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PAPER

Efficient and selective photodimerization of 2-naphthalenecarbonitrile mediated by cucurbit[8]uril in an aqueous solution†

Bin Chen, Su-Fang Cheng, Gui-Hong Liao, Xin-Wei Li, Li-Ping Zhang, Chen-Ho Tung and Li-Zhu Wu*

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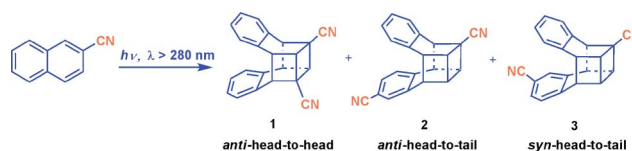
The photodimerization of 2-naphthalenecarbonitrile (**2-NpCN**) in a cucurbit[8]uril (CB[8]) aqueous solution was investigated. Spectroscopic analysis and product distribution reveal that the use of CB[8] as a host inverted the product selectivity from photodimers **2** and **3** in cyclohexane to photodimer **1** in a CB[8] aqueous solution with a large rate acceleration under ambient temperature and pressure.

Optimizing the selectivity of photochemical reactions is one of the most important topics of current research since photochemical reactions generally tend to give more than one product.^{1,2} During the past few decades, many elegant and efficient strategies have been designed towards this goal. The use of supramolecular systems for selective photochemical reactions turns out to be one of the successful approaches because the interactions of substrates with supramolecular systems may alter the photochemical behaviors of the confined substrates, and thus may direct the photochemical reaction to the desired product(s).¹⁻⁷ It is known that photochemical reactions in isotropic solutions and supramolecular systems often lead to different product distributions, or in some cases totally different products.⁵⁻⁷

The cucurbit[*n*]urils (CB[*n*]), a family of pumpkin-like macrocyclic hosts with five, six, seven, eight or ten methylene-bridged glycoluril units, respectively,⁸⁻¹³ feature a hydrophobic cavity and polar carbonyl groups surrounding the portals. The varying cavity and portal size of CB[*n*] are known to show remarkable affinity and selectivity towards hydrophobic or/and positively charged guest molecules in aqueous solutions.¹¹⁻¹³ CB[8] is particularly attractive because it is able to accommodate two aromatic molecules within the cavity, forming a 1 : 2 complex with desired orientations. Kim *et al.* found that the [2 + 2] photodimerization of diaminostilbene dihydrochloride proceeded with a large rate acceleration and a high stereoselectivity inside the cavity of CB[8] in aqueous solution.¹⁴ Ramamurthy *et al.* reported that irradiation of *trans*-1,2-bis(*n*-pyridyl)ethylene dihydrochlorides and *trans*-*n*-stilbazole hydrochlorides, either in CB[8] aqueous media or in the solid state, resulted in high yields of the *syn* photodimer.^{15,16} Sivaguru *et al.* reported that the dimerization of coumarins could take place with a catalytic amount of CB[8] in water, giving rise to the *syn* dimer as the major product.¹⁷⁻¹⁹ Gromov *et al.* showed the stereospecific

[2 + 2] autophotocycloaddition of styryl dyes in CB[8] aqueous solution.²⁰ Inoue and Kim *et al.* investigated the stereoselective photodimerization of 2-anthracene carboxylic acid and α -cyclodextrin-appended anthracene mediated by CB[8].²¹ We have made use of CB[8] as a microreactor to encapsulate 2-naphthalene-labeled poly(ethylene glycol) and alkyl 2-naphtholate, leading to the exclusive formation of photodimers in aqueous solution upon irradiation with light.^{22,23}

In the present work, we wish to report on the photodimerization of 2-naphthalenecarbonitrile (**2-NpCN**) mediated by CB[8] in aqueous solution. **2-NpCN** was reported to form a photodimer in 1971. On the basis of the MS, IR and ¹H NMR spectra, Zweig assigned the 1,4-bridged structure to photodimers.²⁴ Later on, Albini observed that the situation was more complicated.²⁵ In 2008, we re-investigated this reaction and demonstrated that the irradiation of **2-NpCN** in solution with light $\lambda > 280$ nm results in the formation of three rigid cubane-like photodimers, *anti*-head-to-head **1**, *anti*-head-to-tail **2** and *syn*-head-to-tail **3** (Scheme 1).²⁶ Despite of this, the conversion of **2-NpCN** is only 23% after 17 h irradiation in an acetonitrile solution. Moreover, the ratio of **1** to **2** and **3** varies with the solvent used, and **2** and **3** are always present throughout the irradiation. Evidently, the quantum yield and selectivity of the photodimerization leave much to be desired. Herein, we study the CB[8]-mediated photodimerization of **2-NpCN** in aqueous solution. CB[8] was expected to bring two **2-NpCN**s into close proximity with a desirable orientation to facilitate photodimerization with a remarkable selectivity and efficiency.

