition of molar equivalents of water to the ball milling treatment allowed the formation of glucose-graphene co-crystals. Analogously to what happens with drugs, glucose-graphene co-crystals display reasonably dispersion in water, with optimal colloidal stability.



Colloidal stability using UV-Vis absorption spectroscopy at 660 nm of Graphene (Neat 4h) and Graphene Cocrystals. We observe a higher stability in aqueous solutions in the Graphene Cocrystal.

## THEORETICAL RESULTS

*Ab initio* studies of
"Sweet Green Graphene"
Part I
Dr. Antonio M. Rodríguez

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# Unusual ligand rearrangement of a *N*-phosphinoguanidinato ligand

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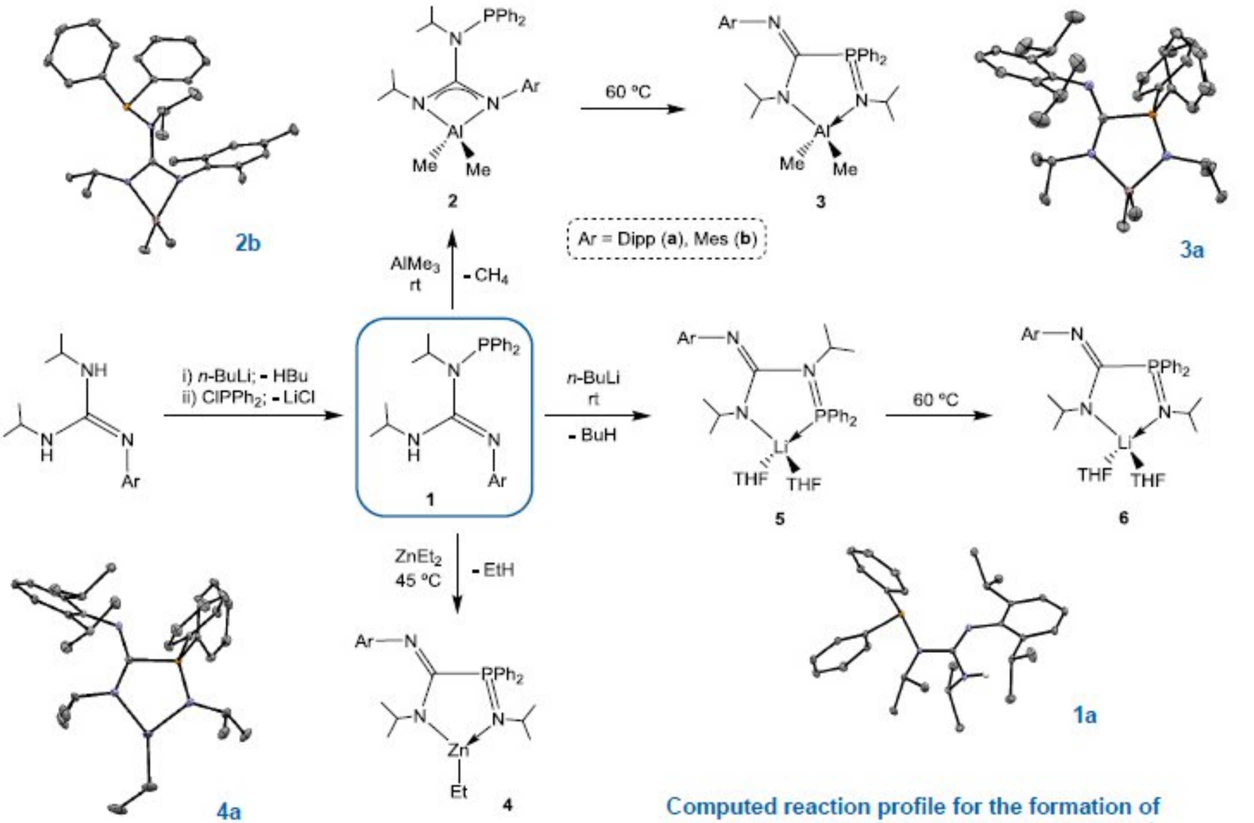
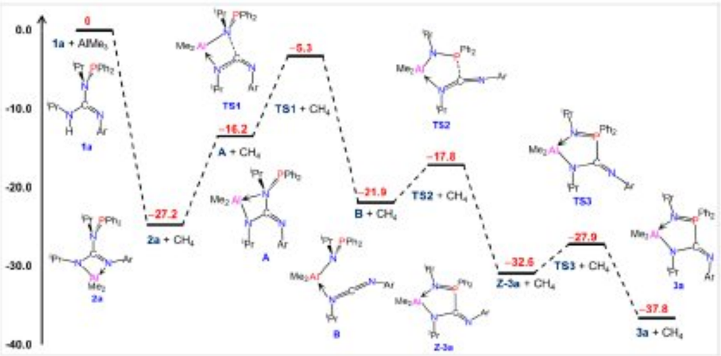
In the last years our group has focused on the development of catalytic methods to prepare guanidines and the coordination chemistry of guanidinato ligands with transition metals and main group elements.[1] As an extension of this work and encouraged by the lack of examples of anionic *N*-phosphinoguanidinato compounds, we aimed to prepare novel *N*-phosphinoguanidines from available trisubstituted guanidines to study their coordination chemistry through protonolysis reactions with different metal alkyls. Here we report the synthesis of *N*-phosphinoguanidines (**1**) and their reactivity towards metal alkyls.[2]



Unexpectedly, the reactions of **1** with metal alkyls lead to the formation of stable phosphinimine-amidinato compounds, after an unprecedented rearrangement under mild conditions of the initially formed *N*-phosphinoguanidinato intermediate.

We proposed a mechanism for this transformation in the reaction of **1a** with $AlMe_3$, supported by DFT calculations, involving cabodiimide de-insertion via a retro-[2,2] cycloaddition followed by a [3+2] dipolar regioselective cycloaddition.

**Computed reaction profile for the formation of complex 3a, Gibbs free energies in kcal·mol⁻¹**



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## FUNDING

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# RICH-SUGAR BIOMASS AS RAW MATERIAL FOR CATALYTIC CONVERSION OF GLUCOSE INTO BIOFUEL

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## ABSTRACT

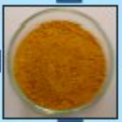
The continuous depletion of fossil fuel reserves has urged the current society to search for new energy feedstocks, not only for the necessity of new fuels, but also for the synthesis of chemicals. In addition, increasing concern about global warming has pointed out that these new energy feedstocks must be obtained according to environmentally friendly processes, which result in a reduction of atmospheric $CO_2$ emissions. Nowadays, biomass has emerged as the most affordable source of a wide range of chemical compounds,[1] being lignocellulosic biomass the main element. Lignocellulosic biomass is rich in carbohydrates, and therefore is a valuable starting point for their catalytic conversion into platform chemicals,[2] mainly 5-hidroxymethyfurfural (5-HMF) and levulinic acid (LA). Both 5-HMF and LA could be transformed into biofuels such as 2,5-dimethylfuran (2,5-DMF)[3] and γ-valerolactone,[4] respectively, via hydrogenation.

In this work we present the catalytic dehydration of glucose under microwave heating and their extraction from rich-sugar biomass, concretely Opuntia Ficus-Indica (OFI), aimed to develop an easy method to obtain 5-HMF and LA selectively from this starting material.

## SUGAR EXTRACTION

1st extraction
Solvent: cyclohexane

Starting material



Cyclohexane-soluble fraction

Cyclohexane extract (1% initial mass). Triacylglycerol fraction. Application in cosmetic or biodiesel synthesis

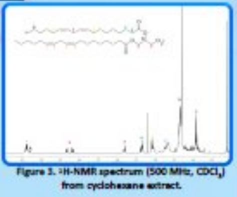


Figure 3. ¹H-NMR spectrum (500 MHz, $CDCl_3$) from cyclohexane extract.

Cyclohexane-insoluble fraction

2nd extraction
Solvent: Ethanol

Ethanol-soluble fraction

Ethanol extract (52% initial mass). Sugar fraction (mainly glucose and fructose). Future transformation into platform chemicals

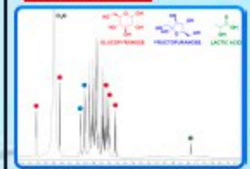


Figure 4. ¹H-NMR spectrum (500 MHz, $D_2O$) from ethanol extract.

Ethanol-insoluble fraction

3rd extraction
Solvent: Water

Water-insoluble fraction

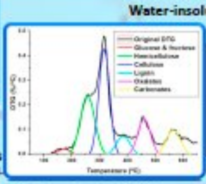


Figure 5. Final residue DTG deconvolution using Origin 9.1.

Final residue (22% initial mass). Insoluble fibres (cellulose, hemicellulose and lignin) and ashes

Water-soluble fraction

Water extract (25% initial mass) Water-soluble molecules such as pectin or starch

## SUPPORTED-CATALYSTS SYNTHESIS[6]

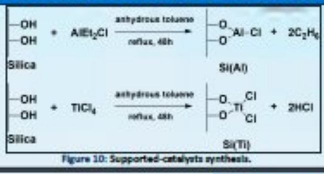


Figure 10: Supported-catalysts synthesis.

## FUTURE TRANSFORMATIONS

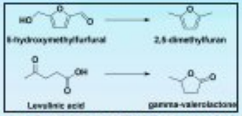


Figure 11: Transformations of 5-HMF and LA into biofuels.

## CONCLUSIONS

- Two biofuel precursors, 5-HMF and LA, were obtained under environmentally friendly conditions, such as microwave heating or water as solvent.
- Catalysts employed were easily recoverable.
- Supported-catalysts inhibited humins formation, which is the main drawback of $AlCl_3{*}6H_2O$.
- Biphasic system emerged as a promising way to obtain 5-HMF, which tend to rehydrate into LA in acidic aqueous conditions.
- An easy method to separate soluble fractions of biomass was developed, in order to apply each fraction in an appropriate way, obtaining the maximum profit.

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## PRELIMINARY CHARACTERISATION

Table 1. Composition of OFI fruit (% w/w, dry matter).[5]

| | PULP | SKIN | SEED |
|---|---|---|---|
| PROTEINS | 5% | 8% | 12% |
| LIPIDS | 1% | 2% | 7% |
| HEMICELLULOSE | 3% | 8% | 5% |
| CELLULOSE | 3% | 29% | 45% |
| PECTIN | 14% | 3% | 4% |
| ASH | 8% | 12% | 6% |
| GLUCOSE | 35% | 21% | 0% |
| FRUCTOSE | 26% | 3% | 0% |
| STARCH | 5% | 7% | 5% |

Pulp and skin were the starting material due to:
- High content in ethanol-soluble carbohydrates (glucose and fructose)
- Low content in fibers (hemicellulose and cellulose)

Fruits

Freeze-dryed, ground and sieved to remove seeds

Starting material

Thermogravimetric analysis

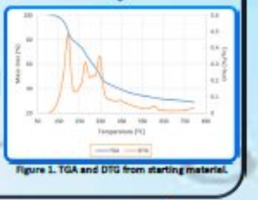


Figure 1. TGA and DTG from starting material.

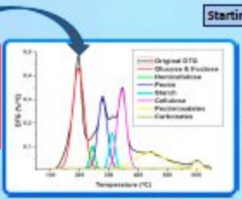


Figure 2. Starting material DTG deconvolution using Origin 9.1.

1st peak, which is the main mass loss, refers to glucose and frutose, corroborating that they are the major component

## MICROWAVE-ASSISTED GLUCOSE DEHYDRATION

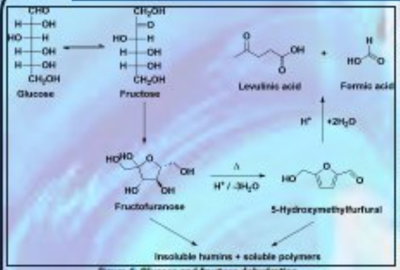


Figure 6: Glucose and fructose dehydration.

