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Carbon Dots as Photoinitiators for Thiol–Ene Reactions

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ABSTRACT

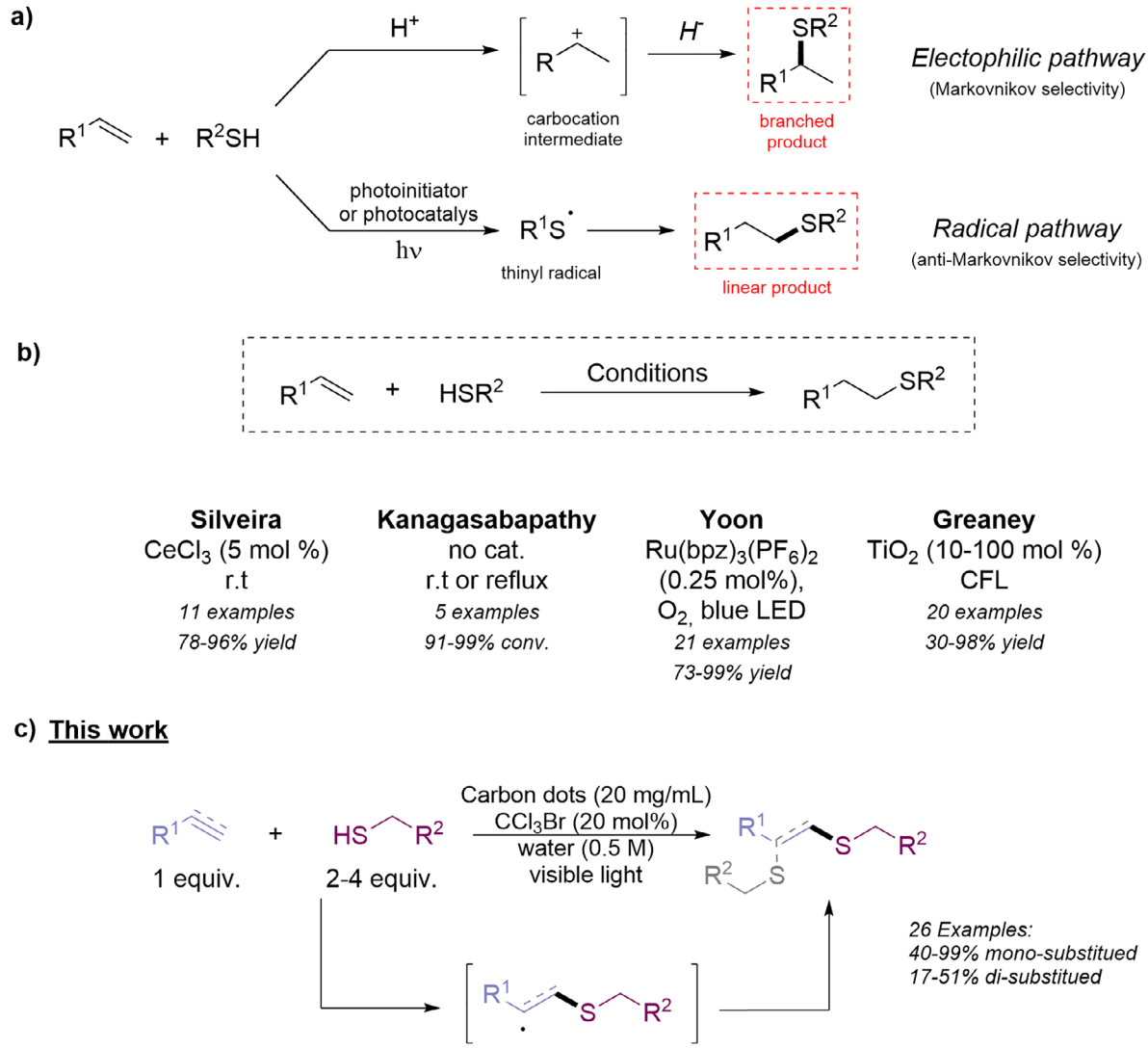
Thiol-ene reactions are highly valuable for the formation of C—S bonds, offering potential applications across various fields. This project presents several advantages over traditional synthesis methods, as a metal-free and eco-friendly approach for the anti-Markovnikov hydrothiolation of olefins, using carbon dots (CDs) as organic and bio-derived photo initiators. This process is characterized by mild conditions, it takes place in aqueous media under visible light, and it is compatible with a broad range of thiols (aliphatic, aromatic, and with different substituents on the ring), alkenes, and alkynes. CDs prepared from citric acid were used for the optimization of the reaction conditions and the scope. Subsequently, the reaction was performed using CDs synthesized with a hydrothermal treatment from industrial fruit waste (raspberries, blackberries, blueberries, pomegranate, and wild strawberries previously extracted with supercritical CO_2). This protocol operates under mild conditions and aligns with the principles of a circular economy, focusing on waste valorization rather than simple recycling. The reaction mechanism was finally probed by performing Stern–Volmer quenching studies in the presence of increasing amounts of CCl_3Br, as well as carrying out clock experiments.

