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EPA EXECUTIVE COMMITTEE

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**New Information
Technologies**

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EDITORIAL**President's Letter**

Dear EPA members,

After several years as editor-in-chief of this newsletter, this is the first contribution as President of our Association. I wish to search to solicit a debate in our Association starting from the page of this newsletter.

The first problem I want to discuss with you is a simple problem, which, in my opinion, has not a simple solution. Our Association shows a stable member number: we have four hundred members. However, I think that, in our experience, there are a lot of researchers working in photochemistry, in fields strictly related to photochemistry, such in the preparation of materials for optoelectronic devices, which are not EPA members. How can we improve the attractiveness of our organization? Usually, a researcher becomes member of a scientific organization because it wants to be a member of a scientific community, sharing problems, opportunities, and challenges. Obviously, the Association offers some benefits: the possibility of offering awards to young scientists that can be used for their career, or to senior scientists to honor a life devoted to the development of a discipline. And, at this purpose, this year we activate to new awards for young scientists and for senior researchers. Is it sufficient?

Being EPA members offers the possibility to have a reduced fee to participate to our international meetings. Furthermore, EPA offers the possibility to publish the scientific results of our work in a Journal (*Photochemical and Photobiological Sciences*) of which it owns a share of ownership. However, we can observe the most of the research results in photochemistry is not published on specialistic journals, such as PPS, but on generalist journals or on open access journals, phenomenon this that shows a rapid increase.

In conclusion, the problem is: how can increase the attractiveness of our organization? What new initiatives can we put in the field? I look forward for your reply. You can use to contribute to the discussion the newsletter or the facebook site. I hope to receive your ideas in this field.

Maurizio D'Auria

Università della Basilicata, Potenza, Italy

EPA STATUTES

The headquarters of EPA have been located in Switzerland where, as a registered non-profit scientific body since 1978, the association is tax exempt. After 40 years, the tax office of the canton of Zurich audited EPA to ensure its compliance with the conditions required for tax exemption. The result of this audit was that EPA can keep its tax-exempt status providing some small amendments to the statutes so that they comply with the current Swiss law. The following changes and additions were formulated by the tax office to the original statutes (in German, approved in 2008 by the General Council):

- If EPA is disbanded, the remaining finances cannot be transferred to just any organisation sharing the same goals: it has to be tax-exempt and in Switzerland (precision added to article 1d).
- An addition was made to explicitly state that EPA does not pursue any commercial aims and does not seek to make a profit (article 2c).
- As a clarification of current and past practices, it was explicitly added to the statutes that EC members act purely in an honorary capacity and are solely reimbursed for actual expenses they incur (article 9g).

Since the General Council had to vote on the statutes anyway, the Executive Committee also suggested some minor modifications in order to better reflect current and past EPA practices.

- Minor changes were made to article 1a with a closer translation into English of the original text in German (no modification to the German, legally binding text).
- The membership status of honorary member was added as article 3f. Frans DeSchryver received an honorary membership by the General Council in 2014 and other honorary memberships had also been awarded in the past. No mention of such membership was however to be found in the statutes.

- Any references to the Vice-President were removed from the statutes (articles 9b and 9d). EPA has effectively not had a Vice-President, at least since the adoption of the new statutes in 2008, but the Past-President has been part of the EC. The Past-President can effectively act as a Vice-President in exceptional cases (article 9d).
- In order to reflect past and current tacit practices, the term limit for the General Treasurer was removed. The Treasurer needs a permanent address in Switzerland and, also given the nature of the work, it did not seem to the Executive Committee in EPA's interest to replace him or her every 6 years.

The General Council, which met on 12th July 2018 in Dublin during the 27th PhotoIUPAC conference, approved unanimously the proposed changes to the statutes.

The complete statutes can be found on EPA's webpage at the following address: www.photochemistry.eu/about-us/statutes/

Alexandre Fürstenberg

University of Geneva, Switzerland

PUBLICATIONS

The European Network LighDyNAmics

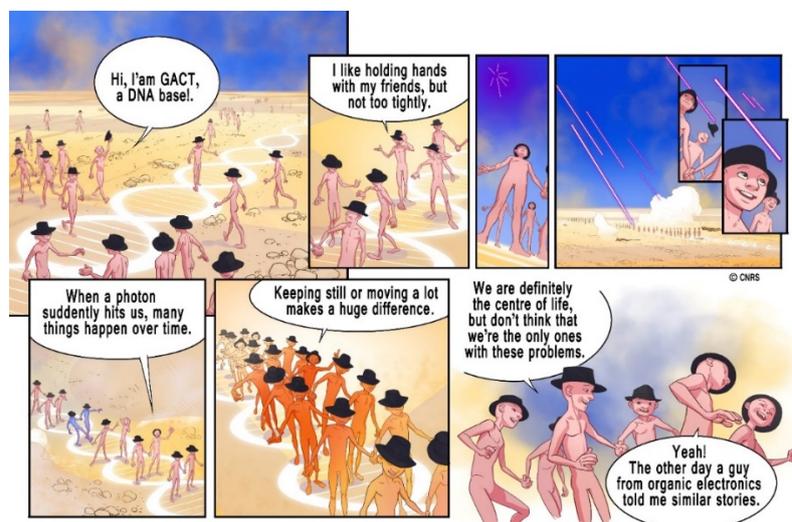
Dimitra Markovitsi

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LightDyNAmics is a photochemistry related project funded by the European Programme H2020 (ETN). It started on April 2018 for a duration of four years. Its title is “DNA as a training platform for photodynamic processes in soft materials”. The main goal of LightDyNAmics is to characterize, at the molecular scale, the dynamical processes induced upon absorption of UV radiation by DNA and to unveil the mechanisms leading to photodamage of the genetic code. This will be achieved by the development of methodologies taking into account factors such as interchromophore interactions, structural complexity, conformational disorder which will be applicable to a broad class of optoelectronic organic materials.

Some key aspects of the project are illustrated in the cartoon below. This type of cartoons had been developed in the past by the project “e-KNOWLEDGE: Network for ICT-enabled non-formal science learning” (2008-2010), funded from the Lifelong Learning Programme of the European Union, to which EPA was associated.

The cartoon characters are named GACT, from the initials of the four DNA bases: Guanine, Adenine, Cytosine and Thymine. GACTs give hands (associate via hydrogen bonds) to form double helices. But even when interconnected, GACTs remain able to perform weak movements, as happens with molecules in soft matter. Upon absorption of UV radiation by DNA (when a photon hits GACTs) the light energy triggers a series of processes which may lead to chemical reactions damaging the DNA; for example, two bases may stuck together (two GACTS become blue) or a GACT may lose an electron, represented by a hat. The processes preceding such reactions strongly depend on the local motions, as is the case of organic materials used in the field of molecular electronics.



LightDyNAMics trains 15 PhD students by crossing the border between theoretical and experimental expertise by performing independent, yet interrelated and complementary research projects focussed on three scientific objectives:

- to develop and optimize a variety of complementary experimental and computational methods for the investigation of multiscale dynamics in multi-chromophore systems;
- to apply these methods to elucidate photoinduced processes in DNA tracts of different size and complexity, from simple nucleobases in the gas phase to genomic DNA, over different timescales from light absorption all the way to the formation of lesions;
- to generalize the acquired knowledge and transfer it to other multi-chromophore systems, soft materials of fundamental and technological interest, identifying the photoinduced processes, their spectral signatures, their yields and dynamics.

Coordinated by R. Improta (Italy), LightDyNAMics brings together 11 academic groups: F. Sandoro (Italy), D. Markovitsi (France), J. Verlet (United Kingdom) J. Plavec (Slovenia), T. Carrell (Germany), J. Šponer (Czech Republic), G. Cerullo (Italy),

S. Quinn (Ireland), M. Gavarelli (Italy) and L. González (Austria). Six companies are also associated either as PhD supervisors (AstraZeneca Limited, Baseclick GmbH, Dynamic Biosensors GmbH) or by offering training to them (E4 Computer Engineering SpA, Molecular Discovery Limited, Prigen s.r.l.). For details see the website: www.lightdynamics.eu.

SPECIAL REPORTS ON PHOTOCHEMISTRY IN SPAIN AND PORTUGAL: A SAMPLE FROM THE 6TH IBERIAN CONFERENCE ON PHOTOCHEMISTRY

Introduction

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In this Thematic Issue we will put the spotlight on the activities of photochemistry groups from Spain and Portugal. The featured contributions represent a sample from the recently held 6th Iberian Conference on Photochemistry at the University of Aveiro (12.9.-14.9.2018). The event was co-organized by Prof. Luis D. Carlos (*Universidade de Aveiro*) and Prof. Sérgio Seixas de Melo (*Universidade de Coimbra*), presenting the Photochemistry Group of the Portuguese Society of Chemistry (SPQ). This conference was held before in Santiago de Compostela (2003), Faro (2007), Granada (2011), Lisboa (2014), and Toledo (2016). It brings together a representative blend of research related to Photochemical Sciences from all over the Iberian Peninsula, with its chemistry community being traditionally very active in this field.

The contributions that follow this brief introduction are from groups based in Coimbra, Lisboa (both *Instituto Tecnico Superior* and *Universidade NOVA*), Madrid, Granada, Valencia, and Barcelona. They cover aspects ranging from luminescent materials, luminescent sensing and bioimaging, to photoreactivity and photodynamic therapy. The next (7th) Iberian Conference on Photochemistry will be held again in Lisboa in 2020. Stay tuned!

New insights on the luminescent properties of Alkynyl-Gold(I) complexes and their supramolecular aggregates

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Alkynyl-gold(I) complexes have become one of the hot topics in the fields of Materials and Supramolecular Chemistry for the past 5-10 years, due to their ability to produce new highly luminescent supramolecular structures.¹

One of the major breakthroughs on this topic was made in 2013, when we published an Au(I)-alkynyl organometallic complex that is capable of spontaneous self-assembly in water, resulting in the formation of long luminescent fibers.² The complex presented a linear structure, with two axial ligands (4-ethynylpyridine and 1,3,5-triaza-7-phosphaadamantane, PTA), with a Au(I) atom in the centre. The major highlight of these results is the fact that hydrogels could be obtained from a such simple structure at very low concentrations (0.015–0.05% weight), and that the position and nature of the ligands can induce unexpected and interesting rheological properties in water.

Later, we reported a similar complex (differing only in the phosphine ligand), which presented a stronger degree of cross-linking during the gelation process.³ Moreover, we were able to form highly homogenous gold nanoparticles upon heating.

An in-depth study on the thermodynamics of the gelation process in these two complexes has demonstrated that intermolecular aurophilic bonds, i.e. bonds between gold(I) atoms of different complex molecules, have a tremendous influence on the self-assembly process that leads to gel formation.⁴

As mentioned previously, self-assembly of Au(I)-alkynyl complexes is highly dependent on a series of factors. In order to gain further insight on the influence of such parameters, a detailed study was conducted on a series of methylated derivatives from the previously studied Au(I)-

alkynyl complexes.⁵ The introduction of a positive charge and the position thereof within the structure of the complex yields aggregates with completely different morphologies. Furthermore, changing solely the counterion results in different supramolecular packaging (when using triflate as counterion, the aggregates presented a square-like 3D shape) or in complete inhibition of aggregation (if the counter-anion induces a significant enough increase in water solubility of the overall complex).