Table 2. Reaction conditions of microwave-assisted dehydration of glucose. Solvent: 2 mL of saturated water with inorganic salt.

| Entry | Catalyst | Catalyst loading $(w_{cat}/w_{glu})$ | Time (min) | Temperature (°C) |
|---|---|---|---|---|
| 1 | $AlCl_3{*}6H_2O$ | 0.5 | 10 | 160 |
| 2 | $AlCl_3{*}6H_2O$ | 0.5 | 10 | 180 |
| 3 | $AlCl_3{*}6H_2O$ | 0.5 | 10 | 200 |
| 4 | $AlCl_3{*}6H_2O$ | 0.5 | 20 | 160 |
| 5 | $AlCl_3{*}6H_2O$ | 0.5 | 30 | 160 |
| 6 | Si(Al) | 1 | 10 | 160 |
| 7 | Si(Al) | 1 | 10 | 170 |
| 8 | Si(Al) | 1 | 10 | 180 |
| 9 | Si(Al) | 1 | 20 | 170 |
| 10 | Si(Al) | 1 | 30 | 170 |
| 11 | Si(Ti) | 1 | 10 | 160 |
| 12 | Si(Ti) | 1 | 10 | 170 |
| 13 | Si(Ti) | 1 | 10 | 180 |
| 14 | Si(Ti) | 1 | 10 | 190 |

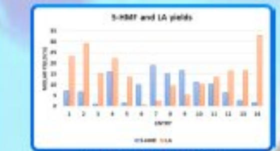


Figure 7: 5-HMF and LA yields.

PRELIMINARY RESULTS

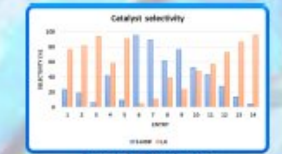


Figure 8: Catalyst selectivity.

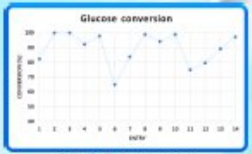


Figure 9: Glucose conversion.

Biphasic system was also tested using cyclopentil methyl eter (CPME) as organic phase

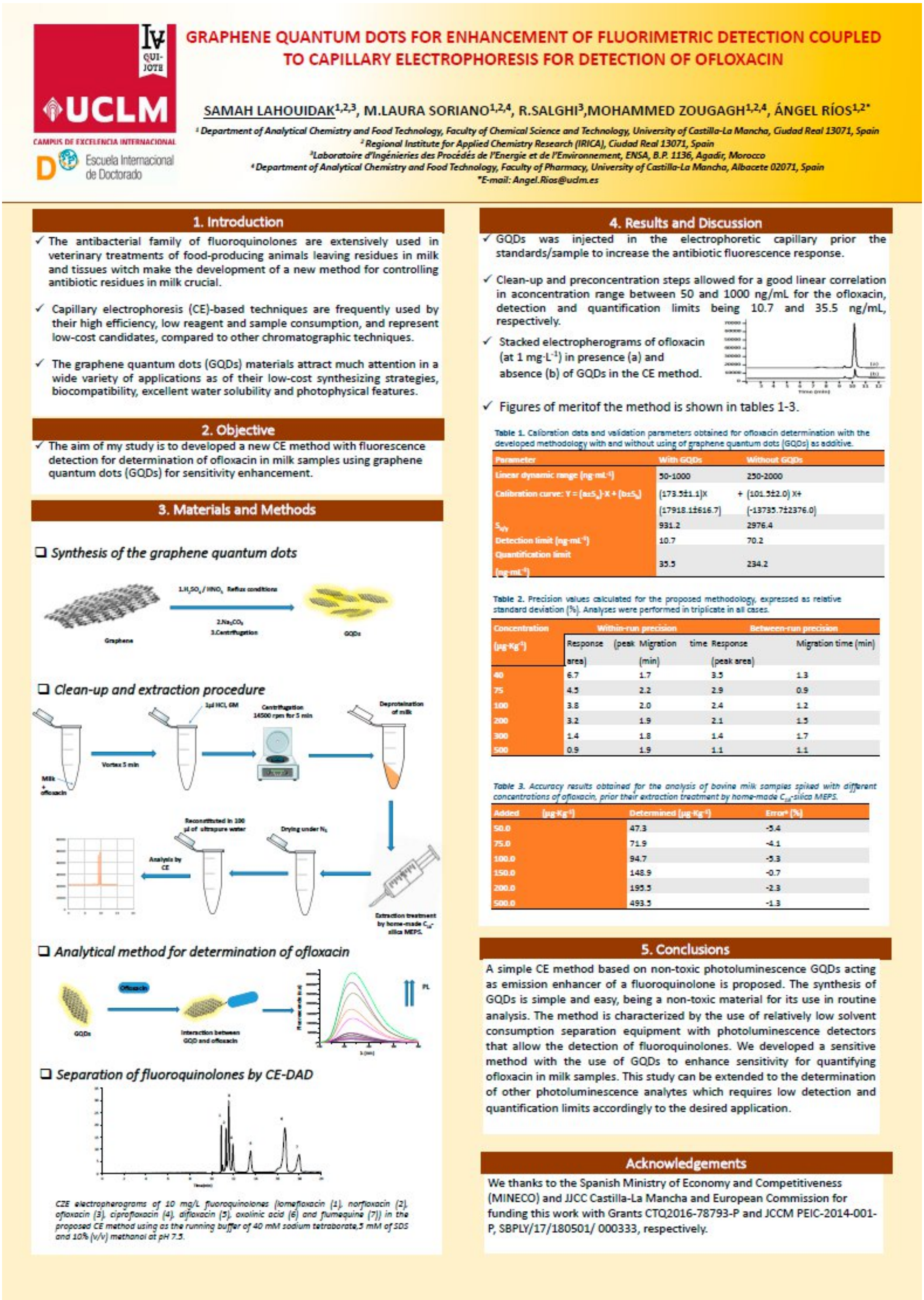
Table 3. Reaction conditions of microwave-assisted dehydration of glucose using biphasic system (1mL $H_2O$ saturated with inorganic salt, 3mL CPME). Reaction conditions: 160 °C, 10 min.

| Entry | Catalyst | Catalyst loading $(w_{cat}/w_{glu})$ | 5-HMF molar yield (%) | LA molar yield (%) | Selectivity 5-HMF (%) | Selectivity LA (%) | Glucose conversion (%) |
|---|---|---|---|---|---|---|---|
| 15 | $AlCl_3{*}6H_2O$ | 0.5 | 29 | 11 | 72.5 | 27.5 | 94.5 |
| 16 | Si(Ti) | 1 | 24 | 2 | 92.3 | 7.7 | 73.1 |

Biphasic system improves yields, selectivity and glucose conversion

## ACKNOWLEDGEMENTS

Thanks to MINECO for financial support (Project Ref: CGL2014-57087-R) and Junta de Comunidades de Castilla-La Mancha (JCCM) (Project Ref: SBPLY/17/180501/000522) for financial support and JCCM, Fondo Social Europeo (FSE) and Iniciativa de Empleo Juvenil (IEP) for the AJHA grant.



# GRAPHENE QUANTUM DOTS FOR ENHANCEMENT OF FLUORIMETRIC DETECTION COUPLED TO CAPILLARY ELECTROPHORESIS FOR DETECTION OF OFLOXACIN

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## 1. Introduction

✓ The antibacterial family of fluoroquinolones are extensively used in veterinary treatments of food-producing animals leaving residues in milk and tissues witch make the development of a new method for controlling antibiotic residues in milk crucial.

✓ Capillary electrophoresis (CE)-based techniques are frequently used by their high efficiency, low reagent and sample consumption, and represent low-cost candidates, compared to other chromatographic techniques.

✓ The graphene quantum dots (GQDs) materials attract much attention in a wide variety of applications as of their low-cost synthesizing strategies, biocompatibility, excellent water solubility and photophysical features.

## 2. Objective

✓ The aim of my study is to developed a new CE method with fluorescence detection for determination of ofloxacin in milk samples using graphene quantum dots (GQDs) for sensitivity enhancement.

## 3. Materials and Methods

### ☐ *Synthesis of the graphene quantum dots*

### ☐ *Clean-up and extraction procedure*

### ☐ *Analytical method for determination of ofloxacin*

### ☐ *Separation of fluoroquinolones by CE-DAD*

*CZE electropherograms of 10 mg/L fluoroquinolones (lomefloxacin (1), norfloxacin (2), ofloxacin (3), ciprofloxacin (4), difloxacin (5), oxolinic acid (6) and flumequine (7)) in the proposed CE method using as the running buffer of 40 mM sodium tetraborate,5 mM of SDS and 10% (v/v) methanol at pH 7.5.*

## 4. Results and Discussion

✓ GQDs was injected in the electrophoretic capillary prior the standards/sample to increase the antibiotic fluorescence response.

✓ Clean-up and preconcentration steps allowed for a good linear correlation in aconcentration range between 50 and 1000 ng/mL for the ofloxacin, detection and quantification limits being 10.7 and 35.5 ng/mL, respectively.

✓ Stacked electropherograms of ofloxacin (at 1 mg·L$^{-1}$) in presence (a) and absence (b) of GQDs in the CE method.

✓ Figures of meritof the method is shown in tables 1-3.

**Table 1.** Calibration data and validation parameters obtained for ofloxacin determination with the developed methodology with and without using of graphene quantum dots (GQDs) as additive.

| Parameter | With GQDs | Without GQDs |
|---|---|---|
| Linear dynamic range (ng·mL$^{-1}$) | 50-1000 | 250-2000 |
| Calibration curve: Y = (a±$S_a$)·X + (b±$S_b$) | (173.5±1.1)X +<br>(17918.1±616.7) | (101.5±2.0) X+<br>(-13735.7±2376.0) |
| $S_{x/y}$ | 931.2 | 2976.4 |
| Detection limit (ng·mL$^{-1}$) | 10.7 | 70.2 |
| Quantification limit (ng·mL$^{-1}$) | 35.5 | 234.2 |

**Table 2.** Precision values calculated for the proposed methodology, expressed as relative standard deviation (%). Analyses were performed in triplicate in all cases.

| Concentration (µg·Kg$^{-1}$) | Within-run precision | | Between-run precision | |
|---|---|---|---|---|
| | Response (peak area) | Migration time (min) | Response (peak area) | Migration time (min) |
| 40 | 6.7 | 1.7 | 3.5 | 1.3 |
| 75 | 4.5 | 2.2 | 2.9 | 0.9 |
| 100 | 3.8 | 2.0 | 2.4 | 1.2 |
| 200 | 3.2 | 1.9 | 2.1 | 1.5 |
| 300 | 1.4 | 1.8 | 1.4 | 1.7 |
| 500 | 0.9 | 1.9 | 1.1 | 1.1 |

*Table 3. Accuracy results obtained for the analysis of bovine milk samples spiked with different concentrations of ofloxacin, prior their extraction treatment by home-made $C_{18}$-silica MEPS.*

| Added (µg·Kg$^{-1}$) | Determined (µg·Kg$^{-1}$) | Error* (%) |
|---|---|---|
| 50.0 | 47.3 | -5.4 |
| 75.0 | 71.9 | -4.1 |
| 100.0 | 94.7 | -5.3 |
| 150.0 | 148.9 | -0.7 |
| 200.0 | 195.5 | -2.3 |
| 500.0 | 493.5 | -1.3 |

## 5. Conclusions

A simple CE method based on non-toxic photoluminescence GQDs acting as emission enhancer of a fluoroquinolone is proposed. The synthesis of GQDs is simple and easy, being a non-toxic material for its use in routine analysis. The method is characterized by the use of relatively low solvent consumption separation equipment with photoluminescence detectors that allow the detection of fluoroquinolones. We developed a sensitive method with the use of GQDs to enhance sensitivity for quantifying ofloxacin in milk samples. This study can be extended to the determination of other photoluminescence analytes which requires low detection and quantification limits accordingly to the desired application.

## Acknowledgements

We thanks to the Spanish Ministry of Economy and Competitiveness (MINECO) and JJCC Castilla-La Mancha and European Commission for funding this work with Grants CTQ2016-78793-P and JCCM PEIC-2014-001-P, SBPLY/17/180501/ 000333, respectively.

XIII YOUNG SCIENCE SYMPOSIUM 2019

# STIMULI RESPONSIVE HYDROGELS FOR DRUG DELIVERY APPLICATIONS

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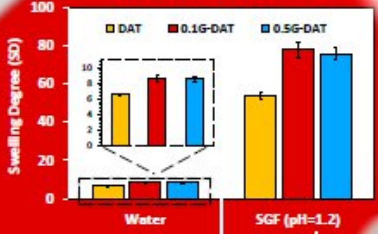
Diaminotriazines (DAT) are resourceful molecules that stand out for their ability to build supramolecular hydrophobic constructions. In addition, these structures respond to environmental changes, which has expanded the scope of DAT-based materials to the field of controlled delivery.[1,2]

In this work, we highlight triazine-based hydrogels as suitable responsive soft materials for drug delivery applications. By incorporating graphene (G) or magnetite nanoparticles (NPs) into the hydrogels, we have proven these materials successfully relase drugs in response to physiochemical stimuli such as magnetic fields, microwave radiation, or environmental pH.