1 | Introduction

The thiol-ene reaction is a coupling process between a thiol and an olefin that produces thioethers. First reported in 1905, this reaction has found numerous applications across various fields, particularly in materials science. The incorporation of thiol groups and the resulting sulfur linkages within polymer backbones can enhance the flexibility of both thermoset and thermoplastic polymers [1]. This process, classified as a "click reaction" [2, 3], found a plethora of applications in organic synthesis and in medicinal chemistry for the preparation of sulfur-containing molecules such as cyclic peptides and thiosugars [4].

Depending on the conditions, the thiol–ene reaction proceeds through three main paths, namely electrophilic, free radical, and nucleophilic mechanisms. In the first case (usually performed under acid-catalyzed conditions), an electrophilic addition occurs onto olefins by following a Markovnikov selectivity [5], thus yielding the branched product (Scheme 1a); the two other pathways afford the corresponding anti-Markovnikov derivative [5]. In the free-radical mechanism, a thiyl radical (in turn released under transition metal catalysis or following a photochemical event) is responsible for triggering the propagation chain, as shown in Scheme 1, path b [5].

As hinted above, the versatility in terms of reaction pathways and substrate scopes makes thiol–ene reactions one of the most investigated "click reaction" [6]. In Scheme 1b some representative examples are depicted, including the $CeCl_3$-catalyzed addition of aromatic thiols to olefins under solvent-less conditions [7] and the catalyst-free coupling occurring in benzene described by Kanagasabapathy and coworkers [8]. In this framework,

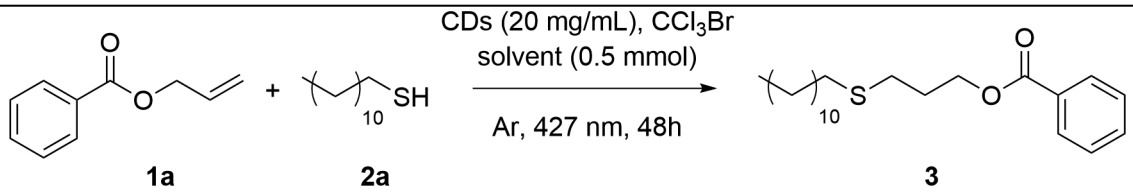


SCHEME 1 | (a) Thiol–ene reaction via electrophilic and radical addition. (b) Significant examples of thermal and photochemical thiol–ene coupling. (c) Our proposal.

significant attention has been given to photocatalyzed conditions, starting from the pioneering works authored by Yoon et al. [9, 10], which involved ($Ru(bpz)_3(PF_6)_2$) as the photoredox catalyst and allowed the preparation of glutathione bioconjugates in water. Greaney and coworkers [11] lately described a visible light-driven TiO_2-catalyzed anti-Markovnikov thiolation of inactivated olefins and styrenes. Starting from these seminal examples, a wide range of light-activated thiol–ene processes has been proposed [12].