Our latest research efforts in controlling the self-assembly of Au(I) complexes have led us to synthesize new alkynyl-gold(I) complexes bearing different alkynyl-chromophores based on polypyridyl units, namely ethynyl-bipyridine and ethynyl-terpyridine (fig. 1A).⁶

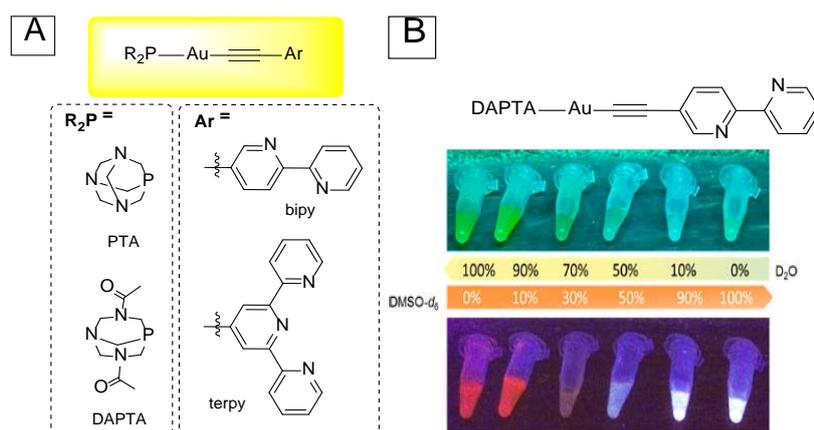


Fig. 1. (A) Chemical structures of the Alkynyl-Au(I) complexes with polypyridyl chromophores; (B) Variation of the color of complex **DAPTA-Au(I)-ethynyl-bipy** in mixtures of DMSO-*d*₆/D₂O under visible (up) and UV light (down).

We found that the complexes undergo self-assembly both in DMSO and water (and mixtures thereof), exhibiting two distinct luminescence profiles, depending on solvent composition (fig. 1B). NMR studies confirm that the complexes display in fact two different intermolecular interaction modes for self-assembly: (i) chain assemblies, which are based mainly on aurophilic interactions, and (ii) stacked assemblies,

which are based on $\text{Au}\cdots\pi$ and $\pi\cdots\pi$ interactions. DFT calculations on the changes in the relative binding energy of complex molecules further support this interpretation of the experimental results.

The presence of metal chelating ligands in the complexes' structure, namely *bipy* and *terpy*, has allowed to exploit the use of external stimuli to control the reversibility of the self-assembly phenomenon. Briefly, the addition of Zn^{2+} induced the disruption of the supramolecular aggregates producing significant changes in their luminescence. Upon addition of a stronger Zn^{2+} chelating agent (sodium ethylenediaminetetraacetate, EDTA), we observed the regeneration of the original supramolecular aggregates and their respective luminescent properties. These results were also confirmed by NMR spectroscopy and SAXS. In particular, these results demonstrate that we are able to modulate the aggregation/disaggregation process by the correct choice of the chemical structure of the chromophore that, in this case, can interact with external cations as a way to control the supramolecular assembly process.

All in all, the formation of luminescent supramolecular structures based on Au(I) alkynyl moieties is an open investigation research field with increasing interest due to the wide range of possibilities that offer any possible change on the chromophores or solubilisation in solvents with different polarity. Applications in materials chemistry, sensors and biological achievements among others have been reported.

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The Photonics and Reactivity Group at the Coimbra Chemistry Centre

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History: The origin of the Photonics and Reactivity laboratory can be traced back to 1973 when Sebastião Formosinho returned to the University of Coimbra after a PhD with Lord George Porter at the Royal Institution, in London. Formosinho had the first pulsed laser installed at the Chemistry Department and founded the group of Photochemistry. Luis Arnaut was a PhD student of Sebastião Formosinho from 1984 through 1988 and after a post-doc with Richard Caldwell at the University of Texas at Dallas, returned to Coimbra in 1989 as Assistant Professor to work in the Photochemistry group. Sebastião Formosinho retired in 2013 and the Photochemistry group originated other groups in the Coimbra Chemistry Centre, including the Photonics and Reactivity Group and the Coimbra LaserLab.

The Coimbra LaserLab is part of a Portuguese roadmap of research infrastructures of strategic interest and member of the consortium LaserLab Europe, the integrated initiative of European laser research infrastructures, supported by H2020. Luis Arnaut is the Director of the Coimbra LaserLab and Carlos Serpa the Deputy Director.

Mission: The Photonics and Reactivity Lab is a place for initiative and creative thinking, to explore less-traveled paths and dare question conventions using the tools of science. The unifying theme is the use of light to open new frontiers. Together with various labs in the Coimbra Chemistry Centre and elsewhere, we design new molecules and customize equipment to explore our original ideas. The first drug patented by a Portuguese university that reached clinical trials was designed in our laboratory. We are also at the origin of a technology introduced in the aesthetics market and tested for medical applications. We cherish innovative solutions and encourage spin-offs of lab members, but believe that long-lasting solutions to human needs must be rooted in fundamental science.

Highlights:

Photoinduced reactivity. The dogma of chemical reactivity is that, for elementary reactions, higher barriers lead to slower reactions. In Nature Communications¹ we recently presented intramolecular electron transfers that become faster as their apparent activation energies increase, and oppose the “inverted region” energy dependence predicted by Marcus. The deficiencies of Marcus theory are exposed and an alternative model for electron transfer is discussed. On-going research calls for a change in paradigm in the theory of electron transfer reactions.

Photodynamic therapy. PDT combines a photosensitizer, light and molecular oxygen to generate cytotoxic reactive oxygen species that kill cells. As explained in an the highly cited paper² (top 1% of the academic field of Biology & Biochemistry), PDT has both a local and a systemic effect. The elimination of the primary tumor and control of metastasis were achieved with a bacteriochlorin photosensitizer named redaporfin and designed by us.³ The mechanisms through which PDT with redaporfin kills cancer cells were presented,⁴ showing that the ER/GA play a role upstream of mitochondria in the lethal signaling pathway.

Photoacoustics and therapy. The ability to produce high-pressure high-intensity ultrasound pulses using nanosecond laser pulses, developed in our laboratory, was applied to the non-invasive delivery of hyaluronic acid as dermal filler for facial rejuvenation. A face-split, randomized, placebo-controlled, double-blind study to investigate passive versus active hyaluronic acid administration⁵ revealed that this procedure is safe and leads to unambiguous facial rejuvenation. Ultrasound pulses generated with picosecond laser pulses are capable of temporarily permeabilize cell membranes and enable gene transfection with negligible cytotoxicity. Photoacoustic transfection of a plasmid DNA encoding Green Fluorescent Protein in COS-7 monkey fibroblast cells attained an efficiency of 5% in 10 minutes at 20 °C (to appear soon).

Protein folding. Protein (miss) folding is at the origin of various neurological diseases. The folding dynamics and energetics of β -helical segments in proteins were described using photoinduced pH jumps with photoacoustic detection,⁶ and revealed the role of salt bridges and

cation- π interactions. The singular fluorescence from aggregated myeloid fibrils has eluded observation, but was unambiguously identified and characterized (to appear soon).

Imaging. The new frontiers of medical imaging and treatment demand the ability to visualize microstructures that target the diseased tissue. By imaging where, when and how much of a marker chromophore appears in the target tissue, it becomes possible to refine basic preclinical research and improve therapy. We presented⁷ a rationally-designed near-infrared fluorophores developed in our group to image 1 mm tumors not visible with the naked eye. Current developments in our group focus on molecular contrast agents for photoacoustic tomography.

Photophysics. The fundamentals of light interaction with matter (and molecules) still raise technical challenges and controversies. The accurate measurement of light doses in PDT was explained.⁸ The transfer of photon momentum when light crosses media with different refractive indexes, manifested in the nanometric deformation of the interface between the two media, was clearly separated from thermoelastic expansion and shown to be consistent with the Minkowski momentum (to appear soon).

Equipment: The laboratory is equipped for (femto, pico and nanosecond) laser-induced transient absorption and emission measurements with UV, visible and near-infrared detection. Time resolved photoacoustic calorimetry both for solutions and thin films and photoacoustic tomography. It also has a cell culture laboratory. Transient 2D-IR spectroscopy will soon be installed. Coimbra research environment allows easy access to multiple microscopy and spectroscopy techniques, molecular synthesis and material preparation, and animal experimentation.

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Applied Photochemistry (and much more!) at Complutense University of Madrid

Guillermo Orellana

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The +25 years old UCM Optical Chemosensors & Applied Photochemistry Group (GSOLFA; <http://www.gsolfa.info/en>), headed by Prof. G. Orellana and with the co-leadership of Prof. María C. Moreno-Bondi, performs R+D activities that span from the molecular engineering of tailored (indicator) dyes to analytical chemistry “in the field”, from photochemical nano(bio)sensors to applications of the photodynamic effect. The combined **experience** of our team members covers photochemistry, molecular spectroscopy, fiberoptic sensing, optical and laser-based analysis, chemical synthesis and HPLC. Longstanding contract research for private and public companies, deep knowledge of the end-user needs, and collaboration with the best national and international groups, back up our many years tradition of applied and basic photochemical research for

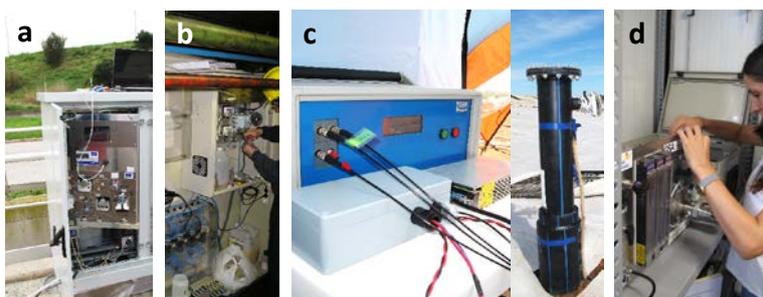


Figure 1. Ru-polypyridyl-based luminescent photochemical sensors at work: a) Microalgae biosensor for waterborne herbicides in a WWTP; b) Iron/surfactants sensors in the cold rolling mill of steel manufacturing plant; c) Humidity/O₂ sensors into a landfill of waste; d) O₂/H₂S/NH₃ sensors in a biomethane production plant.

improving the quality of our life, our environment and the sustainability of industrial processes.

Luminescent photochemical sensors have traditionally been one of the strongest activities of the GSOLFA Group. Numerous publications and patents show our achievements. Thanks to molecular engineering of photoactive indicator dyes, their in-house preparation and detailed photochemical characterization, an in-depth knowledge of chemical optosensing, and a close collaboration with the end-users, many applications have been born in our laboratories and are now (or have been) working in the facilities of relevant companies such as Repsol, Sartorius Stedim (formerly TAP Biosystems), Aqualogy, CENER, Naturgy (formerly Gas Natural Fenosa), Quirón Prevención and Finsa (with EU Life+ support under the LIFESENSEI project) [1]. All photoinduced electron, proton or energy transfer have been “put to work” for the sake of in situ monitoring of chemical species. One of the most interesting achievements so far has been to fabricate luminescent photochemical sensors for gas analysis by direct functionalization, with the indicator dye, of the GaN substrate that forms the basis of most blue and white LEDs [2]. The recent appointment of Prof. Orellana as co-Editor-in-Chief of the prestigious Elsevier’s *Sensors & Actuators B: Chem.* and of Prof. Moreno-Bondi to the editorial board of Springer’s *Anal. Bioanal. Chem.* journals reflect the relevance of the GSOLFA Group contributions to the optical chemical sensor and biosensor fields. Prof. Orellana and his Group have been the recipients of the 2017 UCM Technology and Knowledge Transfer Prize.

In addition to the development of photochemical **(bio)sensors**, the collaborative research with Prof. Moreno-Bondi includes the preparation of **fluorescent**-tagged analytes for *water* and *food analysis* with **molecularly imprinted polymers** (MIPs) or photochemically-active MIPs. The latter are synthetic materials that behave as biomimetic receptors by selectively recognizing a particular analyte. A highly intertwined macromolecular structure grows around a “*template*” molecule that is extracted after polymerization. This creates cavities (binding sites) that are complementary in shape, size and distribution of functional groups to the template molecule, allowing its selective recognition in a similar way to enzymes or antibodies [3].