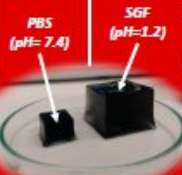
## pH responsive

### pH Swelling studies

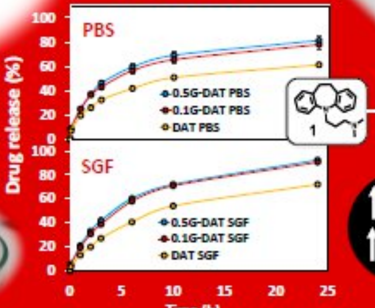


↑Graph ↑SD

↓pH ↑SD



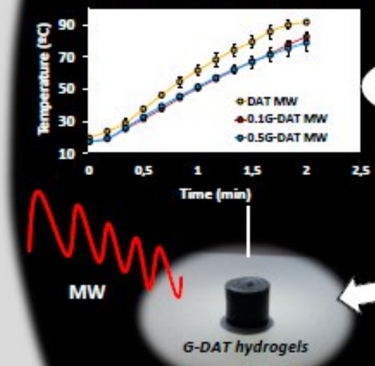
### pH-responsive drug delivery



↑Graph
↑Release

Graphene powder

### MW-temperature dependence

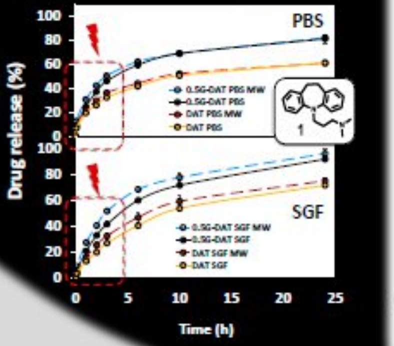


↑Graph ↓Tº(C)

Graphene powder

↑MW ↑Release

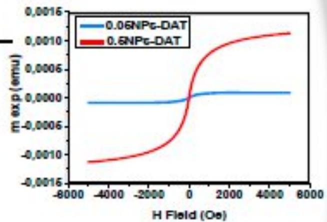
### MW-responsive drug delivery



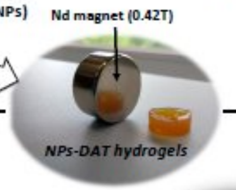
## Microwave responsive

Magnetite NPs:
-Superparamagnetic

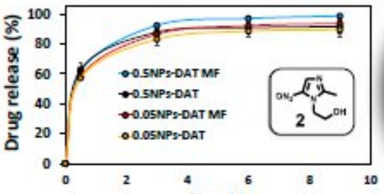
### Magnetic hysteresis loop



Magnetite Nanoparticles (NPs)



### Magnetic-responsive drug delivery



↑MF ↑Release

## Magnetically responsive

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[2] Wang P, Zhang J, Li Y, Wang N, Liu W.. *Mater Lett*. 2015;142:71-74.

*Acknowledgements*
*MINECO*
*Project: CTQ 2014-53600-R*
*Project: CTQ 2017-88158-R*

13 th Young Science Simposium

# GC-MS ANALYSIS OF THE CORKY OFF-FLAVOUR COMPOUNDS FROM PLANKS USED FOR PRODUCING WINE STOPPERS

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## INTRODUCTION

The cork is the closure chosen for wine bottles, and will represent one of the multiple factors that influence its quality. Due to the development of instrumental analytical techniques, such as gas chromatography and mass spectrometry, several scientists attributed to the cork certain off-flavours that sometimes appear in wine, with aromas of moisture and mould[1],[2]. This aroma defect was called "cork taint".

### GOAL

The objective of this work was to identify the corky off-flavour compounds in planks that will be used for producing wine stoppers.

## MATERIAL AND METHODS

**CORK PLANKS**
Gruart La Mancha, S.A.
(Valdepeñas, Ciudad Real)

SAMPLING PLAN

- 6 months
- 9 months
- 12 months

### EXTRACTION AND ANALYSIS OF VOLATILE COMPOUNDS

Dionex ASE 200
(Accelerated Solvent Extraction)

$CH_2Cl_2$ +

**GC-MS**
(Gas Chromatography-Mass Spectrometry)

SIM mode

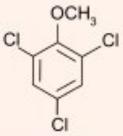
## RESULTS AND DISCUSSION

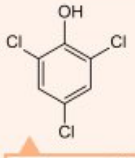
- Several compounds responsible for the sensory defect called "cork taint", as alkylmethoxypyrazines, chloroanisoles, chlorophenols, methylisoborneol and geosmin were detected in the newly harvested cork planks.
- "Cork taint" compounds concentration decreased during their storage (6, 9 and 12 months) outside the cork industry before processing.

**Table 1.** Off-flavour compounds identified in dichloromethane extracts of cork planks obtained by ASE. RI: retention index (DB-5).

| Compound | RI | Nomenclature | Identifier ions (m/z) | Quantifier ion (m/z) |
|---|---|---|---|---|
| MDMP | 1057 | 3,5-dimethyl-2-methoxypyrazine | 109, 138 | 138 |
| IPMP | 1078 | 3-isopropyl-2-methoxypyrazine | 124, 137, 152 | 137 |
| IBMP | 1170 | 3-isobutyl-2-methoxypyrazine | 94, 124, 151 | 124 |
| TCA | 1319 | 2,4,6-trichloroanisole | 169, 197, 212 | 197 |
| TCP | 1335 | 2,4,6-trichlorophenol | 132, 160, 196 | 132 |
| TeCA | 1517 | 2,3,5,6-tetrachlorosanisole | 203, 231, 246 | 203 |
| TeCP | 1552 | 2,3,4,6-tetrachlorophenol | 131,230,232 | 232 |
| TBA | 1619 | 2,4,6-tribromoanisole | 303, 331, 344 | 331 |
| TBP | 1624 | 2,4,6-tribromophenol | 222, 330, 332 | 330 |
| PCA | 1721 | pentachloroanisole | 237, 265, 280 | 265 |
| 2-MIB | 1158 | 2-methylisoborneol | 95, 107, 168 | 95 |
| Geosmin | 1373 | *t*-1,10-dimethyl-*t*-9-decalol | 112, 125, 182 | 112 |

2,4,6-Trichloroanisole





2,4,6-Trichlorophenol

Geosmin

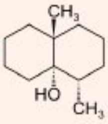
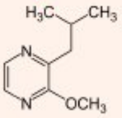
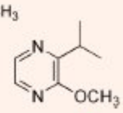


**Table 2.** Concentration (ng/g) of alkylmethoxypyrazines in cork planks during storage time before processing.

| Storage time before proccessing | Samples | MDMP | IPMP | IBMP |
|---|---|---|---|---|
| 6 months | S1 | 2.01 | 0.22 | 0.35 |
| | S2 | 0.32 | | 0.20 |
| | S3 | 0.29 | | 0.43 |
| | S4 | 1.77 | 0.30 | |
| | S5 | 0.55 | | 0.67 |
| 9 months | S1 | 1.71 | | |
| | S2 | | | |
| | S3 | | | |
| | S4 | 0.73 | | |
| | S5 | | | |
| 12 months | | - | - | - |

3,5-dimethyl-2-methoxypyrazine





3-isopropyl-2-methoxypyrazine

**Table 3.** Evolution of the concentration (ng/g) of chloroanisoles and chlorophenols in cork planks during storage time before processing.

| Storage time before proccessing | Samples | TCA | TCP | TeCA | TeCP | TBA | TBP | PCA |
|---|---|---|---|---|---|---|---|---|
| 6 months | S1 | | | 1.80 | 0.44 | 0.62 | 0.59 | 6.27 |
| | S2 | 0.28 | | 0.45 | 0.12 | 0.50 | 0.41 | 2.08 |
| | S3 | 0.19 | | 0.39 | 0.14 | 0.33 | 0.33 | 0.45 |
| | S4 | 0.30 | | 0.24 | 0.19 | 0.47 | 0.49 | 4.27 |
| | S5 | | | 0.36 | 0.28 | | 0.37 | 0.19 |
| 9 months | S1 | | | 0.50 | 0.17 | | 0.46 | 0.31 |
| | S2 | 0.45 | | | | | | 0.23 |
| | S3 | | | | 0.38 | | 0.36 | 0.12 |
| | S4 | | | | 0.25 | | 0.48 | 0.40 |
| | S5 | | | 0.39 | | | 0.52 | 0.18 |
| 12 months | S1 | | | | | | | 0.13 |
| | S2 | | | | | | 0.43 | 0.17 |
| | S3 | | | | 0.24 | | | 0.16 |
| | S4 | | | | | | | |
| | S5 | | | | | | 0.48 | 0.14 |

## CONCLUSION

**These results show the importance of storage stage of raw cork planks before their processing, because of the risk of contamination during these stage and as a result to control the formation of corky off-flavors compounds.**

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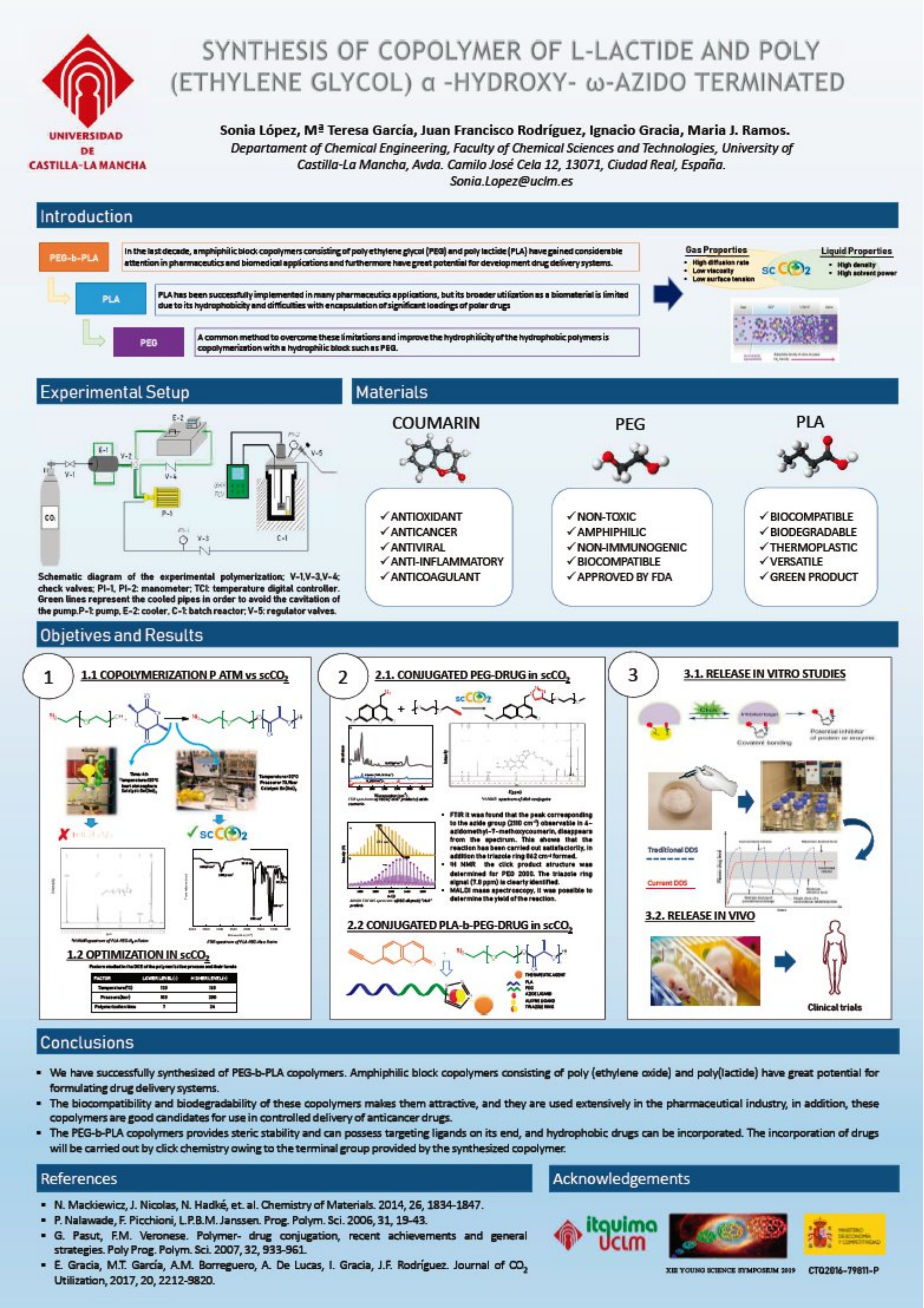
**AKNOWLEDGEMENTS**
The financial support of the assay was developed thanks to Junta de Comunidades de Castilla La Mancha (JCCM) for the project: SBPLY/17/180501/000445.



