

NEWSLETTER

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Past President and PPS matters



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EDITORIAL**President's Letter**

Dear EPA members

Time has passed quickly and I am already reaching the end of my second year as the EPA President. I have been in the Executive Committee (EC) of the EPA since the Ferrara IUPAC meeting in 2010, first as Associate Editor of the EPA newsletter and the last two years as the President. In the two coming years, I look forward to supporting the work of the EC and EPA as Past President. This is becoming official at the assembly of the General Council on July 12th in Dublin during the 27th IUPAC Symposium on Photochemistry held in Dublin (8th-13th of July).

According to the statutes of our Society, the president should be a member of the EC, as he/she should be well aware of the different activities and important issues concerning our Society. The candidate to the next presidency is Maurizio D'Auria from the Università degli Studi della Basilicata, Italy. He has been The Editor of the EPA Newsletter since July 2012. You will find his views on his role as EPA President in the June 2018 issue of the Newsletter. Our Past President, David Worrall, leaves the EC after having been successively in charge of PPS matters, President and Past President for six years in total. Roberto Improta will also step down after six years in his role as head of New Information Technologies. Andrew Keating, in his role of Industry Liaison, has taken on many commitments in his company and he has already stepped down from the EC. We thanks are due to them for their contributions to the committee.

In order for the EC to work efficiently during the last two years, Uwe Pischel was invited to join the EC as Guest Editor of EPA Newsletter for the last two years and he has also been in charge of Public Relations.

The EC have decided that Alexandre Fürstenberg, our treasurer since July 2012, will continue in this role. He has done an excellent job, being wholly involved in this work and would be extremely difficult to replace.

New members will join the EC. As candidates, we propose Uwe Pischel, who has demonstrated his commitment to the EPA, as the Associate Editor of the EPA Newsletter and also to be in charge of Public Relations, as well as Alberto Credi and Norbert Hoffmann who are willing to take over the role of New Information Technologies and the EPA Newsletter Editor, respectively. Candidatures to join the EC to be in charge of the new EPA website are welcome.

My apologies for not being able to keep the EPA website permanently up-to-date. From the beginning of my term, the EC was involved in the building of a new EPA webpage, but after virtually no progress after one year, we decided to change website companies. I am pleased to inform you that we have now finally succeeded in presenting a new webpage. The new EC member in charge of the webpage will be devoted to completing contents and considering suggestions to further improve this webpage, which is, after all, the page of our Society.

In the programme that I presented at the General Assembly in Bad Hofgastein in February 2016, I took over several commitments, some of them not as easy to accomplish as I had thought. But one of the important tasks, from my point of view, was to increase the number of awards for EPA members who are relevant young and senior researchers, as well to researchers well-identified for their service to our Society. The progress on this matter will be presented at the GA at the Photoiupac Conference in Dublin.

This year, EPA has administered or pre-selected three awards in the area of photochemistry, the *EPA-PPS Prize* for the most highly cited paper published in PPS during the foregoing two calendar years, the *EPA Prize for Best PhD Thesis in Photochemistry*

published during the forgoing two calendar years, and the *Porter Medal*, in cooperation with our Inter-American and Asian/Oceanian photochemistry association counterparts, I-APS and APA.

The EPA PhD Thesis has been awarded to Victor Gray from Chalmers University in Göteborg for his work on Triplet-Triplet Annihilation Based Photon Upconversion on fluorescent DNA probes, whereas the EPA-PPS Prize has been awarded to Santi Nonell from Universitat Ramon LLull, Spain for his article on Singlet Oxygen Photosensitization by the Fluorescent Protein Pp2FbFP L30M. The two prizes will be presented during the Thursday afternoon session of the 27th IUPAC International Symposium on Photochemistry in Dublin, chaired by Susan Quinn and Miguel A. Garcia-Garibay.

The Porter Medal has been awarded to Haruo Inoue from Tokyo Metropolitan University and it will be given at the APC 2018 December in Taipei.

The relationship between the EPA and ESP and the RSC is in good shape; I will inform you of interesting development at the General Assembly in Dublin.

I am looking forward to seeing you on the occasion of the PHOTOIUPAC Conference in Dublin.

Julia Pérez Prieto

EPA, President

PUBLICATIONS

Photoluminescence of Graphene Oxide: Effect of pH, Surfactant and Polymer

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Abstract: Graphene oxide (GO), a water soluble precursor for the synthesis of graphene, has drawn tremendous interest in modern science not only for its unique properties but also for the varieties of its applications. Presence of different type of oxygen containing functional groups produces structural defect in graphene. These type of functionalization leads to a finite band gap in GO and it exhibits interesting photoluminescence which is limited in graphene due to zero band gap. The luminescence of GO can be utilized to produce optoelectronic devices, bio- analytical sensors, and bio-imaging setups. The present review aims to describe few selected themes of current interest dealing with tunable luminescence of GO by some external factors, e.g. pH, presence of surfactant, polymer. In view of the recently published data on the photoluminescence of GO, interaction with polymer by forming nano-composite, modulation of band gap by the surfactant and pH of the medium have been discussed to shed light on the tunable optical property of GO. These results helps to understand the tuning of photoluminescence of GO and provide ideas to develop various optoelectronic materials such as light emitting diodes, luminescent bio-trackers etc.

Optical properties and spectral modulation of the GO dispersions due to inherent inhomogeneity of the GO structure containing various domains and functional groups have become popular

research topic for the last ten years.¹⁻⁴ In spite of enormous experimental data on the photoluminescence of GO, exploration of the origin of luminescence and the tuning of the photoluminescence band by different external factor such as pH, addition of surfactant, presence of macromolecules still deserve rigorous experimental study. We have synthesized functionalized GO by modified Hummer's method⁵ and this synthesized GO shows photoluminescence in the UV region which is undoubtedly rare. Accordingly to Eda et al,¹ GO exhibits broad band photoluminescence due to π - π^* transition of the isolated sp^2 domains within the C-O sp^3 matrix and the variation in the band gap of the sp^2 domains. Photoluminescence maxima enables us to calculate the band gap as 3.2-3.6 eV and the size of the sp^2 clusters (~ 3 nm) as well as the average number of the aromatic ring (10) in a cluster.^{4,5} Broad nature of the photoluminescence band indicates the intrinsic structural inhomogeneity of GO and the observed band arises as a result of the result of the overlapping of various emission peaks because of intrinsic inhomogeneous structure.⁶

Dutta et al shows the effect of pH on luminescence spectra of the aqueous dispersions of GO.¹ UV fluorescence of GO obtained by exciting the both π - π^* and n - π^* bands is dependent up on pH of the medium. With increase in pH from 2.5 to 7, GO exhibits ($\lambda_{\text{ex}} = 240$ nm) a 32 nm blue shift a 12 nm blue shift is observed if the GO dispersion is excited at 280 nm (Fig. 1). Quasimolecular study indicates that the center of the fluorescence peak is dependent upon the functional groups present at the surface of GO.¹ The pH dependent emission spectra obtained by exciting the aqueous GO dispersion at 240 nm and 280 nm may be due to the presence of deprotonation of the oxygen containing functional group, such as carboxyl group. Observed blue shift in the emission spectra may rule out the possibility of the fluorescence from phenolic moiety as a red shift is expected due to phenolate anion at higher pH. But it is well known that the emission from the deprotonated form of aromatic carboxylic acid is blue shifted.⁷ According to Zhang et al, pH dependent emission properties is due to protonation and deprotonation of the fluorophore in GO.⁸

The plot of emission maxima against pH of the medium (Fig. 1) indicates that there are two sharp changes with two different slopes in the emission maxima (one at $\text{pH} \approx 3.5$ and another at $\text{pH} \approx 5.5$) in case of excitation of aqueous dispersion of GO at 240 nm but there

is only one such change at $\text{pH} \approx 3.5$ when the sample is excited at 280 nm. So, one may consider that the emission maxima obtained at 390 nm ($\lambda_{\text{ex}} = 240$ nm) in acidic pH (2.5 – 3) is due to dicarboxylic functional groups present at the surface of GO and the fluorescence ($\lambda_{\text{ex}} = 280$ nm) at 370 nm in acidic pH (2.5 – 3) may be due to single carboxylic group embedded in GO. Thus, excitation at 240 nm may be related with the excitation of dicarboxylic functional groups in an aromatic ring but the fluorophore connected with mono carboxylic group is excited at 280 nm.

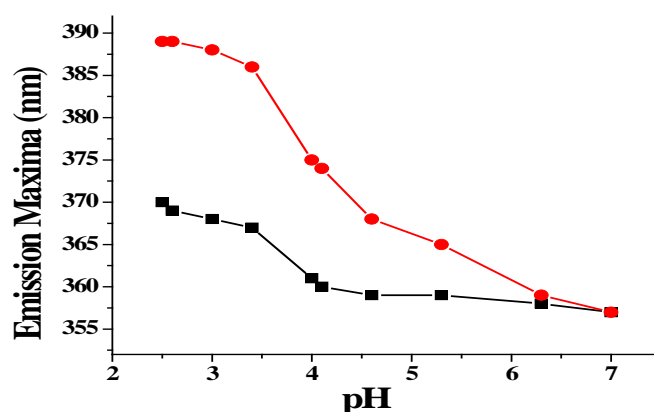


Figure 1. Plot of Emission maxima (nm) vs pH $\lambda_{\text{ex}} = 280$ nm (■), $\lambda_{\text{ex}} = 240$ nm (●)

Interplanar separation between the GO sheets is affected by the repulsion between the deprotonated carboxylic moiety ($-\text{COO}-$) located at the surface of GO and this leads to the increase in interlayer distance and thereby increase in band gap due to the repulsion between the negatively charge groups. Since, the band gap corresponding to the emission of dicarboxylic groups is affected more by the repulsion between higher numbers of $-\text{COO}-$ groups,

the extent of blue shift (≈ 32 nm) is greater in comparison to the blue shift (≈ 12 nm) for the fluorescence due to mono carboxylic group. Therefore, it may be concluded that the blue shift in the emission peaks by changing the pH from 2.5 to 7 is due to the movement of basal planes as a result of weakening of the inter layer π - π stacking interactions by the electrostatic repulsion between the deprotonated carboxylic moieties decorated the GO sheets (Fig. 2).

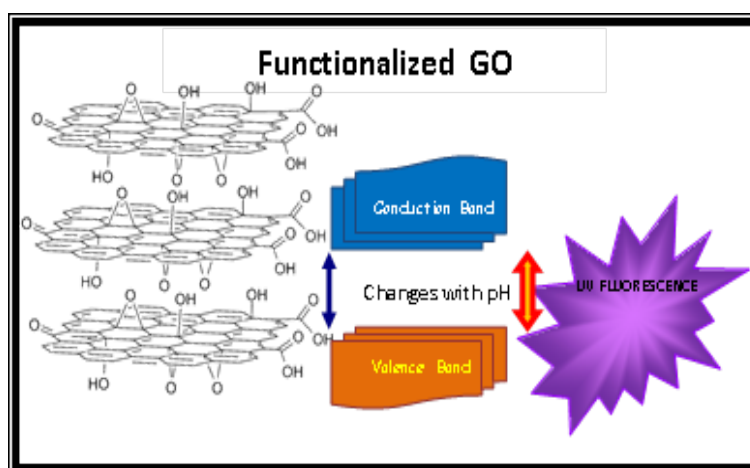


Figure 2. Schematic diagram representing the modulation of band gap and effect of pH on Photoluminescence property of GO

Due to availability of several oxygen containing functional groups (epoxy, hydroxyl, carboxyl) on the surface and sheet edges⁹ and high surface area, GO interacts with many organic, inorganic, biomolecules, polymers¹⁰ and surfactants¹¹⁻¹² to produce several GO based nanomaterials and nano-composites. Adsorption of surfactants on the GO surface plays an important role for many practical applications in Li-ion battery electrodes.¹³ Stability of the aqueous GO system may be enhanced by using surfactants due to the charged head groups of adsorbed ionic surfactants which provide electrostatic repulsion or steric interaction.¹² Although, there are a number of studies on the interaction of the surfactant with GO in water, the

current literatures do not show much focus on the optical properties and spectral modulation of the GO dispersions in the presence of surfactants. P. Saha et al have investigated the effect of an anionic surfactant sodium dodecyl sulphate (SDS) on the photoluminescence of GO in both acidic and alkaline medium.¹⁴ In the acidic medium ($\text{pH} \approx 2$) of GO, the surfactant, SDS, is adsorbed on the GO sheets and the critical surfactant aggregation constant (CSAC) is obtained at a SDS concentration greater than 2 mM. This results a marked 36 nm blue shift of photoluminescence spectrum (Fig. 3).

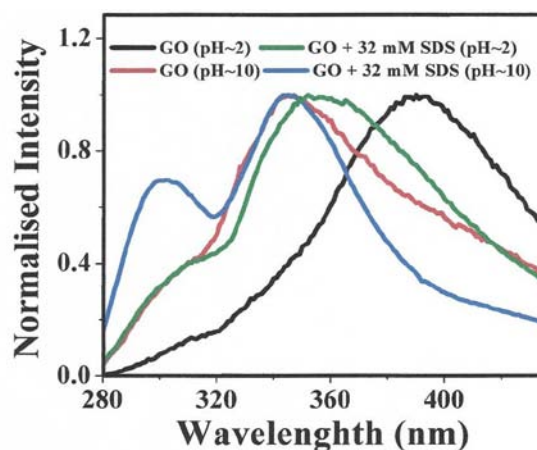


Figure 3. Normalized photoluminescence spectra of aqueous dispersion of GO ($\text{pH} \approx 2$), aqueous dispersion of GO ($\text{pH} \approx 2$) and 32 mM SDS, aqueous dispersion of GO ($\text{pH} \approx 10$), aqueous dispersion of GO ($\text{pH} \approx 10$) and 32 mM SDS ($\lambda_{\text{ex}} = 240 \text{ nm}$)

Adsorption of SDS on the GO sheets as hemispherical micelles (Fig. 4a), at $\text{pH} \approx 2$, modulates the photoluminescence band by providing a nonpolar confined environment. Hence, the acidic dispersion of GO in presence of 32 mM SDS shows a marked blue shift from 390 nm to 354 nm because of restricted solvent relaxation process inside the hemispherical surface micelles on the GO sheets (Fig. 4a). In alkaline medium, the aqueous dispersion of GO exhibits dual emission bands at 303 nm and 347 nm in presence of SDS (Fig. 3).

The negative surface charge and negatively charged carboxylate ions prohibits the SDS adsorption on the GO in alkaline medium ($\text{pH} \approx 10$). But, the repulsion between carboxylate ions on the GO surface increases the spacing between the GO layers⁵ and thereby favours the intercalation of the SDS molecules between the GO sheets (Fig. 4b).