Nanotechnology has brought about advantages to optical sensing such as plasmonic and quantum confinement effects, accelerated

response, dramatic increases of brightness and savings of precious reagents, among others. Molecular imprinting is able to provide the essential analyte recognition without the limitations of biomolecules. Therefore, *combination* of both fields seems a logical step for versatile **advanced optosensors** development. Some recent examples from our Group show the analytical challenges that may be tackled by MIP nanostructures, namely, luminescent core-shell imprinted *nanoparticles* engineered for analyte-targeted Förster resonance energy transfer (FRET)-based sensing, *submicron*-sized MIP *arrays* fabricated by photopolymerization within metal subwavelength apertures, and *self-referencing* fluorescent photochemical nanosensors, to name a few [4].

Photochemical/photophysical strategies such as FRET, SET (surface energy transfer to metal nanoparticles), or fluorescence polarization can also be used to increase the sensitivity and/or the selectivity of analytical methods. We have demonstrated it in the determination of clinical immunosuppressants and food mycotoxins [5].

The GSOLFA Group has also exhibited a lot of activity in the **applications of the photodynamic effect** (i.e. the generation of singlet molecular oxygen, $^1\text{O}_2$, by the combined action of light, O_2 and appropriate photosensitizer dyes) to improve the quality of life of isolated communities in less-favored regions. Successful solar photoreactors have been developed for domestic water disinfection with sunlight, and novel photosensitizers have been developed to fight the cutaneous forms of tropical-endemic leishmaniasis [6].

Finally, I should deeply acknowledge the contribution of the many collaborators, PhD students, contract researchers, postdocs and the other professors in the Group, too numerous to be enumerated here. Without their hard work, ingenuity, talent and enthusiasm, and without the continuous funding of national and European institutions, all the photochemistry and its applications described above would not have been possible.

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Luminescent Biosensors for Biomedical Applications

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The Photochemistry and Photobiology group FQM-247 is settled at the Faculty of Pharmacy of the University of Granada (Spain) and it is composed of professors and researchers of the Physical Chemistry Department and numerous PhD and Master Students.

The research group explores several biological systems and processes, focusing on luminescence emission techniques, developing nanotechnological tools and proposing new diagnostic methodologies for current health and disease challenges. In particular, we develop innovative sensing strategies based on organic fluorophores, lanthanide antennas, quantum dots, fluorescent proteins and nanoparticles, which allow for sensitive, rapid and non-invasive detection of a variety of intracellular analytes of biological relevance *in situ*, e.g.: biothiols, pH levels, phosphate ions, nucleic acids and proteins (Fig. 1), and for the study of enzymatic activities (e.g: proteases and protein kinases).¹⁻⁴ These strategies take advantage of our experience in multicolor single-molecule fluorescence techniques, single-molecule biophysics, and excited-state dynamics. One of the key features of our research is its interdisciplinary dimension, at the interface between chemistry, biochemistry, biology and physics.

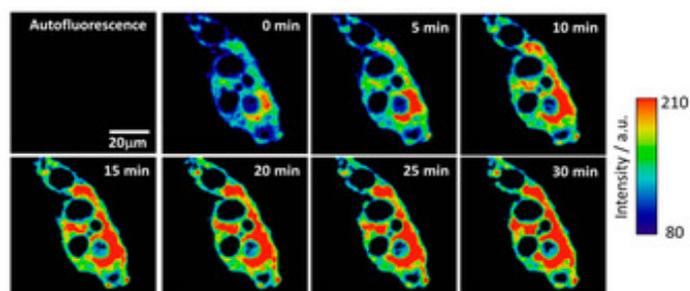


Figure 1. Fluorogenic reaction of DNBS-2Me-4OMe-TM in response to intracellular biothiols. Reproduced from [1], under the terms of the Creative Commons Attribution License. Copyright © 2018 Herrero-Foncubierta et al.

For instance, we employ advanced microscopy and state-of-the-art nanotechnological approaches for understanding how tumoral cells modify their way to use the energy by means of smart adaptation mechanisms. In particular, focusing on tumoral breast cancer cell lines, we are able to identify different metabolic profiles, according to the clinical classifications of these types of tumors. Besides, this knowledge permits us to design potential new drugs aimed to act directly on the tumoral metabolism, improving the current treatments. This transversal research line includes an active collaboration with the Departments of Organic Chemistry and Biochemistry (UGR), the Genyo Center for oncological research, and University of Trento (UNINT).

Current approaches for cancer diagnostics usually are effective in late stages of the disease, lacking early diagnostic tools, which would greatly benefit the prognosis of the patients if available. In this sense, we also develop new diagnostics approaches for the early detection of certain biomarkers of some diseases including cancer or hepatic dysfunction. This would provide means of gaining insights into pathogenesis, while also providing tools for early detection of target deregulation. In particular, we focus on micro-RNAs, which are small fragments of circulating ribonucleic acid found in blood or other biological fluids, as potential biomarkers. We work in the design of novel, simple, portable and affordable instrumentation in conjunction

with companies DestiNA Genomica (Spain) and Optoi Microelectronics (Italy) for the detection and analysis of these important biomarkers in the framework of a European RISE H2020 project.

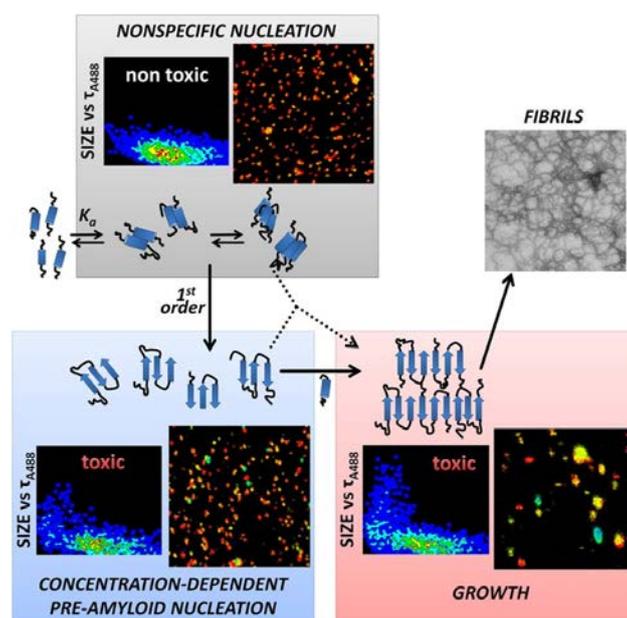


Figure 2. Proposed model of the two-step nucleation mechanism for amyloid aggregation, which is correlated with the supporting single-molecule (SMF-PIE and FLIM-PIE) evidence. Reproduced from [5], under the terms of the Creative Commons Attribution License. Copyright © 2017 Castello et al.

Finally, we employ molecular analysis techniques for studying the underlying mechanism of formation of neurotoxic aggregates, found in Alzheimer disease and other related systems (Fig. 2).⁵ A better comprehension of the foundations by which these protein aggregates, precursors of amyloid fibers, are formed will contribute with valuable information to employ against neurodegenerative diseases.

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Optical and Functional nanoMaterials

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The topics of the four presentations of the Optical and Functional Materials (OM2) group at the 6 JIF in Aveiro span our current scientific interests, which are situated at the intersection of functional nanomaterials and the interaction of materials with light (Fig. 1). We develop and study new nanostructured materials for two main areas of application: i) imaging, sensing and delivery; and ii) energy and optoelectronics.

Regarding the design of nanostructures for imaging, sensing and controlled delivery applications, our toolbox include different types of materials (hybrid nanoparticles, mesoporous materials, functional and stimuli-responsive polymers, gold cluster assemblies, graphene quantum dots and optical sensor materials) and different processes (two-photon absorption, photoluminescence and photo-induced energy and charge-transfer). We combine these materials and processes to develop:

- i) novel fluorescent hybrid silica nanoparticles with diameters from 10 nm to 100 nm, with encapsulated dyes (a range of perylenediimides featuring visible or NIR excitation/emission),^{1,2} and different groups at the surface (for example, for biological targeting),³ exhibiting very high photostability, specificity and brightness;
- ii) advanced hybrid nanocontainers with a core of mesoporous silica of precisely controlled diameter, from 20 nm to 100 nm, and different pore morphologies^{4,5} for modulated on-demand cargo release;^{6,7}
- iii) stimuli-responsive (smart) polymer materials for delivery control and responsive surfaces;⁸⁻¹¹
- iv) smart nanomaterials for boron sensing and scavenging, including novel optical sensing of boric and boronic acids,¹²⁻¹⁴ smart polymer nanoparticles for boron removal and recovery,¹⁵ and boron scavenging nanostructured membranes;¹⁶
- v) gold hybrid plasmonic nanostructures for enhancing the absorption/emission of organic¹⁷ and inorganic^{18,19} luminescent materials; and

vi) push-pull organic molecules and graphene quantum dots with nonlinear emission for multiphoton imaging in biological systems.^{20,21}

We are also interested in nanomaterials for application in energy and data storage, namely:

vii) small acceptor molecules (perylene diimides and others) to promote new exciton diffusion pathways in charge-transfer single crystal interfaces with improved photoconductivity for applications in organic optoelectronics;²²⁻²⁵

viii) polymers with enhanced nonlinear absorption in the near-infrared for high-density volumetric optical data storage;²⁶ and

ix) metal-organic frameworks with photoactive ligands to tune charge transfer pathways for applications in solar cells and electroluminescence.²⁷

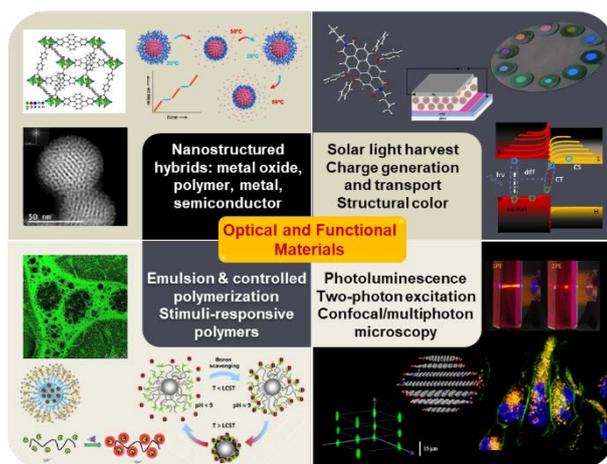


Figure 1. The scientific interests of the Optical and Functional Materials (OM2) group are situated at the intersection of functional nanomaterials and the interaction of materials with light. We develop and study new nanostructured materials for application in mainly two areas: i) Imaging, sensing and delivery; and ii) Energy and optoelectronics.

In the keynote “Bright and stable nanomaterials for imaging and diagnostics” we discussed luminescent nanomaterials, which are used in many applications from advanced imaging techniques to biodiagnostics, etc. The ability to increase the brightness and photostability of these materials have direct impact on their performance, lowering the limit of detection in diagnostic applications and allowing their use in demanding laser scanning imaging techniques. We focus our efforts on two different approaches to obtain materials with large brightness and photostability. The first approach is based on attaching a large number of dyes to a given support, so that the dyes cannot leach out of the structure, and the interaction between the dyes is minimized in order to avoid emission quenching. These supports can be based on polymer chains of controlled architecture, molecular weight and number of dyes,^{12,28} or on silica nanoparticles of precise dimensions.^{4,5} While the polymer supports allow us to tune the number of dyes per support with extreme precision and connect the chain to one biomolecule or another group of interest, the silica nanoparticles are specially effective at protecting the dyes from oxygen and thus present very high brightness and photostability (Fig. 2),^{1,2} and allow us to take advantage of the very rich surface chemistry to attach different functionalities to the surface, as for example, tumour-targeting groups.³ In the second approach, instead of using a large amount of dyes, we are trying to create nanostructures that increase the brightness of each dye. This is done by coupling the dyes (either organic fluorophores¹⁷ or quantum dots^{18,19}) to gold nanoparticles in a way that these building blocks are precisely 5-10 nm part, so that we can take advantage of the field enhancement by the surface plasmon while avoiding quenching of the luminescence by the metal. We have done this either using a polymer brush that completely encapsulates a quantum dot^{18,19} and further attaches to a gold nanoparticle, or by coating a gold nanoparticle with a very thin silica shell to which modified perylenediimide (PDI) dyes were covalently attached.¹⁷ Very recently we have also started working with NIR-luminescent nanostructures containing gold nanoclusters (AuNCs), developing new ways to overcome their poor structural stability so that they can be used as markers in advanced optical imaging (oral contribution “Hybrid gold nanoclusters-polymer nanoparticles as fluorescent probes”, and combining them with plasmonic gold nanoparticles for emission enhancement.