# SYNTHESIS OF COPOLYMER OF L-LACTIDE AND POLY (ETHYLENE GLYCOL) α -HYDROXY- ω-AZIDO TERMINATED

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## Introduction

**PEG-b-PLA** In the last decade, amphiphilic block copolymers consisting of poly ethylene glycol (PEG) and poly lactide (PLA) have gained considerable attention in pharmaceutics and biomedical applications and furthermore have great potential for development drug delivery systems.

**PLA** PLA has been successfully implemented in many pharmaceutics applications, but its broader utilization as a biomaterial is limited due to its hydrophobicity and difficulties with encapsulation of significant loadings of polar drugs

**PEG** A common method to overcome these limitations and improve the hydrophilicity of the hydrophobic polymers is copolymerization with a hydrophilic block such as PEG.

## Experimental Setup

Schematic diagram of the experimental polymerization; V-1,V-3,V-4: check valves; PI-1, PI-2: manometer; TCI: temperature digital controller. Green lines represent the cooled pipes in order to avoid the cavitation of the pump. P-1: pump, E-2: cooler, C-1: batch reactor; V-5: regulator valves.

## Materials

| COUMARIN | PEG | PLA |
|---|---|---|
| ✓ANTIOXIDANT<br>✓ANTICANCER<br>✓ANTIVIRAL<br>✓ANTI-INFLAMMATORY<br>✓ANTICOAGULANT | ✓NON-TOXIC<br>✓AMPHIPHILIC<br>✓NON-IMMUNOGENIC<br>✓BIOCOMPATIBLE<br>✓APPROVED BY FDA | ✓BIOCOMPATIBLE<br>✓BIODEGRADABLE<br>✓THERMOPLASTIC<br>✓VERSATILE<br>✓GREEN PRODUCT |

## Objetives and Results

### 1

### 1.1 COPOLYMERIZATION P ATM vs $scCO_2$

### 1.2 OPTIMIZATION IN $scCO_2$

[illegible]

| FACTOR | LOWER LEVEL(-) | HIGHER LEVEL(+) |
|---|---|---|
| Temperature(ºC) | 110 | 130 |
| Pressure(bar) | 100 | 200 |
| Polymerization time | 7 | 24 |

### 2

### 2.1. CONJUGATED PEG-DRUG in $scCO_2$

- FTIR it was found that the peak corresponding to the azide group (2110 $cm^{-1}$) observable in 4-azidomethyl-7-methoxycoumarin, disappears from the spectrum. This shows that the reaction has been carried out satisfactorily, in addition the triazole ring 862 $cm^{-1}$ formed.
- 1H NMR the click product structure was determined for PEG 2000. The triazole ring signal (7.8 ppm) is clearly identified.
- MALDI mass spectroscopy, it was possible to determine the yield of the reaction.

### 2.2 CONJUGATED PLA-b-PEG-DRUG in $scCO_2$

### 3

### 3.1. RELEASE IN VITRO STUDIES

### 3.2. RELEASE IN VIVO

## Conclusions

- We have successfully synthesized of PEG-b-PLA copolymers. Amphiphilic block copolymers consisting of poly (ethylene oxide) and poly(lactide) have great potential for formulating drug delivery systems.
- The biocompatibility and biodegradability of these copolymers makes them attractive, and they are used extensively in the pharmaceutical industry, in addition, these copolymers are good candidates for use in controlled delivery of anticancer drugs.
- The PEG-b-PLA copolymers provides steric stability and can possess targeting ligands on its end, and hydrophobic drugs can be incorporated. The incorporation of drugs will be carried out by click chemistry owing to the terminal group provided by the synthesized copolymer.

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## Acknowledgements

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13th YOUNG SCIENCE SYMPOSIUM,
FACULTY OF CHEMICAL SCIENCES AND TECHNOLOGIES
**5-7 de Junio de 2019** | Campus Ciudad Real, UCLM

# POTENTIAL OF DIFFERENT OENOLOGICAL PRODUCTS TO INHIBIT 1-HYDROXYETHYL RADICAL IN WINE

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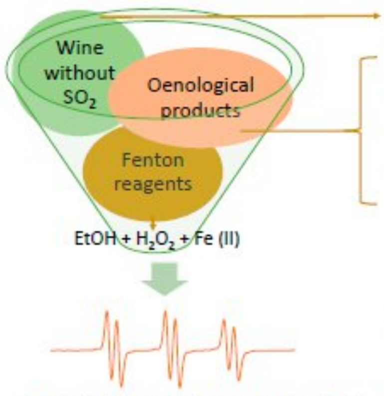
## INTRODUCTION

The elaboration of wines without $SO_2$ is a complex challenge, since the free radicals produced in wine oxidation reactions can cause a great deterioration in its composition and organoleptic characteristics.

Among the free radicals formed during the oxidation of wine, it has been shown that the 1-hydroxyethyl radical is the most abundant and could be the main agent responsible for the oxidation of other organic substances in wine.

The objective of this work was to study the potential of different natural oenological products to inhibit 1-hydroxyethyl radical in wines without $SO_2$.

## MATERIAL AND METHODS

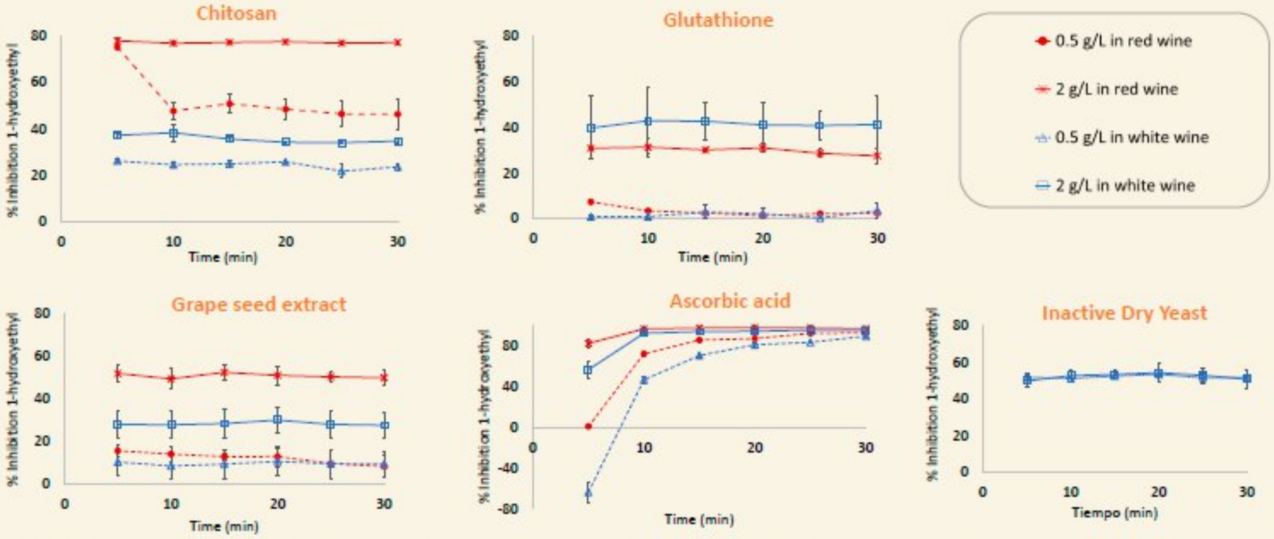


Sauvignon Blanc
Cabernet Sauvignon

Ascorbic acid
Glutathione
Chitosan
Inactive Dry Yeast
Grape seed extract
( 0.5 and 2.0 g /L )

1-HYDROXYETHYL RADICAL ANALYSIS BY ELECTRON PARAMAGNETIC RESONANCE USING POBN AS SPIN TRAP

## RESULTS AND DISCUSSION



- The addition of grape seed extract and chitosan offered higher percentages of radical inhibition in red wine.
- The ascorbic acid at 0.5 g/L showed a pro-oxidant effect at the beginning of the reaction in white wine.
- The effect of the inactive dry yeast to inhibit the radical was only observed in white wine.

**❖ The efficacy of the oenological products studied to inhibit the 1-hydroxyethyl radical in wines without $SO_2$ is influenced by both the concentration used and the type of wine.**

ACKNOWLEDGMENTS: This work has been carried out with the financing of INIA under the projects : RTA2014-00055-C03-01 y RTA2014-00055-C03-02.

# Degradation of antibiotics by ElectroFenton process

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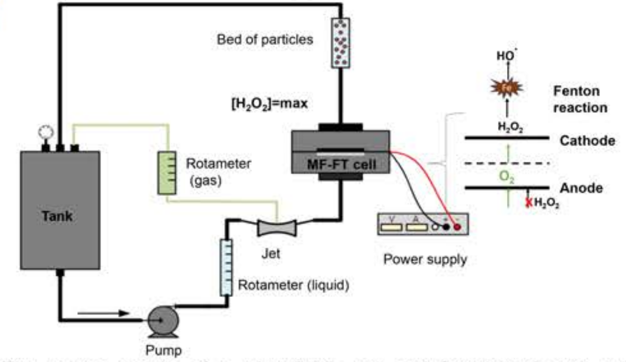
## Introduction

Currently, there is a global environmental problem related to the presence of pharmaceutical compounds in wastewater. One of the main sources of these pollutants are hospitals, which generate a large volume of effluents every day. Among them stand out, not for the volume generated but for its concentration, the urine of the patients that presents a high content of antibiotics, antipyretics, hormones, etc. The conventional processes used in wastewater treatment plants have a low efficiency to eliminate this type of contaminants, causing the emergence of bacteria resistant to antibiotics. Therefore, hospital effluents must be adequately treated to protect both the environment and human health, since there is no legislation governing the concentration limits of drugs in water [5].

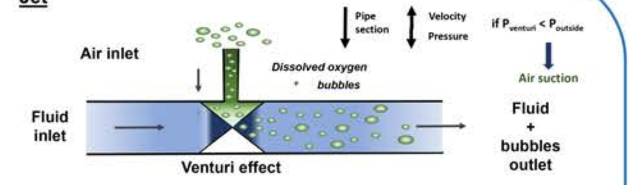
Based on this, in this work the heterogeneous electroFenton process has been chosen for the selective elimination of pharmaceutical compounds in urine medium. Urine is a very complex matrix, which contains organic components and inorganic salts that hinder the process.

## Setup diagram

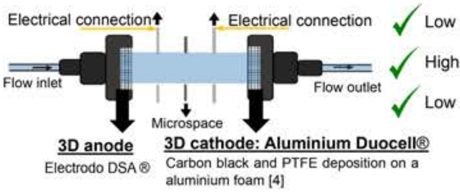


The system consits of a micro-fluidic cell and flow-through with a jet aeration, to supply large amounts of oxygen at low pressure to efficiently generate $H_2O_2$. In addition, it incorporates a fluidized-bed of iron particles (goethita) for catalyze the descomposition of $H_2O_2$ into oxidizing species.

