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doi.org/10.1002/cctc.202300667

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Shining Light on Carbon Dots: New Opportunities in Photocatalysis

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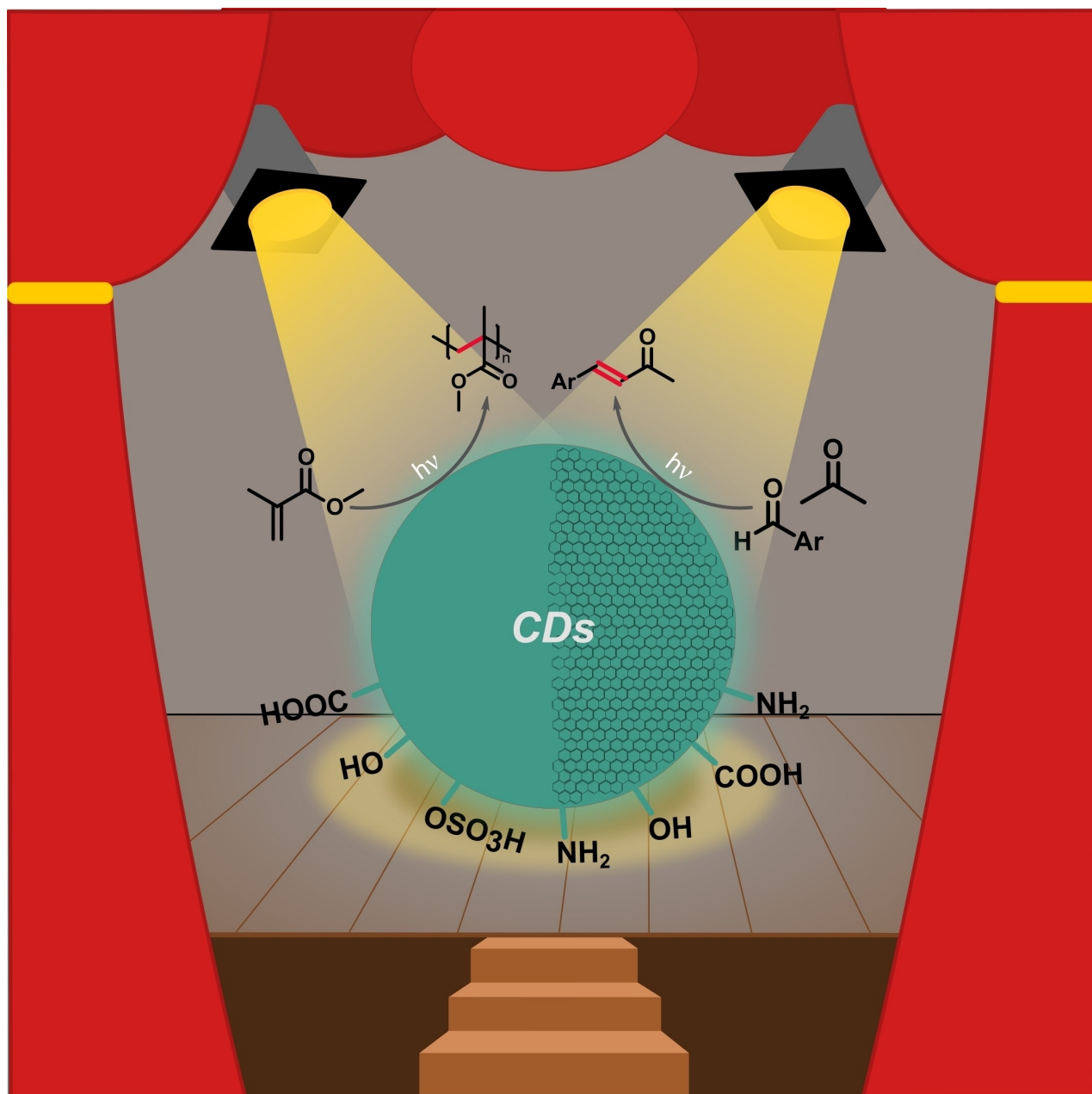
Photocatalysis is an emerging field that exploits light-absorbing catalysts to yield transformations not even achievable in the dark. Considering the drawbacks of metal-based photocatalysts, Carbon Dots (CDs) recently emerged as suitable green alternatives for different photocatalytic reactions. These carbon nanoparticles are easy to prepare, non-toxic and potentially recyclable. Moreover, CDs usually display core-shell structures which are highly tunable *via* synthetic and post-synthetic strategies. This Concept Article focuses on the recent advancements in the CD-based photocatalysis, highlighting the link between their superficial and core composition and their resulting photo-redox abilities, and giving future perspectives on their application in cutting-edge areas.