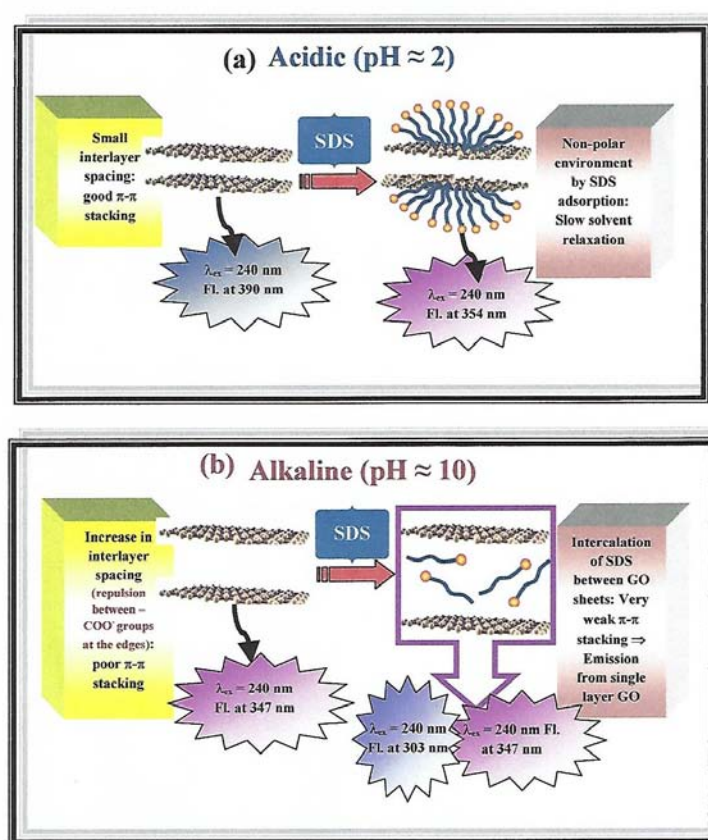


Figure 4. Schematic diagram representing the interlayer spacing between GO sheets and effect of SDS on the emission at (a) acidic ($\text{pH} \approx 2$) (b) alkaline ($\text{pH} \approx 10$)

The intercalation of SDS molecules between successive GO sheets weakens the π - π stacking interaction and this may give rise to largely separated layers of the GO moiety with almost isolated benzoic acid or phenol like species. The SDS intercalated largely separated layers of GO moiety which may be responsible for the fluorescence at 303 nm (Fig. 4b). Thus, the presence of two types of GO moieties, one with weak π - π stacking interaction another with almost disrupted π - π interaction between GO sheets containing benzoic acid or phenol like structure due to the intercalation of SDS, results dual photoluminescence band in alkaline medium (Fig. 3).

GO-based materials with oxygen functionalities on their basal planes facilitate surface modification for making composites with other materials, such as conducting polymers.¹⁵ The polar oxygen containing groups on the surface of GO make it dispersible in aqueous medium and may interact with the polar portion of polymer resulting in intercalated or exfoliated GO-polymer nanocomposites.¹⁶ Conductive polymer like polyaniline (PANI) has brought interest to the researchers because of its facile synthesis and wide environment friendly industrial applications due to desirable electrical, electrochemical and optical properties. In the PANI grafted graphene oxide, GO is actually an excellent template for aniline nucleation and polymerization to form nano-composite through possible interactions like π - π stacking, electrostatic interactions, hydrogen bonding and donor-acceptor interactions. Halder and co-workers have synthesized PANI grafted GO (GO-PANI) with a weight ratio of GO:aniline as 10:1 and studied the pH dependent tunable photoluminescence.¹⁷

Due to the complex nature of the electronic structure of the nano-composite based on GO grafted by PANI, the luminescence behaviour of GO-PANI dispersion shows interesting features dependent upon pH. Observation of dual emission at UV and visible region ($\lambda_{\text{ex}} = 280$ nm) is the most important finding of this work.¹⁷ This indicates the possibility of the presence of two types of emissive species. But at low pH (less than 3) and at high pH (greater or equal to 7), instead of dual emission, single emission band appears. The center of the emission bands at low (less than 3) and high (greater or equal to 7) pH was obtained at 410 nm and 345 nm, respectively. This observation is suggesting the change in relative contribution of two types of fluorophoric moieties at two different pH regions and the 3D Contour plot of the emission of GO-PANI at different pH (Fig.

5) shows that the relative contribution of the emissive species changes with change in the pH of the medium and this leads to change in fluorescence intensities as well as a shift in emission maxima from visible region (410 nm) to UV region (345 nm) with the increase in pH.

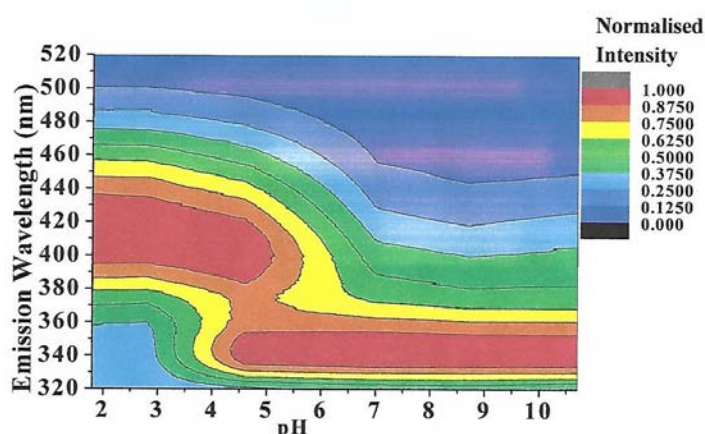


Figure 5. 3D Contour Plot: emission of GO-PANI at different pH (excitation wavelength = 280 nm)

The fluorescence excitation (FLE) spectra monitored at 345 nm and 410 nm shows interesting pH dependent behaviour (Fig. 6a and 6b). In alkaline pH (pH = 10.7), FLE spectra monitored at 345 nm, shows two peaks at 230 nm and 280 nm. The peak at 280 nm is due to $n-\pi^*$ transition of GO moiety (Fig. 6b). But, in acidic pH (pH = 1.8), the peak at 250 nm in FLE of GO-PANI, monitored at 410 nm (Fig. 6a), clearly indicates the formation of a different emissive moiety in the ground state. Absence of significant intensity in the FLE spectrum of GO-PANI in alkaline pH, monitored at 410 nm, suggests that the species emitting at 410 nm has very little contribution at this region of pH (Fig. 6b). Similarly, monitoring emission intensity at 345 nm in acidic pH, FLE spectrum does not exhibit any significant spectral pattern and this may be due to very little contribution of the species emitting at 345 nm at this pH (Fig. 6a). The complete picture of the pH dependent emission band

centered at 410 nm is represented by excitation emission matrices (EEM) of GO-PANI at different pH (Fig. 6c). The EEMs of GO-PANI clearly shows that the emission centered at 410 nm is obtained by the excitation at 250 nm in low pH and this band gradually disappears with the increase in pH.

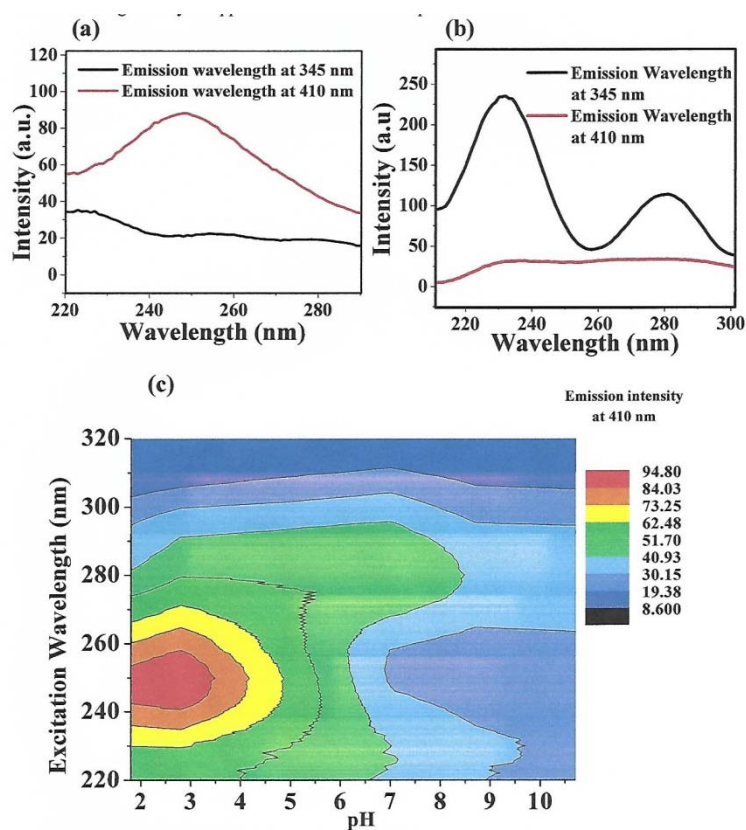


Figure 8. Excitation spectra of the aqueous dispersion of GO-PANI (a) at pH = 1.8 (b) at pH = 10.7 (c) Excitation-emission matrices of GO-PANI at different pH (monitored at 410 nm)

Dual emission band ($\lambda_{\text{ex}} = 280 \text{ nm}$) of the aqueous dispersion (pH = 4.6) of GO-PANI and the different nature of FLE at two different

pH (1.8 and 10.7) clearly suggests the presence of two different fluorophoric species; one of them is formed only when the pH of the medium is acidic. The species emitting at 345 nm is resembled to GO fluorophore, while the second one, emitting at 410 nm, is only appeared in acidic pH. According to Maser and co-workers, electronic properties of reduced GO and an intermediate oxidation state of PANI results a charge transfer interaction between PANI and reduced GO.¹⁸ Since, there is equilibrium between the emeraldine base and emeraldine salt in the aqueous medium containing H^+ ion and this equilibrium is influenced by the pH of the medium. In alkaline medium, PANI exists as emeraldine base form and this is confirmed by the absorption spectrum. With the decrease in the pH, formation of emeraldine salt form with polaron state becomes favorable.¹⁹ In consequence, unstable single electron in the polaron state of PANI transfers charge to the extended conjugated backbone of GO sheets which are capable to store negative charges like graphene layers in graphite intercalation compound. Hence, as a result of charge transfer interaction along with π - π interaction, a ground state complex may be formed in acidic pH. The ground state and the excited state of the fluorophore of the partially charge separated species, becomes stable in aqueous polar medium and this leads a red shifted emission band at 410 nm. But, in alkaline medium, no such polaron states are formed and so usual emission resembled to GO is observed. So, the luminescence a property of GO-PANI nano-composite is changed in acidic pH as a result of electrostatic charge transfer interaction which actually modulates the π -electronic band structure of GO by the formation of a species. Thus, the photoluminescence of GO-PANI is tuned between UV and visible region by changing the pH of the medium and this interesting observation is attributed to the ground state charge transfer interaction by utilizing the polaron state of emeraldine salt form of PANI in the GO-PANI nano-composite.

The observed effect of pH, surfactant and polymer on the photoluminescence of GO shades light in the synthesis of tunable optoelectronic devices in aqueous phase and may open up various aspects of luminescence research of GO based novel functional materials and may provide a further insight on the research of GO based pH sensing materials. The entire work may show a new avenue to the further investigation on the photoluminescence features and

modulation of photoluminescence spectra of GO and thereby will help to develop various kinds of GO based optoelectronic materials.

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The presence of photochemistry in the literature. A personal selection between the articles published in 2018

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I think it is important to verify the impact of our discipline in the scientific literature. The success of photochemistry can be visually observed examining the diffusion of articles related to photochemistry in the most diffused chemical journals.

In the following pages you can find a personal selection of the photochemistry articles appeared on the Journal of the American Chemical Society, on Chemical Communications, and on Chemistry European Journal in the period January-May 2018. You will be able to see the diffusion of the our discipline in several fields of the chemical research.

Journal of American Chemical Society

1. J. Z. Zhang, P. Bombelli, K. P. Sokol, A. Fantuzzi, A. W. Rutherford, C. J. Howe, E. Reisner. Photoelectrochemistry of Photosystem II in Vitro vs in Vivo *J. Am. Chem. Soc.* 140, 6–9. DOI: 10.1021/jacs.7b08563
2. R. Feng, Y. Lu, G. Deng, J. Xu, Z. Wu, H. Li, Q. Liu, N. Kadowaki, M. Abe, X. Zeng Magnetically Bistable Nitrenes: Matrix Isolation of Furoylnitrenes in Both Singlet and Triplet States and Triplet 3-Furylnitrene *J. Am. Chem. Soc.* 140, 10–13 DOI: 10.1021/jacs.7b08957
3. K. M. Blacklock, B. J. Yachnin, G. A. Woolley, S. D. Khare Computational Design of a Photocontrolled Cytosine Deaminase *J. Am. Chem. Soc.* 140, 14–17 DOI: 10.1021/jacs.7b08709
4. Y. Wang, N.-Y. Huang, J.-Q. Shen, P.-Q. Liao, X.-M. Chen, J.-P. Zhang Hydroxide Ligands Cooperate with Catalytic Centers in Metal–Organic Frameworks for Efficient Photocatalytic CO₂ Reduction *J. Am. Chem. Soc.* 140, 38–41 DOI: 10.1021/jacs.7b10107
5. K. Kearney, A. Iyer, A. Rockett, A. Staykov, E. Ertekin Multiscale Computational Design of Functionalized Photocathodes for H₂ Generation *J. Am. Chem. Soc.* 140, 50–53 DOI: 10.1021/jacs.7b10373
6. J. Zhou, K. Wang, B. Xu, Y. Dubi Photoconductance from Exciton Binding in Molecular Junctions *J. Am. Chem. Soc.* 140, 70–73 DOI: 10.1021/jacs.7b10479

7. S.-C. Cheng, K.-J. Chen, Y. Suzuki, Y. Tsuchido, T.-S. Kuo, K. Osakada, M. Horie Reversible Laser-Induced Bending of Pseudorotaxane Crystals *J. Am. Chem. Soc.* 140, 90–93 DOI: 10.1021/jacs.7b10998
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SPECIAL REPORTS ON INORGANIC PHOTOCHEMISTRY

In this Thematic Issue different aspects of the photophysics and applied photochemistry of organometallic complexes and other inorganic materials, among them silicon nanocrystals and CdSe-ZnS quantum dots, are covered. The reader will find examples for the detailed photophysical investigation of fundamental processes, such as photoinduced electron transfer and reversible electronic energy transfer (Prof. Wenger, University of Basel, Switzerland and Prof. McClenaghan, University of Bordeaux/CNRS, France). The deeper understanding of these mechanistic concepts is foreseen to lead to new photosensitizers and nanomaterials with clever photophysical design. The group of Prof. Campagna (University of Messina, Italy) discusses the integration of organometallic complexes in assembled architectures (such as dendrimers or squares) with the aim of arriving at well-defined photoactive systems, for example for the application in photoinduced water oxidation. The contribution by Profs. Ceroni and Bergamini (University of Bologna, Italy) introduces photosensitizer-decorated silicon nanocrystals with potential as light-harvesting antenna systems. Two contributions from Spain (Prof. Gimeo, University of Zaragoza and Prof. Rodríguez, University of Barcelona) focus on the design of gold(I) complexes for a variety of applications ranging from their application in bioimaging to the preparation of luminescent supramolecular soft materials. The contribution of the group of Prof. Knör (Johannes Kepler University of Linz, Austria) provides us with an informative overview of their past and present activities related to Inorganic Photochemistry, with emphasis on the use of artificial photoenzymes in bio-inspired photocatalysis and biomedicine. The diversity of examples and topics in this Thematic Issue illustrate a highly active field with strong implications in many fundamental and applied aspects of photochemistry. Find out!