### Jet



### Micro-fluidic cell and flow-through



**3D anode**
Electrodo DSA®

**3D cathode: Aluminium Duocell®**
Carbon black and PTFE deposition on a aluminium foam [4]

✓ Low ohmic resistance
✓ High mass-transfer
✓ Low energy consumption

## Results

### Influence of the current densitie in the generation of $H_2O_2$

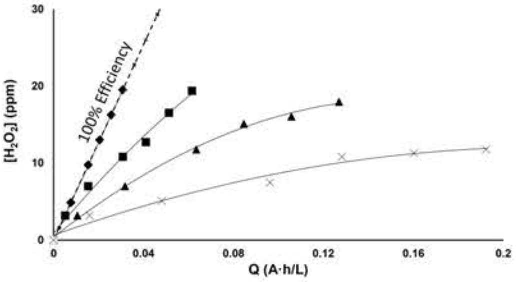


***Figure 1.*** *Hydrogen peroxide generation at different current densities in the MF-FT reactor. ◆j=5 mA/cm³ ■ j= 10 mA/cm³ ▲ j=20 mA/cm³ X j=30 mA/cm³. $Q_v$=140 L/h. $Q_{air}$=100 NL/h. T=25ºC. pH 3. V=2.5 L. P=1 atm.*

### Degradation of Chloramphenicol in urine. Generation of acids.

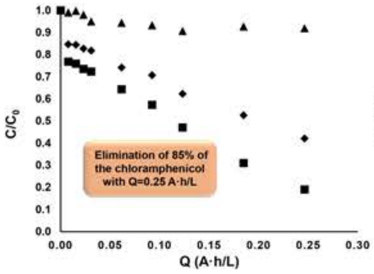


***Figure 2.*** *Chloramphenicol and compounds of the urin in heterogeneous EF . ◆ Chloramphenicol ■ Uric acid ▲ Creatinine j=5 mA/cm³, [Goethita]= 4 g/L. $Q_v$=140 L/h. $Q_{aire}$=100 NL/h. T=25ºC. pH 3. V=2.5 L. P=1 atm. 50 ppm of Chloramphenicol$_0$.*

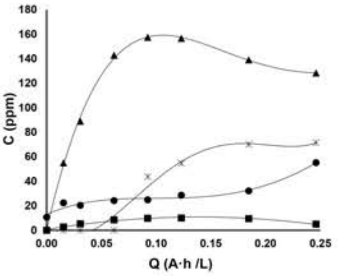


***Figure 3.*** *Carboxylic acid generated in the process.* ▲ Formic acid ● Oxalic acid ■ Oxamic acid, Ж Propionic acid.

## Conclusions

- El jet multiplies the aeration capacity at a given pressure and increases $H_2O_2$ production rate.
- Specific energy consumption of the MF-FT + Jet for the production of $H_2O_2$ is the lowest reported so far (4 kWh/kg $H_2O_2$).
- An excess of applied current density results counterproductive in terms of $H_2O_2$ generation (Figura 1).
- Heterogeneous ElectroFenton allows the elimination of chloramphenicol with a high selectivity, keeping most of the components of the urine unaltered. On the other hand, it is posible to degrade 58% of the chloramphenicol by applying load less than 0.3 A·h /L.

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## Acknowledgements

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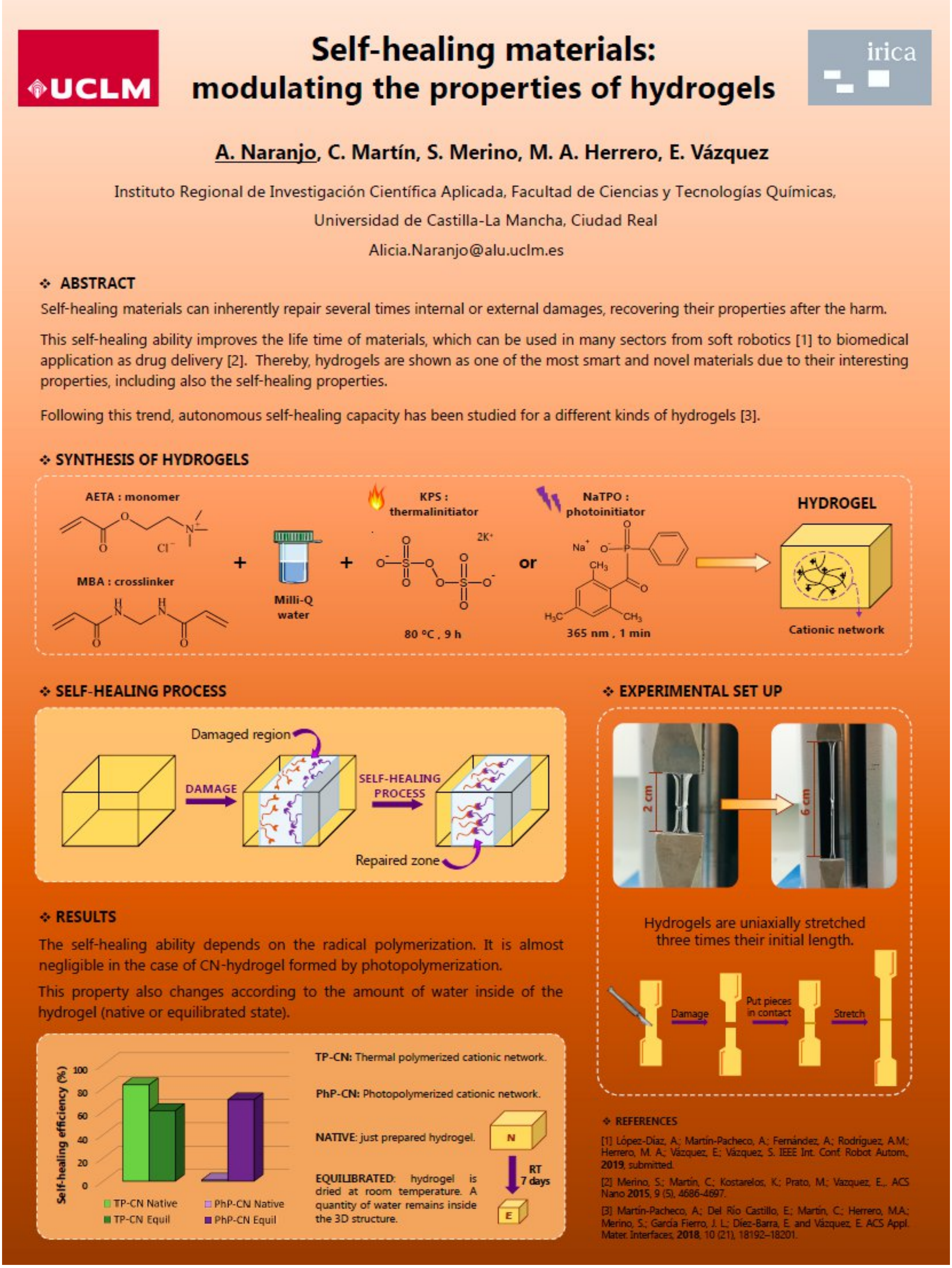

# Self-healing materials: modulating the properties of hydrogels

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## ❖ ABSTRACT

Self-healing materials can inherently repair several times internal or external damages, recovering their properties after the harm.

This self-healing ability improves the life time of materials, which can be used in many sectors from soft robotics [1] to biomedical application as drug delivery [2]. Thereby, hydrogels are shown as one of the most smart and novel materials due to their interesting properties, including also the self-healing properties.

Following this trend, autonomous self-healing capacity has been studied for a different kinds of hydrogels [3].

## ❖ SYNTHESIS OF HYDROGELS

## ❖ SELF-HEALING PROCESS

## ❖ EXPERIMENTAL SET UP

Hydrogels are uniaxially stretched three times their initial length.

## ❖ RESULTS

The self-healing ability depends on the radical polymerization. It is almost negligible in the case of CN-hydrogel formed by photopolymerization.

This property also changes according to the amount of water inside of the hydrogel (native or equilibrated state).

**TP-CN:** Thermal polymerized cationic network.

**PhP-CN:** Photopolymerized cationic network.

**NATIVE**: just prepared hydrogel.

**EQUILIBRATED**: hydrogel is dried at room temperature. A quantity of water remains inside the 3D structure.

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DEPARTAMENTO DE INGENIERÍA QUÍMICA

# Catalytic ammonia decomposition over Ru/SiC

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## INTRODUCTION

Catalytic ammonia decomposition in order to generate hydrogen has been used for energy production. Different catalysts have been **synthesized**, using ruthenium (load 1-7.5% w/w) supported over silicon carbide (SiC).

## EXPERIMENTAL

- **Catalysts synthesis:** incipient wetness impregnation and followed by overnight drying at 80 °C.

| Calcination | |
|---|---|
| $N_2$-Ar (100ml·min$^{-1}$, 50:50) | 500 °C x 2h, 10 °C·min$^{-1}$ |
| **Reduction** | |
| $H_2$-Ar (100ml·min$^{-1}$, 50:50) | 600 °C x 1h, 10 °C·min$^{-1}$ |
| | 400 °C x 1h, 10 °C·min$^{-1}$ |

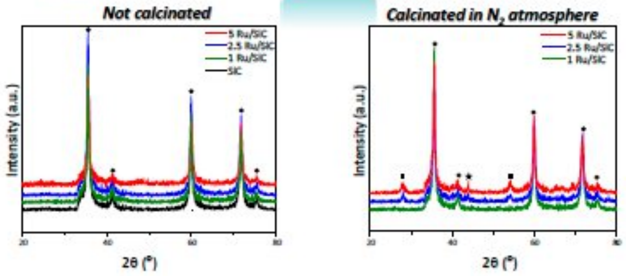
- **Catalytic testing:**

-$NH_3$-Ar: 100 ml·min$^{-1}$, 10:90
-GHSV: 60000 ml/g·h
-Products analyse by Gas Chromatography (GC)

## RESULTS

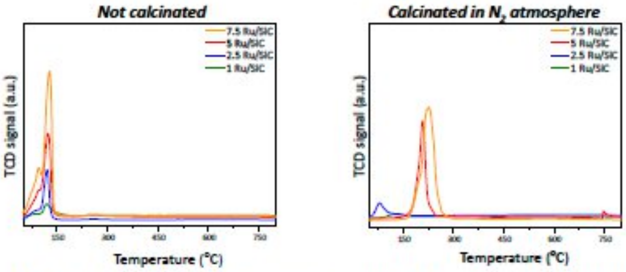
### XRD

• β-SiC support
■ $RuO_2$
★ Ru



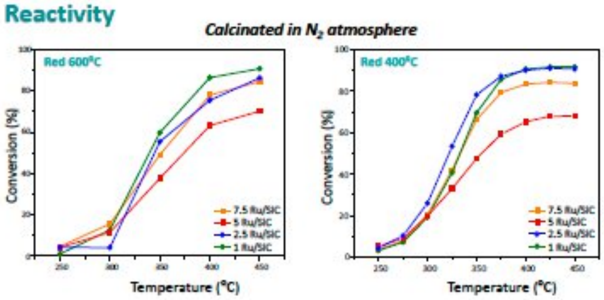
The characterization by XRD (X-Ray Power Diffraction) of the catalysts not calcinated shows that the Ru is very dispersed, only signals of silicon carbide are observed. However, Ru and $RuO_2$ are observed in catalysts calcinated in nitrogen atmosphere.

### TPR-$H_2$

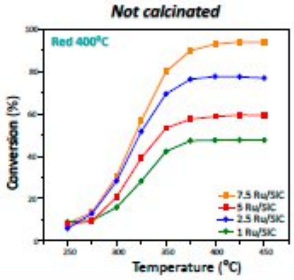


As it can be observed, Ru is very easily reduced ($Ru^{3+}$→$Ru^0$) at 120 °C if it was not calcinated. On the other hand, the catalysts calcinated in $N_2$ show a peak related with reduction of $Ru^{3+}$ at higher temperature (245 °C).

### Reactivity



The decrease in reduction temperature improves the catalytic activity in the catalysts with low loading of ruthenium.
Nevertheless, if the catalysts were not calcinated, the 7.5% Ru/SiC catalysts presents the best behaviour.



## CONCLUSION

In this context, taking into account the **Ru content**, the most favourable catalyst is the one that presents lower load of metal, one step of calcination and reduction at 400 °C. So the best performance was achieved by the 1%Ru/SiC catalyst calcinated in $N_2$. In this case, 95% of conversion was reached at 400 °C.