1. Introduction

Photocatalysis is one of the most fascinating fields of chemical research because it offers the possibility to efficiently convert solar energy into chemical energy.[1] To date, the most used photocatalysts are potentially toxic and expensive metal complexes that require multi-step syntheses and present low stability under certain reaction conditions.[2] Due to these drawbacks, there is mounting interest in the research for eco-friendly alternatives.[3] Recently, a novel class of low-cost carbon derivatives, Carbon Dots (CDs), have drawn increasing attention as a viable alternative to traditional photocatalysts.[4] CDs are quasi-spherical photoactive nanoparticles with dimensions below 10 nm that can be obtained from a variety of economic and readily available carbon precursors.[5] Generally, CDs are core-shell nanoparticles that consist of carbon cores surrounded by shells containing numerous heteroatom functionalities, such as carboxylic acids, alcohols and amines. Furthermore, CDs are green, inexpensive, non-toxic, potentially recyclable, and possess many interesting optical properties. Specifically, CDs show excitation wavelength-dependent emission, high photostability, great up-conversion ability, and excellent redox properties (Figure 1).[5–6] When CDs are exposed to a suitable light source, they may reach an excited state (CDs*). Subsequently, CDs* can be used to initiate operative photocatalytic cycles, as illustrated in Figure 2.[7] Once excited, CDs* can act either as photo-oxidants or photo-reductants, gaining an electron from donor (D; Equation 1) or giving an electron to an acceptor (A; Equation 2), respectively.[8]

$$CDs^* + D \rightarrow CDs^- + D^{\bullet+} \qquad (1)$$

$$CDs^* + A \rightarrow CDs^+ + A^{\bullet-} \qquad (2)$$

The resulting donor or acceptor radical ions may rapidly undergo consequent transformations yielding the final reaction products. Afterwards, the catalytic system may be restored to its ground state (namely CDs) through electron transfer phenomenon, such as a single electron transfer (SET) process with suitable electron acceptors or donors.[9] CDs can also act as photosensitizers promoting organic reactions through energy transfer mechanism.[10]
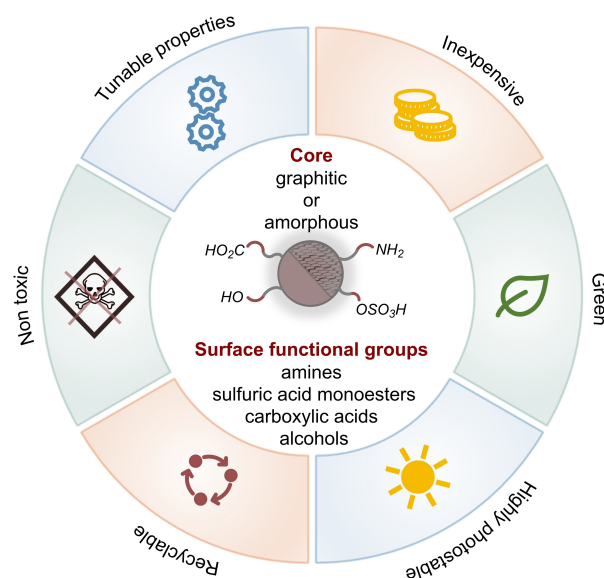
The choice of the starting materials and synthetic conditions employed in the CD synthesis is pivotal to obtain nanoparticles with the desired photocatalytic abilities. In fact, the chemical nature of the chosen precursors influences the structure and consequently the properties of the final materials.[5,11] As an example, by tuning the synthetic conditions, it is possible to obtain CDs with either graphitic or amorphous core. The degree of graphitization influences their excited-state lifetime, which is