Recently, the potential of carbon dots (CDs) in photocatalysis or, more broadly speaking, in sustainable visible light-driven organic synthesis, has been investigated. Such species are zero-dimensional amorphous or graphitic carbon-based nanomaterials, with enhanced water solubility, low toxicity, and photophysical properties that depend on the conditions and the precursors employed in their synthesis. CDs are prepared by following two approaches, namely top-down (where larger carbon structures such as graphite are disrupted via exfoliation or electrochemical etching) or bottom-up [13–22]. In the latter case, materials containing organic molecules undergo thermal decomposition to form differently doped CDs [23]. The latter strategy allows precise control over the composition and structure of the CDs, being often preferred for its simplicity and ability to fine-tune the properties [24]. In the last decade, a plethora of protocols for CD synthesis have been described in the literature, starting from low-value natural sources, including vegetable biomass [25] and seafood waste [26]. Typically, such nanomaterials absorb in the 280–360 nm region with a significant tail in the visible range and mainly emit in the blue and green regions. Doping of the CDs with heteroatoms is a powerful strategy to modulate both photophysical and photochemical behavior of CDs. Nitrogen and sulfur represent efficient doping elements due to their inherent characteristics [27]. The main area of application of such carbon nanomaterials is biomedicine, where their use as biosensors [28, 29], nanocarriers for drugs [30], as well as in theragnosis [31] and photodynamic therapy [32] has been suggested. However, due to their activity as electron donors or acceptors upon irradiation [33], CDs have also been investigated as photocatalysts/photoinitiators in environmental remediation [34], hydrogen production [35, 36], carbon dioxide fixation [37], and even in organic synthesis [38–40]. In this framework, we focused on the design of a visible

TABLE 1 | Optimization table of thiol–ene reaction conditions between allylbenzoate **1a** and dodecanethiol **2a** in the presence of CCl_3Br.



1a **2a** **3**

Entry	Conditions	Yield (%)[a]
1	**1a**:**2a** (1:2), CDs 20 mg/mL, CCl_3Br 20% mol, DMF	83%
2	**1a**:**2a** (1:2), CDs 20 mg/mL, **no CCl_3Br,** DMF	42%
3	**1a**:**2a** (1:2), CDs 20 mg/mL, CCl_3Br 20% mol, **DMSO**	9%
4	**1a**:**2a** (1:2), CDs 20 mg/mL, CCl_3Br 20% mol, **MeOH**	65%
5	**1a**:**2a (1:1),** CDs 20 mg/mL, CCl_3Br 20% mol, DMF	44%
6	**1a**:**2a** (1:2), CDs 20 mg/mL, CCl_3Br 20% mol, **H_2O**	84%
7	**1a**:**2a** (1:2), **no CDs**, CCl_3Br 20% mol, H_2O	< 5%

[a]Isolated yields via column chromatography. Reaction conditions: Solvent solution (1 mL) containing **1a** (0.5 mmol, 0.5 M), **2a** (1-2 mmol), CCl_3Br (20 mol%), a-N-CDs (20 mg/mL), irradiated with a 427 nm LED lamp (40 W) in a Pyrex vessel for 48 h.