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Light-harvesting antennae based on silicon nanocrystals

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Silicon (Si) is a widely used semiconductor: it is abundant, widely available and essentially non-toxic. From the photophysical point of view, it exhibits weak light absorption and emission because it has an indirect bandgap nature. Conversely, Si nanocrystals (SiNCs) in the quantum size range (2-12 nm) are an emerging class of quantum dots¹⁻³ with emission wavelength that can be tuned from the near-infrared (NIR) into the visible by decreasing their size and displaying long-lived emission (tens-to-hundreds of microseconds).⁴ They occupy a niche in the realm of quantum dots, offering several advantages: silicon is abundant, essentially non-toxic and can form robust chemical bonds with ligands at the nanocrystal surface. Surface functionalisation is necessary to prevent nanocrystals' oxidation and to obtain stable colloidal dispersion of SiNCs.

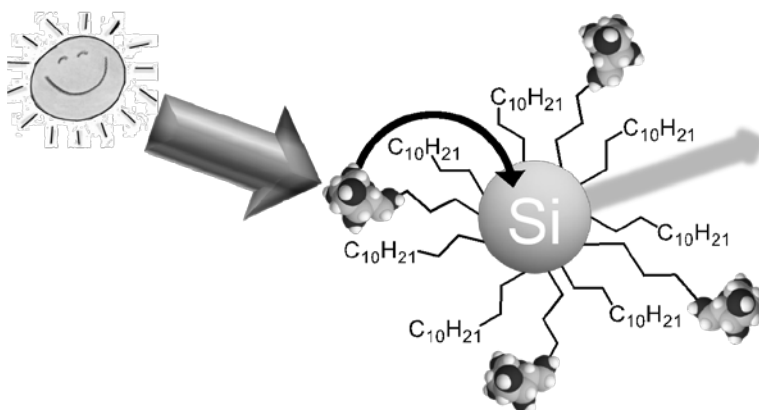


Figure 1. A schematic representation of a light-harvesting antenna based on a silicon nanocrystal core decorated with organic dyes at its surface. Excitation of the dye yields sensitised emission of the SiNCs, according to the working principle of a molecular light-harvesting antenna.

Despite their excellent emission properties, Si nanocrystals are weak light absorbers due to the indirect nature of the band gap of silicon. This limits the luminescence brightness of Si nanocrystals and the photon energy required for photoexcitation is usually significantly higher than the peak emission energy. For example, Si nanocrystals with peak light emission at 800 nm are usually excited with light of wavelength 400 nm to achieve reasonable emission signal. To enhance the light absorption, Si nanocrystals can be decorated with a surface layer containing light-absorbing chromophores that funnel excitation energy to the Si core: this is the working principle of a molecular light-harvesting antenna⁵ (Figure 1) and it is the focus of the ERC StG PhotoSi project.

H-terminated nanocrystals, produced by thermal disproportionation of silicon oxide, were used as a platform for co-passivation with alkylic chains and different organic chromophores, e.g. pyrene,^{6,7} porphyrin,⁸ or benzothiadiazole chromophores.⁹ Excitation of the organic chromophores results in an efficient energy transfer to the nanocrystal core. The investigated hybrid material exhibits high quantum yield also in the NIR spectral region with lifetime in the μ s range.

In the case of the visible light-absorbing 4,7-di(2-thienyl)-2,1,3-benzothiadiazole chromophore, we estimated that an average of 18 chromophores per nanocrystal (diameter = 5 nm) are appended at their surface.⁹ Upon excitation of the chromophore at 515 nm, strong quenching (ca. 95%) of the dye fluorescence is observed. Accordingly, the lifetime of the fluorescent excited state of the chromophore is strongly decreased (ca. 95%), i.e. from 7.9 ns for the free chromophore in toluene to 0.4 ns when bound to SiNCs. Concomitantly, sensitized NIR long-lived emission of SiNCs was observed at 800 nm ($\tau=160 \mu$ s). The efficiency of the sensitization was estimated to be ca. 75% and the brightness was increased of 2 times compared to dodecyl-capped SiNCs upon excitation at 515 nm. The most interesting aspect of the present system is that the 4,7-di(2-thienyl)-2,1,3-benzothiadiazole chromophore exhibit a good two-photon absorption (2PA) cross section with band maxima at 730 and 960 nm, while the two-photon absorption cross section of SiNCs is rather poor. Upon laser excitation at 960 nm a sensitized luminescence of the silicon core in the NIR spectral region was observed and characterized by a close to quadratic power dependence. This system represents an example of light-harvesting

antenna working by both one-photon and two-photon excitation and it combines the 2PA properties of the appended chromophore with the long-lived and oxygen-independent luminescence in the NIR of the SiNCs.

We investigated two main applications of these systems: bioimaging and luminescent solar concentrators.

For bioimaging and optical sensors, long-lived and oxygen-insensitive luminescence is a rare combination of properties, useful to obtain high contrast in images from scattering biological tissues.

In the case of luminescent solar concentrators, SiNCs, embedded in a transparent polymeric slab, absorb UV light and emit in the red and near-infrared spectral region; the emitted light is conveyed to the edges by wave-guide effects and converted into electricity by conventional silicon photovoltaic cells. The optical properties of SiNCs are perfectly fitting with the requirements for an efficient and semi-transparent LSC device because of the apparent large Stokes-shift between absorption and emission. A further improvement is represented by the use of light-harvesting antennae with dyes absorbing in the UV and blue spectral region in order to absorb an higher fraction of sun light without the need to increase the concentration of SiNCs.

Future challenges in the field of light-harvesting antennae based on SiNCs are: (i) a deeper understanding of the mechanism of energy transfer between the chromophores and the silicon core; (ii) an efficient and highly tolerant synthetic strategies which yields highly luminescent SiNCs with a large number of chromophores attached to their surfaces.

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Inorganic Photochemistry in Austria

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Research activities of the Knör group at JKU are covering various aspects of the photophysics and photochemistry of coordination compounds including detailed investigations of organometallic and supramolecular photocatalyst systems, luminescent materials and biologically active light-responsive molecules.

Fascinated by the seminal work of his early mentors Arnd Vogler and Vincenzo Balzani, which significantly shaped the maturing research field in the tradition of Arthur Adamson and Giacomo Giamician, Knör started his independent activities in Inorganic Photochemistry more than 25 years ago. In this period, the novel concept of multielectron transfer sensitizers was introduced [1] in order to better promote the artificial photosynthetic generation of solar fuels (Fig. 1).

In the following years, the crucial advantages of long-wavelength spectral sensitization of photoreactions to fully exploit the potential

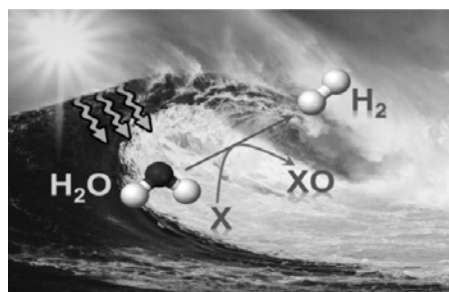


Figure 1. Alternative routes to photocatalytic water splitting and solar energy conversion based on simple salt solutions such as seawater. Light-driven proton reduction providing hydrogen equivalents may be directly coupled to earth-abundant reductants X such as H_2O to achieve a simultaneous photoassisted accumulation of H_2O_2 as an energy-rich multielectron transfer reaction product for powering fuel cells [1].

of solar radiation and diffuse daylight as a sustainable energy source, as well as the rational application of photochemical key-steps in biomimetic catalysis have been systematically elaborated in depth [2]. In this context, Knör became member of the EPA and also a founding member of the now well-established Society of Porphyrins and Phthalocyanines (SPP). He also acted twice as an elected managing board member of the Photochemistry Section of the German Chemical Society (GDCh).

A completely new branch of Inorganic Photochemistry has been introduced with the development of light-driven enzyme model compounds (Artificial Photoenzymes) in the course of the author's habilitation thesis. It could be shown that competitive functional counterparts of native oxidoreductase enzymes, nucleases and even more complex multienzyme reaction centers are readily obtained with this new strategy based on photo-assisted key-steps and full spatio-temporal light-control of catalytic performance [3,4]. Some examples of such bio-inspired processes involving artificial photoenzymes are shown in Fig. 2.

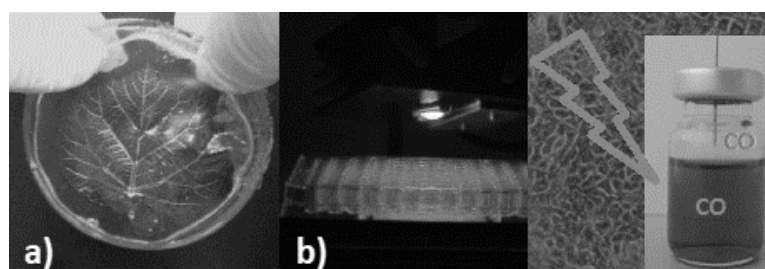


Figure 2. Applications of light-driven enzyme model compounds in bio-inspired photocatalysis and biomedicine:

- a) Flexible hydrogel-based light-harvesting system ("artificial leaf") for solar energy conversion and natural cofactor recycling [3,4].
- b) Photopharmacologic effects on live cells controlled by far-red and NIR-light responsive catalysts replacing biocatalytic functions [4,5].

When Knör was called as the head of the Inorganic Chemistry Institute of Johannes Kepler University in 2006, he also founded the center for photochemical sciences (CNPS) at JKU Linz, which is

well-equipped for performing frontier science projects in photophysics, photochemistry and photocatalysis in Austria.

During the last decade at JKU, continuous efforts were undertaken to improve the performance of artificial photosynthesis, which resulted in the first fully functional counterpart of the complete energy-trapping and solar-to-fuel conversion cascade otherwise only feasible with the natural photosystem I of green plants [3]. The emerging new concept of light-controlled enzyme model compounds was further systematically developed into a rapidly growing branch of biomimetic photochemistry and catalysis [4]. More recently, the unprecedented observation of triggering “spin-forbidden photochemistry” by direct excitation of spectroscopically hidden states with far-red and NIR-light was discovered as a new strategy to overcome some of the major limitations in molecular photomedicine caused by otherwise insufficient light-penetration into tissues [5].

Current national research activities of the Knör group at JKU include photo-controlled release of small bioactive molecules, design of novel light-harvesting chromophores and photoreactive coordination compounds (porphyrins, chlorins, phthalocyanines, corroles, polypyridyls, 1,2-diimines), development of luminescent emitter materials based on metal complexes, coordination polymers and metal-organic frameworks, photochemistry and photocatalytic reactions in biocompatible hydrogels, proton coupled multielectron transfer systems for solar energy conversion and artificial photosynthesis, small molecule activation with homogeneous photocatalysts and photo-biocatalytic hybrid systems, photochemical redox-cofactor recycling, and green chemical photocatalysis for synthesis involving bio-inspired C-H activation, hydrogen atom transfer (HAT) and C-C bond formation. In addition, new tools for the photochemical control of cellular processes and protein translocation are developed within the framework of the interdisciplinary Austrian-wide doctorate college program NanoCell, and a collaboration to establish STED photochemistry (STED = stimulated emission depletion) has recently been started in a project involving the Linz Institute of Technology (LIT).

A longer-term vision of the author's research activities is to promote the newly established research field of bio-inspired photocatalysis with artificial photoenzymes to the next levels of complexity. One of the logical expansions of this versatile concept

requires the control of photocatalytic one-pot multistep reactions using different excitation wavelengths or other orthogonal tools to address a certain set of photocatalysts for driving vectorial substrate conversion cascades resembling natural metabolic pathways. The proof of principle of this latter goal has been successfully demonstrated [4]. Another step forward to mimicking natural systems of higher complexity is the incorporation of artificial photoenzymes into chemical environments such as artificial cell structures, tissue-like environments or synthetic organelles to provide defined three-dimensional architectures with a given light-controllable function. It could already be shown recently for several light-responsive metal complexes, that an efficient immobilization is possible with an optionally shaped carrier matrix made out of soft biocompatible materials such as hydrogels without any loss of the required photocatalytic activity (Fig. 2a).

Thus, studying the photophysics and photochemistry of coordination compounds is meanwhile reaching the fascinating creativity level of optogenetics tools and modern bottom-up synthetic biology approaches. Now, the next generation of young and ambitious scientists is welcome to join this exciting endeavour in order to continue a long and fruitful tradition of basic research in inorganic photochemistry!

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Luminescent gold(I) supramolecular assemblies and applications

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The self-assembly of small molecules by the establishment of non-covalent interactions has received great attention in the past decade as a way to build supramolecular structures with a large number of specific functions and morphologies.¹⁻³ As a result, supramolecular chemistry has matured from a conceptually marvelous scientific curiosity to a technologically relevant science encompassing a broad area of advanced materials.⁴ Within this field, organometallic complexes present a rich chemistry due to the formation of inter- and intramolecular metal...metal bonds (so-called metallophilic interactions). This kind of interactions have been observed to be responsible for the formation of micro- and nano-sized supramolecular assemblies and, in a large number of examples, to be responsible for interesting luminescent properties.⁵⁻⁸ However, differently from conventional fluorescent compounds, which are singlet-state emitters, d⁶, d⁸ and d¹⁰ complexes containing heavy-metals are triplet emitters and as such display phosphorescence. Relativistic effects have shown to be of enormous importance in understanding the chemical and physical properties of the heaviest 6s transition and post-transition metallic elements, mainly gold. The high spin-orbit coupling constant of gold facilitates the access to the low-lying emissive triplet state via the intersystem crossing. This process is even more favoured when Au...Au (aurophilic) contacts are present. These interactions are energetically similar to hydrogen bonds (5-10 kcal·mol⁻¹) and are observed to be directly involved in the increase of the radiative rate constant and an increase on the corresponding luminescence quantum yield.⁹

The correct choice of the ligand (chromophore) coordinated to the metal atoms will determine the resulting (supra)molecular assemblies and their properties. In fact, a large number of chromophores have been studied in the last ca. 20 years in our research group, most of

them containing an alkynyl moiety as a coordinating point to the metal atom. A wide range of different properties and applications can be studied that come from the formation of low molecular weight gelators, sensors or biological applications (within (supra)molecular chemistry field) to the formation of small and homogeneous nanoparticles, encapsulation of nanoparticles in water or hydrogen production (within the field of materials chemistry), as shown in Figure 1.