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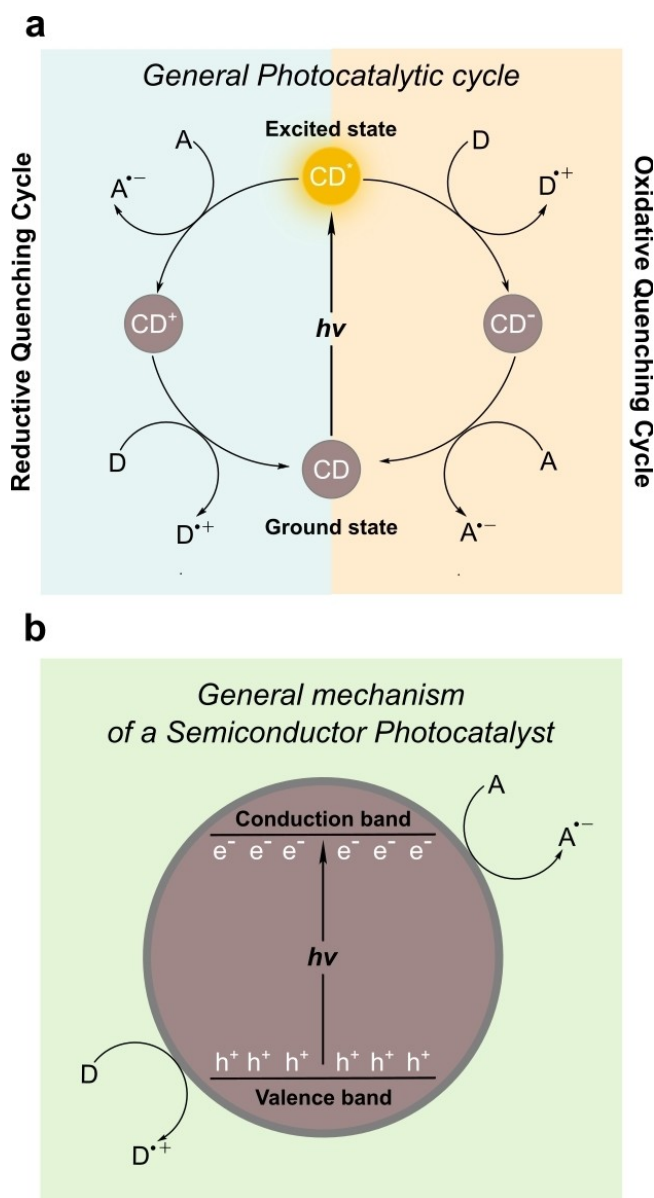   *https://gobbo-group.com/*
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Figure 1. Schematic representation of CDs' cores, surface functionalities, and main characteristics.

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2. Synthesis, Purification and Characterization of Photoactive CDs

Figure 2. a) CDs undergoing a general photocatalytic cycle; b) General photocatalytic mechanism of CDs acting as a semiconductor. D: Electron Donor; $D^{\bullet+}$: Radical Cation Donor; A: Electron Acceptor; $A^{\bullet-}$: Radical Anion Acceptor; e^-: Electron; h^+: Hole.

an important parameter for a photocatalyst since it is correlated to the efficacy of energy transfer phenomenon (*vide infra*).[8,12]
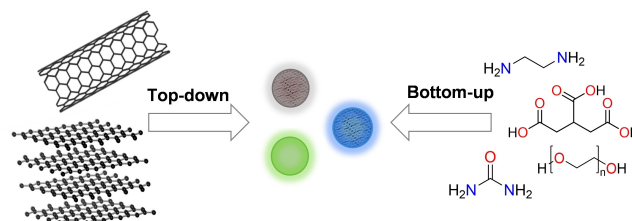
While many reviews are available on environmental and energy conversion applications,[13] this Concept Article aims instead to illustrate how CDs can be used to efficiently harness light irradiation and exploit it to photocatalyze organic transformations. The successful application of CDs as photocatalysts can be achieved by precisely choosing the starting materials, the synthetic strategy and extensively characterizing the final nanomaterials.

The synthetic approaches to prepare CDs are divided into top-down and bottom-up (Figure 3).[14] The former methods (e.g., arc discharge, laser ablation, electrochemical oxidation) employ bulk carbon-based materials and usually produce large quantities of CDs, even though they generally use harsh experimental conditions, such as high pressures and temperatures.[5,13a] Typically, the top-down methods cause more structural defects in the final material, leading to less interesting photoluminescence properties.[5] Focusing instead on the bottom-up methods, they use molecular precursors and normally exploit pyrolysis, microwave or solvothermal treatments to yield CDs.[15] The bottom-up syntheses are more versatile, require milder experimental conditions than the top-down routes, and usually lead to nanoparticles with narrower size distribution.[16] In most bottom-up syntheses, when the temperature is $> 300\,°C$, the formation of CDs with a graphitic core will be favored, whereas when the temperature is $< 300\,°C$, the CDs will have an amorphous structure.[12b] Since many different parameters can be modified in CD synthetic procedures, it should be noted that each of them (e.g., temperature, reaction time, precursors, stoichiometry) can influence the photocatalytic properties of the final CDs. For instance, CDs with different fluorescence emissions (i.e., blue to red) can be obtained by changing just one of the synthetic parameters.[13b]