Scheme 1 Photodimerization of **2-NpCN**.

The inclusion of **2-NpCN** within the cavity of CB[8] was achieved by the sonication of **2-NpCN** (20 μ mol) with CB[8]

Key Laboratory of Photochemical Conversion and Optoelectronic Materials, Technical Institute of Physics and Chemistry, The Chinese Academy of Sciences, Beijing, 100190, China. E-mail: lzhu@mail.ipc.ac.cn; Fax: +86 10-82543580; Tel: +86 10-82543580

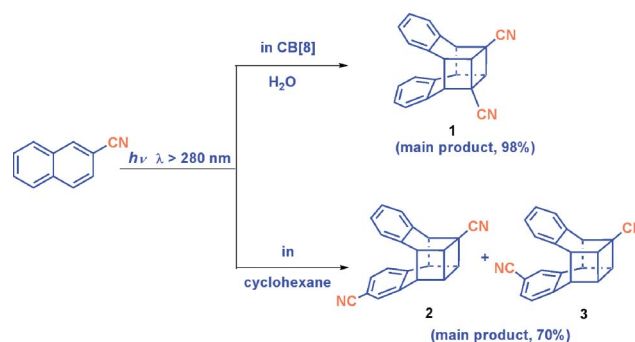
† This article is published as part of a themed issue in honour of Yoshihisa Inoue's research accomplishments on the occasion of his 60th birthday.

(10 μmol) in an aqueous solution for 3 h at 50 $^{\circ}\text{C}$. Filtration through a 0.2 μm membrane then afforded a saturated aqueous solution of the inclusion complex. Generally, the irradiation was carried out in a Pyrex reactor purged with nitrogen at room temperature. A 500 W high-pressure mercury lamp was used as the light source. A quartz jacket with water circulation was used to cool the lamp. A light filter was placed outside the quartz jacket to cut off light below 280 nm so as to protect the photodimer from decomposition. The photodimerization process was monitored by UV-vis absorption spectroscopy. Along with the irradiation, the absorbance of the typical bands for **2-NpCN** quickly decreased. The irradiation was continued until the absorbance no longer changed. Following extraction with chloroform, product analysis was performed by high performance liquid chromatography (HPLC) and NMR spectroscopy. As we described previously,²² photodimers **2** and **3** are too similar to be distinguished using an achiral column. Therefore, an achiral Intersil ODS-3 column was used first to separate photodimer **1** from the mixture of photodimers **2** and **3** (10 \times 250 mm; eluent: acetonitrile : water = 6 : 4; room temperature; flow rate 2.0 mL min^{-1} ; UV detection at 220 nm), and then photodimers **2** and **3** were distinguished by a chiral IA column (Daicel Chemical Industries, Ltd., 10 \times 250 mm; eluent: n-hexane : ethanol = 5 : 5; room temperature; flow rate 1.0 mL min^{-1} ; UV detection at 254 nm). It is significant that the irradiation of **2-NpCN** in the presence of CB[8] is efficient and selective. Generally after 8 h of irradiation, the conversion of **2-NpCN** approached 73% and the yield of the cubane-like photodimers was up to 98% for **1**, 1% for **2** and 1% for **3**, based on the consumption of the starting material (Table 1). It is evident that the yield of photodimer **1** is much improved from 31% in cyclohexane to 98% in an aqueous solution of CB[8], while that of **2** and **3** decreases dramatically. By way of contrast, the irradiation of a **2-NpCN** aqueous solution does not result in any photodimer formation. Compared with the conversion of **2-NpCN** in a cyclohexane solution, which is only 13% after 17 h irradiation, CB[8] clearly templates the photodimerization of **2-NpCN** with a large rate acceleration in aqueous solution. Strikingly, the use of CB[8] as a host inverts the product selectivity from **2** and **3** in cyclohexane to **1** in CB[8] aqueous solution (Scheme 2). The difference in the ratio of **1** to **2** and **3** (*ca.* 49 : 1 in CB[8] aqueous solution but 1 : 2 in cyclohexane) indicates that irradiation of the **2-NpCN** in CB[8] aqueous solution results in photodimerization with a remarkable regioselectivity (*anti*-head-to-head **1** as the main product).

