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“Organic Molecular Materials”

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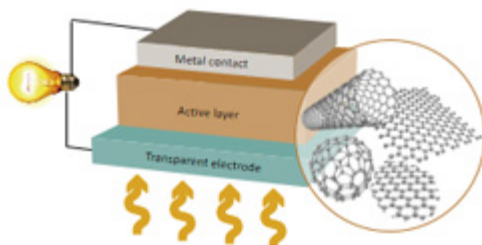
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<http://www.nazariomartingroup.com/index.html>

## ARTICLES 2016

- 1) N. Martín "Carbon Nanoforms for Photovoltaics: Myth or Reality?" *Adv. Energy Mater.* **2016**, 1601102 DOI: 10.1002/aenm.201601102

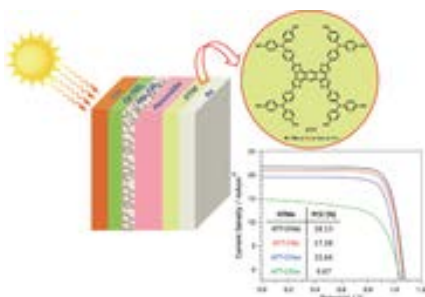
## Abstract



Carbon nanoforms, namely fullerenes, CNTs, graphene and graphene quantum dots reveal appealing properties to be used as interesting active materials for the preparation of photovoltaic devices. The experimental findings evidence that far from being a myth, these carbon materials are becoming a reality with remarkable energy conversion efficiencies.

- 2) I. Zimmermann, J. Urieta-Mora, P. Gratia, J. Aragó, G. Grancini, A. Molina-Ontoria, E. Ortí, N. Martín, M. Khaja Nazeeruddin "High-Efficiency Perovskite Solar Cells using Molecularly-Engineered, Thiophene-Rich, Hole-Transporting Materials: Influence of Alkyl Chain Length on Power Conversion Efficiency" *Adv. Energy Mater.*, **2016**, 1601674 - DOI: 10.1002/aenm.201601674

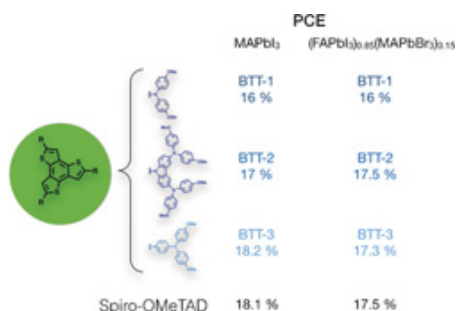
## Abstract



A sulfur-rich polycyclic aromatic hydrocarbon with a flat and rigid  $\pi$ -conjugated structure endowed with triarylamine groups is synthesized for highly efficient perovskite solar cells. The influence of alkyl chains, often introduced to increase the solubility of the hole-transporting materials, is furthermore investigated. Efficiencies as high as 18.1% are obtained.

- 3) A. Molina-Ontoria, I. Zimmermann, I. Garcia-Benito, P. Gratia, C. Roldán-Carmona, S. Aghazada, M. Graetzel, M. Khaja Nazeeruddin, N. Martín "Benzotrithiophene-Based Hole-Transporting Materials for 18.2% Perovskite Solar Cells" *Angew. Chem. Int. Ed.* **2016**, 55, 6270–6274 - DOI: 10.1002/anie.201511877

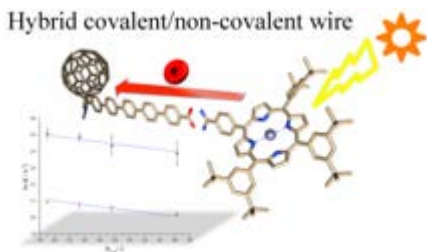
## Abstract



New star-shaped benzotrithiophene (BTT)-based hole-transporting materials (HTM) BTT-1, BTT-2 and BTT-3 have been obtained through a facile synthetic route by crosslinking triarylamine-based donor groups with a benzotrithiophene (BTT) core. The BTT HTMs were tested on solution-processed lead trihalide perovskite-based solar cells. Power conversion efficiencies in the range of 16 % to 18.2 % were achieved under AM 1.5 sun with the three derivatives. These values are comparable to those obtained with today's most commonly used HTM spiro-OMeTAD, which point them out as promising candidates to be used as readily available and cost-effective alternatives in perovskite solar cells (PSCs).