After the synthesis, a fundamental step is the purification of the obtained material. Due to the high temperatures employed in the bottom-up routes, some side reactions are normally triggered. This leads to low molecular weight by-products alongside the desired CDs.[17] These side products must be removed in order to properly characterize the CDs and avoid the attribution of the by-products properties to the nanomaterial.[18] To achieve this, a number of purification strategies have been recently summarized by our research group.[17,19] For example, dialysis can be used to purify aqueous soluble CDs samples, whereas for organic soluble crudes precipitation should be used. In addition, CDs of different size can be further separated by gel electrophoresis or size-exclusion chromatography.[17,19]

A thorough characterization of the obtained nanomaterial is essential, both to assess the successful synthesis of CDs and to investigate their physical-chemical features. Firstly, transmission electron microscopy (TEM), scanning electron microscopy (SEM)

Figure 3. Schematic representation of the synthetic routes yielding CDs.

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and atomic force microscopy (AFM) are employed to examine CD size, size distribution, morphology, and to identify whether it presents a crystalline structure with a graphitic or amorphous core.[20] The identification of CD core type can also be corroborated using X-ray diffraction (XRD) or Raman spectroscopy.[5] Another particularly useful technique is X-ray photoelectron spectroscopy (XPS), which provides the elemental composition and information on the type of bonds present in the sample.[21] This technique can also be functional for the characterization of doped CDs as it allows for the quantification of the doping agent.

Another essential information to acquire is the type and number of CDs surface functionalities, that can influence CDs photocatalytic behavior. The appropriate choice of starting materials can lead to the formation of different groups on CD surface. These moieties can be exploited for post-synthetic modifications. In particular, the functionalization can improve the charge carrier separation prolonging the CD excited-state lifetime, as well as promote the interaction with specific substrates, since superficial moieties enable the absorption of a wide range of molecular species onto CD surface.[5,16, 22] For example, the evaluation of the type and number of acid/base sites can be performed by standard pH titration. The number of primary and secondary aliphatic and aromatic amines can instead be quantified by $^{19}F\{^1H\}$-NMR spectroscopy analysis using *p*-fluorocinnamaldehyde, as recently described by our research group,[11] whereas a Kaiser test (KT), a quantitative and selective colorimetric test, can be used for the determination of primary amines specifically.

Concerning the optical and electrochemical properties of CDs, there is increasing evidence that their emission as well as their photo-redox properties are influenced by (i) sp^2-conjugated core domains, (ii) surface functionalization, and (iii) heteroatom doping.[23] Concerning heteroatom doping, this can be used to improve charge carriers separation and prevent their recombination. Heteroatom doping can also slow down the kinetics of the redox reaction, contributing to trap the photo-generated electrons at surface sites.[22b] Usually, nitrogen-doped CDs show improved catalytic performances, as this atom has a similar size to the carbon atom and integrates into the π orbitals system, raising the highest occupied molecular orbital (HOMO) level and narrowing the bandgap.[24] Instead, boron is generally used as an electron-deficient dopant, because empty π orbitals of boron atoms produce a decrease in charge density.[22b,25] Moreover, some examples of multi-doped CDs have also been reported. These CDs usually show increased photoluminescence quantum yield (QY) and general better performances in the chosen photocatalytic application.[13b] Metal-doped CDs are also reported, mainly derived from organometallic complexes or metallic salts.[25–26] In addition, some organic solvents, such as formamide, can as well act as dopants, since during the synthesis is probable that they decompose and their by-products become incorporated into the CD structure.[27]