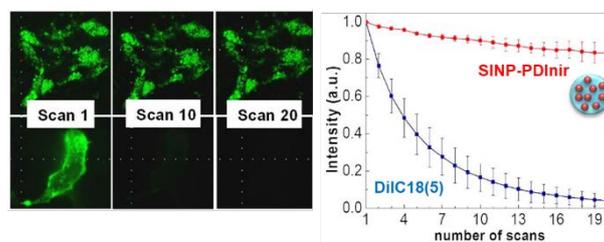


Figure 2. Consecutive laser scanning confocal images of HEK293 cells with internalized silica nanoparticles emitting in the NIR (SiNP-PDI_{nir}) and the DiIC18(5) membrane marker, excited with a HeNe 633 nm laser (image size $62.5 \times 62.5 \mu\text{m}$, 5.12 s per scan) show only a small decrease in intensity for the SiNP-PDI_{nir}.

In the invited lecture “Perylenediimides as building blocks for functional nanomaterials” we highlighted the versatility of this family of molecules in the development of functional nanomaterials. PDIs show interesting properties, such as near-unity fluorescence quantum yield, excitation in the visible region, strong and reversible electron-accepting character, and high electron mobility. The synthesis of PDIs derivatives, starting from the commercially available perylene-3,4,9,10-tetracarboxylic acid dianhydride, allows the selective introduction of substituents in the imide group (ruling the solubility, aggregation and immobilization) or in the bay region (tuning the electronic and optical properties). The bay substituents also induce a twist in the PDIs planar structure that can go up to 40° , and we have used this to develop new exciton diffusion pathways in single-crystal organic semiconductor.^{24,25} The bay substituents also induce changes in the photophysical properties with fluorescence maxima covering the visible and NIR region.^{1,2} Additionally, PDIs can be introduced in different nanostructures through specific imide substituents (Fig.3).⁷

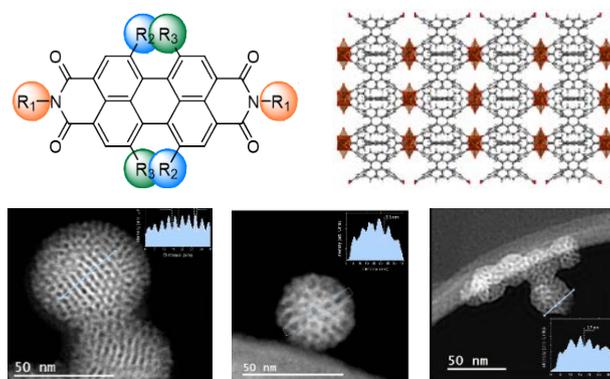


Figure 3. General structure of perylene-3,4,9,10-tetracarboxylic diimide derivatives synthesized in our laboratories, with different bay substituents (R_2 and R_3), ranging from electron donor to electron acceptor groups that tune the optical and electronic properties, and imide substituents (R_1) that allow the incorporation in different nanostructures (top left). Metal-organic framework (top right) and mesoporous silica nanoparticles (bottom, scale bar: 50 nm) with PDIs incorporated.

In the oral communication “On the origin of the emission of carbon nanodots: molecule-like fluorophores vs nanographene-like clusters” emphasis was laid upon our recent study on the nonlinear emission spectroscopy of nitrogen doped carbon dots and graphene like molecules.^{21,29} We showed that the emission spectrum of the carbon quantum dots depends on the excitation mode (linear vs non-linear excitation). At the same excitation energy, nonlinear excitation results in excitation-wavelength independent emission, while upon linear excitation the emission is excitation-wavelength dependent. The spectral dispersion observed upon linear excitation was assigned to a trivial effect of the heterogeneity of the chemical and structural environment (e.g. distortion from planarity induced by defects and the presence of amine groups, cluster size and shape) of the emitting sites within the dots. The independence of the nonlinear emission on the excitation wavelength was discussed in terms of selective excitation of isolated sp^2 clusters bearing electron donor and acceptor groups on the

edge, embedded in the dots and involved in π - π stacking interactions with clusters in adjacent layers. Our comprehensive study provides an extra piece of the puzzle of the origin of photoluminescence emission in carbon dots (Fig. 4).

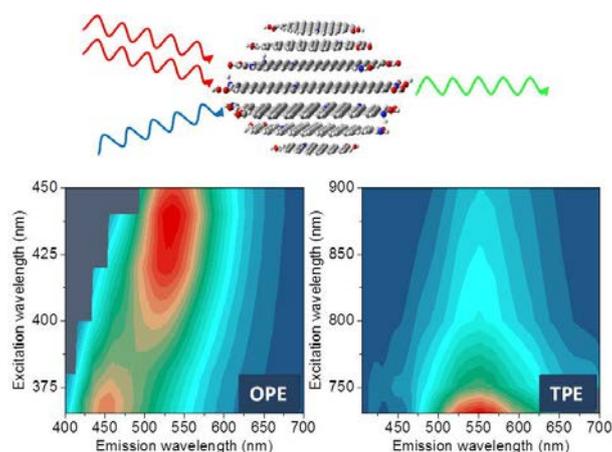


Figure 4. Contour plots of the emission spectrum under one-photon (OPE) and two-photon (TPE) excitation, illustrating the excitation mode dependence of the emission of the carbon quantum dots.

At the OM2 we embrace the challenge of research for a better society starting from the very basic light-matter interactions to the establishment of clear design guidelines for optically responsive (nano)materials with application in biomedical research, optoelectronics and solar energy harvesting.

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NIR-responsive nanohybrids for efficient energy transfer

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Co-doped lanthanide nanoparticles (UCNPs), such as NaYF₄: Yb³⁺, Er³⁺, can convert two or more low-energy photons into high-energy photons due to their intra-configurational 4f electron transitions.¹ UCNPs usually emit ultraviolet-visible (UV-Vis) light upon excitation with near-infrared (NIR) light.¹ Moreover, UCNPs exhibit long luminescence times, high stability, no photoblinking or photobleaching, and biocompatibility. Thus, UCNPs are widely studied as energy donors in resonance energy transfer (RET) processes for many applications (Figure 1).²

However, three main requirements are needed to obtain efficient RET from UCNPs to an energy acceptor (EA) i.e. an organic dye, after excitation of the nanoparticle with NIR light: i) spectral overlap between UCNP emission and EA absorption; ii) highly emissive UCNPs; and iii) EA proximity to the UCNP surface.²

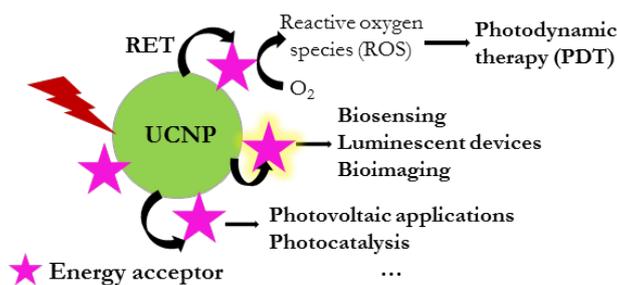


Figure 1- Schematic representation of resonant energy transfer (RET) processes based on UCNPs.

Firstly, the spectral overlap can be maximized by carefully choosing the dopants of the UCNPs to modulate the emission wavelengths and therefore match the EA absorption with the UCNP emission.^{1, 2} Secondly, we demonstrated that it is possible to control the shape of the UCNPs and their final size by changing the temperature at which the reactants are added and the temperature of the reaction, respectively.³

Moreover, the donor-acceptor distance can be modified through surface chemistry.¹ In order to modify the UCNP periphery to obtain efficient RET from UCNP to an EA, we developed two different strategies.

One strategy was based on the direct attachment of a photosensitizer (PS), specifically a BODIPY derivative (BDP) provided with a carboxylate group with a high affinity to the NaYF₄: Yb, Er NP surface.⁴ This method facilitated a high BDP loading and avoided the PS leaching from the nanohybrid.⁵ To improve the water dispersibility of the nanohybrid, the UCNP surface was simultaneously modified with a polyethylene glycol derivative (PEG-SH), leading to a water-dispersible UC@PEG-BDP nanohybrid.⁵

With the purpose of evaluating the nanohybrid capability for PDT, we carried out *in vitro* studies using SH-SY5Y human neuroblastoma-derived cells. About 50% of cell death was detected after irradiation at 975 nm (45 min, 239-mW cm⁻²) of UC@PEG-BDP incubated SH-SY5Y cells.⁵ In addition, toluidine-stained semithin section of the cells displayed abundant signs of cells damage, i.e., large vacuoles (black arrows in Figure 2A), swelling or picnotic cells. Most importantly, no damage was observed in cells irradiated in the absence of UC@PEG-BDP and in UC@PEG-BDP incubated cells in the absence of NIR light (Figure 2A). These results evidenced the high capability of the UC@PEG-BDP nanohybrid to generate reactive oxygen species, and mainly, singlet oxygen (¹O₂), thus, opening the way to use it in PDT.

The second strategy uses a water-soluble macrocycle, in particular cucurbit[7]uril (CB[7]). CB[7] interacts with metallic cations via the two carbonyl portals on the edges.⁶ We demonstrated that CB[7] was adsorbed on the surface of naked NaYF₄:Yb,Er NPs (UC_n) spontaneously, thus leading to bright and water-dispersible UC_n@CB nanohybrids.⁷ The application of these nanohybrids as scaffolds for EA was demonstrated by using two kinds of EA: i) methylene blue (MB), a well-known PS,⁷ and ii) CH₃NH₃PbBr₃ perovskites NPs (PK).⁸

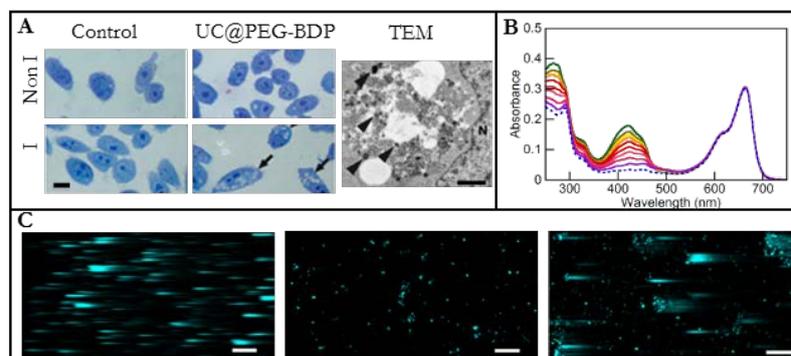


Figure 2- A) Semithin sections of SH-SY5Y cells (left) and UC@PEG-BDP incubated cells (centre) non-irradiated (top) and irradiated (bottom) with a 975 nm laser diode. Scale bar: 10 μm . Right: TEM images of UC@PEG-BDP incubated cells upon NIR-irradiation. Scale bar: 1 μm B) Absorption spectrum of an aqueous dispersion of UC@CB@MB and DPBF (green line) and its evolution after NIR-irradiation (up to 70 min). C) Confocal images at $\lambda_{\text{exc}} = 975$ nm, $\lambda_{\text{em}} = 420\text{-}500$ nm, and a dwell time of $2 \mu\text{s}\cdot\text{pixel}^{-1}$ of naked UCNP (left), UC_n@CB@PK_{CB} (centre), and UC_{OLA}@PK_{OCA} (right). Scale bar: 50 μm .

We showed that MB interacts with the free carbonyl portal of UC_n@CB, leading to UC_n@CB@MB nano hybrids.⁷ The high efficiency of the nano hybrid for ¹O₂ generation under NIR irradiation, confirmed by using diphenylbenzofuran (DPBF) as a probe (Figure 2B),⁷ proved the potential of these nano hybrids in PDT.