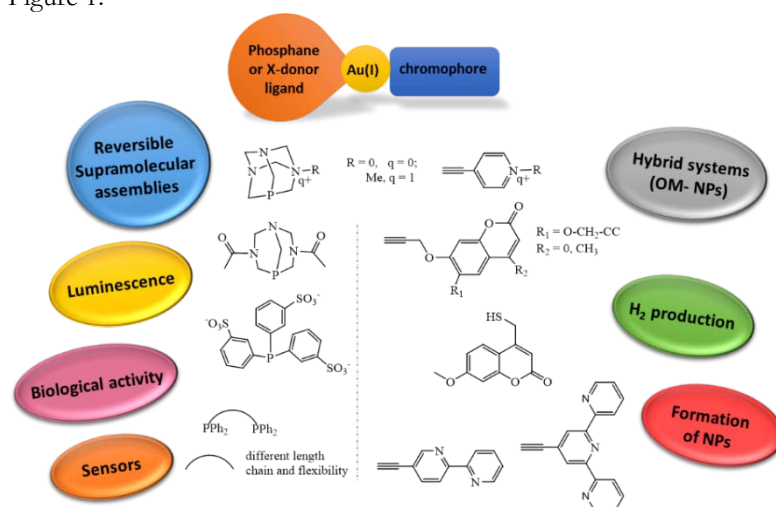


Figure 1. Schematic representation of the recent gold(I) complexes studied in our research group and some of their potential applications.

Starting from (supra)molecular assemblies, the formation of luminescent organometallic hydrogelators was described by us in 2013, based on a complex with very simple chemical structure, PTA-Au(4-pyridylalkynyl), being PTA = 1,3,5-triaza-7-phosphatricyclo[3.3.1.1^{3,7}]decane.¹⁰ This was the starting point from a deeper investigation on this field that was followed by different modifications on the chemical structure of the new systems developed in our group. These modifications were based on changes on the chromophore but also, maintaining the same chromophore but with the introduction of a positive charge by methylation of a nitrogen atom either from the phosphine or from the pyridyl unit. Interestingly, the formation of very long fibers (up to hundreds of

micrometers) was detected for some of the neutral complexes. The involvement of π - π interactions, together with hydrogen bonds and aurophilic contacts was evidenced by NMR and absorption/emission techniques and, in some cases, by the resolution of the X-ray crystal structure¹¹ and supported by theoretical calculations.¹² On the other hand, as can be seen in Figure 2, the formation of positively charged structures, induces a radical change on the supramolecular morphology with different shapes and sizes (rods, vesicles or squares instead of fibers) and with different luminescent properties (yellow, green and red) and with solvatochromic behaviour in solution.^{13,14}

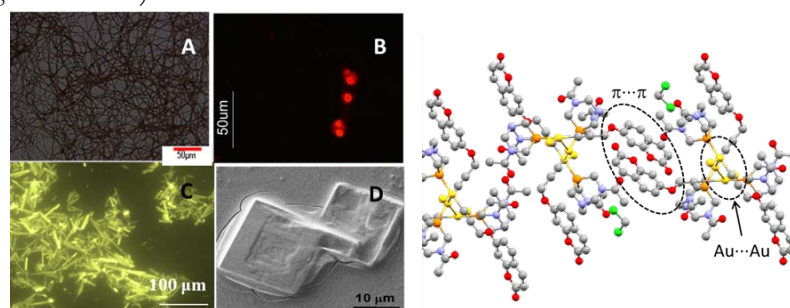


Figure 2. Left: Optical (A), Fluorescence (B, C) and scanning electronic microscopy images of gold(I) supramolecular structures that are neutral (A) and positively charged (B-D). Right: x-ray crystal structure of a gold(I) complex containing a coumarin chromophore and 3,7-diacetyl-1,3,7-triaza-5-phosphabicyclo[3.3.1]nonane (DAPTA) phosphine showing some of the weak intermolecular interactions.

The ideal goal for supramolecular chemists working in this area is the possibility to control the resulting aggregation/disaggregation process. This has been perfectly achieved very recently with a series of bipyridyl/terpyridyl gold(I) complexes which were able to aggregate/disaggregate in a reversible way by means of the introduction of a metal cation to coordination to the N-donor chromophores and removing it by the presence of a cryptand agent.¹⁵ Changes on the supramolecular morphologies have been also detected as a result of molecular recognition processes,¹⁶ which is one of the applications of these type of structures that is also the basis of the resulting biological properties as antitumoral, antimalarial and anti-arthritis drugs.^{17,18}

In the frontier with materials chemistry we have demonstrated that the well-defined alignment of the molecules within the supramolecular hydrogelating network are the key point for the formation of very small and homogeneous nanoparticles upon reduction of Au(I) to Au(0) induced by electron beam irradiation or temperature (Figure 3A).¹⁹ The same hydrogelators have been used as encapsulating agents removing NPs from organic to aqueous solution being demonstrated the key role of metallophilic interactions in the successful achievement of this process, since this process is successful always when a metal atom able to establish $\text{Au(I)} \cdots \text{M}$ contacts is present at the surface of the nanomaterial.²⁰

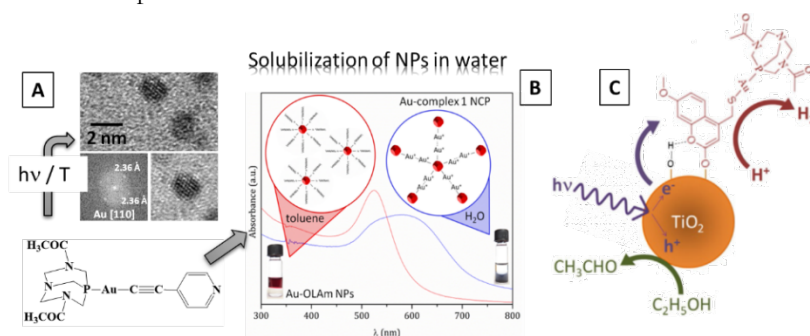


Figure 3. (A) Formation of Au(0) NPs from a Au(I) hydrogelator; (B) solubilisation of NPs in water due to the establishment of metallophilic interactions; (C) H_2 production by using a Au(I) complex supported on TiO_2 .

Also in the frontier between organometallic/supramolecular chemistry and materials science, we have developed the first example of a gold(I) complex supported on TiO_2 able to produce H_2 with better results than those well-known and previously used that are Au(0)/ TiO_2 materials. These results open a new field of interdisciplinary research both for organometallic and materials chemists.²¹

All in all, we can see that luminescent Au(I) organometallic chemistry is a very promising research field that is increasing on importance in

the last years and that is involved in very different topics and that promotes interdisciplinary research with a great variety of applications.

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Understanding the behavior of group 11 emitters for the design of complexes with potential applications in medicine and material science

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Communication

Introduction

Research interest in emissive complexes is related with their potential use, for instance in the fabrication of different devices as sensors, OLEDs or solar cells as well as diagnosis fluorescent microscopy agents in medicine. Our interest is focused in understanding the emissive processes in order to design suitable systems as it is resumed in the examples below.

Building block strategy for modulable emissive complexes

Triplet harvesting leads to an increment of the efficiency of OLEDs devices. The presence of heavy atoms in the molecule and/or emissive thermally activated delayed fluorescence processes (TADF) may lead to the desired effect.¹ Thus understanding the emissive process represents an important task in the design of useful species. The use of a building block strategy allows the modification of small parts of the compound and helps in the understanding the origin of the emissive process. That is the strategy selected by us with these aims. We present four different emissive systems which show how to change the origin and/or the luminescent properties.

System I (Fig. 1) combines a weak blue fluorophore ligand *nido*-carborane diphosphane and displays gold...gold interactions (which have been demonstrated as a possible origin of emissive behavior).²

All the complexes display red emissions in the solid state both at room temperature, but in frozen solutions and/or at 77K an additional band at lower energies (ca 500 nm) is also present. TDDFT studies for some of the complexes have led to the conclusion that the red emission has been related to the calculated

first triplet transition and has been assigned to a ligand *nido*-carborane (L) to the interacting gold centres (MM) charge transfer (LMMCT). The same diphosphane has been used as part of system II (Fig. 1)³ together with a carborane-dithiolate ligand, supporting $d^8 \cdots d^{10}$ interactions. The complexes are emissive in the solid state at 77K. TDDFT studies reveal two different origins for the emissions; when the d^8 metal is platinum the emission is due to a charge transfer transition from the *nido*-carborane cage to the metal ligand “ P_2PtS_2 ” system and when the d^8 metal is nickel or palladium to a charge transfer transition from the d^8 metal to the coordination core “ $P_2M(d^8)S_2$ ”. The results for both systems indicate that slight changes in one of the building block (the monophosphane or d^8 metal) may modify the origin of the emissions.

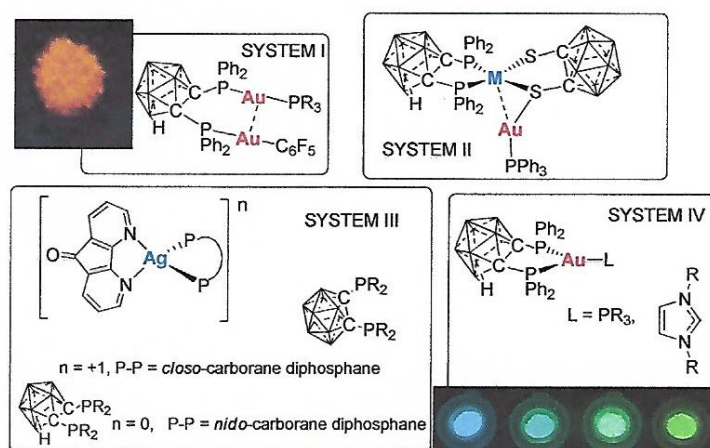


Figure 1. Different studied systems

A different fluorophore, 4,5-diazafluoren-9-one (DAFO) has been used in the synthesis of tetranuclear silver derivatives with different diphosphanes (Fig. 1, system III).⁴ In the solid state at room temperature the cationic complexes with the dppe and the *closo*-carborane diphosphanes are emissive, whereas those with the *nido*-carborane diphosphanes are not. Lifetimes for these complexes are in the nanosecond range (as for the free ligand), thus intraligand transitions highly modified by coordination to the metal may be proposed as the origin of the luminescence. A different system is

formed by three-coordinate gold(I) complexes of the *nido*-carborane diphosphane with different ancillary ligands such as phosphines and *N*-heterocyclic carbenes (NHC) (Fig. 1, system IV) for which the luminescence is originated by a Jahn-Teller distortion in a T-shape of the excited state. These complexes show almost quantitative quantum yields.^{5,6} TDDFT calculations for the NHC system showed a LMCT transition (diphosphane \rightarrow Au).

Gold and light in medicine

Progress towards more efficient medicines requires being able to perfectly understand their inner interplay within the cells. This is one of the greatest challenges in medicine as it would allow the development of specific treatment for each particular disease. Within this context, our research group has centered their efforts towards the development of emissive anticancer gold complexes. It is well known that Au(I) complexes present antiproliferative properties towards a wide range of tumor cells.⁷ However, and despite all the efforts made for many researchers, there is not yet an agreement on the action mechanism of these types of drugs. Our approach to tackle this problem is to be able to track the gold complex by fluorescence microscopy, which is a non-invasive technique that has as unique requirement the use of luminescent species. Unfortunately, bioactive Au(I) complexes do not normally present the right photophysical properties to be used with fluorescence microscopy. Therefore; an emissive tag needs to be grafted in the right derivatization in order not to disrupt the bioactive properties of the drug. Several strategies could be followed for that, a) the use of organic chromophores or b) selecting phosphorescence metallic species.⁸ Both of them present a series of advantages and disadvantages. For instance, organic fluorophores are normally commercially available and their chemistry to be incorporated into the metallodrug is well-known. However, they can present problems when it comes to their photophysical properties for cell imaging, as typically their excited state lifetimes and Stokes shifts values are limited and in consequence the use of time-gating techniques is restricted. Moreover, their excitation and emission maxima are too energetic, which could promote damage into the biological sample and poor light penetration. In the case of emissive metal complexes, the use of time gating techniques is more accessible as they generally bear long lifetimes. Moreover, their large

stokes shifts prevents from quenching phenomena by reabsorption of the emission and their emissive properties can be easily modulated by different functionalization. On the contrary, one of the major disadvantages is their coupling with the metallodrug itself. Synthesizing heterobimetallic complexes could be sometimes tricky and the synthetic procedures must be perfectly planned beforehand, *ie.* either the synthesis of the two metallic complexes and posterior coupling reaction, or a stepwise synthetic route.

Herein we present two luminescent metallodrug reported by our group as examples of both strategies, a luminescent acridine gold(I) derivative⁹ and a heterobimetallic Re(I)/Au(I) species.¹⁰ In both cases, fluorescence microscopy showed the biodistribution of both metallodrugs, mainly in the lysosomes and cytoplasm, respectively (Fig. 2). Once the biodistribution of the drug is ascertain, further analysis for the elucidation of the mechanism of action can be done with a greater precision.

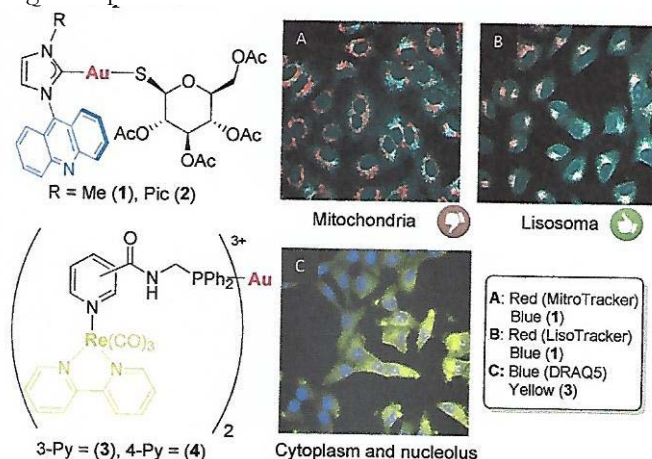


Figure 2. Example of emissive and bioactive gold complexes.