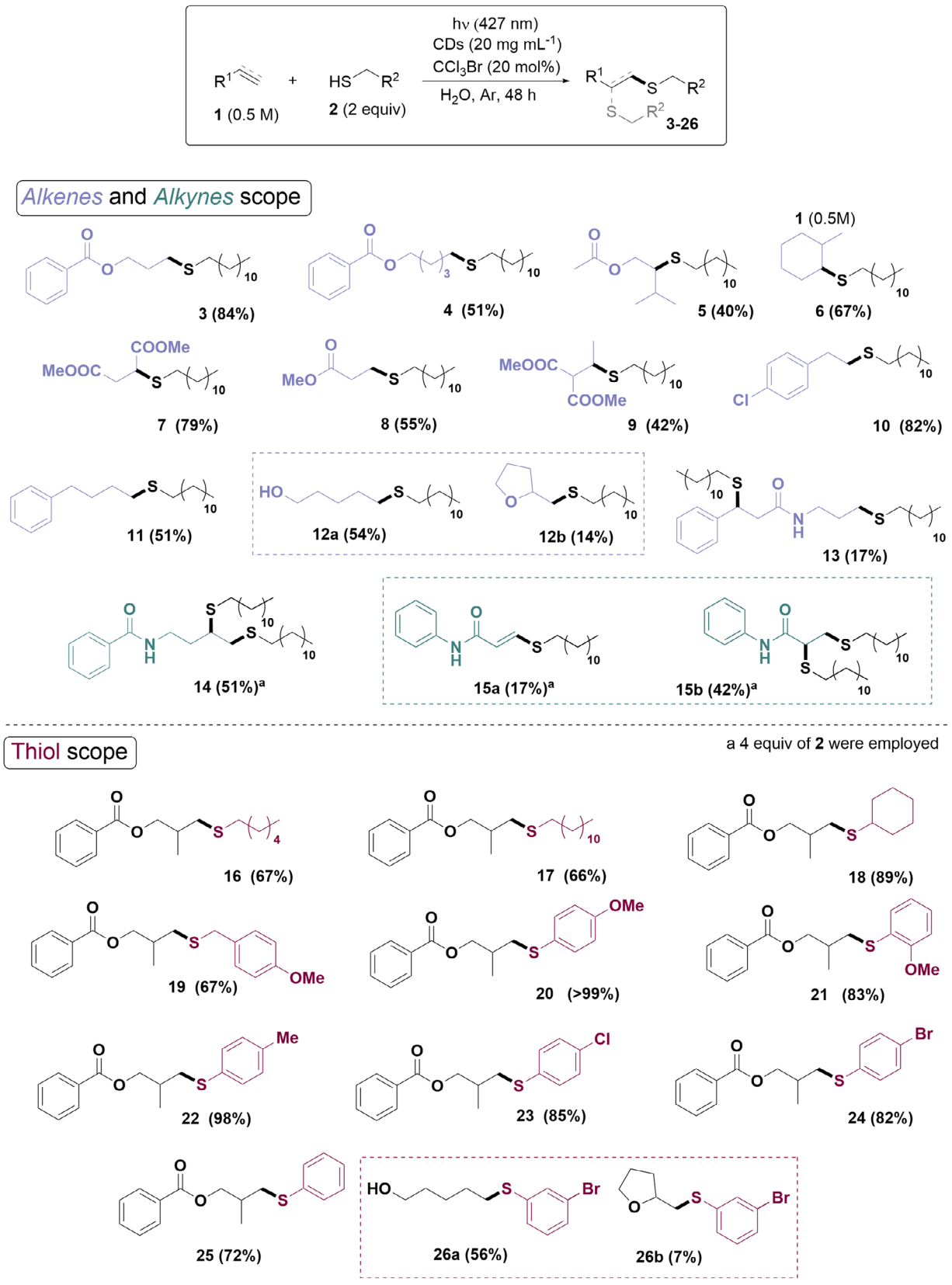
light-triggered, metal-free anti-Markovnikov hydrothiolation of alkenes and alkynes in aqueous mixture, by exploiting CDs as bio-derived photocatalytic initiators. The results obtained with citric acid-derived nitrogen-doped CDs (a-N-CDs) employed in our previous investigation [40, 41] have been then compared to those prepared by adopting agro-food waste (fruit pomace furnished by the company Rigoni di Asiago s.r.l., Italy) as the precursors.

2 | Results and Discussion

To test our idea, we initially investigated the reaction between allylbenzoate **1a** and dodecanethiol **2a** with a-N-CDs and CCl_3Br irradiated with a 40 W Kessil Lamp with an emission at 427 nm. The optimization of the reaction conditions (parametric analysis of solvents, the presence of CDs, and the amount of starting substrates) was carried out. As apparent from Table 1, the best results arising from this preliminary screening have been observed by irradiation of an argon equilibrated solution of **1a** (0.5 M) and dodecanethiol (1 M, equiv) in DMF in the presence of a-N-CDs (20 mg mL^{-1}) and CCl_3Br (20 mol%) for 48 h at 427 nm (40 W Kessil lamp) under stirring. Under these conditions, the desired product **3** was isolated in 83% yield (Table 1, entry 1). When carried out in the absence of CCl_3Br as co-catalyst, the reaction yield drops to 42% (entry 2). Other reaction media, including DMSO and methanol, offer less satisfactory performances with respect to DMF (entries 3,4), while the use of 1 equivalent of **2a** in DMF afforded the desired compound **3** in only 44% yield. We then decided to focus on water, the sustainable solvent par excellence, in which a comparable yield of **3** (84% yield) was obtained. Additional experiments (Supporting Information) exclude the need for a freeze-pump-thaw cycles prior to irradiation.