In addition, CB[7] was used to assemble NaYF₄: Yb, Tm and CH₃NH₃PbBr₃ NPs effectively.⁸ As a consequence, an extremely efficient RET from the UCNP to PK (ca. 100%) was observed after NIR irradiation of the UC_n@CB@PK_{CB} nano hybrid. We envisaged that confocal microscopy could be useful to visualize the assembly efficiency and the homogeneity of the sample. The images of small aggregates of the UCNPs at short dwell times ($2 \mu\text{s}\cdot\text{pixel}^{-1}$) displayed a tail in the fast-scan direction. Interestingly, we could estimate the luminescence decay values from the intensity profiles obtained by drawing a line along the tail of the aggregates.⁸ Figure 2C evidences the homogeneity of the sample and the drastic shortening of the UC_n@CB@PK_{CB} photoluminescence lifetime. For comparative

purposes, we synthesized a similar nanohybrid via ligand chain interdigitation between oleate-capped UCNPs (UC_{OLA}) and octylammonium-stabilized PKs (PK_{OCA}). However, the images showed the low homogeneity of the $UC_{OLA}@PK_{OCA}$ sample.⁸

These results indicated the suitability of the CB[7] strategy to synthesize NIR-nanohybrids for highly efficient RET processes as well as the use of confocal microscopy to estimate UCNPs emission lifetimes and the sample luminescence homogeneity.

Acknowledgements: to MINECO and MECD for the financial support (projects CTQ2011-27758, CQT2014-60174-P, partially co-financed with FEDER funds, JdC and RyC to MGB and FPU to Laura Francés-Soriano) and UE/FP7-PEOPLE, PCIG09-GA-2011-294263). These studies have been performed at the ICMOL, UVEG (Spain).

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ABSTRACT OF THESIS ON PHOTOCHEMISTRY

Novel photocatalytic approaches for ecosustainable synthesis

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Over the past decades, **photochemical synthesis**, namely organic synthesis that uses light as source of energy to break and forge bonds, has vehemently re-emerged as an important theme of research in ecosustainable organic chemistry. Thanks to the possibility to excite a precise functional group in a (complex) reaction mixture and the peculiar reactivity of the so-formed excited states, photochemistry has become very attractive for both synthetic researchers and industrial practitioners. An added value to this activation manifold is represented by **photocatalysis**: in these reactions, the substrate does not absorb light directly, instead low-energy light is absorbed by a purposely added molecule (the photocatalyst, PC) that converts this energy directly into chemical energy for substrate activation, thus limiting competing undesired processes. This activation can occur according to three different mechanisms: Single-Electron Transfer (SET), Hydrogen-Atom Transfer (HAT) or Energy Transfer (ET) (see Figure 1). **In my Ph.D. thesis**, I studied their application for the **development of novel ecosustainable synthetic protocols** under the supervision of Prof. Maurizio Fagnoni at the University of Pavia.

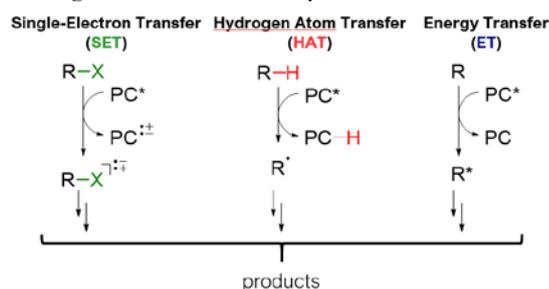


Figure 1. Possible mechanisms for substrate activation in photocatalysis.

Photocatalysis via Single-Electron Transfer (SET)

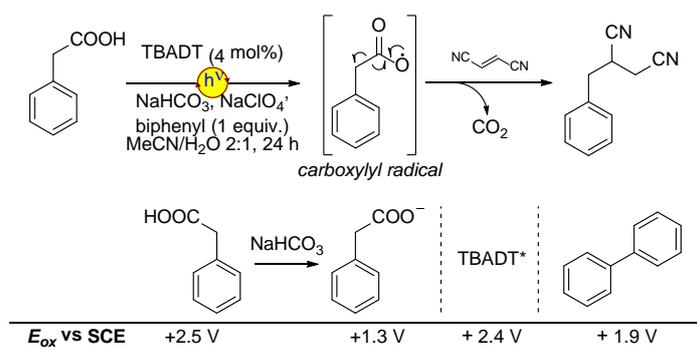
I developed **two synthetic protocols** for the generation of highly reactive intermediates via a photocatalyzed SET mechanism.

The **first study** focused on α -arylacetic acids as precursors of benzyl radicals to achieve the photocatalyzed benzylation of electron-poor olefins (see Figure 2, top).¹

The reaction proceeded smoothly in the presence of catalytic amounts of a W-based photocatalyst, TBADT (tetrabutylammonium decatungstate, $(\text{Bu}_4\text{N})_4[\text{W}_{10}\text{O}_{32}]$) under UV light ($\lambda_{\text{exc}} = 310 \text{ nm}$).² In the presence of a suitable base, i.e. NaHCO_3 , arylacetic acids were converted into the corresponding anions. Thanks to their lower oxidation potential ($E_{(\text{RCOO}^-/\text{RCOO}^-)} = +0.9\text{-}1.2 \text{ V vs SCE}$, compared to $+2.5 \text{ V vs SCE}$ for undissociated phenylacetic acid), carboxylates were readily oxidized by the excited state of TBADT (TBADT*, $E_{(\text{TBADT}^*/\text{TBADT}^*)} \sim +2.4 \text{ V vs SCE}$) to afford carboxylyl radicals. However, this redox step turned out to be strongly reversible, with adverse effects on reaction yields. We solved this *empasse* by introducing biphenyl ($E_{(\text{BP}^+/\text{BP})} = +1.9 \text{ V vs SCE}$) as a redox mediator.³ Thus, TBADT* was reductively quenched by biphenyl affording the radical cation of the latter: thanks to the stability and the oxidizing power of this intermediate, back electron transfer was inhibited, thus promoting the desired reactivity. Once generated, carboxylyl radicals broke apart yielding benzyl radicals, which were trapped by electrophilic olefins (e.g. fumaronitrile, Figure 2, top) in a Giese reaction. The reaction tolerated several substituents on the aromatic ring (whether electron-donating or electron-withdrawing) and could be successfully extended to heteroaromatic analogues.¹

The **second study** focused on the development of unconventional precursors of acyl radicals. A possible approach is the direct oxidation of aldehydes, however this process is highly challenging ($E_{(\text{RCHO}^+/\text{RCHO})} > +2 \text{ V vs SCE}$), so that an alternative approach is needed. A possible solution relies on the use of an electroauxiliary moiety in place of the formyl hydrogen: accordingly, in this work acylsilanes were used as a source of acyl radicals under photocatalytic conditions (see Figure 2, bottom) thanks to their lower redox potential ($E_{(\text{RCOTMS}^+/\text{RCOTMS})} < +1.5 \text{ V vs SCE}$) and favourable mesolytic cleavage of the $\text{C}(\text{sp}^2)\text{-Si}$ bond (see Figure 2, bottom).⁴

- Photocatalyzed generation of benzyl radicals (Ref. 1)



- Photocatalyzed generation of acyl radicals (Ref. 4)

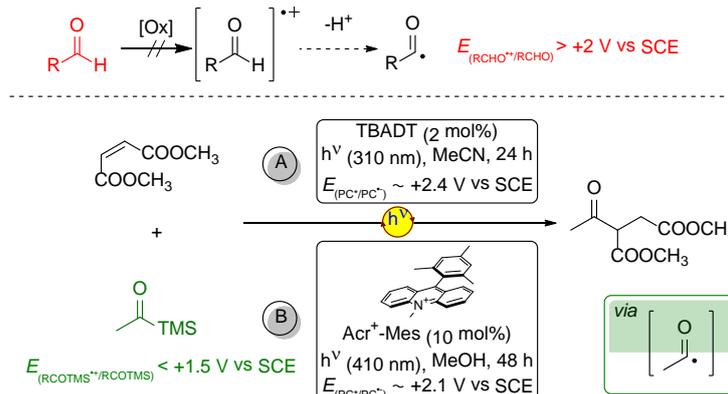


Figure 2. Generation of benzyl radicals and acyl radicals via photocatalyzed SET.

Being complementary to studies already present in the literature, our protocol allowed to generate aliphatic acyl radicals under mild conditions both under UV (Figure 2, bottom: condition A) and visible light irradiation (Figure 2, bottom: condition B), as well as under flow and sunlight-promoted conditions. In the reaction, the excited state of the photocatalyst (either TBADT or 9-mesityl-10-methylacridinium tetrafluoroborate) was responsible for the oxidation of the acylsilane to afford the corresponding radical cation. This unstable intermediate undergoes a mesolytic cleavage to produce a positively charged trimethylsilyl moiety and an acyl radical. The latter was trapped by

electrophilic olefins (e.g. dimethyl maleate, Figure 2, bottom) to afford Stetter adducts in high yields.⁴

Photocatalysis via Hydrogen Atom Transfer (HAT).

HAT is a chemical transformation consisting of the **concerted movement** of two elementary particles, i.e. a proton and an electron, between two molecules in a single kinetic step.⁵ In the perspective of photocatalysis, this transformation involves a substrate containing an X-H bond and the excited state of a photocatalyst. The net result is the generation of the reduced state of the photocatalyst and, most importantly, a neutral radical X•, which can be used in organic reactions.

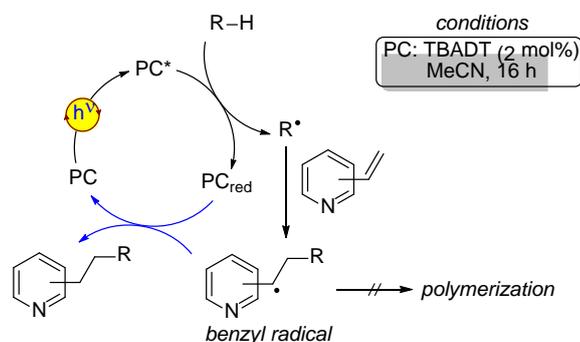


Figure 3. Synthesis of alkylpyridines from vinylpyridines via photocatalyzed HAT (Ref. 6).

In particular, the addition of radicals onto vinylaromatics to form benzyl derivatives has always represented a challenging task, mainly due to competitive polymerization. A photocatalytic approach could represent a godsend since it could hamper undesired processes due to the efficient interception of the radical adduct in the closure of the photocatalytic cycle (Figure 3, blue arrows).

In my thesis, I designed a synthetic strategy for the synthesis of alkylpyridines from vinylpyridines based on the use of TBADT as the photocatalyst.⁶ Its excited state is known to cleave homolytically (in a high chemo- and regioselective fashion) C-H bonds in a variety of organic derivatives ($R-H$) through a Hydrogen Atom Transfer (HAT) process.^{2,7} Moreover, its reduced state is a moderate reductant, suitable to quench the benzyl radical generated during the reaction (see Figure 3).

This approach fits well in the literature, being the first synthetic protocol for the functionalization of styrene-like molecules by neutral radicals generated via a photocatalyzed HAT. Additionally, alkyipyridines are important building blocks for the preparation of compounds with biological activity, but also valuable molecules due to their commercial applications.⁶

Photocatalysis via Energy Transfer (ET).

During my visiting period at the University of Wisconsin-Madison, in Prof. Tehshik P. Yoon's research group, I had the opportunity to study the synthetic potentialities of Energy Transfer as a pathway for substrate activation in photocatalysis. Yoon's group has studied in depth the use of triplet excited states of transition-metal complexes (TMCs) for visible-light sensitization in organic reactions.⁸⁻¹⁰ One of the most promising reactions was the [2+2] photocycloaddition to obtain cyclobutanes.

I reasoned that, given the importance of cyclobutanes in natural molecules with biological activities, the chance of late-stage diversification of this core would be extremely desirable. Accordingly, I started looking for a compatible and versatile synthetic handle to be introduced in the substrate that could bear conditions for the photocatalyzed [2+2] cycloaddition and I came across the class of vinyl boronate esters.