The optical properties of these nanoparticles are primarily investigated by UV-Vis spectroscopy. In general, most CDs present a main absorption band below 300 nm, attributed to the π–π* transitions of C=C bonds, and another tail band ranges between 300–400 nm, which is linked to the n-π* transitions of C=O bonds.[25] After light irradiation, the photo-generated carriers are first stored in the carbon backbone. The storage efficacy increases with the increasing degree of graphitization.[28] Moreover, the presence of a graphitic core leads to increased photostability and longer wavelength emission.[27] Consequently, the photoluminescence properties of CDs, namely fluorescence, phosphorescence, and up-converted photoluminescence, are attractive features for many applications, even though fluorescence is the most common and exploited light-emitting process.[29] Generally, CDs display excitation-dependent emission spectra and large Stokes shifts.[23] These characteristics are unusual among molecular fluorophores and semiconductor nanoparticles and they can be ascribed to the intrinsic heterogeneity of the sample.[23] To gather more information about the emission properties of CDs, it is useful to determine the photoluminescence quantum yield, i.e., the ratio between the number of emitted photons and the number of absorbed photons.[28] It has been shown that CDs synthesized at lower temperatures tend to possess higher photoluminescence QYs, whereas at higher temperatures there is a decrease in the number of surface functionalities, an increase in the graphitization degree, and a consequent lowering in the photoluminescence QYs (i.e., with an high graphitization degree, CDs present a longer excitation lifetime which can lead to non-radiative relaxation mechanisms).[20] Furthermore, QY can influence CD photostability as recently showed by Javed *et al.* In their minireview it is highlighted that CDs with low to moderate QY show good photostability, whereas CDs with high QY are more prone to photobleaching.[30] Another important parameter is the photoluminescence lifetime of the emissive state. This is the time available to the excited state to undergo energy and electron transfer processes or other quenching events.[23] In particular, CDs can undergo fluorescence quenching. This is a non-radiative process by which they transfer energy to another species called quencher (Q). The interaction between CDs and quencher can occur *via* collision or complexation.[31] The most commonly used method to evaluate fluorescence quenching is through the Stern-Volmer (SV) equation (Equation 3):

$$I_0/I = 1 + K_{SV} \times [Q] \qquad (3)$$

where I_0 is the luminescence intensity in the absence of the quencher, I is the intensity in the presence of the quencher Q, and K_{SV} is the quenching constant. K_{SV} is determined from the slope of the resulting curve and correlates the excited-state lifetime τ_0 to the quencher rate coefficient K_Q of CDs.[10a]

The electrochemical properties are primarily investigated by cyclic voltammetry (CV). CV is the most widely used technique to determine the energy gap between the lowest unoccupied molecular orbital (LUMO) and the HOMO of CDs. In particular, this energy gap needs to be equal to or lower than the photon source energy for the CDs to work as photocatalysts.[32] CV gives information about the ground state redox potentials of CDs, whereas it is interesting to investigate the excited state proper-

ties of the system as well. To study these latter ones, a commonly employed method is to use the Rehm-Weller equation (Equation 4), which correlates the excited-state potentials to the ground-state potentials.[33] In detail, the equation is:

$$E_P^* = E_P + E_{0,0} + w_r \qquad (4)$$

where E_P^* is the excited-state reduction or oxidation potential, E_P is the ground-state reduction or oxidation potential, $E_{0,0}$ is the energy gap between the zeroth vibrational levels of the ground and excited states, and w_r is the work function describing the charge generation and separation during the electron-transfer process.[34]

3. Carbon Dots in Photocatalysis

CDs are promising for important advancements in photocatalysis due to their non-toxicity, high cost-effectiveness, and potential recyclability. Particularly, recyclability is an essential feature for a green photocatalyst. Indeed, methodologies to recover CD-based photocatalysts (e.g., precipitation or extraction) are reported depending on their physical-chemical characteristics.[7b,16,35]

Even though the literature describes many examples of CD-based composites which are efficient photocatalytic systems for diverse applications,[13b] the aim of this section is to discuss the most recent and cited examples of metal-free CDs as sole photocatalysts, that is as a peculiar type of fully organic photocatalytic nanomaterial (Table 1). In fact, in the last few years progress has been made in engineering the surface functionalities of CDs by employing suitable starting materials. Consequently, metal-free and heteroatom-rich CDs, owning enhanced physical-chemical properties, have emerged as green multi-catalytic platforms for a wide range of photochemical transformations.[16]

One of the first examples of CDs employed as photocatalysts was reported by Kang and co-workers. This case study regarded the application of CDs-**A** in the aldol condensations between acetone **1a** and aromatic aldehydes **2** to give the α,β-unsaturated compounds **3** (Figure 4).[7b] The authors synthesized the water-soluble CDs-**A** through the electrochemical ablation of graphite rods. The crude was purified by filtration and centrifugation obtaining nanoparticles of about 5 nm with a graphitic core (lattice spacing 0.212 nm). The CDs-**A** contained many hydroxyl groups on their surface (0.75 mmol/L), capable of forming hydrogen bonds with the aromatic aldehydes **2**. Under light irradiation, CDs-**A** enhanced the electrophilicity of **2** thus accelerating the aldol condensation. For the light-driven transformations, the scope of the reaction provided various *para*-substituted aromatic α,β-unsaturated ketones **3**. These products were achieved in good to excellent yields (63–99%).

