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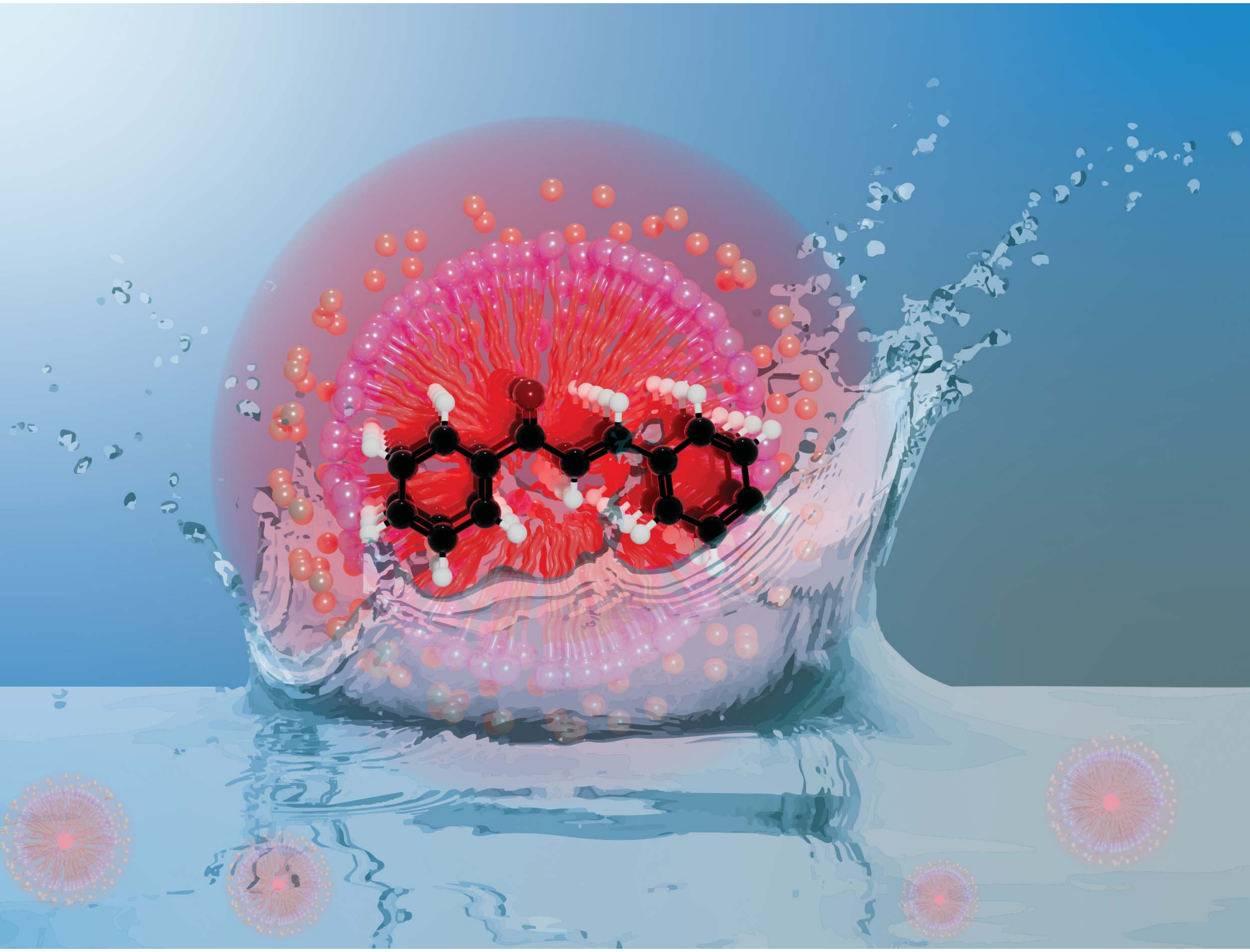

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Long-lived room temperature phosphorescence in aqueous micellar systems: application in cyclobutane synthesis

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The micelle-induced excimer formation of simple chalcone is investigated. An extraordinarily long-lived NIR-emission displaying an average lifetime of 19.22 μs was recorded. The emission was induced by the unique micro-environment provided by the micelles and could not be observed simply by increasing the concentration in organic solvent. Using visible light overlapping with the recorded excitation spectrum, an intermolecular [2 + 2] photocycloaddition could be promoted, illustrating the synthetic potential of the concept. The incorporation of chalcone into the micelles displayed no apparent change in size or shape of the micelles as illustrated by TEM-images and diffusion NMR studies. In addition to providing a unique and essential environment for the present study, aqueous micelles represent an alternative to organic solvents for sustainable synthesis.