- 4 S. Vela, S. Bauroth, C. Atienza, A. Molina-Ontoria, D. M. Guldi, N. Martín "Determination of the Attenuation Factor ( $\beta$ ) in Hybrid Covalent/Non-Covalent Molecular Wires" *Angew. Chem. Int. Ed.*, **2016**, *55*, 15076–15080 - DOI: 10.1002/anie.201608973

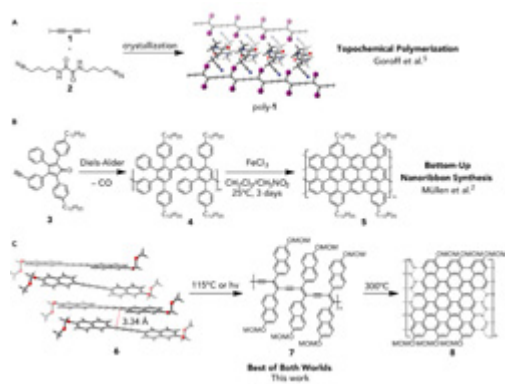
Abstract



Hybrid covalent/supramolecular porphyrin–fullerene structures were synthesized as highly efficient molecular wires with a remarkably low attenuation factor ( $\beta=0.07\pm 0.01 \text{ \AA}^{-1}$ ). Hydrogen-bonding interactions and p-phenylene oligomers of different lengths are responsible for efficient electron transfer in the molecular wires.

- 5 P. J. Evans, N. Martín "Graphene Nanoribbons via Crystal Engineering" *Chem*, **2016**, *1*, 16–31 - DOI:10.1016/j.chempr.2016.06.012

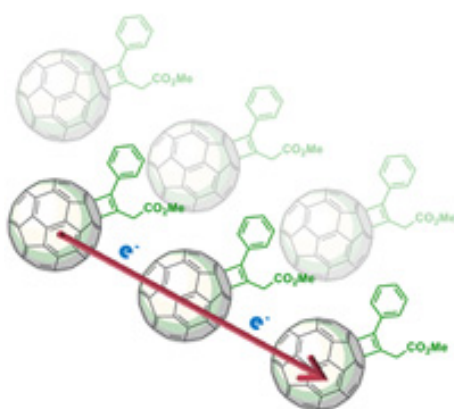
Abstract



In this issue of Chem, Rubin and coworkers have developed a new approach for the bottom-up synthesis of graphene nanoribbons by efficiently combining crystal engineering and topochemical polymerization.

- 6 S. Reboredo, R. M. Girón, S. Filippone, T. Mikie, T. Sakurai, S. Seki, N. Martín "Cyclobuteno[60]fullerenes as efficient n-type organic semiconductors" *Chemistry - A European Journal*, **2016**, *22*, 13627 – 13631 - DOI:10.1002/chem.201602813

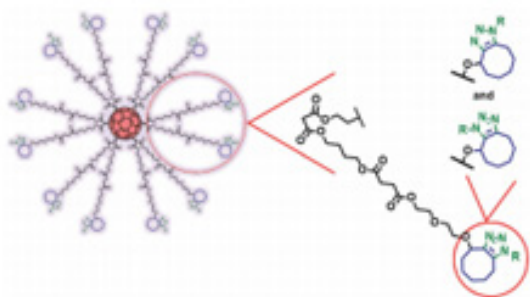
Abstract



Cyclobuteno[3,4:1,2][60]fullerenes have been prepared in a straightforward manner by a simple reaction between [60]fullerene and readily available allenates or alkynoates as organic reagents under basic and mild conditions. The chemical structure of the new modified fullerenes has been determined by standard spectroscopic techniques and confirmed by X-ray diffraction analysis. Some of these new fullerene derivatives exhibit a remarkable intrinsic electron mobility (determined by using flash-photolysis time-resolved microwave conductivity (FP-TRMC) measurements), which surpasses that of the well-known phenyl-C61-butyric acid methyl ester, thus behaving as promising n-type organic semiconductors.