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Reversible Electronic Energy Transfer: From Covalent Molecular Dyads to Nanomaterial Hybrids

Nathan D. McClenaghan

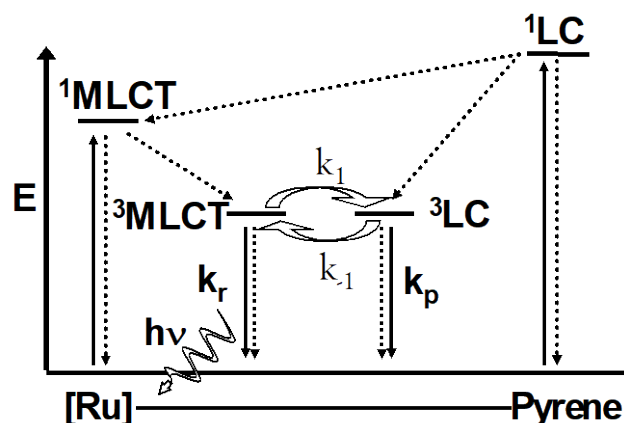
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Electronic energy transfer between two chromophores is both a successful and a widespread phenomenon, which is well understood and formalised according to Förster and Dexter mechanisms.^{1,2} At least this is the case for energetically-downhill unidirectional electronic energy transfer. In contrast, when specific energetic and kinetic properties are satisfied, reversible energy shuttling between two proximal chromophores is possible, which results in a new set of photophysical properties for the bichromophoric molecular system. Considering Scheme 1, $k_1 > k_{-1} \gg k_r \gg k_p$.^{3,4}

More specifically, in order to observe Reversible Electronic Energy Transfer (REET) between two chromophores, lower-lying excited states should be thermally accessible one from another ($\Delta E \leq 1000 \text{ cm}^{-1}$ at ambient temperature), while interchromophore transfer should be relatively rapid with respect to other de-excitation pathways. In this way energy can shuttle back and forth between chromophores before ultimately being emitted from the chromophore with the inherently shorter lifetime.

This situation can be conveniently attained in bichromophore dyads, particularly in inorganic-organic hybrids. Indeed, this is exemplified by a prototype system where $\text{Ru}(\text{bpy})_3^{2+}$ (bpy = 2,2'-bipyridine) is tethered to pyrene.⁵ Their lowest lying excited states $^3\text{MLCT}$ and $^3(\pi-\pi^*)$, respectively, are quasi-isoenergetic while the natural lifetimes of the two chromophores is rather different. Indeed, organic triplets typically decay on the millisecond timescale while MLCT states decay on the microsecond timescale, thus further leaving sufficient time for energy to transfer back and forth between chromophores setting up a dynamic excited state. In this way, red MLCT emission appears as delayed emission due to the pyrene unit, which serves as an energy reservoir that globally transiently stocks energy, and ultimately funnels it back to the emissive energetic unit. As well as prolonging observed luminescence lifetimes, this can also

be a means to change the nature of the excited-state of consequence, for example, in photosensitizer systems. In addition to a range of covalent $\text{Ru}(\text{bpy})_3^{2+}$ -pyrene dyads, further prototype systems involving copper, iridium, platinum, rhenium units and a range of organic chromophores have equally been developed.^{3,4,6,7}



Scheme 1. Jablonski-Perrin diagram for a $\text{Ru}(\text{bpy})_3^{2+}$ -pyrene type molecule where interchromophore REET is operational.

Beyond covalent dyads, non-covalent assemblies harnessing REET were envisaged to give new function to molecular ring-on-thread assemblies. In a first example, the enhanced photo-induced disassembly of a supramolecular donor-acceptor complex comprising a tetracationic cyclophane threaded by an electron rich thread, compared to a prototype “molecular piston” was demonstrated.^{8,9} Here REET served to facilitate threefold the photoinduced electron transfer process, which served to destabilize the complex and assure dethreading. Addition of oxygen prompted the rethreading, thus resetting the system.

As well as governing the operation of a simple molecular machine, REET was further used to report on conformational changes, exemplified by a supramolecular double helix-on-thread structure.¹⁰ While the conformationally-free bichromophoric thread showed REET between the photoactive ends, as it threaded inside a double helix sheath the ends were distanced (circa 2 nm) and consequently REET and lifetime varied significantly. Thus a

prototype lifetime-based conformational probe is demonstrated which, as it relies on triplet-triplet energy transfer, is highly distance dependent.

Previously we sought to extend the scope of REET and instill it in molecule-nanomaterial hybrids, with the aim of demonstrating reversible molecule to material transfer, in spite of the differing natures of molecular and nanomaterial electronic structures.¹¹ More precisely, we decorated the surface of red-emitting core-shell CdSe-ZnS quantum dots (QDs) with pyrene ligands. The thick passivating shell proved a barrier for molecule-nanomaterial communication and the chromophores proved orthogonal, as such the nanosystem acted as a ratiometric oxygen sensor due to a significant oxygen sensitivity of the pyrene chromophore, in contrast to the oxygen-insensitive QD.¹¹ Subsequently, a related system minus the ZnS shell allowed unidirectional transfer from quantum dot to a lower-lying molecular triplet.¹² More recently, in two separate reports, REET between molecule and nanomaterial was finally realized with energetically matched pyrene-CdSe hybrids, to prolong the QD emission from the 10s of nanoseconds to beyond the 100s of microseconds, without recourse to changing the nanomaterial composition.^{13,14}

In conclusion, REET is a process that can be introduced in a wealth of bichromophoric systems, on respecting the simple energetic and kinetic guidelines outlined above. As such, it should continue to prove applicable in a range of molecules and nanomaterials contributing to the realms of photosensitizers and stimulus responsive systems.

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From Photoinduced Electron Transfer to Charge Accumulation and New Photosensitizers

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Photoexcitation of donor-bridge-acceptor compounds commonly induces the transfer of single electrons, leading to the formation of electron-hole pairs.¹ Recently, the Wenger group synthesized and explored a series of new molecular systems that go conceptually beyond simple charge-separation (Fig. 1). This includes long-range electron transfer reactions that are coupled to multiple proton transfers,² circular electron transfer,³ and multi-electron transfer leading to charge accumulation.⁴ In some cases, proton-coupled electron transfer (PCET) was found to play a key role, especially with regard to the light-driven accumulation of multiple redox equivalents in absence of sacrificial donors or acceptors.⁵ In other cases, we have been able to perform light-initiated multi-electron reductions via photoredox catalysis, by merging photoinduced electron transfer with thermal hydrogen-atom transfer.⁶ Another important focus of current research in the Wenger group is the development of new photosensitizers based on earth-abundant transition metals (Fig. 2).⁷ Recently, we discovered that chelating diisocyanide ligands give access to luminescent Cr(0) and Mo(0) complexes that are

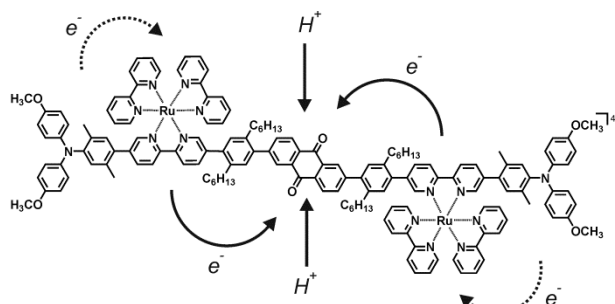


Figure 1. A molecular pentad in which two electrons and two protons can be accumulated upon photoexcitation of the two Ru(II) photosensitizers.

isoelectronic to $\text{Fe}(\text{2,2'-bipyridine})_3^{2+}$ and $\text{Ru}(\text{2,2'-bipyridine})_3^{2+}$.^{8,9}

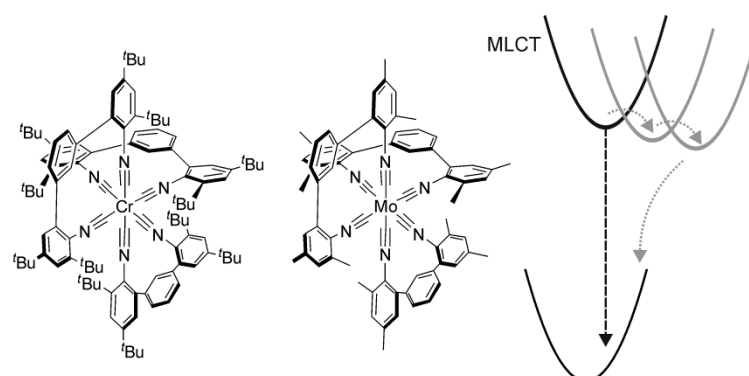


Figure 2. Luminescent Cr(0) and Mo(0) complexes with chelating isocyanide ligands.

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Multicomponent Photochemistry: Organized Systems for Photoinduced Intercomponent Processes

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The group of Molecular and Supramolecular Photochemistry at the University of Messina has been active for long time on the photochemical and photophysical properties of multicomponent species. In recent years, our attention has been focused on artificial photosynthesis and on luminescence sensing. As far as artificial photosynthesis is concerned (for space reason, here we will not report on our work concerning luminescence sensing), along several years we have developed light- and redox-active dendrimers made of Ru(II) and Os(II) polypyridine subunits and studied their properties as light-harvesting artificial antennae, by taking advantage of the properties of the specific subunits and the energy gradient across the dendrimer arrays, derived by a suitable molecular design and synthetic strategies which allow a topological control of the structure.¹ The high efficiency of energy migration within the dendrimer arrays is due to sub-ps time constant of down-hill energy transfer and across iso-energetic subunits, whereas ultrafast long-range electron transfer occurs through the entire structure.² On the basis of former results, we are now employing the metal dendrimers as photosensitizers for photoinduced water oxidation, a key process to be solved for obtaining an efficient water splitting. Indeed, metal dendrimers like the first generation tetranuclear compound $[\text{Ru}\{\mu\text{-}2,3\text{-dpp}\}\text{Ru}(\text{bpy})_2\}_3]^{8+}$ (**4**; 2,3-dpp = 2,3-bis(2'-pyridyl)pyrazine; bpy = 2,2'-bipyridine) absorb much more visible light than the usually used $[\text{Ru}(\text{bpy})_3]^{2+}$ -type monomeric species, essentially because their absorption extends to the red region (Fig. 1). After having demonstrated the higher efficiency of **4** compared to $[\text{Ru}(\text{bpy})_3]^{2+}$ for photoinduced water oxidation, using as catalyst both heterogeneous and molecular species,³ we studied in more detail mechanistic aspects and demonstrated that **4** works in a so-called "anti-biomimetic"

manner: in fact, its MLCT excited state first oxidizes the catalyst, successively transferring the added electron to a sacrificial agent (or electrode).⁴ Interestingly, aggregation in solution between the light-harvesting metal dendrimer and the water oxidation catalysts takes place, and promotes the desired ultrafast processes. The "anti-biomimetic" mechanism was later demonstrated, in collaboration with the Ferrara photochemistry group, to effectively work for photoinduced charge injection into nanostructured TiO₂.⁵

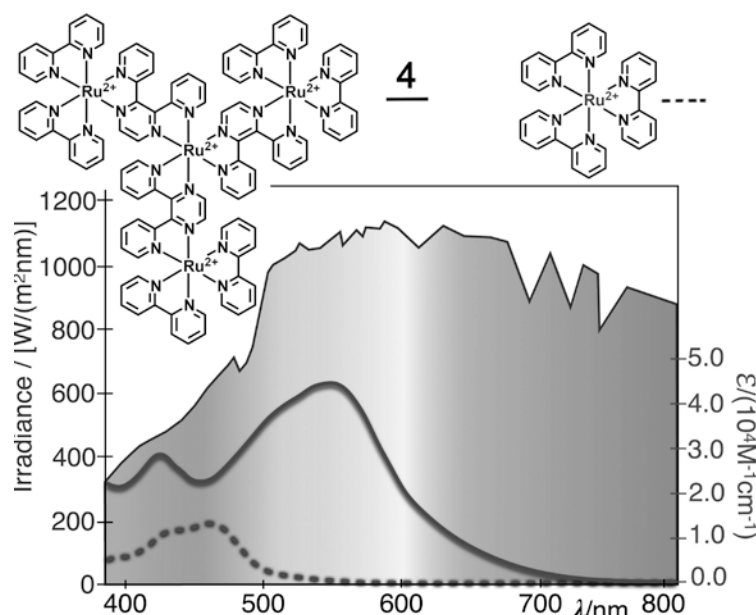


Figure 1. Overlap between solar spectrum and the absorption spectra of **Ru4** (solid line) and [Ru(bpy)₃]²⁺ (dashed line). Charges omitted.

The good light absorption and favorable oxidation potential for water oxidation of the metal dendrimers based on 2,3-dpp as bridges is paid by their low excited-state energy. We are at the moment exploring other multicomponent Ru(II) dendrimers having higher-energy MLCT states. The new designed dendrimers will be suitable for photoreduction of CO₂, within an ongoing project in collaboration with Ishitani's group at TokyoTech.

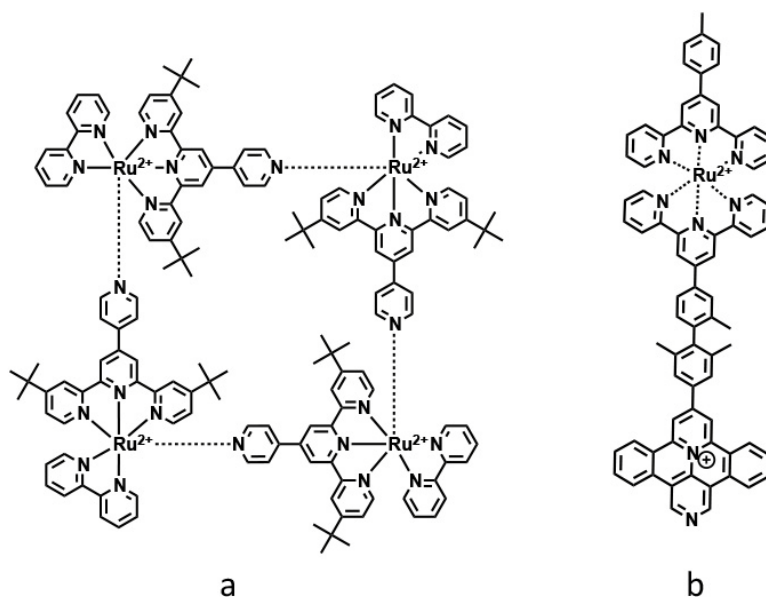


Figure 2. (a) Structural formula of a luminescent tetraruthenium molecular square prepared by photochemical route⁶ and (b) a representative bichromophoric species having selectively addressable chromophores (see text). Charges omitted.

Another line of research is aimed to build up luminescent molecular squares by photo-induced reaction, in collaboration with Garry Hanan at the University of Montréal. We have recently prepared - by a photochemical route - a luminescent tetraruthenium square by taking advantage of photolability of coordinated pyridines and the availability of terpyridine ligands bearing supplementary pyridine arms (Fig. 2a).⁶ We are now investigating in detail the mechanism of molecular square formation, in particular its dependence on concentration and light intensity, as well as the ability of encapsulating small molecules and performing light-induced processes.