Introduction

Aggregation induced emission (AIE) emerged at the beginning of this century and enabled various luminescence applications, but synthetic applications beyond simple energy transfer, for example to oxygen, are still lacking.[1–7] In AIE restriction of intramolecular motion (rotation and vibration) within the aggregates inhibits non-radiative decay pathways, enabling emissive behavior.[8,9] In the last decades many applications in materials and bioimaging have been developed.[10–12] It Is noteworthy that aggregation typically is restricted to larger molecules designed to aggregate, while small molecules relevant for synthesis rarely aggregate. Favored aggregation of AIE active luminogens (AIEgens) in hosts can lead to emissive behavior already at low concentrations, opening up new application avenues.[13] Examples of suitable hosts include cyclodextrins,[14,15] cucurbiturils,[16] biomolecules,[17] polymeric host structures[18,19] and more.[20–22] Micelles have also been studied as suitable hosts to enable AIE and luminescent micelles have been disclosed.[23] The formation of luminescent micelles often proceeds *via* aggregation of amphiphiles decorated with luminogens. These designed amphiphiles show good performance in biological systems due to their emission properties (Fig. 1a, left).[24–28]

Another approach is based on micellar encapsulation of AIEgens (Fig. 1a, right). Hydrophobic molecules are preferably located in the micellar core and the resulting reduction of dimensionality and effective solvent can induce AIE.[29,30] This concept has previously been explored to investigate micelle formation at very low concentrations spectroscopically, because emission is only visible upon micelle formation.[31–33] Close packing has been taken advantage of in the field of solid state photochemistry.[34,35] Through close packing, reaction media based on micelles may have the potential to provide alternatives to solid state type photochemistry by creating similar conditions in solution. To the best of our knowledge, current applications of micellar AIE in the context of synthesis is limited to a single example of reductive dehalogenation where a mechanism based on FRET is proposed to start the photocatalytic reaction.[36]

Inspired by the unique ability of micelles to induce AIE, we set out to explore the synthetic potential of micelle-induced close packing of small molecules. Simple *E*-chalcone **1a** was selected as a suitable small molecule as it contains π-stacking aromatic groups. Additionally, chalcones are biologically relevant[37–40] and have previously been shown to engage in photochemical reactions.[35,41–43] We aimed to induce excimer formation of simple **1a** in a micellar reaction medium and initiate an intermolecular [2 + 2] photocycloaddition reaction using visible light (Fig. 1b). An additional advantage to the present concept is the usage of water as the solvent. It has become increasingly urgent to develop more sustainable methods and, in this context, reaction media based on micelles are interesting as an alternative to organic solvents for the pharmaceutical industry.[44,45]