- 7 J. Ramos-Soriano, J. Juan Reina, A. Pérez-Sánchez, B. M. Illescas, J. Rojo, N. Martín "Cyclooctyne [60]Fullerene Hexakis Adducts: a Globular Scaffold for Copper-Free Click Chemistry" *Chem. Commun.*, **2016**, *52*, 10544-10546 - DOI: [10.1039/C6CC05484F](https://doi.org/10.1039/C6CC05484F)

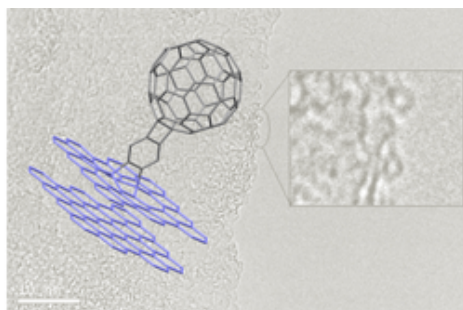
Abstract



The synthesis of a new highly symmetric hexakis adduct of C<sub>60</sub> appended with 12 cyclooctyne moieties has been carried out. This compound has been used for the copper-free strain-promoted cycloaddition reaction to a series of azides with excellent yields. This strategy for the obtention of clicked adducts of [60]fullerene is of special interest for biological applications.

- 8 D. García, L. Rodríguez-Pérez, M. A. Herranz, D. Peña, E. Guitián, S. Bailey, Q. Al-Galiby, M. Noori, C. J. Lambert, D. Pérez, N. Martín "A C<sub>60</sub>-aryne building block: synthesis of a hybrid all-carbon nanostructure" *Chem. Commun.*, **2016**, *52*, 6677-6680 - DOI: [10.1039/C5CC10462A](https://doi.org/10.1039/C5CC10462A)

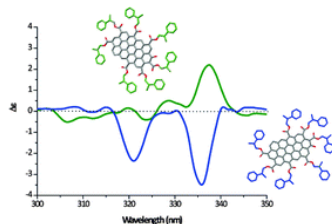
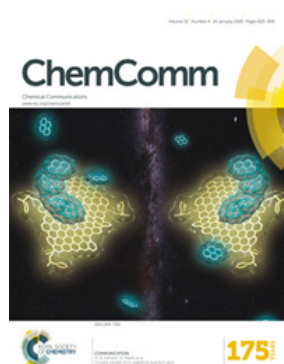
Abstract



The synthesis of a [60]fullerene-benzyne building block and its further chemical cycloaddition reaction with graphene has resulted in a new all-carbon hybrid material which has been characterized by TGA, FTIR and Raman spectroscopies, XPS as well as AFM and TEM. Based on computational studies, the formation of both [2+2] and [4+2] cycloadducts on the graphene surface is feasible.

- 9 M. Vázquez-Nakagawa, L. Rodríguez-Pérez, M. A. Herranz, N. Martín "Chirality transfer from graphene quantum dots" *Chem. Commun.*, **2016**, *52*, 665-668- DOI: [10.1039/c5cc08890a](https://doi.org/10.1039/c5cc08890a)

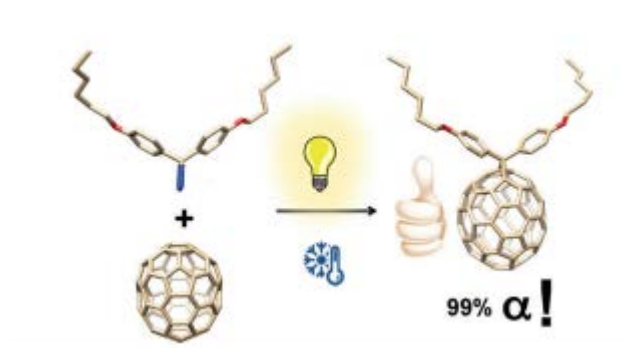
Abstract



Chiral graphene quantum dots were prepared by acidic exfoliation and oxidation of graphite, dialysis, and esterification with enantiomerically pure (R) or (S)-2-phenyl-1-propanol. Circular dichroism studies support the formation of supramolecular aggregates with pyrene molecules, where a transfer of chirality occurs from the chiral graphene quantum dots to the pyrene.

- 10 S. Vidal, M. Izquierdo, W. Kit Law, K. Jiang, S. Filippone, J. Perles, H. Yan, N. Martín "Photochemical Site-selective Synthesis of [70]Methanofullerenes" *Chem. Commun.*, **2016**, 52, 12733-12736 DOI: 10.1039/c6cc06072b