In 2015, MacFarlane and colleagues reported for the first time the use of sulfur-doped CDs as photo-switchable acid catalysts for ring-opening reactions.[36] This work opened a route towards the development of similar light-sensitive and recyclable green photocatalysts. In 2019, Sarma and co-workers

Table 1. Examples of CDs as sole photocatalysts for organic transformations. EDA: Ethylenediamine.

CDs	Precursors	Synthetic Method	CDs Characteristics	Application
A	Graphite rods	Electro-chemical ablation	Graphitic core Size: 5.40 ± 0.72 nm Absorption band max: 240 nm	Aldol condensations
B	Glucose, Sulfuric acid	MW Carbonization	Graphitic core Size: 2–6 nm Emission band max: 424 nm	C—C couplings
C	Arginine, EDA	MW Hydro-thermal method	Amorphous core Size: 2.47 ± 0.84 nm Emission band max: 365 nm	Perfluoro–alkylations
D	Sodium alginate, EDA	Hydro-thermal method	Amorphous core Size $<$ 10 nm Absorption band max $\approx$ 350 nm	Photo-polymerizations
E	Glucosamine hydrochloride, 1,3-diamino benzene	Hydro-thermal method	Amorphous core Size: 7.5 ± 1.5 nm Emission band max: 530 nm	C—C couplings
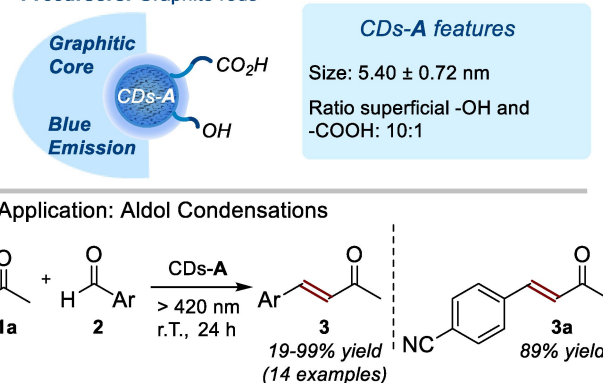
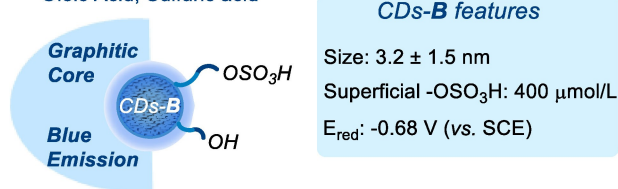**Figure 4.** Aldol condensation catalyzed by CDs-**A**.

synthesized sulfur-doped CDs-**B** that may act as dual photocatalysts, namely both as photo-oxidants and reductants, in dehydrogenative cross-coupling reactions.[35] In brief, the synthesis of CDs-**B** consisted of *(i)* microwave-assisted carbonization of glucose in oleic acid at 180 °C for 7 minutes, *(ii)* treatment of the material with fuming sulfuric acid to introduce sulfur-based functionalities. The resulting graphitic nanoparticles possessed carboxylic and sulfuric acid monoesters groups (0.5 mmol/L and 0.4 mmol/L, respectively) on their surface and had a size between 2–6 nm. Moreover, the authors determined the presence of 400 μmol/L of -OSO₃H groups on the surface of CDs-**B** by standard titration method. These recyclable CDs-**B**

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catalyzed the reaction between benzyl hydrocarbons **4** and different nucleophiles **5** (Figure 5). Specifically, CDs-**B**, owing to their reductive properties, promoted the formation of the hydroperoxyl intermediate of substrate **4**. Then, sulfur-based moieties of the catalysts facilitated the coupling between nucleophiles **5** and substrates **4**. This led to products **6** with good to excellent yields (68–99%).