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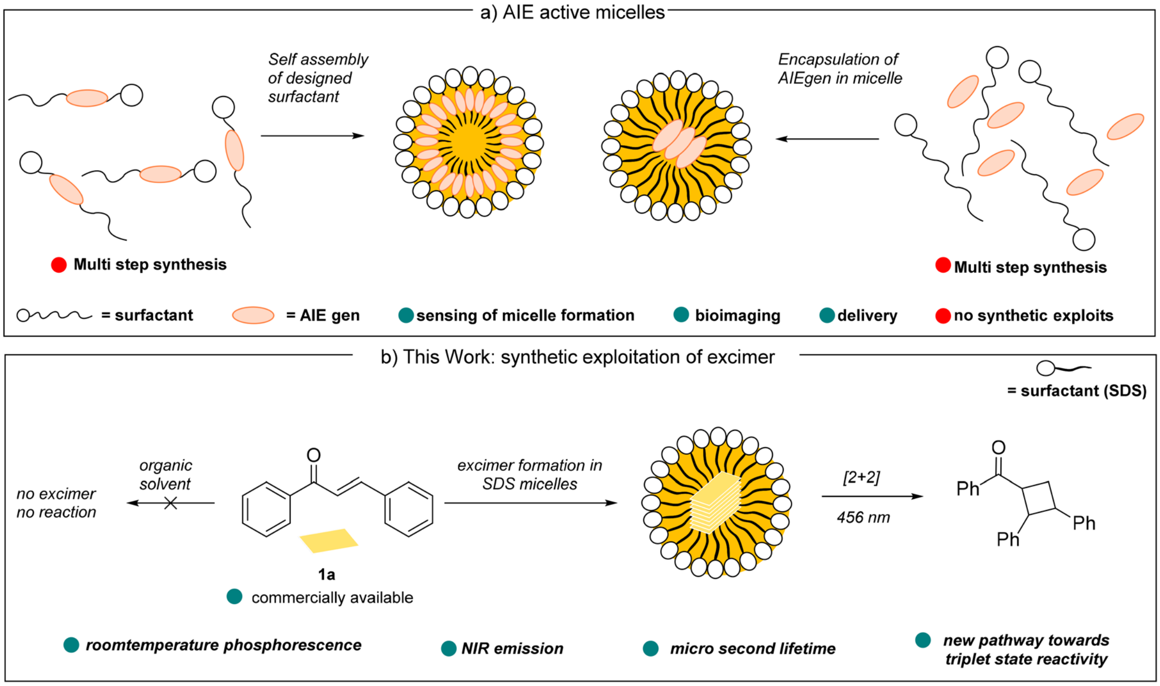


Fig. 1 a) AIE active micelles, designed luminogen surfactants (left) and encapsulation of AIEgen (right). (b) This work on micelle-induced close packing of *E*-chalcone **1a** to induce unique emission behavior and photochemical [2 + 2] photocycloadditions.

Results and discussion

The spectroscopic properties of **1a** were investigated in Sodium dodecylsulfate (SDS)-micelles, due to the lack of solubility in water (see SI), and compared to organic solvent. We initially chose, SDS as a cheap and commercially available surfactant which has previously been applied in spectroscopy and micellar catalysis.[46–50] The formation of SDS micelles loaded with **1a** was achieved by simply stirring the surfactant and **1a** in water. Unexpectedly, in micellar solutions at high concentrations of **1a** in SDS a broad emission band occurred in the near infrared (NIR) region (Fig. 2a). In particular, by increasing the concentration of **1a** from 0.35 mM to 35 mM in SDS solutions (4% by weight in water) a broad emission with maximum around 700 nm upon excitation in the range between 400 and 470 nm arises (Fig. 2a and b). The applied SDS concentration (0.14 M) is well above the critical micelle concentration (CMC = 0.8 m M),[51] and has been used for synthetic application by our group.[47,48] Interestingly, the emission is strictly dependent on the excitation wavelength, and it is not detectable upon irradiation in the UV-region (Fig. S2), which is consistent with the excitation spectrum (Fig. 2b). Comparatively, in isotropic solvents like dichloromethane (DCM) and MeOH no NIR emission is detected (Fig. S3). The absorption properties do not change significantly in different solvents (Fig. 2c). The UV/vis absorption maxima are around 310–315 nm, with a slight red shift in MeOH, hexafluoroisopropanol (HFIP) and also in the micellar medium. No additional absorption bands can be observed around 400–470 nm even at high concentration (Fig. S4). These results indicate that the NIR emission in micelles is highly concentration dependent and only forms upon close packing induced by micelles. To get further insights into the origin of the emission band, luminescent lifetime experiments were carried out. Fig. 3d reports the recorded radiative decay with an average lifetime of 19.2 µs, indicating the phosphorescence as radiative mechanism, thus suggesting a triplet character of the excited state. The multiexponential decay observed can be rationalized by considering multiple species in the solution.