#### Abstract



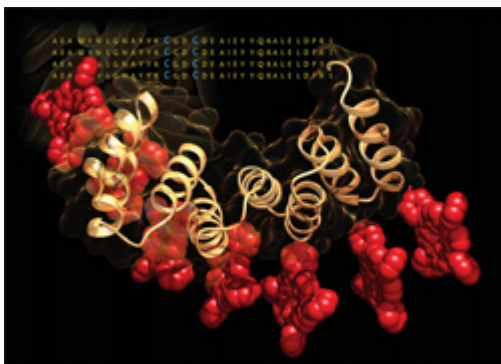
Methanofullerenes such as the well-known [70]PCBM are commonly synthesized under harsh conditions to obtain the product as a mixture of site-isomers (namely  $\alpha$ ,  $\beta$  and minor  $\gamma$ ) due to the D<sub>5h</sub> symmetry of the C<sub>70</sub> cage. We report the first site-selective synthesis of [70]methanofullerenes under light irradiation and low temperatures, thus avoiding time-consuming and highly expensive HPLC separations. Pure major site-isomers  $\alpha$ -[70]PCBM and  $\alpha$ -[70]DPM have been thus efficiently prepared including the crystal structure of 5b. Photovoltaic preliminary results revealed a slightly beneficial performance for  $\alpha$ -pure [70]PCBM site-

isomer devices.

- 11 S. H. Mejías, J. López-Andarias, T.I. Sakurai, S. Yoneda, K. P. Erazo, S. Seki, C. Atienza, N. Martín, A. L. Cortajarena "Repeat protein scaffolds: ordering photo- and electroactive molecules in solution and solid state" *Chem.Sci.*, **2016**, 7,4842-4847-DOI:10.1039/c6sc01306f

#### Abstract

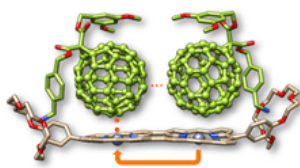
The precise control over the organization of photoactive components at the nanoscale is one of the main challenges



for the generation of new and sophisticated macroscopically ordered materials with enhanced properties. In this work we present a novel bioinspired approach using protein-based building blocks for the arrangement of photo- and electroactive porphyrin derivatives. We used a designed repeat protein scaffold with demonstrated unique features that allow for the control of their structure, functionality, and assembly. Our designed domains act as exact biomolecular templates to organize porphyrin molecules at the required distance. The hybrid conjugates retain the structure and assembly properties of the protein scaffold and display the spectroscopic features of orderly aggregated porphyrins along the protein structure. Finally, we achieved a solid ordered bio-organic hybrid thin film with anisotropic photoconductivity.

- 12 L. Moreira J. Calbo, J. Aragón, B. M. Illescas, I. Nierengarten, B. Delavaux-Nicot, E. Ortí, N. Martín, J.F. Nierengarten "Conjugated Porphyrin Dimers: Cooperative Effects and Electronic Communication in Supramolecular Ensembles with C60" *J. Am. Chem. Soc.* - DOI: 10.1021/jacs.6b07250

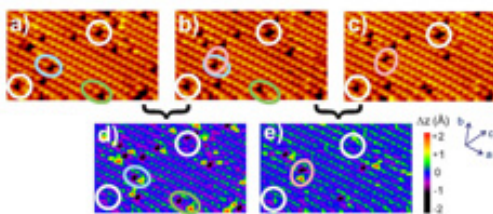
#### Abstract



Two new conjugated porphyrin-based systems (dimers 3 and 4) endowed with suitable crown ethers have been synthesized as receptors for a fullerene-ammonium salt derivative (1). Association constants in solution have been determined by UV-vis titration experiments in CH<sub>2</sub>Cl<sub>2</sub> at room temperature. The designed hosts are able to associate up to two fullerene-based guest molecules and present association constants as high as  $\sim 5 \times 10^8 \text{ M}^{-1}$ . Calculation of the allosteric cooperative factor  $\alpha$  for supramolecular complexes [3·12] and [4·12] showed a negative cooperative effect in both cases. The interactions accounting for the formation of the associates are based, first, on the complementary ammonium-crown ether interaction and, second, on the  $\pi$ - $\pi$  interactions between the porphyrin rings and the C60 moieties. Theoretical calculations have evidenced a significant decrease of the electron density in the porphyrin dimers 3 and 4 upon complexation of the first C60 molecule, in good agreement with the negative cooperativity found in these systems. This negative effect is partially compensated by the stabilizing C60-C60 interactions that take place in the more stable syn-disposition of [4·12].