In collaboration with Franco Scandola at the University of Ferrara we have recently revisited the superexchange mechanism for photoinduced electron transfer, using linearly-arranged dinuclear metal complexes. We have highlighted that photoinduced charge

separation and charge recombination can take advantage of different virtual states for enhancing donor-acceptor electronic coupling.⁷ This allowed us to suggest that, depending if photoinduced charge separation takes place via oxidative or reductive electron transfer, for obtaining long-lived charge-separated states one has to avoid spacers having components that are (relatively) easy to reduce or to oxidize. We are further proving the theory by investigating a series of bichromophoric compounds with different spacers, prepared by Philippe Lainé in Paris (See Fig. 2b for a representative compound), by separately exciting each chromophore and following the photoinduced intercomponent decay processes. The different rate constants for charge separation (oxidative and reductive, depending on the excited subunits), as well as the possibility to accumulate charge-separation states, which appears to be a function of the excited chromophore, are explained according to the new aspects highlighted by our hypothesis.

The formerly-mentioned aggregation of metal dendrimers based on 2,3-dpp bridging ligands with suitable water oxidation catalysts draw our attention to the possible self-aggregation of such large multicomponent species. Actually, we demonstrated – using different methods, including small angle X-ray scattering, SAXS - that in acetonitrile solution, already at low concentration, self-aggregation occurs, probably taking advantage of the dendrimer structure allowing interactions between different arms (the *branches* of the *molecular tree*). The consequence is the occurring of inter-dendrimer energy transfer, occurring in the ps timescale,⁸ that indicates an interesting parallelism with the antenna systems of natural photosynthesis: actually, in such metal dendrimers intra-dendrimer sub-picosecond energy transfer is followed by inter-dendrimer energy transfer (Fig. 3), similarly to what happens in LH2 and LH1 of some photosynthetic bacterial photosynthetic organisms. An interesting next step would be the incorporation in this aggregates of water oxidation catalysts.

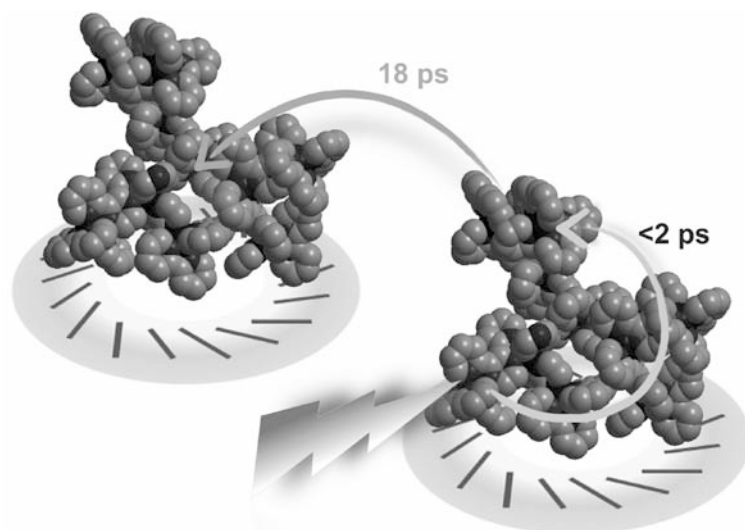


Figure 3. Schematization of the intra-dendrimer (< 2 ps) and aggregation-induced inter-dendrimer (18 ps) energy transfer processes occurring in decanuclear metal dendrimers.⁸

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ABSTRACT OF THESIS ON PHOTOCHEMISTRY

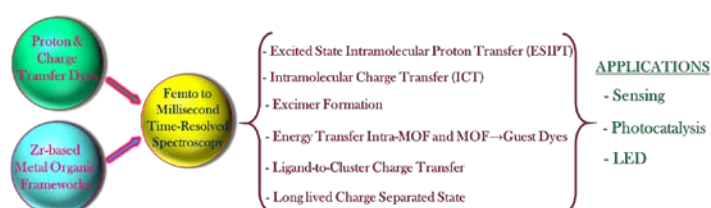
Dynamics of Proton, Charge & Energy Transfers in Solutions and Within Metal-Organic Frameworks: Toward Sensing and Nanophotonic Applications

Mario Gutierrez Tovar

Nowadays, the Society faces new challenges, as getting renewable and efficient sources of energy, new materials and smart devices to protect the earth, and to save time and energy. Some of these challenges reside in fields, spanning from efficient and low-cost illumination systems (LEDs), to photocatalysis (i.e. H₂ production and water purification), drug photodelivery and sensing of specific compounds (i.e. volatile organic compounds, explosive molecules, etc). To face these challenges when making new materials, one has to elucidate the Structure-Function-Dynamics relationship. To this end, laser-based techniques are being the right and most powerful tools, as recognized by awarding its achievement several Nobel Prizes in Chemistry, Physics and Medicine.

Encouraged with the new developments in materials research and available ultrafast spectroscopies; in my Ph.D. thesis, **I explored from femto to millisecond (fs-ms) regime the photophysics and photochemistry of: I) new molecules showing excited state-intramolecular proton-transfer (ESIPT) and intramolecular charge-transfer (ICT) reactions, and II) a new family of Zr-based metal-organic frameworks (MOFs) in suspensions, solid state and polymeric matrices (Scheme 1).** Not of less importance, I used four time-resolved techniques (ps-time-resolved single-photon counting, fs-emission up-conversion, ns-flash photolysis, and fs-UV-visible-midIR absorption spectroscopies), allowing me to characterize the photo-events and

their photoproducts from fs to ms time window. The research led new knowledge proposing new applications in sensing, photocatalysis and LEDs. Below, I shortly describe the findings.



Scheme 1. Illustration of the studied systems using fs-ms time-resolved techniques for elucidating their rich photobehavior and proposed applications in photonics.

To begin with, I have studied the photophysics and photochemistry of new ESIPT dyes and become very familiar with the ultrafast techniques and involved photoevents in solutions:

- **Part I: (2'-hydroxyphenyl)benzoxazole (HBO) Derivatives**

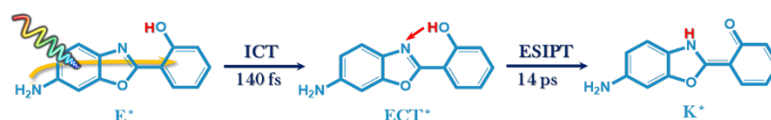


Figure 1. Illustration of the ICT and ESIPT processes taking place in 6A-HBO in DCM (Ref. 1).

One of the most important issues in the potential-energy surfaces of ESIPT reactions, is the presence or not of an intramolecular charge-transfer (ICT) event, and how both processes are coupled and affected by the environment. To explore these issues, I studied in the first part of my Ph.D. work, the fs-ns emission spectroscopy of (2'-hydroxyphenyl)benzoxazole (HBO) and two amino-derivatives (5A-HBO and 6A-HBO). The amino group in the

derivatives produces an increment in the electron density of the dye owing to a photoinduced ultrafast ICT process. This provokes a decrease in the -OH 's acidity at S_1 , and a slowing down of the ESIPT process. For example, the ESIPT in the parent molecule, HBO, in dichloromethane (DCM) takes place in ~ 150 fs, while in its 6A-HBO derivative, an initial and ultrafast fast ICT (140 fs) reaction occurs followed by a slow ESIPT in 14 ps (Figure 1, Ref. 1).

The environment, especially the basicity of the solvents, strongly influences the ESIPT process. For example, for 6A-HBO in DCM it is irreversible, while in acetonitrile (ACN) it is reversible. In methanol (MeOH), it is assisted by the solvent, and in acetone it happens via tunneling (Figure 2, Ref. 1-2).

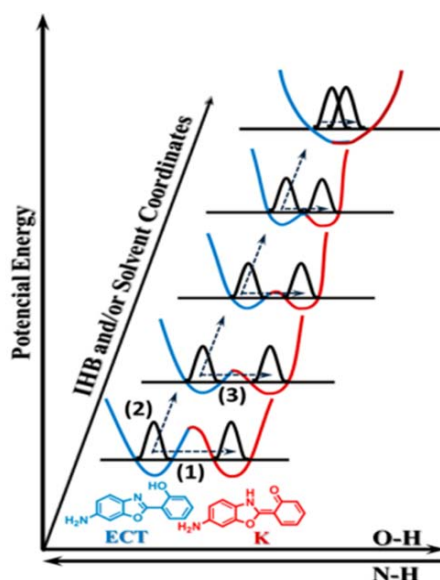


Figure 2. Schematic illustration of the potential energy surfaces where proton motion of 6A-HBO in solution takes place (Ref. 2).

The position of the amino group in the dye molecular frame strongly influences the photodynamics. When the amino group is in

the 6-position, the ESIPT is favored than when it is in the 5-position. Remarkably, this dependence is also affected by the solvent nature. For example, in ACN, the ESIPT does not happen for 5A-HBO, while for 6A-HBO it occurs in a reversible way (Figure 3, Ref. 2-4). In a less basic solvent (DCM), the ESIPT is reversible for 5A-HBO and irreversible for 6A-HBO.

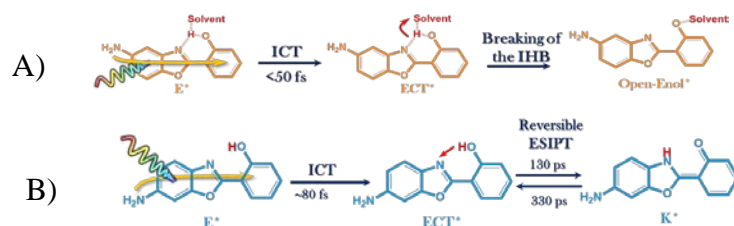


Figure 3. Illustration of the photoprocesses occurring in the excited of (A) 5A-HBO and (B) 6A-HBO in ACN (Ref. 2-3).

- **Part II: Metal Organic Frameworks (MOFs)**

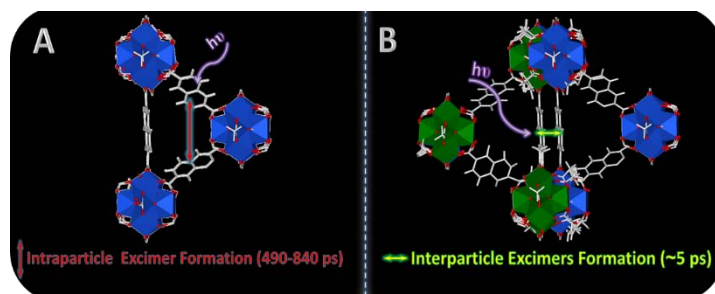


Figure 4. Schematic representation of the A) intraparticle and B) interparticle excimer formation (Ref. 5).

In the second and larger part of my Ph.D. work, I explored the spectroscopy and photodynamic of a new series of Zr-based MOFs (Zr-NDC, Zr-NADC (2-35%), Zr-NDC/Tz and Zr-

NDC/CN) and their interaction with different guest molecules, in suspensions, solid and polymeric films. I studied the steady-state and fs-ms time-resolved emission of monomers and excimers of the naphthalene linkers in Zr-NDC MOF. It was reported, for the first time, on inter- and intraparticle excimer emissions in suspensions and solid-state (Figure 4, Ref 5).

Upon functionalization of the naphthalene linkers with amino-groups (Zr-NADC), we observed energy transfer from the naphthalene linkers to the amino-functionalized ones, competing with excimer formation (Figure 5, Ref 6). Both Zr-NDC and Zr-NADC MOFs exhibit an ultrafast Ligand-to-Cluster Charge Transfer (LCCT) with the subsequent generation of a long-lived charge-separated state (Ref. 7). The formation of this state is paramount for using these materials as photocatalysts. Figure 6 illustrates the photophysical scheme of representative examples of the studied MOFs.

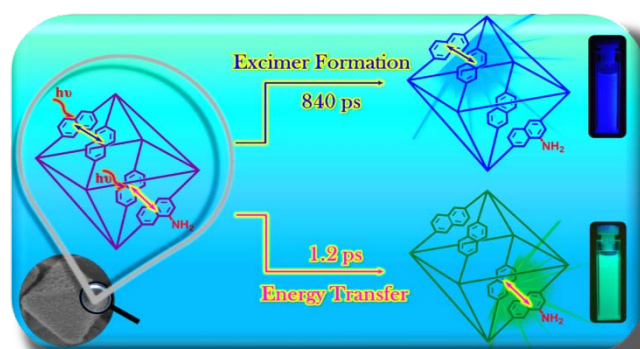


Figure 5. Competition between excimer formation (2-10% of NADC linkers) and energy transfer (20-35% of NADC linkers) processes in Zr-NADC MOFs (Ref. 6).

Taking advantage of the porous structure of Zr-NDC MOF, we have encapsulated three different organic fluorescent molecules into their pores and studied the spectroscopy and dynamics of the formed composites at different dye loadings. We observed and characterized energy transfer from the excited MOFs to the trapped guests, and used it to fabricate hybrid materials that

emit multicolor and cool white light with a high quantum yield (Figure 7, Ref. 8).

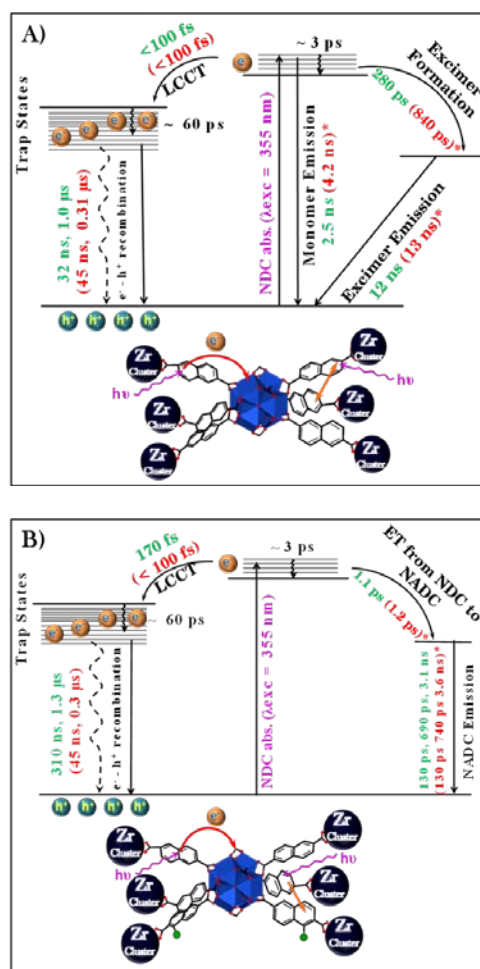


Figure 6. Illustration of the studied processes in photoexcited A) Zr-NDC and B) Zr-NADC MOFs in DCM (Green) and N,N'-dimethylformamide (DMF) (Red), (Ref. 7).