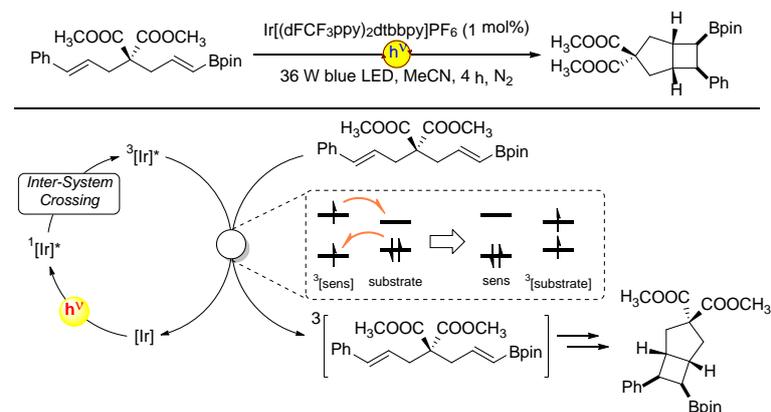


Figure 4. Visible-light promoted [2+2] cycloadditions of vinyl boronate esters.

The scope of intramolecular photocycloadditions between styrenes and vinyl boronate esters to achieve cyclobutylboronates (see Figure 4) is wide and the reaction tolerates both electron-donating and electron-withdrawing groups, heteroaromatic rings and olefins. With the hypothesis that these products could serve as branching points for the rapid diversification of these novel scaffolds in medicinal chemistry campaigns, the group is currently attempting chemical manipulation of the boronate functionality.

In conclusion, SET, HAT and ET are synthetic methodologies that complement each other in photocatalyzed organic reactions and their study has allowed to pursue the three most important objectives in organic synthesis: ecosustainability, selectivity and efficiency.

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The Porphyrin Triplet State: from porphyrin-2,2':6',2''-terpyridine conjugates to photocatalysis

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The doctoral work here presented deals with three separate projects, all sharing a main theme: porphyrins. It has been carried out under the supervision of Catherine E. Housecroft and Edwin C. Constable at the Department of Chemistry of the University of Basel. It is available under the creative commons license at edoc.unibas.ch.

Part I deals with the synthesis of a new porphyrin-bis-terpyridine ruthenium complex and its application as a light harvester in regards to dye-sensitized solar cell applications (DSCs).^[1]

Part II moves the focus onto photocatalysis. A four-fold symmetry water-soluble free-base porphyrin catalyst has been prepared and employed as sensitizer to promote triplet molecular oxygen to its singlet state. Singlet oxygen is active against simple aminoacids, an evidence that made us interested in assessing whether photocatalytic activity would be retained in microorganisms. We stepped into photodynamic therapy by applying our photocatalyst to expose *E. coli* liquid cultures to photochemically-generated singlet oxygen.^[2]

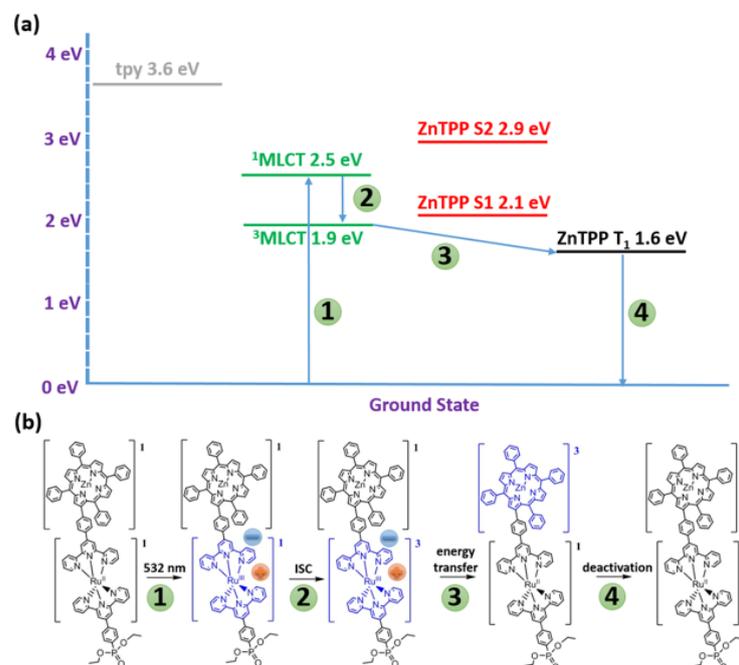
Part III deals with the application of photocatalyst employed in Part II to organic synthesis. The singlet oxygen reactivity is screened towards aliphatic and aromatic sulfides. We collected examples on commercially available substrates, both symmetric and asymmetric, of natural and pharmaceutical origin. The main goals were the assessment of catalyst's site-specificity, tolerance to coexisting functional groups and dependence on diverse core structures (unpublished results).

Part I: Porphyrin-bis-terpyridine Ruthenium Dyad

In Scheme 1 is depicted the dyad we prepared and tested as dye for light harvesting applications. Phosphonic esters are known to bind to nanoparticulate titania through an esterification mechanism analogous to the one of carboxylic esters, resulting in covalent links of the type P-O-Ti. Confident that our dye would anchor, we used solid state absorption spectroscopy to check whether or not a TiO₂

transparent electrode soaked in a solution of the dye had incorporated it. Encouraged by this positive result, we decided to build a solar cell based on our dye. Despite the fact that the electrode had incorporated the dye, the photoconversion efficiency is too low for the dye to be used in solar cell applications. This unexpected result triggered our interest in gaining a deeper understanding of the energy-transfer processes that dissipate the energy gained through excitation.

Scheme 1. (a) Energetic level diagram for the dyad (b) energy-transfer scheme upon $^1\text{MLCT}$ excitation at 532 nm in the dyad. For the middle



species in the scheme, the negative charge could be on either terpyridine (tpy) domain. ISC = intersystem crossing.

Transient absorption experiments on the free dye revealed that no matter if the initial excitation energy resides on $\{\text{Ru}(\text{tpy})_2\}$ or zinc tetraphenylporphyrin $[\text{Zn}(\text{TPP})]$, eventually energy moves to the porphyrin triplet state T_1 within tens of picoseconds. This state represent the lowest excited state and returns to the ground state by means of thermal deactivation.

Part II: Antimicrobial Activity of Singlet Oxygen

The term *oxidative stress* has been coined to indicate the action of reactive oxygen species (ROS), such as singlet oxygen, on living organisms. The only defence mechanism against singlet oxygen seems to appear in plants, whit light-harvesting complexes (rich in chlorophylls) having a triplet state that well suits the energetic demand for oxygen sensitization. Packed in the supramolecular assembly, molecules of β -carotene offer a protection against it; in fact, carotene's triplet state lies lower than oxygen's singlet state and inverse energy transfer quenches the reactive species. As the amount of ROS increases, more and more damage accumulates, until the point at which the organism undergoes the so-called programmed death or apoptosis. The fact that an organism would program its own death if properly stimulated has opened the way to the use of singlet oxygen as a therapeutic tool. A whole new field has developed, and nowadays photodynamic therapy has found medical applications.

The major part of the dyes used in oxygen sensitization is of synthetic origin and light-independent side effects cannot be excluded *a priori*, creating the need for the dye confinement from cytoplasmic media. The use of carriers like polymersomes seemed appealing, since it ensures dye encapsulation, but allows the diffusion of reactive species. To test our hypothesis, we determined the correlation between the colony-forming units (CFU, number of bacteria able to multiply) of *E. coli* in presence of photocatalyst-loaded polymersomes upon irradiation and in dark conditions (Fig. 1). Singlet oxygen generation induces oxidative stress, which ultimately leads to the degradation of a plethora of biomolecules and death of the microorganism. CFU values were drastically reduced only under the combined action of irradiation and sensitizer. As expected, exposure to empty polymersomes did not influence bacterial growth and photocatalyst-loaded polymersomes had no effect in the dark.

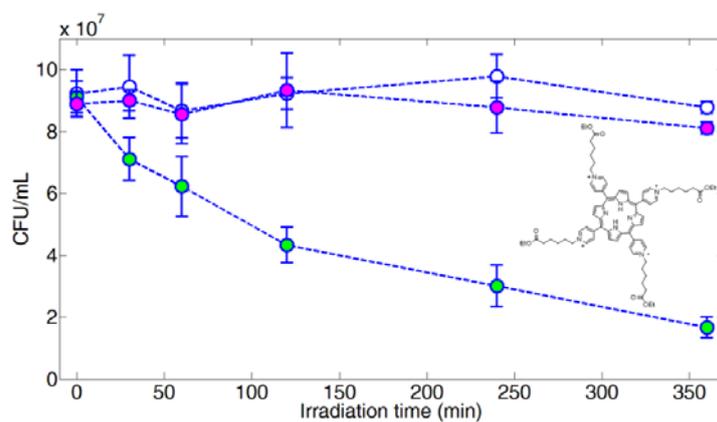
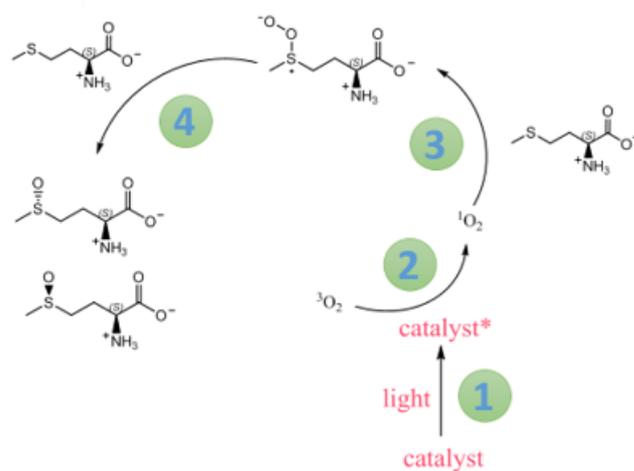


Figure 1. CFU for *E. coli* culture upon addition of photocatalyst-loaded polymersomes and irradiation with a LED red source ($\lambda_{\text{max}} = 660 \text{ nm}$) (green circles), no irradiation nor addition of external factors (empty circles), no irradiation but addition of photocatalyst-loaded polymersomes (magenta circles). (inset) Molecular structure of the photocatalyst employed.

Part III: Singlet Oxygen in Photocatalysis

A well-established reaction in organic chemistry is the oxidation of thioethers. Depending on the oxidant, both the sulfoxide and the sulfone can be easily obtained in high yields. The advantage of running such reaction photochemically is the selective formation of the sulfoxide, coupled to a quantitative yield and absence of by-products. The reaction proceeds as illustrated in Scheme 2 and both diastereoisomers are expected to be formed in equal amounts.



Scheme 2. Mechanism for the photocatalytic oxidation of thioethers.

Interestingly, when the photocatalyst described in Part II was added (0.25 mol%) to chiral substrates, diastereomeric excess to purity was observed in the product (Figure 2). The examples we investigated are taken from the flavour- (grapefruit-smelling isothiocineole and passion fruit-smelling 2-methyl-4-propyl-1,3-oxathiane) and pharma industry (antiretroviral drug lamivudine and antibiotic cephalixin). Oxygen addition to the sulfur atom seems to proceed counterintuitively if we think in terms of steric hindrance (see what happens to oxathiane). A clear mechanism is not yet at hand at this point, but further elucidatory studies are being conducted.