- 13 C. Urban, R. Otero, D. Ecija, M. Trelka, N. Martín, J. M. Gallego, R. Miranda "Collective Concerted Motion in a Molecular Adlayer Visualized through the Surface Diffusion of Isolated Vacancies" *J. Chem. Phys.*, 2016, 145, 154706 DOI:10.1063/1.4964862

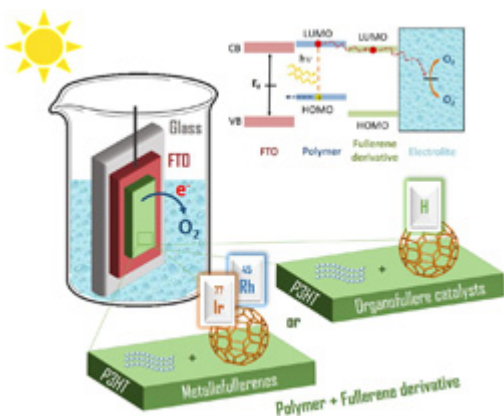
#### Abstract



We have measured STM movies to study the diffusion of individual vacancies in a self-assembled layer of a tetrathiafulvalene derivative (exTTF) on Au(111) at room temperature. The diffusion is anisotropic, being faster along the compact direction of the molecular lattice. A detailed analysis of the anisotropic displacement distribution of the single vacancies shows that the relative abundance of double jumps (that is, the collective motion of molecular dimers) with respect to single jumps is rather large, the number of double jumps being more than 20% of the diffusion events. We conjecture that the relative abundances of long jumps might be related to the strength of the intermolecular bonding and the misfit of the molecular overlayer with the substrate lattice.

- 14 R. M. Girón, J. Marco-Martínez, S. Bellani, A. Insuasty, H. C. Rojas, G. Tullii, M. R. Antognazza, S. Filippone, N. Martín "Synthesis of Modified Fullerenes for Oxygen Reduction Reactions" *J. Mater. Chem. A*, **2016**, *4*, 14284–14290 - DOI: 10.1039/C6TA06573B

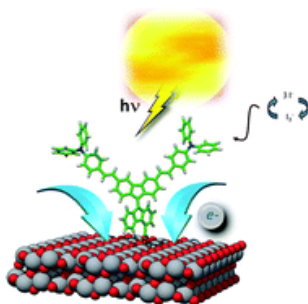
Abstract



The oxygen reduction reaction (ORR) is a key process common in several energy converting systems or electro-chemical technologies such as fuel cells, metal–air batteries, oxygen sensors, etc., which is based on the use of expensive and scarcely available platinum metal. In the search for carbon-based catalysts for ORRs, two different classes of new fullerene hybrids and metal-free fullerene derivatives endowed with suitable active sites have been prepared by highly selective metal- and organo-catalyzed synthetic methodologies. Along with their classical behavior as electron acceptors in polymer-based photo-electrochemical cells, the new fullerene derivatives are able to efficiently catalyze ORRs by using no metals or very low amounts of metals. Remarkably, the activity of metal-free fullerenes has proved to be as high as that observed for metallofullerenes bearing noble metals, and up to ten-fold higher than that of PCBM.

- 15 M. Wielopolski, M. Marszalek, F. G. Brunetti, D. Joly, J. Calbo, J. Aragón, J.-E. Moser, R. Humphry-Baker, S. M. Zakeeruddin, J. L. Delgado, M. Grätzel, E. Ortí, N. Martín "Synthesis and Optoelectronic Properties of Chemically Modified Bi-fluorenylidenes" *J. Mater. Chem. C*, **2016**, *4*, 3798-3808 - DOI: 10.1039/C5TC03501E

Abstract



The development of new light harvesting materials is a key issue for the progress of the research on organic & hybrid photovoltaics. Here, we report a new class of organic sensitizers based on the bi-fluorenylidene moiety as  $\pi$ -linker within the donor– $\pi$ -linker–acceptor (D– $\pi$ –A) scheme. The new dyes are endowed with electron donor and electron acceptor units at strategic positions in order to improve their electronic and light-harvesting properties. The comprehensive study of these compounds through the use of different experimental and theoretical techniques, provides an in-depth understanding of their electronic and photophysical properties, and reveal their interest as photovoltaic materials.