The interaction of CB[8] with **2-NpCN** in aqueous solution was confirmed by UV-vis, fluorescence and ^1H NMR analysis. With the addition of CB[8] into an aqueous solution of **2-NpCN**

Table 1 Conversion and yield for the photodimerization of **2-NpCN** in the absence and presence of CB[8] at room temperature, respectively

Entry	Medium	Irradiation time/h	Conversion (%)	Photodimer yield (%)		
				1	2	3
1	Cyclohexane	17	13	31	38	31
2	Acetonitrile	17	23	85	8	7
3	H ₂ O	8	0	0	0	0
4	CB[8]/H ₂ O	8	73	98	~1	~1



Scheme 2 Photodimerization of **2-NpCN**.

(40 μM), the solubility decreased, therefore causing a decrease in the absorption (Fig. 1). The fluorescent changes of **2-NpCN** toward CB[8] are pronounced. In the absence of CB[8], **2-NpCN** shows the monomer emission of naphthalene derivatives with a maximum at 354 nm. The progressive addition of CB[8] to the **2-NpCN** aqueous solution led to a gradual growth with a maximum at 410 nm, which is typical excimer emission of a 2-naphthalene chromophore (Fig. 2a).²³ The excitation spectra for the monomer and excimer emissions, monitored at 354 and 410 nm, respectively, are similar. However, the spectrum for the latter is slightly but evidently red-shifted (Fig. 2b), indicating that the excimer originates from the pair of naphthalene groups that exist prior to excitation.

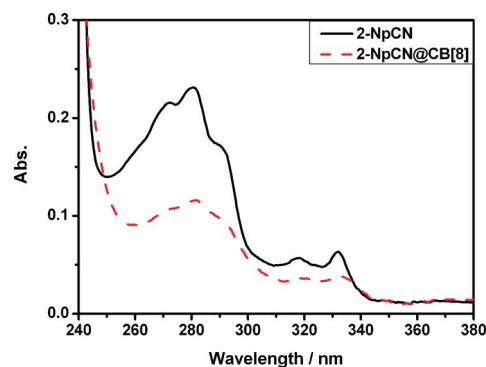


Fig. 1 UV-vis spectra of **2-NpCN** in the absence (black) and presence of 0.5 equiv. CB[8] (red) in H₂O at room temperature (the concentration of **2-NpCN** is 40 μM , 1 cm quartz cell).

The ^1H NMR spectra of **2-NpCN** in the absence and presence of CB[8] are shown in Fig. 3. The encapsulation-induced upfield chemical shifts for the aromatic naphthoate resonances are consistent with the inclusion of the naphthalene groups in the shielding hydrophobic cavity. Because no separate peaks for the free and the bound guests are observed, the rates of the encapsulating process are fast on the ^1H NMR spectroscopy time scale.

From these results, it is clear that the environment of **2-NpCN** during irradiation is a major factor in both the efficiency and selectivity of the photodimerization. The high regioselectivity of the CB[8]-mediated photodimerization of **2-NpCN** is illustrated in Scheme 3. The cavity of CB[8] accommodates two molecules of **2-NpCN** and aligns them in a geometry that is favorable for the occurrence of photodimerization. As described above, the irradiation of **2-NpCN** in solution results in the formation of three rigid cubane-like photodimers, *anti*-head-to-head **1**, *anti*-head-to-tail **2** and *syn*-head-to-tail **3**. Herein, there are three kinds