## ACNOWLEDGMENTS

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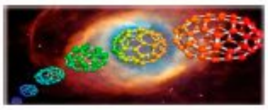

# 2-(2-HYDROXYPHENYL)-4,6-STYRYLPYRIMIDINES. SYNTHESIS AND OPTICAL PROPERTIES

**Rodrigo Plaza-Pedroche**, **Verónica Muñoz-Canales and Julián Rodríguez-López**

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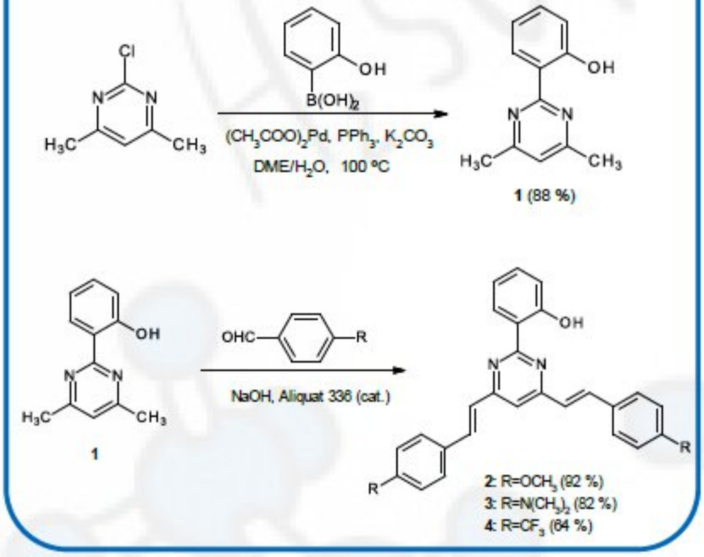
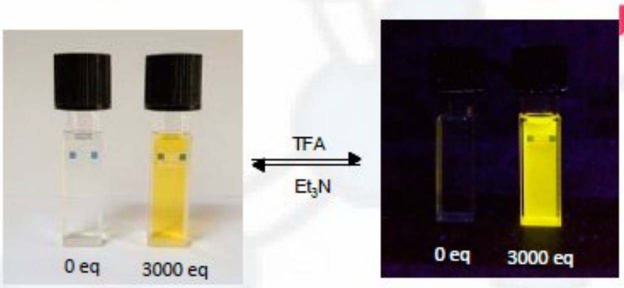
## ABSTRACT

In this work, a series of 2-(2-hydroxyphenyl)-4,6-styrylpyrimidines has been efficiently prepared by a combination of Suzuki and aldol condensation reactions. Their optical absorption and emission properties were studied in different solvents and media. The abilities of these molecules to function as colorimetric and luminescence pH sensors were demonstrated with color changes and luminescence switching upon the introduction of acid.

## SYNTHESIS



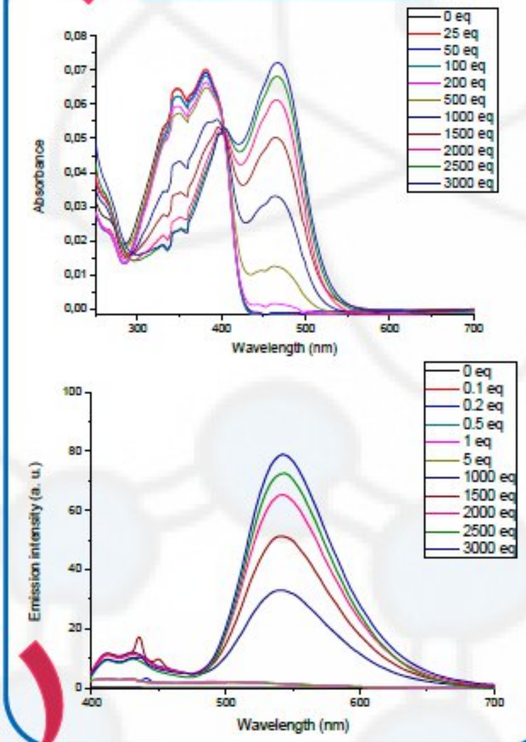
## CHANGES IN THE COLOR OF A $CH_2Cl_2$ SOLUTION OF 3 AFTER ADDITION OF TFA UNDER VISIBLE (LEFT) AND UV LIGHT (RIGHT)



Visible

Uv ($\lambda_{em}$= 365 nm)

## TITRATION CURVES OF A $CH_2Cl_2$ SOLUTION OF 3 WITH TFA



## CONCLUSIONS

The route of synthesis was straightforward. The compounds display reversible color changes and luminescence switching upon addition of acid that could enable the development of new colorimetric and luminescence pH sensors.

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# Membrane-less electrolyzer: a new concept for $H_2$ production via alcohol electrochemical reforming

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## INTRODUCTION

The increasing demand of worldwide energy joint to the environmental problems encourages the development of clean and renewable energy such as hydrogen.

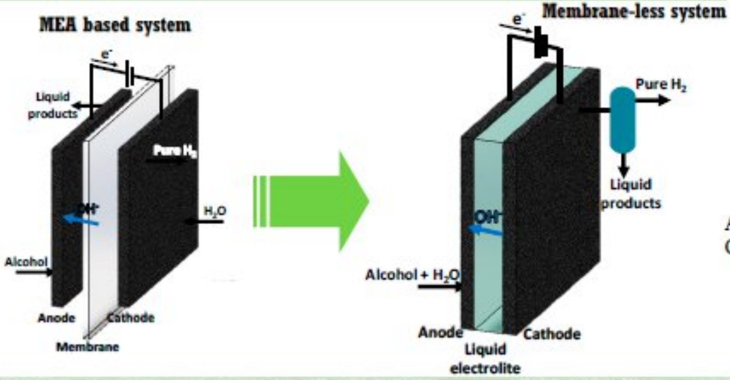
**WATER ELECTROLYSIS**

- ✓ Most stablished technology
- ✗ High cell voltages → high energy consumption [1]

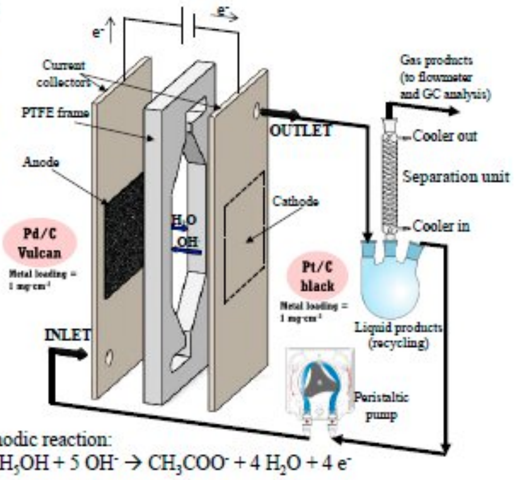
**ALCOHOL ELECTROCHEMICAL REFORMING**

- ✓ High energy contained → lower energy consumption

**SIMPLE AND INTEGRATED FREE CARBONACEOUS $H_2$ PRODUCTION SYSTEM**



## EXPERIMENTAL



Anodic reaction:
$C_2H_5OH + 5\,OH^- \rightarrow CH_3COO^- + 4\,H_2O + 4\,e^-$

Cathodic reaction:
$2\,H_2O + 2\,e^- \rightarrow H_2 + 2\,OH^-$

## RESULTS

### Proof of concept

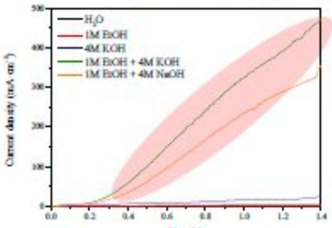


Figure 2. Linear sweep voltammetry measurements in a 0-1.4 V range. Scanrate = 10 mV·s-1, T = 85 °C.

**ELECTROCATALYTIC ACTIVITY**
⬇
**ETHANOL ELECTRO-OXIDATION CONTRIBUTION**
✚
**LIQUID ELECTROLYTE**

- OH- TRANSPORTATION PATHWAY
- HIGH IONIC CONDUCTIVITY

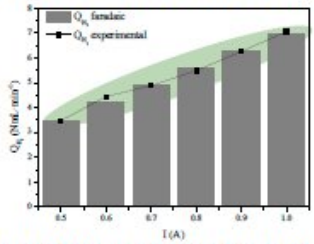


Figure 3. Galvanostatic transients: $H_2$ production rates vs current applied. T = 25 °C

**PURE AND FARADAIC HYDROGEN**

Figure 4. Gas chromatograph of the outlet gas phase from measurement obtained at 1 A galvanostatic transient (Figure 3).

### Influence of the membrane-less electrolyzer conditions

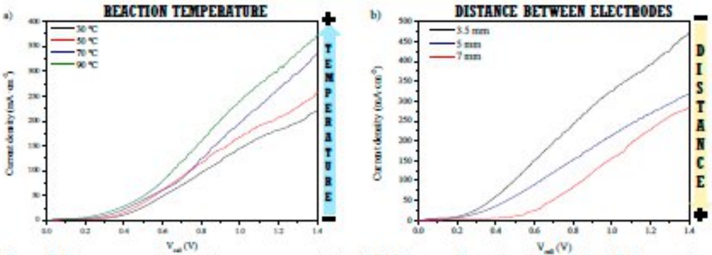


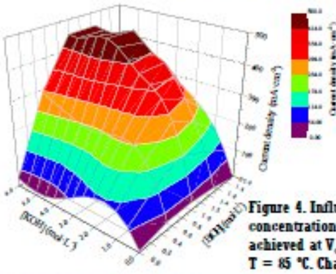
Figure 3. Linear sweep voltammetry measurements in a 0-1.4 V range. Scanrate = 10 mV·s-1. a) Influence of the temperature. b) Influence of the chamber width.

**⬆ TEMPERATURE ⬆ ELECTROCATALYTIC ACTIVITY**

- INCREASE OF THE IONIC CONDUCTIVITY OF THE ELECTROLYTE
- ENHANCEMENT OF THE KINETICS OF THE ELECTROCHEMICAL REACTIONS

**⬇ DISTANCE ⬆ ELECTROCATALYTIC ACTIVITY**

- DECREASE OF THE OHMIC LOSSES OF THE SYSTEM



**0 mol·L-1 — ETHANOL CONCENTRATION — 2 mol·L-1**

INSUFFICIENT FUEL SUPPLY
MASS TRANSFER LIMITATIONS

LOW IONIC CONDUCTIVITY OF THE SOLUTION
UNAVAILABILITY OF OH- IONS AT CATALYST SITES

**6 mol·L-1 — KOH CONCENTRATION — 0 mol·L-1**

HIGH SOLUTION VISCOSITY
LARGE OH- COVERAGE OF THE ACTIVE SITES

LOW IONIC CONDUCTIVITY OF THE SOLUTION

Figure 4. Influence of EtOH and KOH concentration vs. current density achieved at $V_{cell}$ = 1.4 V. T = 85 °C. Chamber width = 3.5 mm.

**OPTIMIZED EXPERIMENTAL CONDITIONS**
1 mol·L-1 EtOH + 4 mol·L-1 KOH
TEMPERATURE = 85 °C
CHAMBER WIDTH = 3.5 mm

## CONCLUSIONS

This membrane-less configuration may simplify the further scale up, enhancing the stability of the system due the lack of a polymer electrolyte membrane. Under optimized conditions, pure hydrogen can be obtained with a lower energy consumption ( 16.9 $kWh·kg^{-1}_{H2}$) than reported in previous studies.

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## ACKNOWLEDGMENTS

GOBIERNO DE ESPAÑA
MINISTERIO DE ECONOMÍA Y EMPRESA

# Organogels from 2*H*-benzo[*d*]1,2,3-triazole derivatives. Application in crystallization of drugs.

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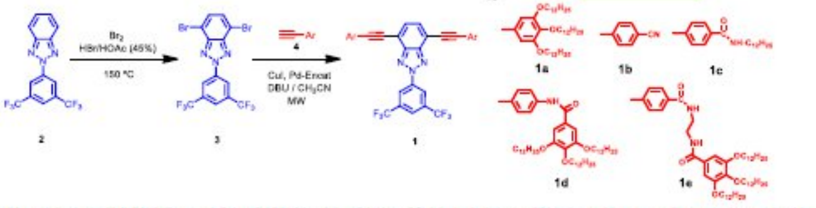
## Abstract



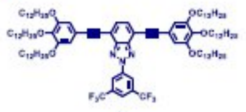
**Scheme 1.** Synthesis of 2-(3,5-bis(trifluoromethyl)phenyl)-4,7-bis(phenylethynyl)-2*H*-benzo[*d*]1,2,3-triazole derivatives (**1**) by a Sonogashira cross-coupling reaction between **3** and **4**.

2*H*-Benzo[*d*]1,2,3-triazole derivatives have been previously tested as **optical waveguides**[1], **OFETs**[2], and **polymers in bioimaging**[3]. In this work, benzotriazole derivatives with a range of chemical functionalities form **organogels**. Noteworthy, bis-amide derivative **1e** gave robust thermally stable gels in a range of solvents down to 0.1 wt%.

The gels have also been showed to be useful **vehicles to crystallize pharmaceutical drugs**, resulting in a change in the polymorphism in the case of sulfathiazole.