In the same year, our group reported the use of amine-rich nitrogen-doped CDs for photochemical perfluoroalkylation of unsaturated organic compounds (Figure 6).[37] These nitrogen-doped CDs-**C** were produced by the hydrothermal treatment of arginine and ethylenediamine (EDA). After filtration and dialysis, CDs-**C** were obtained with an amorphous core. Because of their photo-reductive nature, the excited-state reduction potential of CDs-**C** was estimated by using Rehm-Weller equation, which
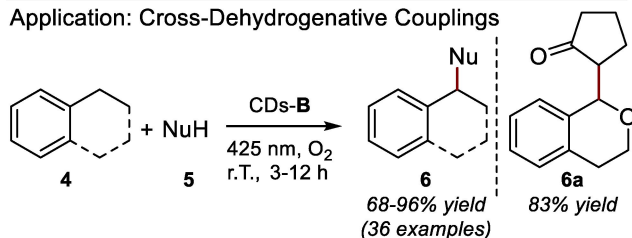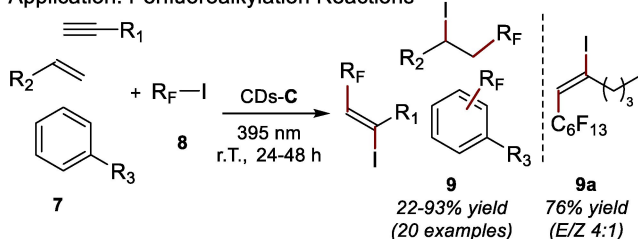
was found to be -2.2 V (vs. saturated calomel electrode, SCE). Therefore, upon irradiation at 395 nm CDs-**C** reach an electronically excited state acting as strong reducing agents. The excited CDs-**C** may undergo SET events with the radical source **8**, promoted *via* halogen-bond between the two species. This process generated the radical intermediates, which then reacts with a variety of organic substrates **7**. Finally, perfluorinated products **9** were delivered up to 90% yield.

In 2020, Kutahya *et al*. reported the first free radical and controlled photo-polymerization using CDs as biocompatible photocatalysts. Specifically, they described the synthesis of nitrogen-doped CDs-**D** to promote a controlled atom transfer radical photo-polymerization (photo-ATRP).[10b] In this work, CDs-**D** were synthesized *via* hydrothermal method from sodium alginate and EDA. These nanoparticles had an amorphous core, and XPS showed the presence of sodium carboxylate and amide moieties on the CDs-**D** surface (elemental composition: Na $\approx 8\%$, C $\approx 72\%$, N $\approx 5\%$, O $\approx 13\%$). These nanoparticles were employed to reduce a Cu(II) complex for the polymerization of methyl methacrylate (MMA) **10** in the presence of ethyl α-bromophenylacetate, as the alkyl halide source. This resulted in the formation of polymer **11** with narrow dispersity (Figure 7).

Since the field of dual photocatalysis has grown in interest in recent years,[39] the application of CDs in combination with a metal complex led to mild light-driven dual-catalytic processes. An important example in this research area is the work of Pieber and colleagues. They employed amine-rich CDs-**E** to photocatalyze different carbon-heteroatom cross-coupling reactions (C−O, C−S, C−N) in the presence of Ni(II) complexes.[40] Through hydrothermal method, glucosamine hydrochloride and 1,3-diaminobenzene were allowed to react to yield CDs-**E**. The obtained amorphous green-emitting CDs-**E** contained several amino functional groups (9303 µmol/g by KT). These superficial groups promoted the interaction between the CDs-**E** and the Ni complexes, overcoming the short lifetime limitation of the nanomaterial.[40] The cross-coupling reaction then proceeded *via* photoreduction of the Ni(II) complex in the presence of CDs-**E**. An example of this class of reactions is reported in Figure 8,

Carbon Dots as Photoxidative and Acidic Catalysts

Precursors: Glucose, Oleic Acid, Sulfuric acid

Graphitic Core
Blue Emission
CDs-**B**
OSO₃H
OH

CDs-B features
Size: 3.2 ± 1.5 nm
Superficial -OSO₃H: 400 µmol/L
E_{red}: -0.68 V (*vs*. SCE)

Application: Cross-Dehydrogenative Couplings

CDs-**B**
425 nm, O₂
r.T., 3-12 h

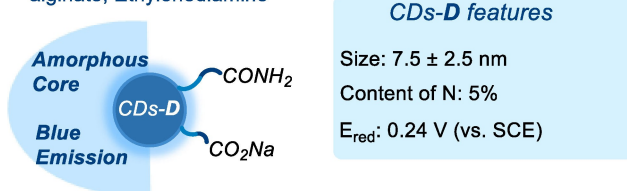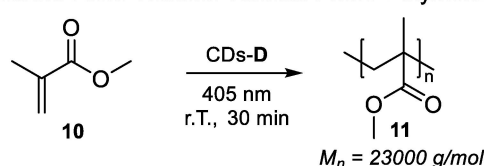**4** + NuH **5** → **6** 68-96% yield (36 examples) | **6a** 83% yield

Figure 5. Cross-dehydrogenative coupling catalyzed by CDs-**B**. Nu: Nucleophile.