As the emission only appears at high concentrations, we investigated aggregate formation of **1a** as a potential origin for the long lived NIR emission. To assess this proposed excimer state, a large amount of **1a** was dissolved in tetrahydrofuran (THF) and injected in water under sonication, inducing the formation of nanoparticles. The formed nanoparticles show the same emission as the micellar solution of **1a** (Fig. 2e) supporting the hypothesis of excimer formations in the micellar medium. Noteworthy, the NIR emission does not occur in organic solvents by increasing concentration of **1a**, nor in the solid state (See S5). Consequently, the unique microenvironment formed by the micelles appears critical in reaching the emissive state in solution. The micelles are a sustainable alternative to organic solvents and are used to encapsulate the organic molecules in water for later synthetic application.[52]

The data show that the emission is strictly connected to the dense packing conditions obtained in the micellar environment, similar to the condensed phase.[53] Photophysical studies on the investigated *E*-chalcone **1a** have been previously reported in literature,[54,55] but radiative transitions generating emission bands in the NIR region have not been observed prior to this study. Furthermore, literature data show that chalcones exhibit a triplet-state biradical with twisted structure with long triplet

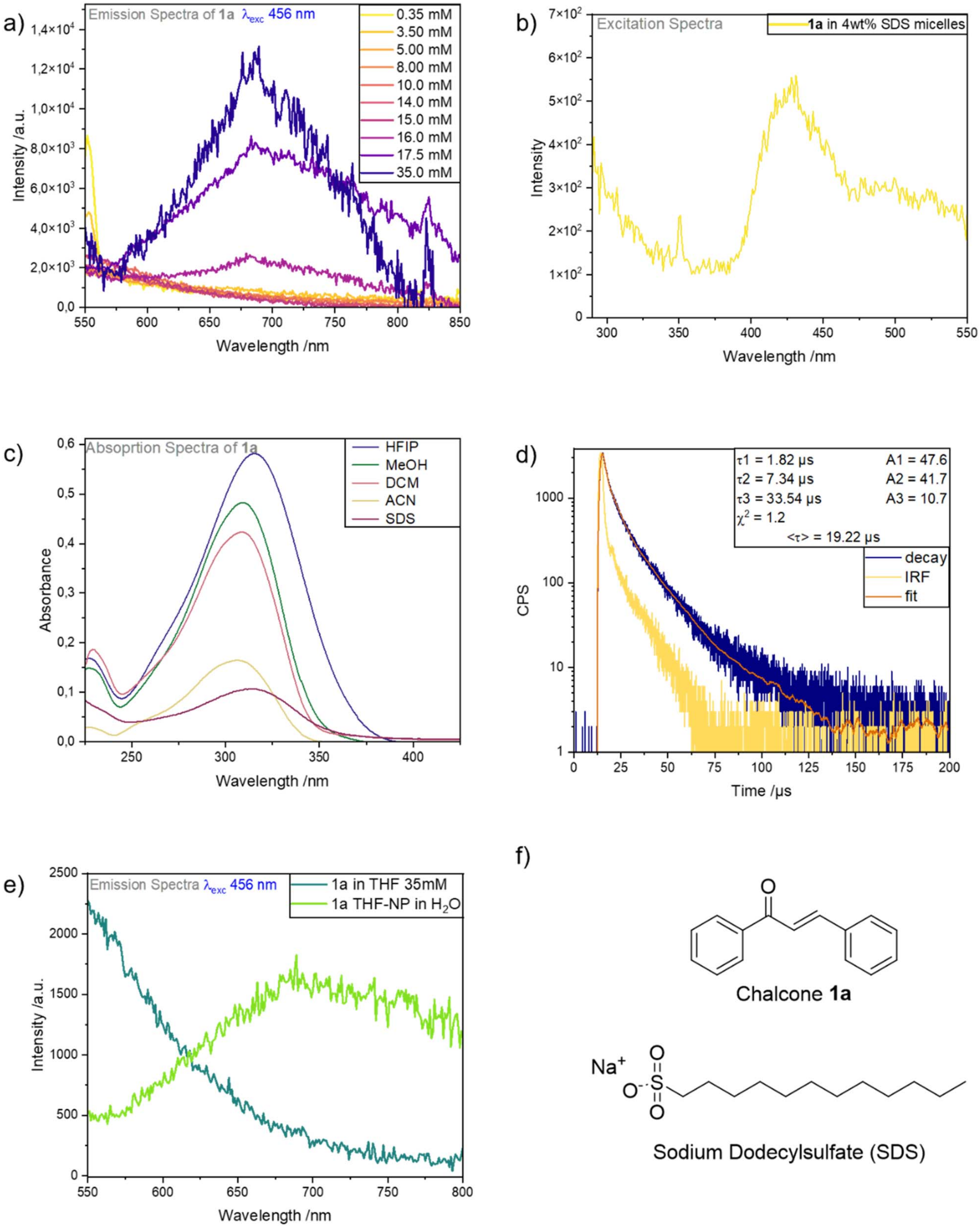


Fig. 2 (a) Emission spectra of **1a** in different concentrations in 4 wt% SDS solution. The band at 550 nm is the Raman signal of the solvent. 