## Results and discussion

### Gelation tests



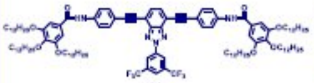
1a

**2% wt**

1. 1,4-butanediol
2. 2-propanol
3. 1-pentanol
4. Methanol

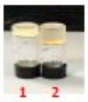
**1% wt**

1. 1-pentanol
2. 1,4-butanediol

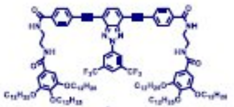


1d

**2% wt**

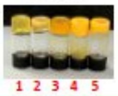


1. DMF
2. Diethylene glycol



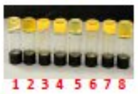
1e

**2% wt**



1. DMF
2. Ethanol
3. Methanol
4. 1-butanol
5. 1-propanol

**1% wt**



1. Ethanol
2. Methanol
3. Hexane
4. 2-butanol
5. 1-propanol
6. 1-butanol
7. 2-propanol
8. 1,4-butanediol

Gelation tests were carried out for compounds **1** using **thirty different** solvents and at **different concentrations** (2% wt, 1% wt and 0.5% wt). Gelators were dissolved in 0.5 mL of the respective solvent through gentle heating followed by sonication for 1 min until complete dissolution. The vials were then kept undisturbed at room temperature and were checked after 4, 24, 48 and 72 hours using **the simple tube inversion test to assess** the flow properties of the resulting mixture.

**0.5% wt**

1. Ethanol
2. 1-propanol
3. 1-butanol
4. 2-butanol
5. 2-propanol
6. 1,4-butanediol

### CGC

The critical gelation concentration (CGC) is the minimum amount of gelator which is needed to obtain a gel in a specific solvent.

1a → 0.7-1.7 % wt

1d → 1.5-1.8 % wt

1e → 0.1-1.8 % wt SUPERGELATOR

### $T_{sol}$

The gel to sol phase transition temperature, (Tsol) is the temperature when the gel breaks down, becoming solution again.

1a → 62-105 °C

1d → 45-53 °C

1e → 70-178 °C

The strong hydrogen bonds between the protons of N–H groups and interdigitation between the long alkyl chains may explain the strength of these gels and the high values of $T_{sol}$ in **1e.**

### IR experiments

In **1e**, the **C=O stretching frequency** of the amide group appears at 1682 cm$^{-1}$, **increasing** highly **the intensity** in the case of the gel. **N-H amide stretching** appears at 3256 cm$^{-1}$ in the compound in the solid state, but in the dried xerogel it can be observed a surprising **change**, not only **of the intensity**, but also in **the morphology** in the band (Figure 1). This can be attributed for **the strong hydrogen bonds** between amide groups responsible of the gel formation.

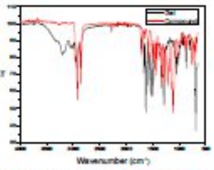


**Figure 1.** Spectra for compound **1e** (red) and the dry xerogel (black)

### Rheology experiments

Gels of **1a** and **1e** proved to be particularly robust by rheology experiments with **high mechanical resistance**. The additional amide groups in **1e** apparently results in more robust fiber formation. In the case of **1a** the gels are opaque suggesting larger, more crystalline particles and the high yield stress may indicate the presence of large solid particles rather than an elastic gel network. This difference is also reflected in the **higher σ values** for **1e** compared to **1a** in 1,4-butanediol (1585 vs 914 Pa)

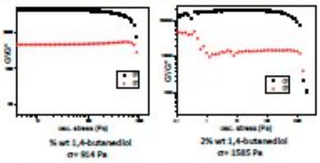


**Figure 2.** Stress sweep experiments for **1a** (left) and **1e** (right).

### Drug crystallization

**Sulfathiazole** crystals undergo a morphological change when crystallized in the absence or presence of **1e** gel. Thus, when sulfathiazole crystallizes in solution of 1-propanol, needle-shaped crystals corresponding to polymorph II are observed. On the other hand, when crystallization is performed in the presence of organogel **1e**, block shaped crystals corresponding to polymorph I are observed. This **change in the polymorphism**[4] is able to change the drug properties and its possible pharmacological application.

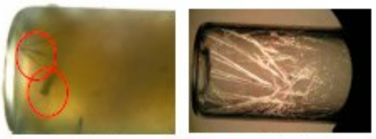


**Figure 3.** Crystals of sulfathiazole grown in **1e** gel in 1-propanol (left) and by solution evaporation (right).

## Conclusions

- ✓ **2*H*-benzo[*d*]1,2,3-triazole derivatives 1a, 1d,** and **1e** form **organogels** in specific solvents.
- ✓ Compound **1e** proved to be an effective gelator of a wide range of solvents even at low concentrations, in some cases acting as a **supergelator** (e.g. ethanol, critical gelation concentration 0.1% wt).
- ✓ These gels also displayed **high thermal stability** and **significant mechanical resistance**, especially **1e**.
- ✓ Gels of **1e** were used in the crystallization of the pharmaceutical drugs. In the case of **sulfathiazole** the gel induces a **change in the polymorphism**. These gels offer significant scope for expanding the rage of current polymorph discovery methods particularly in the pharmaceutical industry.

## References

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[2] I. Torres-Moya, I. Arrechea-Marcos, C. Tardío, J. R. Carrillo, A. Díaz-Ortiz, J. T. López Navarrete, M. C. Ruiz Delgado, P. Prieto, R. Ponce. RSC Advances, **2018**, *8*, 21879-21888.

[3] R. Vázquez, M. J. Martinez-Tomé, Z. Kahveci, I. Torres-Moya, A. Falco, R. Mallavia, C. Reyes. Polymers, **2018**, *10*, 938.

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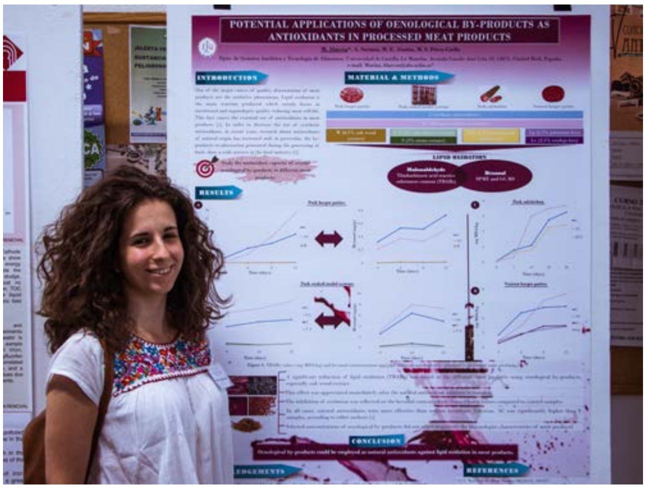

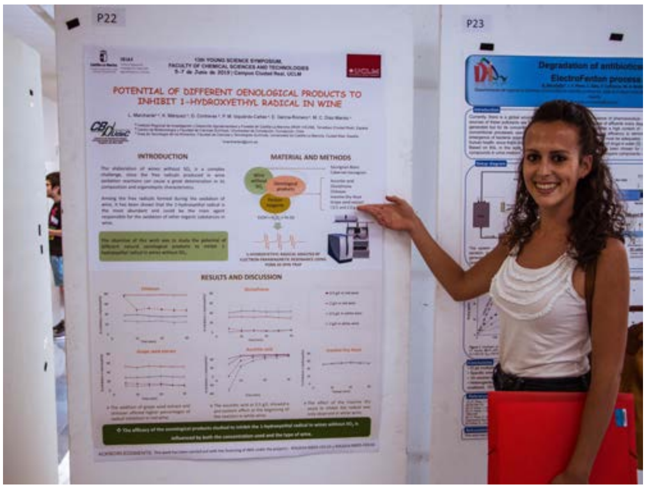

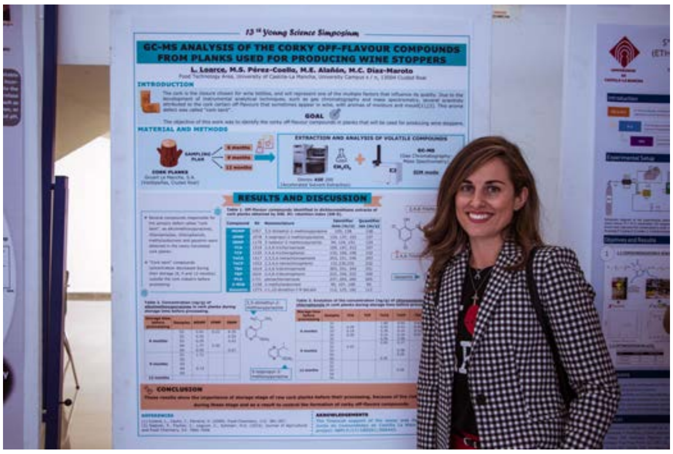

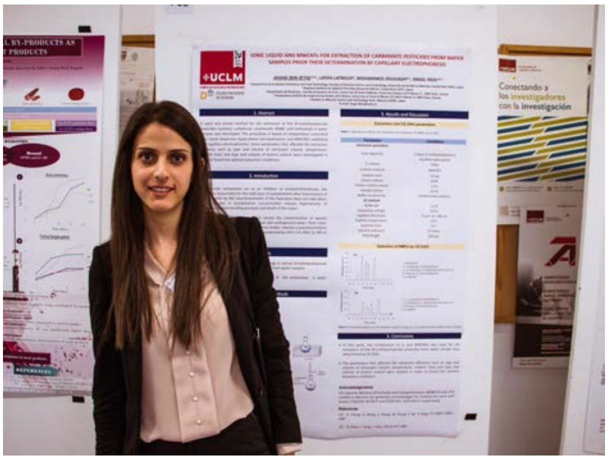

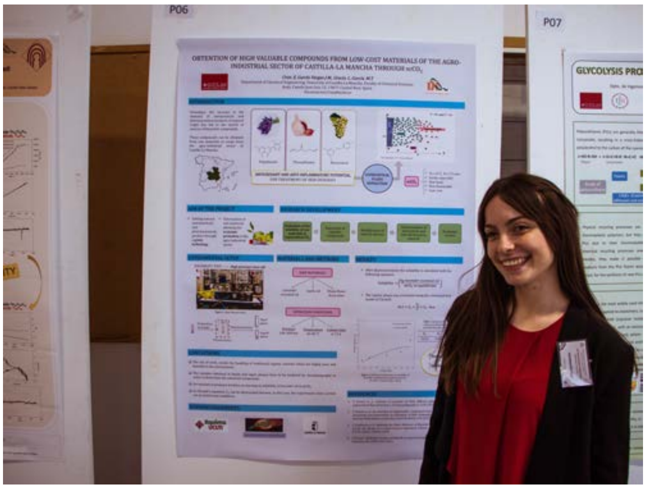

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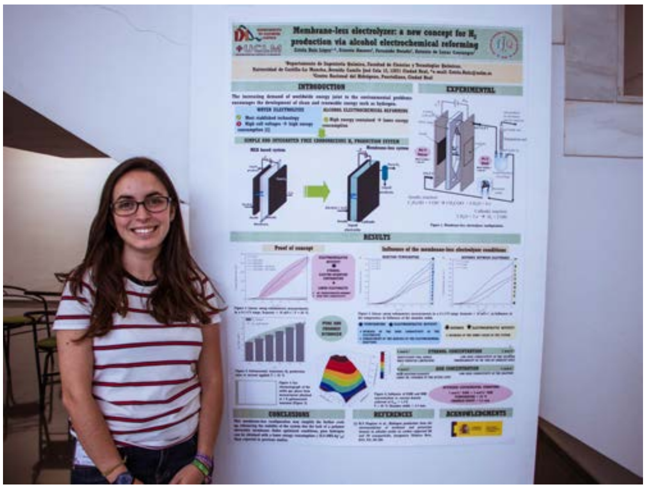