- 16 N. Martín "Journal of Materials Chemistry C Top Picks web collection: ‘‘The Many Faces of Carbon’’" *J. Mater. Chem. C*, **2016**, *4*, 6198-6199 -DOI: 10.1039/c6tc90085b

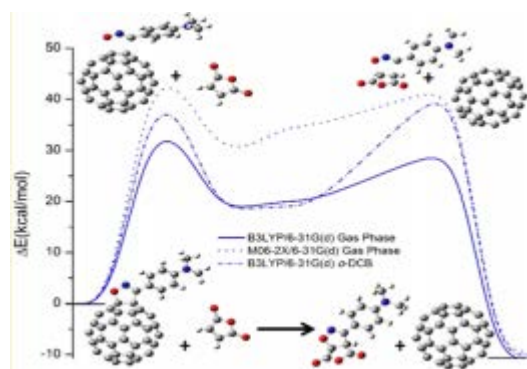
Abstract



Nazario Martín introduces a Journal of Materials Chemistry C Top Picks web collection on “the many faces of carbon” (<http://rsc.li/carbon>).

- 17 C. Morera-Boado, M. Martínez Gonzalez, R. A. Miranda-Quintana, M. Suarez, R. Martínez-Álvarez, N. Martín, J. M. García de la Vega "Theoretical Study on the Mechanism of the Thermal RetroCycloaddition of Isoxazolinofullerenes" *J. Phys. Chem. A*, **2016**, *120*, 8830–8842 - DOI: [10.1021/acs.jpca.6b07297](https://doi.org/10.1021/acs.jpca.6b07297)

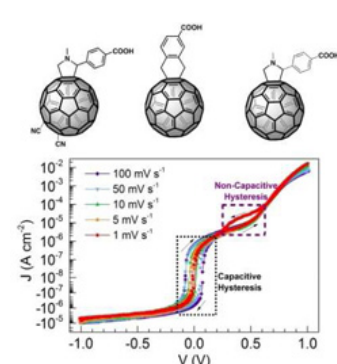
### Abstract



The retro-cycloaddition thermal reaction of isoxazolino[4,5:1,2][60]fullerenes to pristine fullerene seems to be guided by the electronic nature of the substituted nitrile oxide 1,3-dipole in the isoxazoline ring. Trapping experiments proved that the reaction mechanism occurs by thermal removal of the nitrile oxide 1,3-dipole in a process that is favored in the presence of a big excess of a highly efficient dipolarophile such as maleic anhydride. Theoretical gas phase calculations carried out at the B3LYP/6-31G(d) and M06-2X/6-31G(d) levels of theory underpin the experimental findings and predict that compound 1c, bearing the p-(CH<sub>3</sub>)<sub>2</sub>N-Ph substituent on the isoxazoline ring and with a remarkable experimental conversion efficiency in just 12 h, showed the lowest activation energy. Solvent calculations have predicted the same behavior in gas phase. Different approaches such as electrostatic natural population analysis and Houk's distortion/interaction model have been applied to understand how the electronic nature of these substituents affects the retro-cycloaddition reaction process. Analysis of the values of the condensed Fukui functions and dual descriptor shed light on the mechanism of the retro-cycloaddition reaction.

- 18 M. Valles-Pelarda, B. Clasen Hames, I. García-Benito, O. Almora, A Molina-Ontoria, R. S. Sánchez, G. Garcia-Belmonte, N. Martín, I. Mora-Sero "Analysis of the Hysteresis Behavior of Perovskite Solar Cells with Interfacial Fullerene Self-Assembled Monolayers" *J. Phys. Chem. Lett*, **2016**, DOI: [10.1021/acs.jpcclett.6b02103](https://doi.org/10.1021/acs.jpcclett.6b02103)

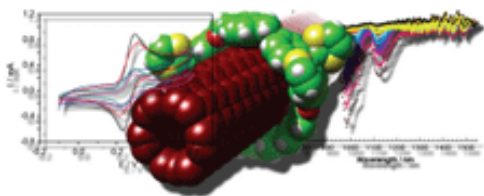
### Abstract



The use of self-assembled monolayers (SAMs) of fullerene derivatives reduces the hysteresis of perovskite solar cells (PSCs). We have investigated three different fullerene derivatives observing a decrease on hysteresis for all the cases. Several processes can contribute to the hysteresis behavior on PSCs. We have determined that the reduced hysteresis observed for devices with SAMs is produced by a decrease of the capacitive hysteresis. In addition, with an appropriated functionalization, SAMs can increase photocurrent even when no electron selective contact (ESC) is present and a SAM is deposited just on top of the transparent conductive oxide. Appropriated functionalization of the fullerene derivative, as introducing -CN groups, can enhance cell performance and reduce hysteresis. This work paves the way for a future enhancement of PSCs by a tailored design of the fullerene molecules that could actuate as an ESC by themselves.