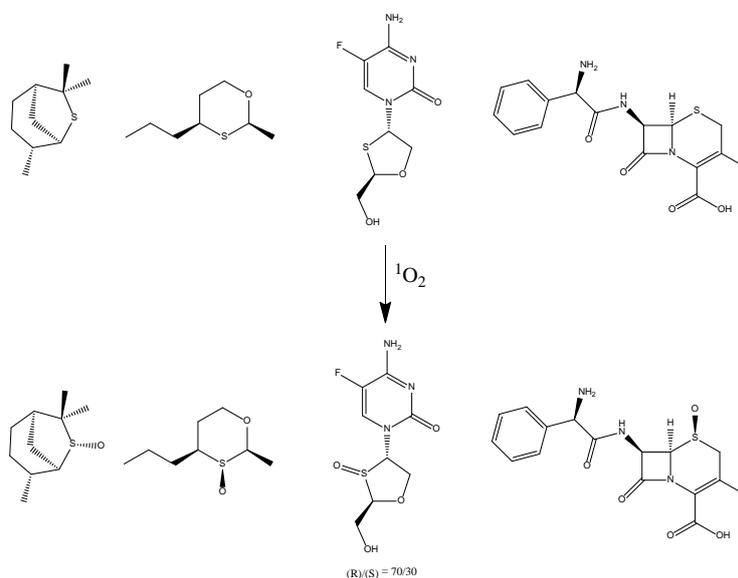


Figure 2. (top, from left to right) isothiocineole, 2-methyl-4-propyl-1,3-oxathiane, lamivudine and cephalexin; (bottom) solely observed products.

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CONFERENCE REPORT

Conference Report on the 27th IUPAC International Symposium on Photochemistry

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The 27th edition of the IUPAC International Symposium on Photochemistry was held from the 8th to the 13th of July 2018 on the campus of the University College Dublin in Ireland. The event was co-chaired by Professor Susan Quinn (University College Dublin) and Professor Miguel García-Garibay (UCLA, USA). This biennial conference is a main event in every photochemist's agenda and, in compliance with previous symposia, it attracted a large amount of leading scientists in the field from all over the world. Over 500 delegates from 41 countries (Figure 1) contributed to the success of the event. 8 plenary and 200 oral sessions were presented. During six days, the participants enjoyed a perfect organization which enabled a fruitful exchange on the latest developments around topics such as organic, inorganic, and supramolecular photochemistry, luminescent probes, photochemical synthesis, biomimetic and artificial

photosynthesis, solar energy conversion and photovoltaics, photobiology, bioimaging, photoswitching and photochromism, photoactive (nano)materials, nonlinear and ultrafast processes, spectroscopy, and environmental photochemistry, to name a few. This collection corresponded well with the motto of the 27th PhotoIUPAC: "Tackling the world's challenges with photochemistry".

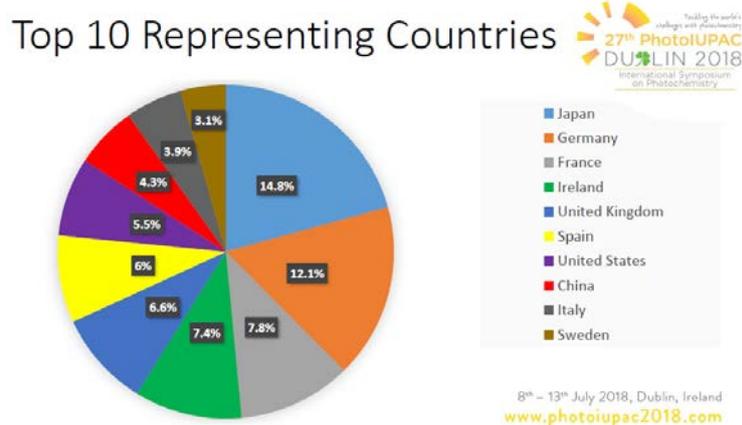


Figure 1. Most representing countries at the 27th PhotoIUPAC conference.

The conference showcased the research of some of the most emblematic leaders in the field. In total eight Plenary Lectures were presented. These covered topics such as molecular photovoltaics (Professor Michael Grätzel, EPFL, Lausanne, Switzerland and Professor Laura Herz, University of Oxford, UK), solar energy conversion and hydrogen/oxygen production (Professor Tetsuro Majima, Osaka University, Japan), theoretical photochemistry (Professor Ana Krylov, University of Southern California, Los Angeles, USA), photonic control of charge transfer (Professor Julia A. Weinstein, The University of Sheffield, UK), enantioselective catalysis in photochemical transformations (Professor Thorsten Bach, Technical University Munich, Germany), photofunctionalization of macromolecular systems (Professor Christopher Barner-Kowollik, Queensland University of Technology, Brisbane, Australia and Karlsruhe Institute of Technology, Germany), and chemically controlled activation of fluorescent probes (Professor Gonzalo Cosa, McGill

University, Quebec, Canada). The Plenary Lectures were complemented by 16 Invited Lectures covering topics such as photoswitches, photoactive (nano)materials and crystals, systems for photorelease, luminophores for bioimaging, transient spectroscopy of DNA binders and photoprocesses in DNA assemblies, photocatalysis, fluorescence in biological systems, etc. Further, *ca.* 180 Contributed Lectures and more than 200 posters provided the opportunity to catch up with the latest on photochemical research.

The conference was also the stage for the award of two EPA Awards: the Best PhD thesis for Dr. Victor Gray (Chalmers University of Technology, Gothenburg, Sweden) the EPA-PPS Prize for Professor Santi Nonell (Ramon Llull University, Barcelona).

The program offered space for several social activities such as excursions or opportunities to get together during evenings and know the beautiful and active city of Dublin. Plus the running FIFA World Cup offered plenty of excuse for a pub visit. Without doubt the PhotoIUPAC 2018 symposium will be remembered as the perfect continuation of a long tale of success which started 52 years ago. Many thanks to the Co-chairs and their countless members of the Organizing Team for flawless assistance, the assembly of a very balanced scientific program, and a wonderful time in Ireland. This has set the standards for the 28th IUPAC International Symposium on Photochemistry to be held in Amsterdam in July 2020 (<https://www.aanmelder.nl/iupac2020>), chaired by Professor Fred Brouwer.

Conference report: 26th Lecture Conference on Photochemistry, Garching/Munich

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From September 10 to 12, 2018, the *26th Lecture Conference on Photochemistry* of the GDCh Division of Photochemistry was held at the Garching campus of the TU Munich. More than 270 participants from universities, research institutes and industry joined this meeting to discuss the most recent results and developments of photochemical and photophysical research.

The scientific program was an impressive testament to the fact that photochemistry is still a highly interdisciplinary research area. Hence, the diverse aspects of photochemistry were covered in 12 invited lectures, 29 short oral contributions and more than 90 poster presentations, that ranged from the traditional disciplines, namely synthesis as well as photophysical, spectroscopic and mechanistic studies, to applications in materials science, biology and medicine.

After the opening remarks of the organizer, Thorsten Bach (TU Munich), the scientific program was inaugurated with a lecture from Nobel laureate Ben Feringa (University of Groningen). As plenary speaker in the session "Photochemical Switches", he gave an account of his seminal work on the control of molecular motion with light. In this context, the increasing relevance of photoswitches in life sciences was impressively documented in the session "Optogenetics - Photopharmacology" that was introduced by Dirk Trauner (New York University) with a presentation about photochemical control of biological processes by photoswitchable azobenzene-lipid combinations.

The subject area "Photochemistry of New Materials" was headed by Tanja Weil (MPI for Polymer Research, Mainz) who presented the fascinating properties of fluorescent nanodiamonds and demonstrated how the knowledge of the photophysical processes may be used for the optimization of material properties.

The topic "Spectroscopy und Technology" was represented by Johan Hofkens (KU Leuven) with a lecture about the remarkable methods and applications of single molecule microscopy ("The power of the ONE"). Specifically, he showed the great potential of this method to

be used for super resolution microscopy with exemplary studies on perovskite nanoparticles.

It is good tradition of this meeting to integrate an "Industrial Photochemistry" session with contributions from the chemical industry. This year, Xenia Beyrich-Graf (BASF SE, Ludwigshafen) opened the session and gave an instructive insight how to improve the properties of LED lamps significantly with fluorescent organic color converters. In another presentation, Rolf Klein (TechnoScriptum, Groß-Umstadt) reported about the use of plastic additives to adapt absorption properties that enable more efficient and versatile laser plastic welding applications. In the concluding lecture of this session, Daniel DiRocco (Merck Sharpe and Dohme, Rahway) showed that the development and optimization of photocatalytic syntheses is by now also a well-established research field in industrial chemistry.

Kevin Booker-Milburn (University of Bristol) presented in the session "Synthetic Organic Photochemistry" how photoreactions can be performed efficiently in organic synthesis at very large scale with *batch* and *flow* methods.

Finally, two large sections were dedicated to the current trend in photochemistry to induce photoreactions with visible light and photocatalysis. As the opener, Tehshik P. Yoon (University of Wisconsin-Madison) presented remarkable examples for stereocontrol in photochemical synthesis with visible light by combination of chiral Lewis acids and transition metal-photocatalysts. Along the same lines, Paolo Melchiorre (University of Tarragona) showed that the combination of visible-light photocatalysis and asymmetric organocatalysis leads to novel enantioselective photoreactions.

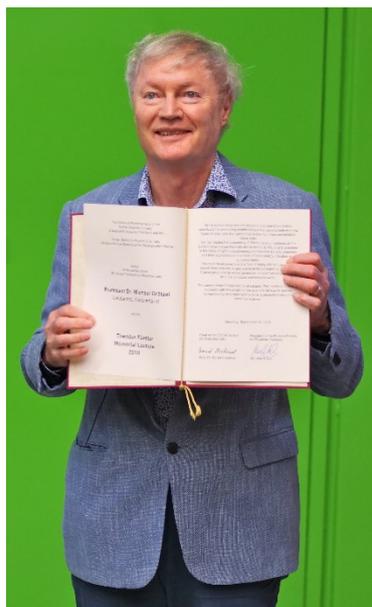
In the segment "Photoredox-Catalysis" Eric Meggers (University of Marburg) gave a lecture about the large synthetic potential of chiral-at-metal complexes as asymmetric catalysts for stereoselective photoreactions and discussed the relevant structures and mechanisms. In addition, David W. C. MacMillan (Princeton University) presented novel reactions and synthetic routes that are initiated by photoredox catalysis with visible light.

A highlight of the conference was the presentation of the "Theodor Förster Memorial Lecture", a prize that is awarded by the *Gesellschaft Deutscher Chemiker* and the *Deutsche Bunsen-Gesellschaft für Physikalische Chemie*. This year, Prof. Dr. Michael Grätzel, École Polytechnique Fédérale de Lausanne (EPFL), received this award in appreciation of his important contributions to photochemistry and materials science.

Specifically, Prof. Grätzel was honoured for his outstanding scientific impact in the area of photovoltaics, specifically his pioneering contributions in the development of a new type of solar cells that work with excellent performance at lower light intensities. After the award ceremony, Prof. Grätzel presented the *Theodor Förster Memorial Lecture 2018* with the title "Molecular Photovoltaics and Perovskite Solar Cells".

The most outstanding poster presentations of the conference were also honoured. Thus, from the many excellent contributions with high scientific quality and professional layout the posters of Monika Schildhauer (LMU München), Veit Georg Hänsch (HKI Jena), and Christoph Kerzig (Universität Basel) were awarded with the poster prize.

The *27th Conference on Photochemistry* will be held in September 2020 at the University of Kiel.



Prof. Dr. Michael Grätzel, awardee of the *Theodor Förster Memorial Lecture*



Awardees of the poster prize; from left to right: Monika Schildhauer, Veit Georg Hänsch, Dr. Christoph Kerzig, Prof. Dr. Burkhard König (Chair).

Picture credits: Bernd Strehmel

The CNRS-Summer School PHOTOCHEM on Preparative and Applied Photochemistry from 16th to 21st September 2018 in Anglet, France.

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Over the last decade, photochemistry has seen a remarkable comeback with significant contributions to green chemistry, organic synthesis, waste treatment, chemical engineering, continuous-flow technologies and new light sources.¹ At the same time, the neglect of this research area by academia as well as industry has caused an urgent need for training in photochemical principles, reactor engineering and applications. Historically, France has been a key location in photochemical research and manufacturing.^{2,3} The first comprehensive Summer School on Photochemistry, chaired by Dr Karine Loubière, was thus organized as an École Thématique of the Centre National de la Recherche Scientifique (CNRS). The event took place between September 16th and 21st 2018 at the Atlantic seaside location of Anglet in the Southwest of France (Fig. 1).⁴



Figure 1. Logo of the CNRS-Summer School PHOTOCHEM.