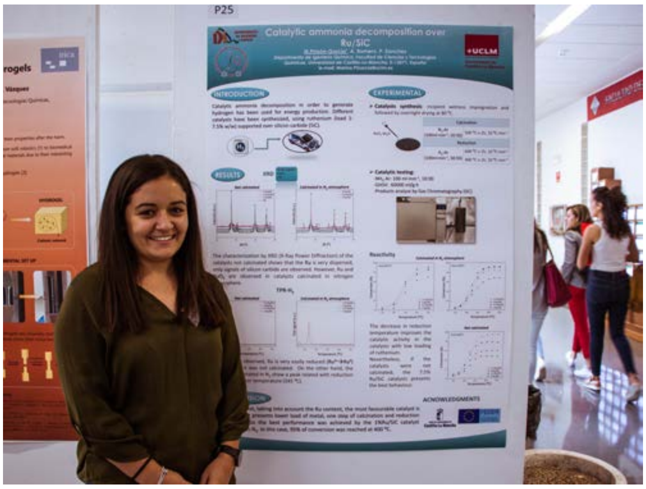

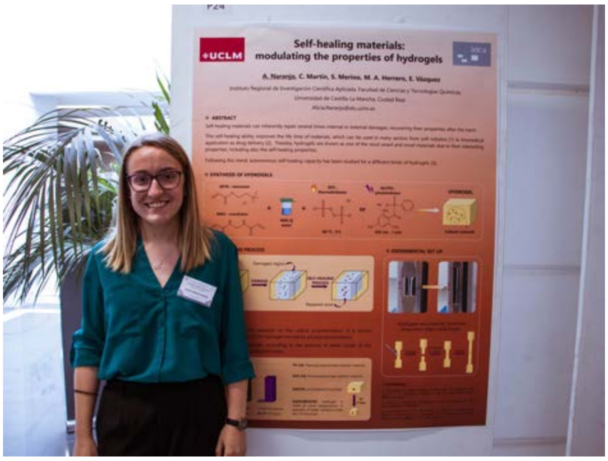

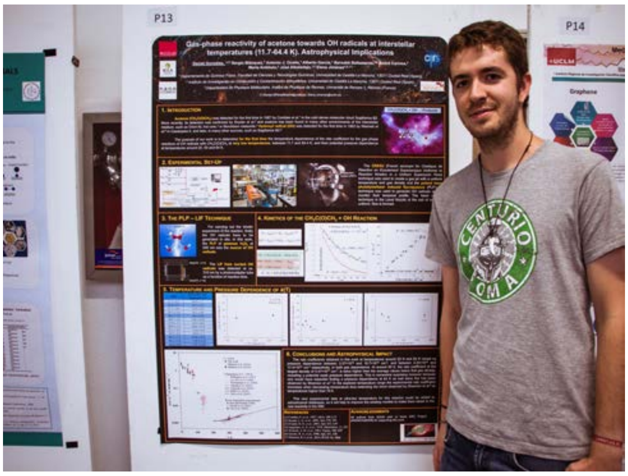

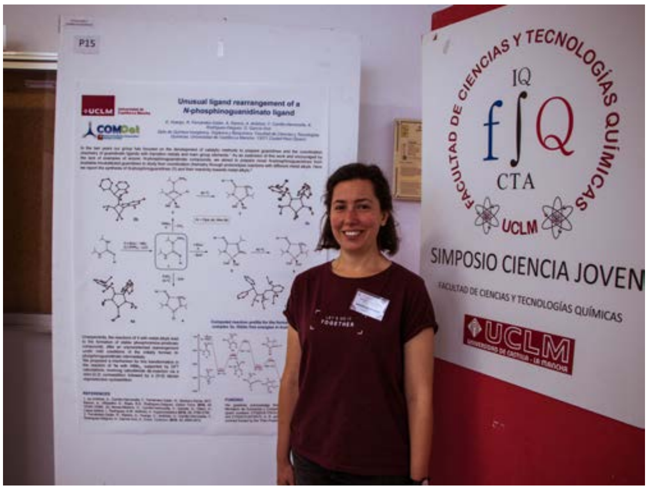

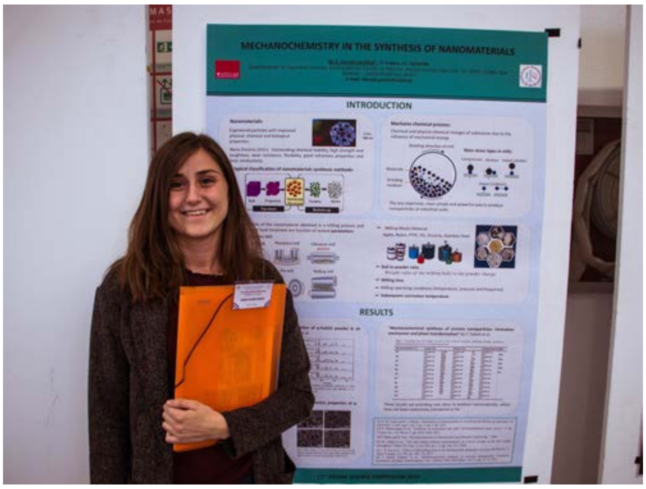

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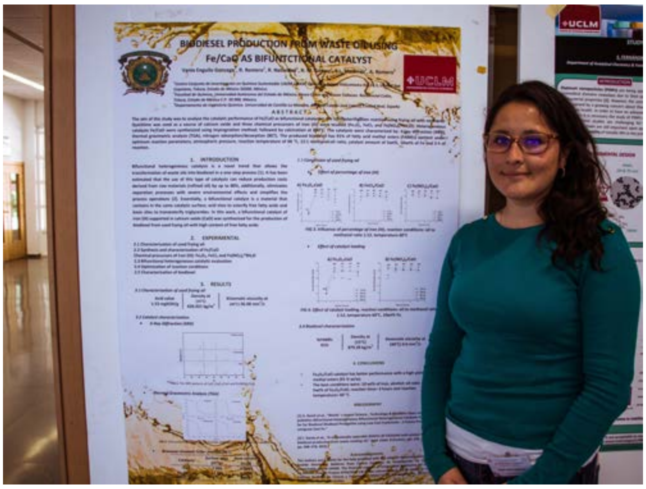

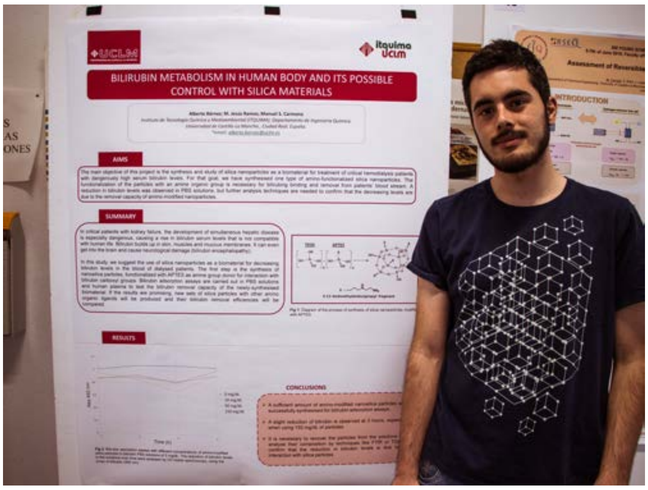

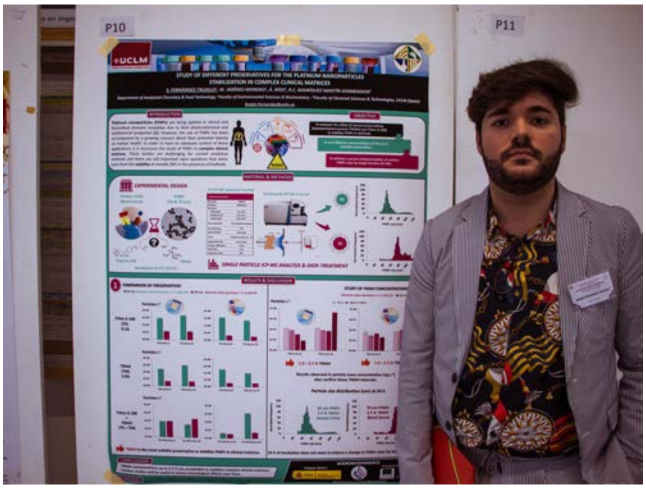

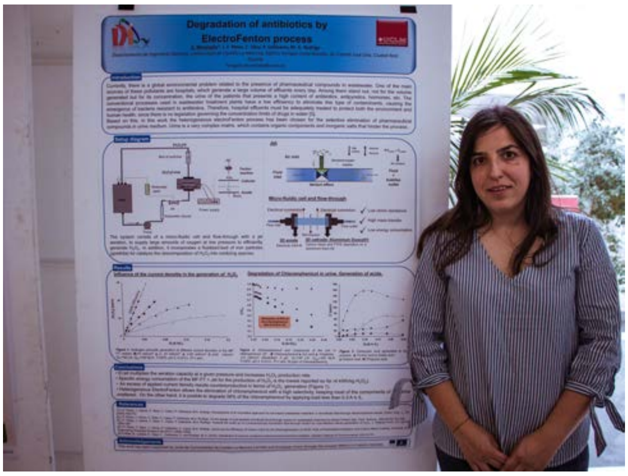

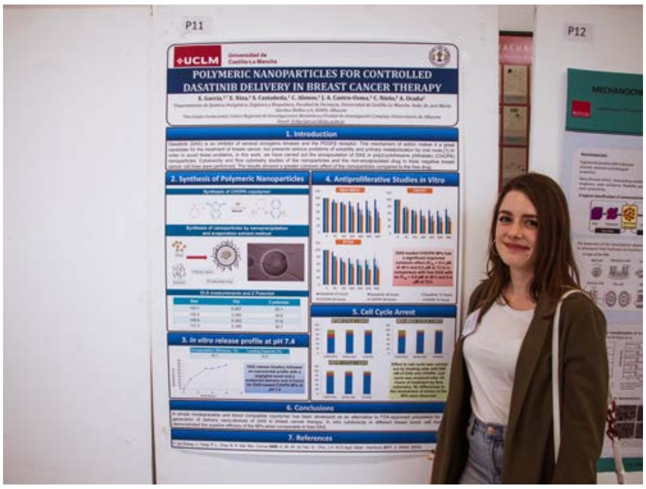

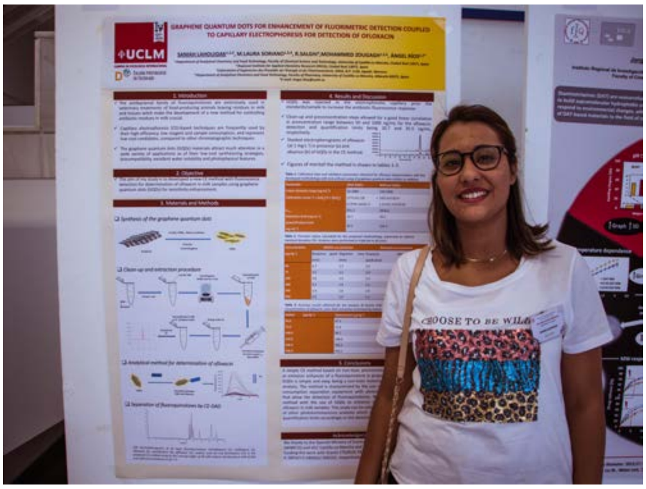

# ACTO CONMEMORACIÓN SISTEMA PERIÓDICO

Durante el segundo día del XIII Simposio de Ciencia Joven, tuvo lugar un acto de conmemoración del Sistema Periódico moderado por D. Ángel Ríos, decano de a Facultad de Ciencias y Tecnológías Químicas, y con la participación de:

- Pedro Lozano Rodríguez (Universidad de Murcia): “Modelos y ejemplos. La experiencia en la Universidad de Murcia”

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Como en ediciones anteriores, el comité organizador del XIII Simposio de Ciencia Joven hizo entrega del IV Premio Ciencia Joven en el acto de clausura del evento. En esta edición, el premio ha sido otorgado a José Fernando Pérez con el trabajo titulado "New electroFenton reactors for wastewater treatment". También se hizo entrega del II Premio Ciencia Joven al mejor póster a Jorge Leganés Bayón, con el póster titulado "Stimuli responsive hydrogels for drug delivery applications".

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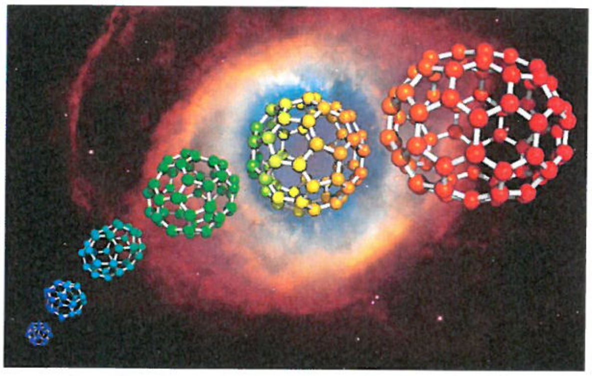

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