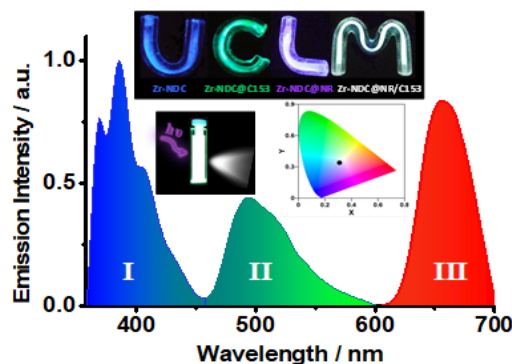


Figure 7. Multicolor (blue= Zr-NDC, green= C153@Zr-NDC, red= NR@Zr-NDC) and cool white light (NR/C153@Zr-NDC) emissions. The inset is a photo of the real emission and the CIE coordinates of the NR/C153@Zr-NDC composite material (Ref. 8).

To worthy note, it was showed that the simultaneously doping of the MOF with Nile Red (NR) and coumarin 153 (C153) allows to get a three-band emission spectrum producing white light (Figure 7). These findings were patented at international level for MOF-LED fabrication (see CV). To design a MOF-LED, we first explored Zr-NDC and C153@Zr-NDC MOF materials dispersed in polycarbonate (PC) films (Ref. 10). In both polymeric layers, the excimers formation in Zr-NDC as well as the energy transfer in C153@Zr-NDC was determined. With the result published, we fabricated the first Zr-MOF-LEDs and explored their electroluminescence properties (Figure 8, Ref. 11). We also demonstrated that both, the dye encapsulation and the presence of defects in the MOF structure have considerable influence in the device performance.

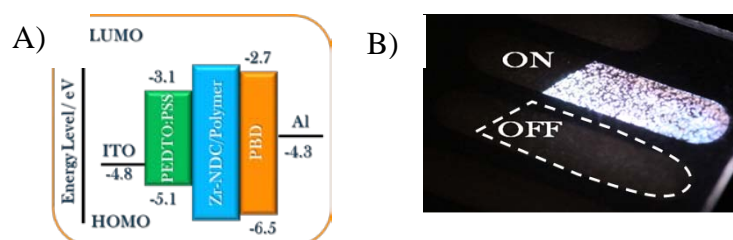
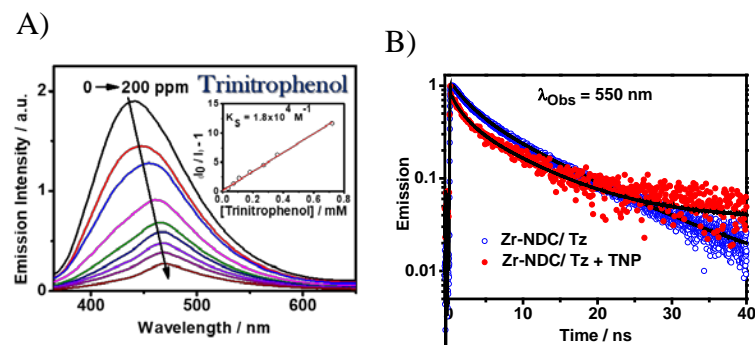


Figure 8. A) Schematic representation of the MOF-LED device structure, showing the used materials and their energy levels. B) Photo of a working LED device in which the luminescent layer is made with a film of Zr-NDC MOF dispersed in a PVK polymeric matrix (Ref. 11).

Finally, we explored the spectroscopy and photodynamic of new Zr-NDC/Tz and Zr-NDC/CN MOFs, and showed how the presence of several nitroaromatic like-explosive molecules affect their photobehavior. For example, the interaction with tens ppm of trinitrophenol (TNP) produces a strong emission quenching, explained in terms of charge transfer through intermolecular H-bonds between the Tz or CN moieties of the MOFs and the -OH group of TNP (Figure 9). Furthermore, I have shown the large selectivity and several-times use of these MOFs for TNP (Ref. 12).



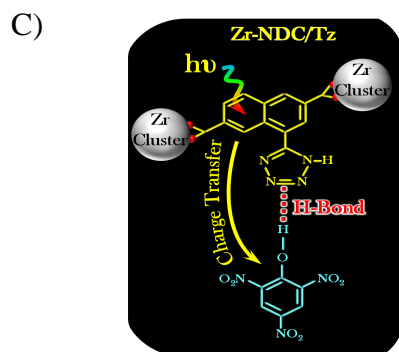


Figure 9. A) Emission spectra and B) time-resolved decays of Zr-NDC/TZ in presence of the explosive TNP. C) Representation of the H-bonds interactions between Zr-NDC/TZ and TNP (Ref 12).

In summary, I combined steady-state and four time-resolved spectroscopic techniques to get a deeper understanding of the photophysical processes involved in the excited state of new PT dyes and new Zr-MOFs. I also spent 3 months in the group of Prof. Majed Chergui (École polytechnique fédérale de Lausanne, EPFL, Swiss), where I investigated the properties of the charge carriers in a photoexcited perovskite using time-resolved X-Ray absorption spectroscopy (Ref. 9). My Ph.D.'s work has crystallized in **13 articles in peer scientific journals, and 1 international patent**. The results were disseminated in 24 national and international conferences: 8 posters, and 16 oral contributions. Not less important, the Ph.D. thesis allowed me to be awarded with 6 national and international prizes.

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Upconversion nanoparticles: synthesis and applications

Thesis of Laura Francés Soriano, Universitat de València (Spain)

Supervisors: Prof. Julia Pérez-Prieto, Dr. María González-Béjar

In the last decades *nanochemistry* has reached great interest due to the importance of developing innovative and unique materials at nanometric scale, specifically nanoparticles. The properties of nanomaterials differ from those at a larger scale, so nanochemistry open innovative ways in science to develop materials with new properties and novel performance.

Upconversion nanoparticles (UCNPs) are lanthanides inorganic based nanocrystals, such as NaYF_4 co-doped with lanthanides cations (such as Er^{3+} , Yb^{3+} , Tm^{3+} ...). UCNPs display excellent chemical, thermal and photostability, good biocompatibility, narrow bandwidth, long luminescence times, no photoblinking and no photobleaching. Most importantly, they can emit in the visible after their excitation at the near-infrared (NIR) because of their intra-configurational 4f electron transitions. Hence, UCNPs are of great interest in many fields such as security, photocatalysis or sensing and specially in biomedicine (bioimaging, photodynamic therapy...) due to the high penetration depth of the NIR light in tissues.

Nanochemistry plays a crucial role in architecting UCNPs, firstly, in the development of new synthetic routes and/or control of the reaction parameters in order to achieve monodisperse and uniform UCNPs with high UC efficiency. Secondly, nanochemistry is important for surface engineering, since it deals with the convenient modification and functionalization of the UCNPs surface to provide them with desired properties and functionalities.

This thesis is focused in the synthesis of new nanomaterials based on NaYF_4 co-doped nanoparticles with exceptional properties and their further derivatization. Texture and phase recognition analysis (TPRA) based on electron nanodiffraction technique is used to characterize the geometry of UCNPs synthesized by the high temperature co-precipitation strategy which uses stoichiometric amounts of NH_4F . Here, we confirmed experimentally that despite the apparently different shapes of samples (hexagons, rods, and cubes), all the

nanocrystals are actually β -phase hexagonal prisms. This is of relevance since many biological features of nanostructures, such as cellular internalization and cytotoxicity, are governed by their geometry. In addition, reproducibility in biological experiments is paramount.

In addition, water-dispersible, ca. 30 nm-sized $\text{NaYF}_4: \text{Er}^{3+}, \text{Yb}^{3+}$ UCNP, capped with a polyethylene glycol (PEG) derivative and highly loaded with a singlet oxygen photosensitizer, specifically a diiodo-substituted Bodipy (IBDP), was synthesized. The photosensitizer, bearing a carboxylic group, was anchored to the UCNP surface and, at the same time, embedded in the PEG capping; the combined action of the UCNP surface and PEG facilitated the loading for an effective energy transfer and, additionally, avoided photosensitizer leaching from the nanohybrid (UCNP-IBDP@PEG).

The effectiveness of the nanohybrids in generating singlet oxygen after NIR excitation with a continuous wavelength laser was evidenced by using a probe molecule. In vitro assays demonstrated that the UCNP-IBDP@PEG nanohybrid was taken up by the SH-SY5Y human neuroblastoma-derived cells showing low cytotoxicity. Moreover, ca. 50% cancer cell death was observed after NIR irradiation (45 min, $239 \text{ mW}\cdot\text{cm}^{-2}$).

Steady-state and time-resolved emission studies on this nanohybrid, and on its hydrophobic analogous, shows that the Yb^{3+} metastable state, formed after absorption of a NIR photon, can decay via two competitive energy transfer processes: sensitization of IBDP after absorption of a second NIR photon and population of Er^{3+} excited states.

Moreover, spontaneous adsorption of cucurbit[n]uril, CB[n] ($n = 6, 7$, and 8), on the surface of naked $\text{NaYF}_4: \text{Er}^{3+}, \text{Yb}^{3+}$ gave rise to UCNP@CB[n] exclusion complexes. These complexes proved to be highly stable as well as highly emissive under near-infrared excitation. By using two tricyclic basic dyes (specifically, methylene blue and pyronin Y) as a proof of concept, we demonstrate that the UCNP@CB[n] ($n = 6, 7$) nanohybrids can form exclusion complexes with this type of dyes via the CB carbonyl free portal, i.e., UCNP@CB@dye hybrids, thus making it possible to locate a high concentration of the dyes close to the UCNP and, consequently, leading to efficient energy transfer from the UCNP to the dye.

Furthermore, CB[7] was used to assemble two different nanoparticles, UCNPs and $\text{CH}_3\text{NH}_3\text{PbBr}_3$ perovskite nanoparticles (PK). This innovative strategy allows to anchor the perovskite nanoparticles firmly and closely to the naked UCNPs surface, thus leading to $\text{UC}_n@\text{PK}_{\text{CB}}$ nanohybrids.

A commercial multiphoton laser scanning confocal microscope is used to demonstrate the successful assembly. This technique proves to be useful to evaluate luminescence lifetime in the range of several tens of μs and allows visualization of the extraordinarily efficient nontrivial resonance energy transfer from the upconversion nanoparticle to the perovskite after NIR excitation of the nanohybrid as well as the homogeneity of the $\text{UC}_n@\text{PK}_{\text{CB}}$ sample. The considerable photostability of the perovskite in these nanohybrids is demonstrated by prolonged irradiation of the nanohybrid under UV light as well as under NIR light.

Last but not least, UCNPs were capped with a thin polymer shell by replacing the oleate ligand of hydrophobic UCNPs by multidentate thiolate-grafting of $\text{P}(\text{MEO}_2\text{MA-co-SEMA})$ copolymers. The presence of the 2-(2-methoxyethoxy)ethyl side chains of MEO_2MA extending out of the nanohybrid made them water-dispersible and thermosensibles. The $\text{UCNP}@\text{P}(\text{MEO}_2\text{MA-co-SEMA})$ nanohybrids exhibited an enhanced emission by up to a factor of 10, as compared with that of their hydrophobic precursor in dichloromethane and even in water (a factor of 2).

Their thermoresponsiveness was modulated by the pH; this is consistent with the presence of some thiol groups at the nanohybrid periphery. Remarkably, the nanohybrid emission, as well as its stability, was almost independent of the aggregation state (in the basic-acid and temperature range studied here). The formation of stable water-dispersible UCNPs with enhanced emission, together with their amphiphilic and temperature-responsive polymer coating, is promising for building multifunctional nanostructures for intracellular imaging, therapy, and drug delivery.

Fluorescent molecular rotors: from working principles to visualization of mechanical contacts

Tomislav Suhina

Visualization and the ability to precisely measure mechanical contacts between solid surfaces is a subject of considerable interest in physics. This is because mechanical contacts are intimately related to friction, which is a process of immense importance as it occurs everywhere around us. Friction is defined as a force that resists relative motion between objects in contact. Without friction, we would not be able to move nor survive. Although we find numerous examples where it is useful, friction causes huge energy losses and damage in materials caused by wear. According to recent estimates, friction is held responsible for 30 % of the world's total energy consumption. In spite of its importance, our fundamental understanding of friction and its relation to the microscopic contact area is still very limited. Although a significant amount of theoretical work was conducted on this subject, experimental observation of the real contact area presents a significant challenge. This thesis describes development and detailed characterization of a method that overcomes experimental difficulties related to monitoring contacts between surfaces in contact, and enables us to visualize and measure contacts before, during, and after frictional processes with diffraction limited resolution.

To tackle the problem, we developed a method based on fluorescent molecular rotors. For this purpose, molecules are designed to contain functional groups that allow us to immobilize them on solid surfaces, such as microscope glass cover slips. These molecules are weakly fluorescent in low-viscosity solvents, because internal rotational motions result in a rapid decay of the excited state, such that the emission of the fluorescence photon is not fast enough to compete effectively. When such motion becomes hindered due to confinement, as in solvents of high viscosity or polymer matrices, the molecules remain in their excited state much longer and fluoresce. Similar confinement, in our case, occurs in contact points between functionalized glass surface and an arbitrary object, and this allows us to visualize and measure contacts with fluorescence microscopy.

In Chapter 1 we introduce the topic of this thesis, and discuss the role that the real contact area plays in understanding friction. In this Chapter we also introduce fluorescent molecular rotors, their application, and the origin of their response towards confinement in viscous liquids and polymer matrices. In Chapter 2 we describe experimental methods and data analysis procedures that are used throughout this thesis.

In Chapter 3 we demonstrate the validity of our approach for visualizing the contact area by immobilizing dicyanomethylenedihydrofuran (DCDHF) molecular rotors on glass cover slips, and forming contacts between functionalized cover slips and a PMMA bead. With a confocal microscope with a rheometer attached to it, we are able to exert controlled normal forces with the bead, and simultaneously monitor the fluorescence response from glass to which we covalently attached the molecular rotors. Contact-induced confinement of surface-bound DCDHF molecular rotors results in strong fluorescence enhancement, and enables us to visualize and measure contact area between the two objects under a range of loads. Approximating the measured contact area as circular allows us to confirm the validity of our experimental results by comparing them with the ones predicted by the widely-used Hertz theory of non-adhering elastic contacts, which assumes perfect smoothness of both surfaces in contact. We find excellent agreement between the two. In addition, we are able to detect the presence of fine structure within the zone of contact, which is caused by the microscopic roughness of PMMA bead. During our basic photophysical characterization of this molecule, we observe fluorescence quantum yield trends which suggest environment polarity is an important parameter in excited-state decay kinetics of this molecule, in addition to microviscosity. Furthermore, we found fluorescence decays of our DCDHF molecular rotor to be nonexponential in some solvents. Both observations indicate that photophysical behavior of these molecules is more complex than previously reported for the same chromophore, and motivated us to conduct a detailed photophysical characterization of this chromophore (Chapters 4 and 5).