The main objective of the CNRS-Summer School was to offer high quality and multidisciplinary training in photochemistry. The scientific program was primarily intended for professionals in chemistry related areas, but in particular to:

- Chemists, especially those involved in green and sustainable chemistry, as the provided training in photochemical technologies and engineering enabled them to select the most suitable reactor, light source and operating conditions for any future applications.
- Chemical engineering specialists, who wanted to complement their professional skills with a fundamental knowledge of photochemical concepts and applications, and the subsequent impact on reactor engineering (considering radiative transfer in the reaction medium).
- All current (or potential future) users of photochemistry, particularly in research areas outside organic synthesis (health, materials, environment etc.).

The event was split into 5 modules with a total of 19 talks over 4 ½ days, delivered by 16 professionals from academia and industry, and a workshop & demonstration session. The topics covered fundamental concepts, organic synthesis, technologies, engineering and related applications of photochemistry (Table 1).

Module 1: Fundamental concepts of photochemistry	
1.1	Light absorption, electronically excited states, absorption spectra <u>Prof Thierry Pigot</u> , Univ. Pau Pays de l'Adour, IPREM, Pau, France
1.2	Photophysical deactivation of excited states, energy transfer and photochemical reactions <u>Dr Sylvie Lacombe</u> , CNRS, IPREM, Pau, France
Module 2: Applications of photochemistry in organic synthesis	
2.1	Structural diversity and molecular complexity through organic photochemical reactions <u>Dr Norbert Hoffmann</u> , CNRS, ICMR, Reims, France
2.2	Photoredox catalysis <u>Prof Louis Fensterbank</u> , Sorbonne Univ., IPCM, Paris, France
2.3	Sensitized photooxidations <u>Dr Esther Oliveros</u> , CNRS, France
2.4	Photopolymerization processes: State of the art, trends, perspectives <u>Prof Jacques Lalevée</u> , Univ. Haute-Alsace, IS2M, Mulhouse, France
2.5	Industrial photochemistry: An appraisal <u>Prof André M. Braun</u> , Karlsruhe Institute of Technology, Germany
Module 3: Photochemical technologies	
3.1	Laboratory-scale photochemistry: Reactors and light sources <u>A/Prof. Michael Oelgemöller</u> , James Cook Univ., Townsville, Australia
3.2	Safety issues of industrial photochemistry and adequate responses <u>Alexander C. Peschl</u> , CEO, Peschl Ultraviolet GmbH, Mainz, Germany

3.3	Solar Photochemistry <u>A/Prof. Michael Oelgemöller</u> , James Cook Univ., Townsville, Australia
3.4	Microstructured technologies and flow photochemistry <u>Dr Karine Loubière</u> , CNRS, LGC, Toulouse, France and <u>A/Prof. Michael Oelgemöller</u> , James Cook Univ., Townsville, Australia
3.5	LED and OLED Technologies <u>Laurent Massol</u> , CEO, LED Engineering Development, Montauban, France
3.6	3-6 Practical workshop and demonstrations various exhibitors
Module 4: Photochemical reactor engineering	
4.1	Design and dimensioning of industrial photochemical equipment <u>Prof André M. Braun</u> , Karlsruhe Institute of Technology, Germany
4.2	Physics and place of radiative transfer in the modeling of photoreactors for conception and optimization purposes <u>Prof Jean-François Cornet</u> , SIGMA Clermont, Institut Pascal, Clermont-Ferrand, France
4.3	Integral formulation and Monte Carlo method for the simulation of radiation transfer in photoreactors of any geometry <u>Dr Jérémi Dauchet</u> , SIGMA Clermont, Institut Pascal, Clermont-Ferrand, France
4.4	Influence of hydrodynamic conditions in photoreactor modeling and its application in flow photochemistry <u>Dr Karine Loubière</u> , CNRS, LGC, Toulouse, France
Module 5: Environmental, energy, material and medical applications	
5.1	Environmental and energy applications of photocatalysis <u>Dr Nicolas Keller</u> , CNRS, ICPEES, Strasbourg, France
5.2	Photoswitchable materials and systems <u>Dr Rémi Métivier</u> , CNRS, PPSM, Cachan, France
5.3	Photodynamic therapy in medicine <u>Prof Gilles Lemerrier</u> , Univ. Reims Champagne-Ardennes, ICMR, Reims, France

Table 1. Program and speakers of the CNRS-Summer School on Photochemistry.

A highlight and well received event of the PHOTOCHEM Summer School was the practical workshop, which enabled all participants to experience photochemical reactors and light sources for themselves and to interact with experts from industry, research and education. The displays included home-made laboratory equipment as well as commercial devices. The participants were split into small

groups and subsequently rotated through the 6 stations over a 2 hours period (Table 2).

<p>Station 1: Laboratoire de Génie Chimique (LGC) (Robbie Radjagobalou and Dr Jean-François Blanco) <i>Polymeric tube LED-driven microreactors for laboratory photochemistry</i></p>	
<p>Station 2: Corning (Dr Marc Winter) <i>Laboratory and mesoscale microstructured photoreactors</i></p>	
<p>Station 3: Institut des Sciences Analytiques et Physico-Chimique pour l'Environnement et les Matériaux (IPREM) (Dr Mickaël Le Behec) <i>Reactor for standardized tests of photocatalytic materials for NO_x removal</i></p>	
<p>Station 4: Peschl Ultraviolet GmbH (Alexander C. Peschl) <i>Industrial photochemical technologies and their design</i></p>	
<p>Station 5: LED Engineering Development (Laurent Massol) <i>LEDs and LED panels</i></p>	
<p>Station 6: Laboratoire de Chimie de Coordination (LCC/ENSIACET) (Dr Odile Dechy-Cabaret) <i>Teaching and training of microscale organic synthesis: A common chemistry/chemical engineering tool</i></p>	

Table 2. Program and exhibitors of the practical workshop and demonstrations.

The Summer School was fully booked with 55 participants, of which 20% came from industry (Arkema, Minakem, Corning, Firmenich and Pechl UV), 53% were academic researchers, 20% were PhD students or postdoctoral fellows and 7% were engineers and technicians (Fig. 3). Upon registration, all participants were asked to provide a short paragraph on their research background & interests. 76% of the participants came from a Chemistry discipline, but 23% had a Chemical Engineering background. While the majority of the participants came from France, 4 participants came from Germany, Switzerland or Australia, respectively.



Figure 3. CNRS-Summer School Picture.

Overall, the CNRS-Summer School PHOTOCHEM was a huge success with lively discussions and engaging interactions between participants and speakers. It clearly demonstrated a strong interest in all aspects of photochemistry. The Summer School delivered high-level teaching and training of photochemical principles, technologies, photoreactor engineering and applications to industrial and academic participants. It is hoped that the Summer School will be repeated in the foreseeable future and will be offered to a broader European audience.

Acknowledgements. The CNRS-Summer School was financially supported by the Centre National de la Recherche Scientifique (CNRS), the Institut National Polytechnique de Toulouse (INP Toulouse), the Agence Nationale de la Recherche (ANR, PICPOSS proposal ANR-15-CE07-0008), the Laboratoire de Génie Chimique (LGC), the CRITT Génie des Procédés et Technologies Environnementales (CRITT GPTE), the **Institut de Chimie**

Moléculaire de Reims (ICMR), the International Consortium on Eco-conception and Renewable Resources (INCREASE), the Laboratoire de Chimie de Coordination (LCC), the Université de Pau et des pays de l'Adour, the Institut des Sciences Analytiques et Physico-Chimique pour l'Environnement et les Matériaux (IPREM), the Société Française de Génie des Procédés (SFGP) and the Société Chimique de France (SCF). The authors thank all members of the scientific committee for their support. MO also thanks the Australian Research Council for support (ARC, DP130100794).

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AWARDS

Abderrazzak Douhal, professor at the “Universidad de Castilla la Mancha” (Spain) awarded by the Japanese Photochemistry Association for his work on Photochemistry of MOFs

The Japanese Photochemistry Association (JPA), in its annual scientific meeting (5-7 of September 2018, Kwasei Gakuin University, JAPAN), has awarded Abderrazzak Douhal, Professor of Physical Chemistry at the University of Castilla la Mancha (UCLM, Toledo, Spain), the Elsevier Lectureship Award in its 5th international edition. The JPA’s award recognizes the work of Prof. A. Douhal for his research on Zr-based metallic organic frameworks (MOFs) photochemistry and applications in lighting and photosensing. MOFs are being intensively studied due their potential applications in several research fields of science and technology, like for example catalysis, gas separation and storage, photonics, drugs delivery, and photosensing. At the JPA’s meeting, Prof. A. Douhal has delivered a lecture showing the recent advances in the photochemistry and photophysics of MOFs with possible applications in LEDs, photodetection of nitroaromatics-based explosives, and drugs delivery.



Figure 1. Professor Hitoshi Tamiaki, President of the JPA, and Prof. Abderrazzak Douhal at the awarding ceremony.

Abderrazzak Douhal graduated and received Ph.D. degrees in chemistry from the University of Kadi Ayyad (Marrakech). He later worked at the Institute of Physical Chemistry of CSIC (Madrid), at the Institute for Molecular Science (Okazaki, Japan) and at University of Paris-Sud/CNRS (France). He was a visiting researcher at California Institute of Technology (Pasadena, USA), collaborating with Prof. Ahmed H. Zewail (Chemistry Nobel Prize winner, 1999). Since 1998, he is heading the Femtoscience and Microscopy research group at the Faculty of Environmental Sciences and Biochemistry at the UCLM. His current research focuses on studying photoevents of molecules and materials in molecular cages and pores of nanosystems of relevance in nanoscience and nanotechnology, using ultrafast laser-based techniques.

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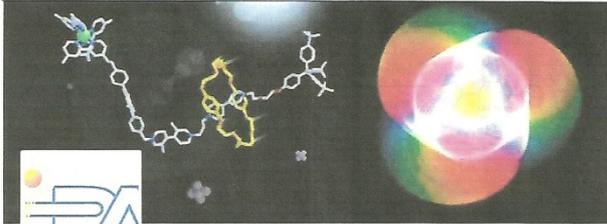
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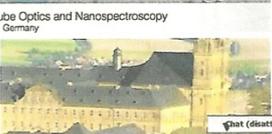
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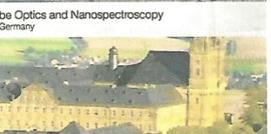
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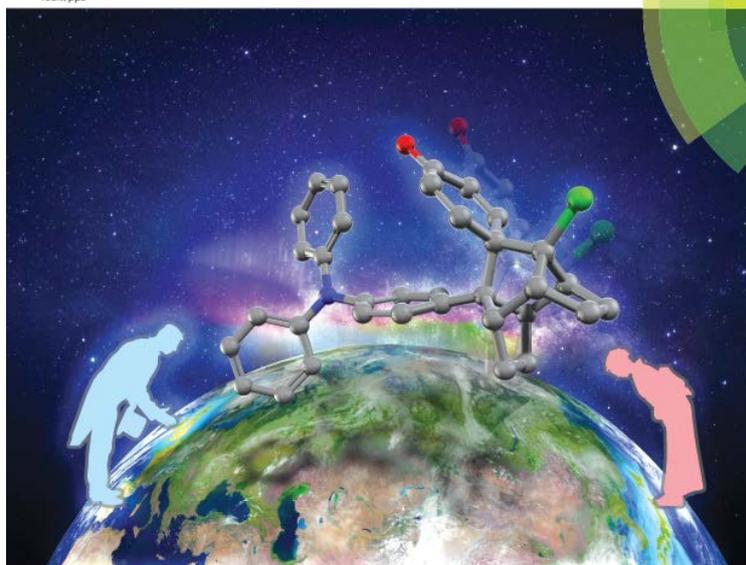
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