Carbon Dots as Photoreductive Catalysts

Precursors: Arginine, Ethylenediamine

Amorphous Core
Blue Emission
CDs-**C**
NH₂
NH₂

CDs-C features
Size: 2.5 ± 0.8 nm
Superficial -NH₂: 1350 µmol/L
E_{red}: -2.2 V (vs. SCE)
QY: 0.17 [a]

Application: Perfluoroalkylation Reactions

CDs-**C**
395 nm
r.T., 24-48 h

7 + R_F−I **8** → **9** 22-93% yield (20 examples) | **9a** 76% yield (E/Z 4:1)

Figure 6. Perfluoroalkylation reaction catalyzed by CDs-**C**. R_F: Perfluorinated radical source. [a] QY of CD-**C** with quinine sulphate as standard reported in.[38]

Carbon Dots as Photoreductive Catalysts

Precursors: Sodium alginate, Ethylenediamine

Amorphous Core
Blue Emission
CDs-**D**
CONH₂
CO₂Na

CDs-D features
Size: 7.5 ± 2.5 nm
Content of N: 5%
E_{red}: 0.24 V (vs. SCE)

Application: Atom Transfer Radical Photo-Polymerization

CDs-**D**
405 nm
r.T., 30 min

10 → **11** M_n = 23000 g/mol

Figure 7. Photo-ATPR catalyzed by CDs-**D**. M_n: Average number molar mass.

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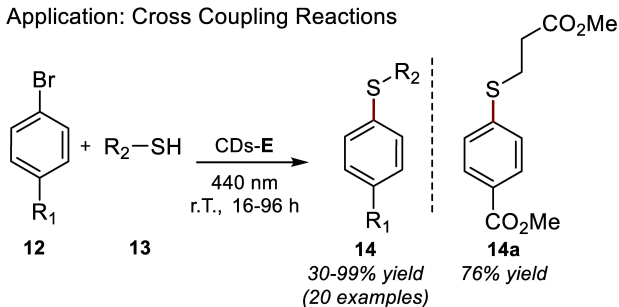**Chemistry Europe**
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Carbon Dots as Photoreductive Catalyst

Precursors: *Glucosamine hydrochloride, 1,3-diaminobenzene*
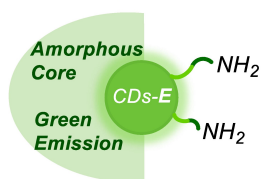
CDs-E features
Size: 7.5 ± 1.5 nm
Superficial -NH_2: 9303 umol/L
E_{red}: -0.26 V (vs. SCE)
QY: 0.33 [a]

Application: Cross Coupling Reactions

Figure 8. Cross coupling reaction catalyzed by CD-E. [a] QY of CD-E with fluorescein as standard reported in.[41]

which shows the reaction between aryl halides **12** with thiols **13** under blue light irradiation mediated by Ni(dtbbpy)Br$_2$ (dtbbpy = 4,4′-di-tertbutyl-2,2′-dipyridyl) as metal complex. In the optimized conditions, the coupling products **14** were produced up to quantitative yields. This work highlights how the punctual choice of the starting materials can lead to a CD-based photocatalyst with excellent photo-redox properties.

4. Summary and Outlook

In the last decade, CDs have been chosen as versatile candidates to act as photocatalysts due to their ease of synthesis and functionalization, and their unique photo-redox properties. This Concept Article focuses on the recent achievements in the application of CD-based catalysts in photocatalysis, by describing some representative examples. While encouraging results on the use of CDs in photocatalysis have been achieved so far, the main challenge still remains gaining control over the synthesis-structure-properties relationship. For instance, a punctual comprehension of the synthetic mechanism yielding CDs should be achieved, and more rigorous methodologies to characterize the CDs properties must be developed. Filling this gap not only will allow for the synthesis of CDs with specific photocatalytic properties, but could also lead to the design and synthesis of novel nanomaterials for specific applications such as photodegradation of pollutants,[42] stereo-controlled photocatalysis,[16,25] and enzyme-mimicry.[43] Among these, enzyme-mimicking CDs, known as nanozymes, have recently gained increasing interest.[43] To date, only a few examples of photoactive CD-based nanozymes have been reported.[44,45] Remarkably, the development of new light-active

nanozymes could result in *in vivo* clinical applications, including bio-sensing, bio-imaging, and drug delivery.[46]

To conclude, we believe that a comprehensive understanding of the chemical nature and the photo-reactivity of CDs will help to obtain high-performing catalytic platforms to convert light energy into chemical green energy exploitable in everyday life.

Acknowledgements

Conflict of Interests

The authors declare no conflict of interest.

Keywords: Carbon dots · Green chemistry · Nanostructures · Photocatalysis · Synthetic methods

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