Chapter 4 describes steady-state and time-resolved spectroscopic measurements conducted on the DCDHF-based chromophore used in Chapter 3. The combination of these powerful laser spectroscopy techniques with quantum-chemical TD-DFT calculations enables us

to learn more about the excited-state dynamics of DCDHF molecular rotors in low, medium, and high polarity solvents. We show that both single and double bond rotations can cause excited state decay in the case of DCDHF rotors. In non-polar solvents fluorescence is quenched by rotation about a dicyanomethylene double bond, which results in momentary excited-state decay due to a conical intersection. In a sufficiently polar environment rotation about a formally single bond leads to a nonfluorescent internal charge-transfer state. In medium-polar solvents such as ethyl acetate, formation of dark charge-transfer state is reversible, and this results in delayed fluorescence which manifests itself in nonexponential fluorescence decays reported in Chapter 3. As polarity of the environment increases further, formation of the charge-transfer state becomes irreversible. We detect this species directly using time-resolved transient infrared spectroscopy in the polar solvent dimethylsulfoxide, while in non-polar toluene intermediate species is not detected.

In Chapter 5 we perform additional mechanistic studies on the same molecule using quantitative fluorescence and transient absorption spectroscopies. These methods enable us to examine and quantify the influence of solvent polarity on the photophysical behavior of this type of molecular rotors. The obtained experimental data support the model with two polarity-responsive excited-state deactivation barriers proposed in Chapter 4. In low-viscosity solvents the presence of two excited-state deactivation pathways leads to fast excited state decay and weak fluorescence both in solvents of low polarity and in solvents of high polarity. In solvents of intermediate polarity, the fluorescence quantum yield, however, is remarkably high. Finally, pump-probe measurements in the visible spectral range reveal the spectra of the intermediate dark state in three (polar) solvents, while no intermediate state can be observed in case of low-polarity solvents. Thus, these results provide strong support for the model that was proposed in Chapter 4.

Chapter 6 describes a detailed photophysical characterization of a molecular rotor based on meso-substituted boron-dipyrromethane (BODIPY) framework. We again use visible and IR pump-probe spectroscopies combined with TD-DFT calculations, and we show that fluorescence deactivation of this molecule takes place through a fast and irreversible process which does not involve intermediate electronic states. Our data indicate that nonradiative excited-state deactivation of BODIPY molecular rotors is practically independent

of solvent polarity, but strongly governed by viscoelastic/free volume properties of the local environment in both low- and high-viscosity regimes.

In Chapter 7 we introduce a new DCDHF-based molecular rotor with extended π -conjugation, which results in a significant red shift of the absorption and emission spectra relative to the previously introduced DCDHF and BODIPY molecular rotors. We design this molecule in order to reduce the potential risk of exciting fluorescent impurities that may be present on our surfaces. In this chapter we compare the photophysical behavior of these molecules in solutions, immobilized on a glass surface, and under contact-induced confinement by means of steady-state and time-resolved spectroscopies. While the fluorescence of the two examined molecular rotors based on dicyanomethylenedihydrofuran accepting unit is significantly enhanced within the contact zone, the BODIPY-based molecular rotor unexpectedly does not show confinement-induced response, in spite of the fact that its fluorescence correlates with changes of solution viscosities and becomes greatly enhanced in polymer matrices. We attribute this observation to different modes of distortion that lead to the nonradiative decay channel. Based on the response of other two molecular rotors, we show that in the contact zones the probe molecules are strongly confined but still have some freedom to move. The nanoscale environment resembles a viscous liquid like glycerol, and remains homogeneous within contacts.

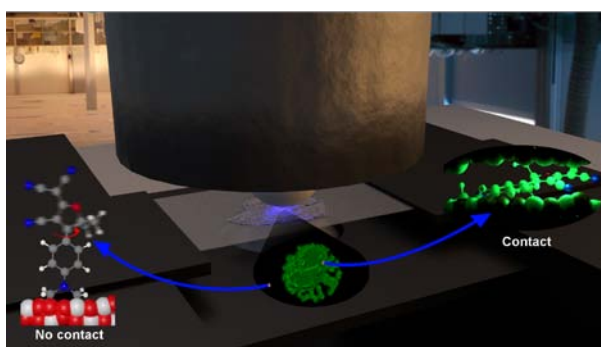


Figure 1. Schematic representation of the contact area measurement and its working principle.

Chapter 8 describes an application of our method to study the relation between friction and the real contact area. We find that frictional force is directly proportional to the real area of contact between a glass cover slip and a polystyrene bead. The contact area, however, does not grow linearly with the applied normal force and results in breaking of first Amontons' law of friction, which states that the force of friction is directly proportional to the applied load. With the help of simulations from our collaborators we find that this is because both elastic interactions and plastic deformations play an important role in deformations of the asperities that are present within the zone of contact between the objects.

CONFERENCE REPORT

CECP 2018 Conference Report



From Sunday, February 4 to Thursday, February 8, 2018, 85 photochemists from 17 different countries came together to share their results and experiences at the Congress Centre of Bad Hofgastein/Austria. It was a wonderful atmosphere and all participants really enjoyed the meeting. The scientific organization has been done by the international scientific committee: Mohamed **Sarakha**, Clermont-Ferrand, F, Dominik **Heger**, Brno, CZ, Heiko **Ihmels**, Siegen, D, Jacek **Waluk**, Warsaw, PL, Oliver **Wenger**, Basel, CH, Maurizio **Fagnoni**, Pavia, I, and as a guiding member: Stephan **Landgraf**, Graz/A for EPA Austria (ZVR: 050416508). The conference started on Sunday with the get-together including a buffet and an opening lecture.

From Monday to Wednesday there were two sessions per day with one plenary lecture in each session (6 plenary lectures and an experimental lecture of Amitabh **Banerji**, Cologne, D), as well as short talks (28 oral presentations), and a poster session every evening (41 poster presentations). Plenary lectures have been given by Andreas **Steffen**, Würzburg/D, "Influence of metal-metal interactions on the excited states in d¹⁰ coinage metal complexes", Martin **Goez**, Halle/D, "Accessing the super-reductant eq through sustainable photoionizations", Elena **Selli**, Milan/I, "Photocatalytic materials: spectroscopic characterization in relation to photoactivity", Gonzalo **Angulo** vs. Arnulf **Rosspeintner**, Warsaw/PL/Geneva/CH, "Flies On the Storm: a Dialogue About Diffusion In Photochemistry" (Tandem lecture), Marcello **Brigante**, Aubièrre/F, "Photochemical generation of inorganic radicals:

Environmental applications and polluted water remediation", and Radek **Cibulka**, Prague/CZ, "Flavins - not only cofactors but also efficient photocatalysts".



Fig. 1: Plenary speakers of **CECF 2018**, from left to right Steffen, Goetz, Selli, Angulo (first row), Rosspeintner, Brigante, Cibulka, and Banerji (second row).

In order to keep the scientific level of the meeting on an internationally high one the scientific committee selected 6 plenary lectures and 28 (out of 34 applications) short talks. Four of these short talks have been presented as tandem talks with two presenters from the same working group. Additional talks on activities of EPA and the IUPAC Subcommittee Photochemistry have been presented on Wednesday afternoon. All poster applications were accepted after checking by the local committee.

The local organizing committee has been formed by: Stephan Landgraf (local organizer from EPA Austria) and Sabine Richert. Additional help from Heidi Schmitt (also for the conference photos) is also gratefully acknowledged. The variety of different accommodations is available in Bad Hofgastein from private rooms up to hotels with a high comfort. Even during high season rooms are available in appropriate number if booked early enough. Contact

person for accommodation: Carina Schönegger, tourist office.
Official web site of the meeting: www.cecp.at

The key idea of the CECF meeting is to bring together young and experienced photochemists from all photochemical fields. Therefore everything was done to remove all hindrance to join the meeting. Additionally the evening should be undisturbed by the dinner. So four evening buffets were organized for all participants. The costs were included in the conference fee. So for students the conference itself has been free of charge. Young researchers up to 4 years after PhD, all attendees from Eastern European countries, and retired researchers could join for a reduced fee, too. A reduced fee for EPA members has been offered (30 € for full members and 15 € for students). For **CECF 2020** held in February, 2020, in Bad Hofgastein a small increase of the fees has to be accepted to compensate inflation.

The location of the Conference Centre of Bad Hofgastein allows a perfect access to physical activities during the afternoon break. Downhill and cross-country skiing, as well as the thermal bath and spa, offer a variety of possibilities to enjoy the region or to relax.



Fig. 2: Participants of CECF 2018 located on an Europe map.

Together with the scientific program an ideal combination is present at this place.

Finally 92 persons registered for the meeting till the beginning of the conference. Since 2006 there have been some fluctuation in the distribution of participants with a mean values slightly above 100. Most of the participants come from Germany, but there also significant attendance from France, Switzerland, Poland, Italy, Hungary, Sweden, Croatia, and Czech Republic (with 5 participants and more).

The meeting is organized on a non-profit base. All travel grant applications have been accepted by the local organizer. 5 attendees have been supported by this procedure.

Statistics: PL: 1, HR: 1, H: 1, RUS: 1, UA: 1.

CECP 2018 Awards for Young Scientists

There are two prices for young scientists to encourage them to present their best results at the CECP meeting. All oral and poster presentations have been evaluated by the international scientific committee. Both prices have been awarded at the end of the meeting at the closing ceremony.

CECP 2018 Award for best oral presentation:

O8 Julien Christmann, Mulhouse, F

"Evidencing the noticeable role of back electron transfer in polymerization kinetics initiated by a triazine-based type-II photoinitiating system"



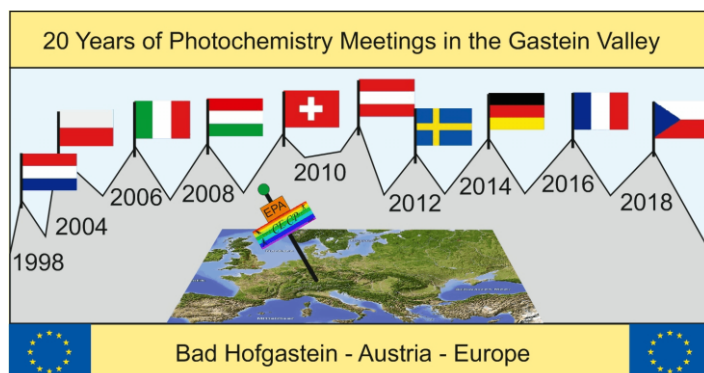
CECP 2018 Award for best poster presentation:

P23 Gaowa Naren, Göteborg, S

“A Photocontrolled RGB Emitting System”



Last but not least we celebrated 20 Years *“Photochemistry meetings in the Gastein valley”* with our “Bauernbuffet” on Wednesday evening.



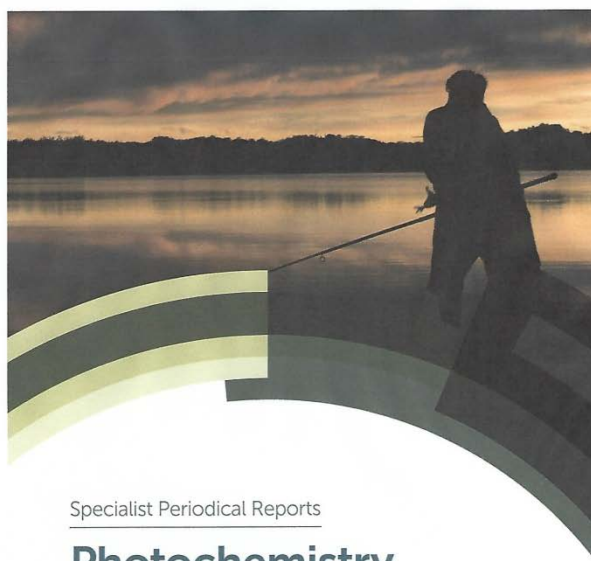
Tab. 1: Former CECF awards:

Year	Award for best oral presentation	Award for best poster presentation
2006	David Bailey , Bremen/D	Katja Draxler , Konstanz/D
2008	Dominik Wöll , Konstanz/D	David Carteau , Bordeaux/F
2010	Simone Draxler , Munich/D	Sabine Richert , Graz/A
2012	David Bléger , Berlin/D	Franziska Graupner , Munich/D
2014	Fillipo Monti , Bologna/I	Jesper Nilson , Göteborg/S
2016	Anne Fuhrmann , Berlin, D	Maria Pszona , Warsaw, PL

The winner of *CECF 2014 Award for best poster presentation* got one new tablet PC sponsored by Gilden Instruments.

The winner of *CECF 2016 Award for best poster presentation* got one new tablet PC sponsored by Peschl Ultraviolet and Ekspla.

BOOK ON PHOTOCHEMISTRY



Specialist Periodical Reports

Photochemistry

Volume 46

Edited by Angelo Albini and Stefano Protti



PHOTOCHEMISTRY Vol. 46:**Preface**

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Part 2. Highlights

Chapter Name: Two-photon responsive chromophore for uncaging reactions

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Chapter Name: Controlled release of volatile compounds using the Norrish-type II reaction

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Chapter Name: Recent advances in the design of light-activated tissue bonding

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Chapter Name: Photoresponsive molecular probes targeting nucleic acid secondary structures

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Chapter name: Transition metal complexes in ECL based biosensing techniques

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Chapter name: Photochemical bond activation at metal centres: a snapshot into selectivity.

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Chapter Name: Aromatic hydrocarbons as catalysts and electron shuttles in light-induced electron transfer reactions

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Chapter name: Photochemical and Photocatalysed Multicomponent Reactions

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Chapter name: Asymmetric Catalysis of Triplet-State Photoreactions

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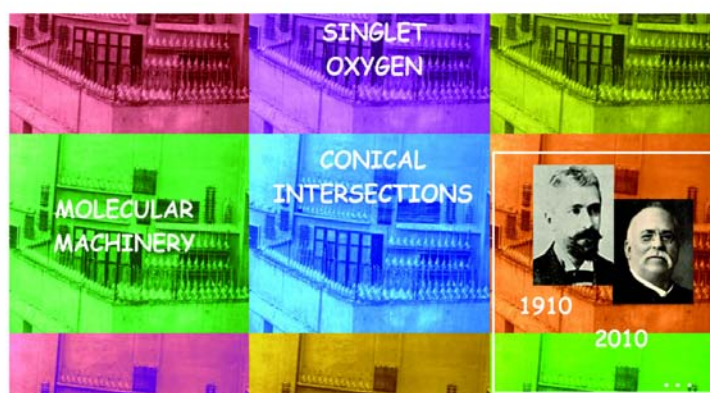
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