With these results in hand, we investigated the scope of the optimized procedure on substituted alkenes and alkynes with different thiols in water, as summarized in Scheme 2. Dodecanethiol was successfully exploited for the formation of the C—S bond without any effect of the alkyl chain length (compare results obtained for compounds **3,4**), for the preparation of both hindered (**5**) and cyclic products (**6**). Electron-poor olefins (**7–9**) and styrenes (**10**) were also isolated in good yields. Notably, in the thiolation of 4-pentenol, a mixture of linear product **12a** (54% major) along with a minor amount of tetrahydrofuran **12b** (14% yield) was observed, while irradiation of *N*-allyl cinnamamide in the presence of dodecanethiol afforded the double functionalized compound **13**. The process was also extended to alkynes, where a double vicinal hydrothiolation of the triple C—C bond was observed, as apparent from products **14** and **15b**. In the last case, a minor amount (17% yield) of vinyl sulfide **15a** was obtained. We thus explored the scope of thiols. Notably, both alkyl (**16–18**), benzyl (**19**), and differently substituted aromatic (**20–26**) thiols were successfully employed as the functionalizing agents. With thiophenols, C—S bond formation was achieved in up to quantitative yields (see results obtained for compounds **20, 22**). As already reported above, when using 4-pentenol as the starting substrate, a mixture of 5-thioaryl pentanol (**26a**, 56% yield) and 2-(thioaryl)methyl-tetrahydrofuran (**26b**, 7%) was isolated.

With the aim of valorizing agrifood waste in a circular approach, we focused on waste feedstocks (i.e., different fruit pomaces: raspberry, blueberry, blackberry, and pomegranate) as precursors for the synthesis of the CDs. For the sake of comparison, CDs were also prepared from the residues obtained by $scCO_2$ extraction of wild strawberries. Optical and morphological properties of CDs have been spectroscopically characterized by UV–Vis and by transmission electron microscopy (TEM) to confirm the presence of dispersed nanometric material (see an example in Figure 1, where the highlighted nanoparticles have a diameter of 13, 18, and 22 nm). The photophysical properties of the prepared CDs, including λ_{em}, quantum yield, and lifetime values for the observed photoluminescence, are available in Table S2. We initially compared the photocatalytic performances of CDs obtained from raspberries under different reaction conditions by focusing on the preparation of **16** as the model reaction. The hydrothermal treatment of the wild raspberry residues involves heating at 180°C 2 g of substrate in 20 mL of MillQ water in a sealed autoclave for



SCHEME 2 | Scope of the thiol–ene reaction with-N-CDs as photoinitiator.

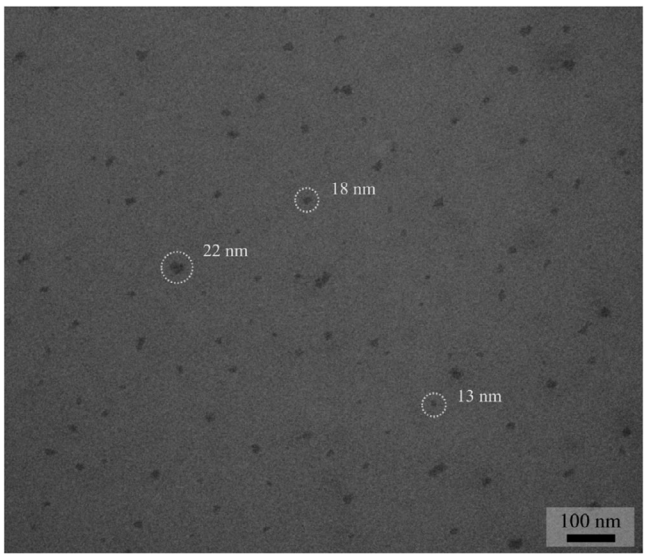


FIGURE 1 | TEM image of CDs obtained from a 6 h hydrothermal treatment of raspberries.