- 19 E. Martínez-Periñán, A. de Juan, Y. Pouillon, C. Schierl, V. Strauss, N. Martín, Á. Rubio, D. M. Guldi, E. Lorenzo, E. M. Pérez "The mechanical bond on carbon nanotubes: diameter-selective functionalization and effects on physical properties" *Nanoscale*, **2016**, *8*, 9254–9264, DOI:10.1039/c6nr01182a.

#### Abstract

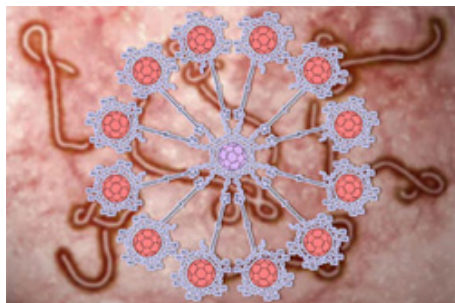


We describe the functionalization of SWNTs enriched in (6,5) chirality with electron donating macrocycles to yield rotaxane-type mechanically interlocked carbon nanotubes (MINTs). Investigations by means of electron microscopy and control experiments corroborated the interlocked nature of the MINTs. A comprehensive characterization of the MINTs through UV-vis-NIR, Raman, fluorescence, transient absorption spectroscopy, cyclic voltammetry, and chronoamperometry was carried out. Analyses of the spectroscopic data reveal that the

MINT-forming reaction proceeds with diameter selectivity, favoring functionalization of (6,5) SWNTs rather than larger (7,6) SWNTs. In the ground state, we found a lack of significant charge-transfer interactions between the electron donor exTTF and the SWNTs. Upon photoexcitation, efficient charge-transfer between the electron donating exTTF macrocycles and SWNTs was demonstrated. As a complement, we established significantly different charge-transfer rate constants and diffusion coefficients for MINTs and the supramolecular models, which confirms the fundamentally different type of interactions between exTTF and SWNTs in the presence or absence of the mechanical bond. Molecular mechanics and DFT calculations support the experimental findings.

- 20 A. Muñoz, D. Sigwalt, B. M. Illescas, J. Luczkowiak, L. Rodríguez-Pérez, I. Nierengarten, M. Holler, J. Remy, K. Buffet, S. P. Vincent, J. Rojo, R. Delgado, J. Nierengarten, N. Martín, "Synthesis of giant globular multivalent glycofullerenes as potent inhibitors in a model of Ebola virus infection" *Nature Chemistry*, **2016**, *8*, 50-57 - DOI: 10.1038/NCHEM.2387 *Chemical & Engineering News*, Volume 93, Number 45, November 16 - 2015 pg. 30

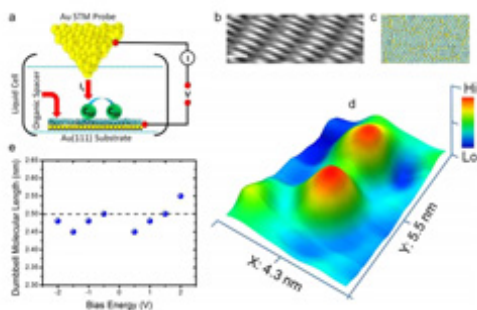
#### Abstract



The use of multivalent carbohydrate compounds to block cell-surface lectin receptors is a promising strategy to inhibit the entry of pathogens into cells and could lead to the discovery of novel antiviral agents. One of the main problems with this approach, however, is that it is difficult to make compounds of an adequate size and multivalency to mimic natural systems such as viruses. Hexakis adducts of [60]fullerene are useful building blocks in this regard because they maintain a globular shape at the same time as allowing control over the size and multivalency. Here we report water-soluble tridecafullerenes decorated with 120 peripheral carbohydrate subunits, so-called 'superballs', that can be synthesized efficiently from hexakis adducts of [60]fullerene in one step by using

copper-catalysed azide-alkyne cycloaddition click chemistry. Infection assays show that these superballs are potent inhibitors of cell infection by an artificial Ebola virus with half-maximum inhibitory concentrations in the subnanomolar range.

## Abstract



Predicting the electronic framework of an organic molecule under practical conditions is essential if the molecules are to be wired in a realistic circuit. This demands a clear description of the molecular energy levels and dynamics as it adapts to the feedback from its evolving chemical environment and the surface topology. Here, we address this issue by monitoring in real-time the structural stability and intrinsic molecular resonance states of fullerene (C<sub>60</sub>)-based hybrid molecules in the presence of the solvent. Energetic levels of C<sub>60</sub> hybrids are resolved by in situ scanning tunnelling spectroscopy with an energy resolution in the order of 0.1 eV at room-temperature. An ultra-thin organic spacer layer serves to limit

contact metal-molecule energy overlap. The measured molecular conductance gap spread is statistically benchmarked against first principles electronic structure calculations and used to quantify the diversity in electronic species within a standard population of molecules. These findings provide important progress towards understanding conduction mechanisms at a single-molecular level and in serving as useful guidelines for rational design of robust nanoscale devices based on functional organic molecules.