456 nm was chosen to exclude ground state absorption of **1a**. (b) Excitation spectrum of **1a** ($c = 35.0$ mM in SDS/water). (c) Absorption spectra of **1a** in different solvents. Concentration **1a**: 4 μM. Spectra recorded at 23 °C. (d) Lifetime of emission at 700 nm ($\lambda_{exc} = 465$ nm). (e) Emission spectra of **1a** in THF (35 mM) and nanoparticle of **1a** fabricated through reprecipitation of THF solution of **1a** (35 mM and 10 μL) in water (3 ml) at room temperature ($\lambda_{exc} = 456$ nm). (f) Structures of **1a** and SDS. All spectra were recorded at room temperature under air.

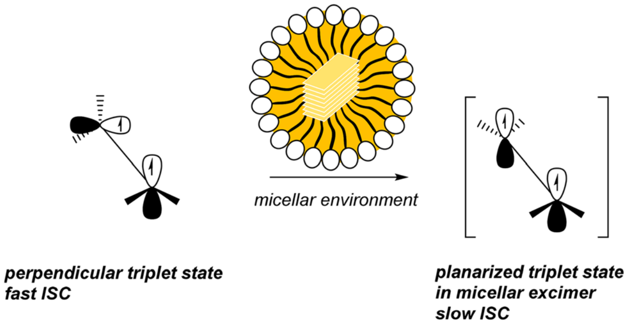


Fig. 3 Spatial confinement in the micellar environment potentially inducing a strong planarity to the single biradical species.

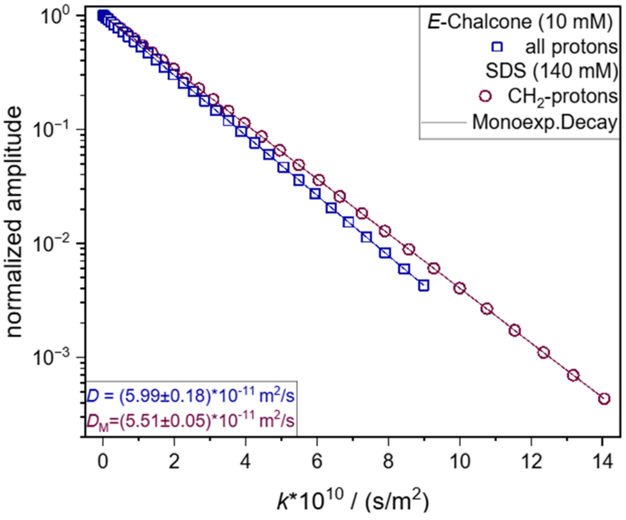


Fig. 4 Self-diffusion echo attenuation curves of 10 mM *E*-Chalcone **1a** and SDS surfactant in a 140 mM SDS mixture. SDS = sodium dodeylsulfate.

state lifetimes (up to 500 ns) depending on the nature of substitutions on the phenyl rings, but there are no reports of radiative decays in the microseconds scale.[54] Noteworthy, the longest lifetime is associated with an increasing planarity of the biradical, which results in an increasing energy gap between triplet and ground states that are normally quasi-degenerate.[56,57] Based on literature data, the unsubstituted *E*-chalcone **1a** investigated in our work should exhibit a triplet state with lifetime of 12 ns in MeOH.[54] Thus, we propose that the spatial confinement in the micellar environment induces either strong planarity to the single biradical species (Fig. 3), or formation of planar aggregates with AIE character (as supported by the experiment with nanoparticles of **1a**). These hypotheses are in accordance with the presence of multiple emissive species. The weakness and broadness of the NIR band can be explained by the high flexibility of aldehydes and ketones in the excited state.