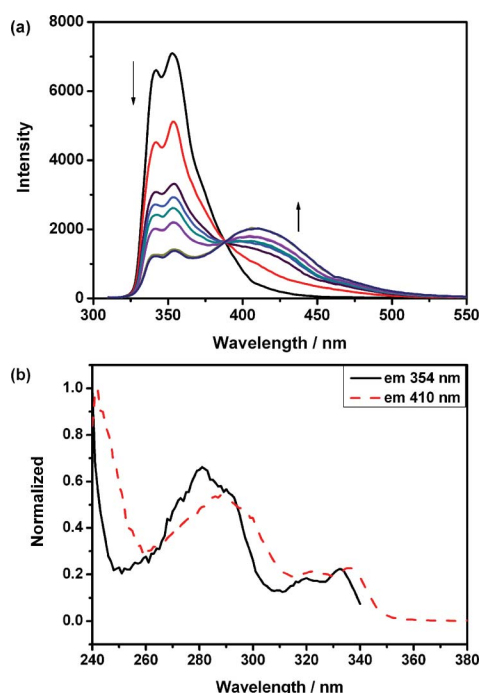


Fig. 2 (a) Fluorescence spectra of **2-NpCN** (5 μM) with various concentrations of CB[8] in H_2O at room temperature (0, 0.2, 0.4, 0.5, 0.6, 0.8, 1.0, 2.0 and 2.5 equiv. of CB[8], $\lambda_{\text{ex}} = 296 \text{ nm}$). (b) Excitation spectra of **2-NpCN** in the presence of CB[8] monitored at $\lambda_{\text{em}} = 354 \text{ nm}$ (—) and 410 nm (---), respectively.

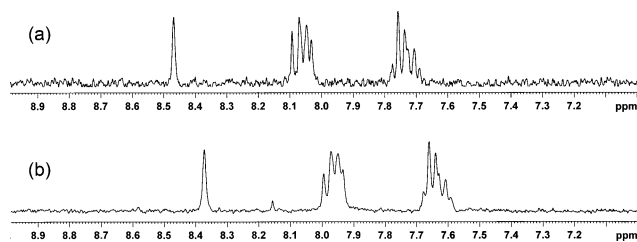
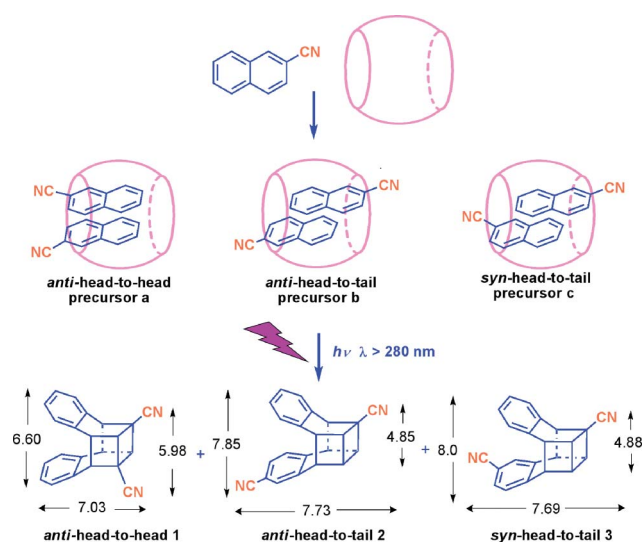


Fig. 3 ^1H NMR spectra of **2-NpCN** (a) in the absence and (b) in the presence of CB[8] in D_2O .

of isomers, precursors **a**, **b** and **c** (Scheme 3), orientating the two carbonitrile groups to the portal of the barrel-shaped CB[8] suitable for the photodimerization. On the other hand, the size of **1** ($5.98 \times 6.60 \times 7.03 \text{ \AA}^3$) from its crystal structure suggests that it could tightly fit in the cavity of CB[8] ($6.90 \times 6.90 \times 9.10 \text{ \AA}^3$), while that of **2** ($4.85 \times 7.85 \times 7.73 \text{ \AA}^3$) and **3** ($4.88 \times 8.00 \times 7.69 \text{ \AA}^3$) are too large to be accommodated into the 6.90 \AA cavity of CB[8] for their formation, which appears to play a crucial role in determining the selectivity of the photodimerization.

In summary, we have demonstrated that CB[8] can mediate the photodimerization of **2-NpCN** in aqueous solution. Spectroscopic characterization and product distribution reveal that CB[8] encapsulates two molecules of **2-NpCN** and aligns them to favour cubane-like photodimer **1** formation with a large rate acceleration under ambient temperature and pressure, whereas no photodimers could be detected in a host-free aqueous solution.



Scheme 3 CB[8]-mediated photodimerization of **2-NpCN**.

Acknowledgements

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