## PUBLICATION ANALYSIS

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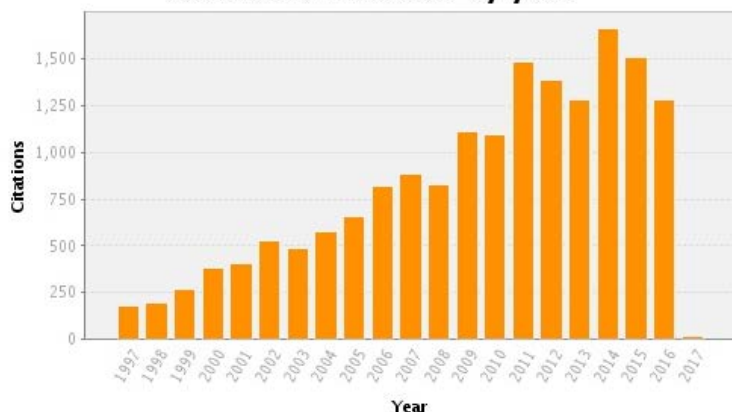
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## CONFERENCE PRESENTATIONS 2016

TÍTULO CONGRESO: 6th EuCheMs Chemistry Congress

TÍTULO CONFERENCIA: Current Challenges in Fullerenes Chemistry. “Honoring to Harry Kroto”

LUGAR : Seville (Spain) Fecha: September 11-15, 2016

ENTIDAD ORGANIZADORA: ANQUE

CARACTER: nacional x internacional (Congress Plenary)

TÍTULO CONGRESO: 6th EuCheMs Chemistry Congress

TÍTULO CONFERENCIA: Giant multivalent glycofullerenes as potent inhibitors of Ebola virus infection

LUGAR : Seville (Spain) Fecha: September 11-15, 2016

CARACTER: nacional x internacional (Plenary)

TÍTULO CONGRESO: POLYMAT Spotlights: Approaches to Aromatic 2D and Porous Polymers

TÍTULO CONFERENCIA: Glycofullerenes as active materials for the Ébola virus infection

LUGAR : San Sebastian (Spain) Fecha: June 21-24, 2016

ENTIDAD ORGANIZADORA: POLYMAT. University of the Basque Country

CARACTER: nacional x internacional (Plenary)

TÍTULO CONGRESO: 229th ECS Meeting

TÍTULO CONFERENCIA: Sugar/Nanocarbon Hybrids As Multivalent Inhibitors of Artificial Ebola Virus Infection

LUGAR : San Diego (USA) Fecha: May 29-June 3, 2016

ENTIDAD ORGANIZADORA: Electrochemical Society USA  
CARACTER: nacional      x internacional      (Invited)

TÍTULO CONGRESO: 229th ECS Meeting  
TÍTULO CONFERENCIA: Chirality Transfer from Graphene Quantum Dots  
LUGAR : San Diego (USA)      Fecha: May 29-June 3, 2016  
ENTIDAD ORGANIZADORA: Electrochemical Society USA  
CARACTER: nacional      x internacional      (Invited)

TÍTULO CONGRESO: 229th ECS Meeting  
TÍTULO CONFERENCIA: Fullerenes for Catalysis: C60 As Molecular Vector in Hydrogen Transfer Reactions  
LUGAR : San Diego (USA)      Fecha: May 29-June 3, 2016  
ENTIDAD ORGANIZADORA: Electrochemical Society USA  
CARACTER: nacional      x internacional      (Invited)

TÍTULO CONGRESO: International Winterschool on Electronic Properties of Novel Materials  
TÍTULO CONFERENCIA: New Organic Compounds for Photovoltaic Devices  
LUGAR : Kirschberg (Austria)      Fecha: February 13-20, 2016  
ENTIDAD ORGANIZADORA:  
CARACTER: nacional      x internacional      (Invited)

## MOMENTS TO SHARE - 2016

### Christmas Group (21/12/2016)





60th birthday Prof. Nazario Martín (25/03/2016)