Next, we investigated the size and appearance of the micellar assemblies including the influence of the presence of the substrate under synthetically relevant reaction concentrations. TEM images of concentrated SDS solution (2 wt%, 69 mM) and SDS in the presence of **1a** (15 mM) revealed spherical micelles of approximately 3.5 nm to 5 nm radius and 5 nm to 10 nm radius respectively (Fig. S10). This is in accordance with literature data on the micellar phases of SDS determined *via* small-angle X-ray scattering.[58] Due to limited contrast and resolution of the obtained TEM images we turned to diffusion NMR to obtain precise values for mean micelle size and influences of **1a** on the micellar solution.

The size of micelles can be estimated by assuming a spherical shape and applying the Stokes–Einstein equation, which relates the hydrodynamic radius R_H of a diffusing object to its diffusion coefficient D_S in solution (see eqn (1)).

$$R_H = \frac{k_B T}{6\pi\eta D_s} \qquad (1)$$

The diffusion coefficients of SDS and **1a** in the micellar solution were obtained from linear fits of the spin echo decays (see example in Fig. 4). Due to fast exchange, the surfactant diffusion coefficient D_S is a weighted average of the diffusion of micelles (D_M) and monomers. For the analysis of micellar size we make use of the fact that the influence of monomers is negligible, as the SDS concentration (140 mM) is large compared to the *cmc*, *i.e.* $D_M = D_S$. In comparison to the SDS diffusion in chalcone-free aqueous solution ($D_S = (5.74 \pm 0.17)$ 10^{-11} m^2 s^{-1}, compare Fig. S15), the SDS diffusion coefficient in presence of 10 mM **1a** is unaltered, with $D_M = (5.51 \pm 0.17)$ 10^{-11} m^2 s^{-1}. It provides a hydrodynamic radius of the micelles of $R_H = (3.61 \pm 0.06)$ nm. This size is somewhat larger than the length of an extended surfactant molecule and reflects the tendency of the system to form non-spherical, slightly elongated micelles. We can conclude that SDS micelles are rather robust upon loading with substrate **1a**, keeping their packing parameter and shape, which can be attributed to the dominating electrostatic interactions by repelling head groups.

The diffusion coefficient of **1a** in micellar solution, $D_{1a} =$ (5.99 ± 0.17) 10^{-11} m^2 s^{-1} at 10 mM of **1a**, is only slightly larger than that of SDS. This implies that almost all **1a** is incorporated in the micelle, which is consistent with the poor solubility of **1a** in water, as well as with the localization of similarly hydrophobic compounds in micellar solutions.[59]

This study demonstrates unique emission behavior of **1a** including an extraordinarily long excited state lifetime of 19.2 µs which motivated us to explore the synthetic potential.[60] We set out to investigate [2 + 2] photocycloadditions occurring through the micelle-induced triplet excimer species. We selected activated alkenes as coupling partners to obtain an intermolecular [2 + 2] photocycloaddition. This type of reaction has been previously investigated under direct irradiation with UV-light[61] and visible light under neat or heterogeneous conditions,[43,53] or triplet energy transfer catalysis[42,62,63] including various activation strategies.[64–66] Interestingly, the previous report using visible light at 456 nm irradiation in organic solvent employs an Ir-based photocatalyst. Whereas the present study reveals a protocol that does not rely on a photocatalyst, but rather micelle-induced packing which enables the formation of an excited state species exhibiting unique properties beneficial for synthesis. While a micellar environment has previously been shown to provide beneficial effects in a synthesis context[52] such as substrate activation[47] or oxygen tolerance for triplet states,[67] the current study entails an application of micelle-induced close packing. After optimization (see SI Table S2) the combination of 1.0 equivalent of **1a** with 6.0 eq. of styrene **2a** in 4 wt% SDS in water under blue light irradiation ($\lambda_{\max}$ = 456 nm) was found to successfully deliver the desired cyclobutane **3a** in 68% isolated yield and in a isomeric ratio (*i.r.*) of 71 : 20 : 5:2 : 1 : 1, where the major isomer is the expected *trans–trans* isomer (Fig. S23 and 24).[65] We primarily observe formation of the head-to-tail regioisomer which indicates a triplet energy pathway rather than an electron transfer process as the latter would involve an unlikely primary radical intermediate. This reasoning has previously been used to exclude electron transfer in [2 + 2] photocycloadditions.[68,69] Prior to isolation, the micellar mixture was diluted with brine and then extracted with dichloromethane. Control experiments confirmed that blue light is necessary (Table S2 entry 3–7), and that micellar media were superior to classical organic solvents where limited or no reactivity is observed (entry 11–15). Additionally, SDS proved to be optimal in comparison to other commercially available anionic, cationic and non-ionic surfactants (entry 16–22).

The applicability of the long-lived triplet state in [2 + 2] cycloadditions of various *E*-chalcones **1** and coupling partners was evaluated using the optimal reaction conditions (Fig. 5) on differently substituted substrates. Electron donating or withdrawing groups, as well as halogen substitution of **1** are tolerated. The corresponding cyclobutanes with 1,1-diphenyl ethylene are formed in moderate to good yields with varying isomeric ratios (**3b–3i**, Fig. 5). Next, replacement of the simple phenyl ring with a more complex benzo[d][1,3] dioxole was successful (**3j**). A 2,3-dimethylbutadiene was introduced efficiently, resulting in the corresponding cyclobutane **3k** in a yield of 49%. Simple styrene coupling partners instead of 1,1-diphenyl ethylene resulted in product mixtures with multiple diastereomers formed in significant quantities. Furthermore,

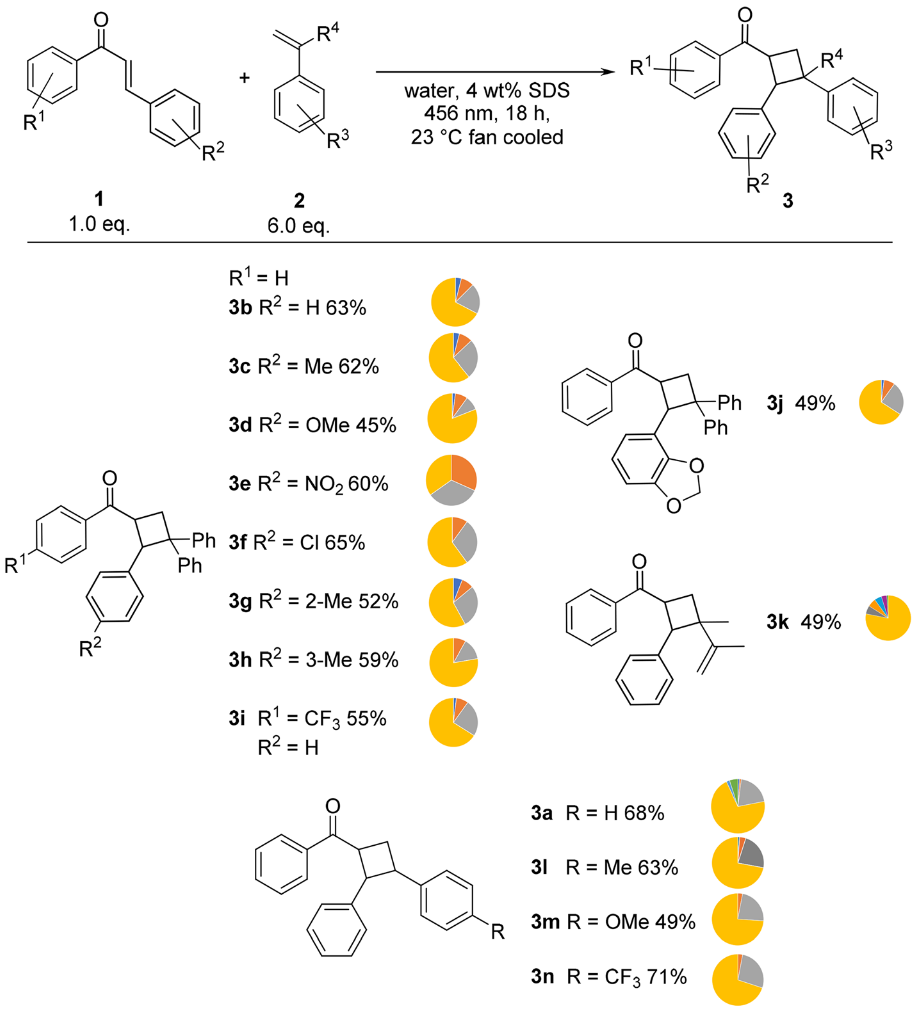
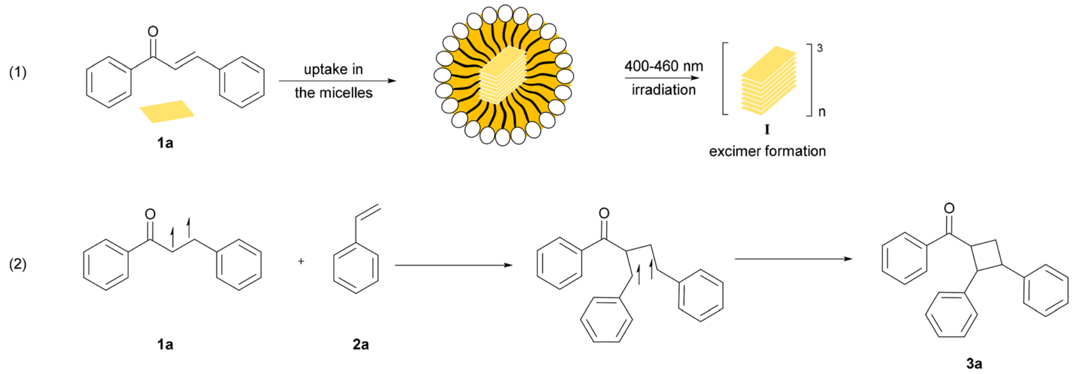


Fig. 5 Overview of the micelle-induced [2 + 2] photocycloaddition. Reactions were performed on a 0.2 mmol scale in 6.0 mL of 4 wt% SDS solution in water and irradiated with 456 nm blue LED for 18 h. The reaction mixture was cooled with a fan, and the temperature was measured to be 23 °C. Isolated yields are given, isolated isomeric ratio was determined using GC-FID and is indicated by sector charts with yellow indicating the major isomer. Values can be found in the SI.



Scheme 1 Mechanistic proposal for the triplet state reactivity of chalcone enabled by micellar system.

similar R_f values of the dimer of **1a** and the cyclobutane products resulted in challenging separations. Nonetheless, electron rich and electron poor styrenes could be converted into product and isolated (**3a**, **3l–3n**). A more comprehensive list of substrates showing significant reactivity can be found in the SI.

Mechanistic proposal

Based on these results the following mechanism can be proposed: Upon uptake of chalcone in the micelles, the close packing conditions in the micelles, similar to the condensed phase, result in a change of the photophysical properties of the mixture. Planarization of **1a** in a multimer leads to a degeneration of the triplet and ground state, as well as slower ISC from the triplet to the ground state. This now allows excitation by irradiation with 400–470 nm light yielding Excimer **I** with an emission around 700 nm and a long lifetime. The triplet state **1a** can now engage in [2 + 2] photocycloaddition with styrene **2a** to form the cyclobutane products **3a** (Scheme 1).

Conclusion and outlook

We have developed a photochemical protocol for application in synthesis that takes advantage of the close packing of molecules induced by micelles. In contrast to methods based on solid state chemistry, this protocol is induced through a micro-heterogeneous solution of aqueous micelles. The use of a micellar medium enables an extraordinarily long excited state lifetime of simple chalcone. The developed protocol for [2 + 2] photocycloadditions successfully enabled cyclobutane formation using visible light (456 nm). Interestingly, no Ir-based photocatalyst is required for the visible light-based protocol and the aqueous micelles not only enable the reactivity displayed in the present study, but also allow for more sustainable alternatives to organic solvents. Thus, our protocol contributes to advancing sustainable synthesis.

The close packing of simple and small molecules enabled by micelles has the potential to induce unique photochemical behaviour such as shifts in absorption and emission and uniquely long lifetimes which is beneficial for synthesis. Hence, we believe that using the disclosed protocol with different small molecules, visible light can be used to obtain long lived excited states that are currently unexplored, delivering unknown synthetic applications.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting the findings in this article are presented in the manuscript or available in the SI. Supplementary information is available. See DOI: **https://doi.org/10.1039/d5ta05567a**.

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