6 or 24 h; in selected experiments, diethylenetriamine was used as *N*-dopant (Supporting Information). As shown in Table 2, the synthesis afforded compound **16** in the 53%–67% yield range, and best results have been obtained for *N*-doped CDs obtained with a 6 h hydrothermal treatment (entry 3).

Accordingly, we prepared *N*-doped CDs from the other fruit residues; the only exception being the residue obtained by extraction of wild strawberry pomaces previously extracted with $scCO_2$, which have been treated under hydrothermal conditions for 24 h to obtain the CDs. The worst results were obtained with the extracted wild raspberry residues (Table 2, entry 4), while **16** was isolated in good yields (58%–59%) when using blueberries and pomegranate-derived N-CDs (entries 1 and 3). In any case, CDs obtained from blackberries gave the best performances (entry 2, 75%).

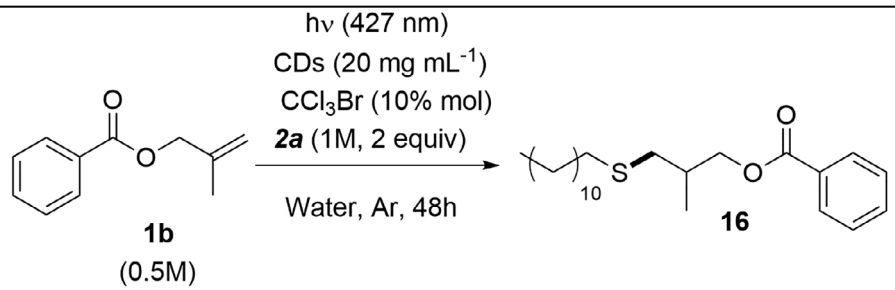
Coherently with previous results [13–18], Stern–Volmer investigation plots [13–18], on all the a-N-CDs revealed a diffusional value ($>10^{10}$ M s^{-1}) for the measured k_q for both CCl_3Br and dodecanethiol (Supporting Information), pointing ito the interaction of such compounds with the photoexcited CDs. Furthermore, irradiation of a solution of dodecanethiol (**2a**) and *N,N*-diallyl derivative **1n** results in the formation of product **27** arising from a 5-exo-trig cyclization, providing further evidence for a radical-based mechanism (Scheme 3).

Based on these results and on the available literature [13–18, 41], we proposed the mechanism depicted in Scheme 4, where the photoexcited CDs* generate the radical anion intermediate $CCl_3Br^{-\bullet}$ (paths a and b), which in turn releases trichloromethyl radical by losing Br^- (path c). $CCl_3^{\bullet}$, in turn, abstracts a hydrogen atom from thiol **2**, to form the electrophilic thiyl radical intermediate (path d); the species is then trapped by the olefin **1a**, and the resulting alkyl radical is finally converted into the desired product (path e), while restoring thiyl radical $RS^{\bullet}$.

3 | Conclusions

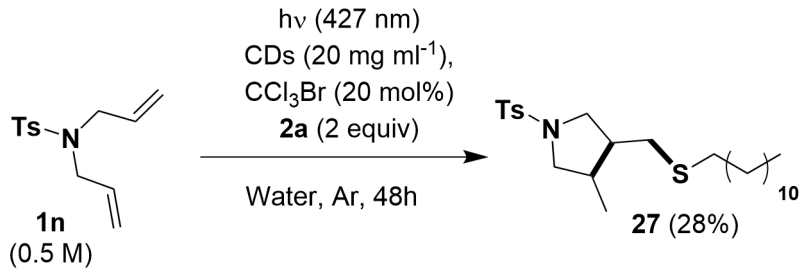
As hinted in the introduction section, thiol–ene reactions are powerful tools for building C—S bonds and hold broad potential across multiple fields. In the present study, we optimized the

TABLE 2 | Thiol–ene reaction coupling of **1b** and **2a**. Comparison between different wild raspberry-derived CDs (upper part); Thiol–ene reaction coupling of **1b** and **2a**. Comparison between different bioderived N-CDs (lower part).

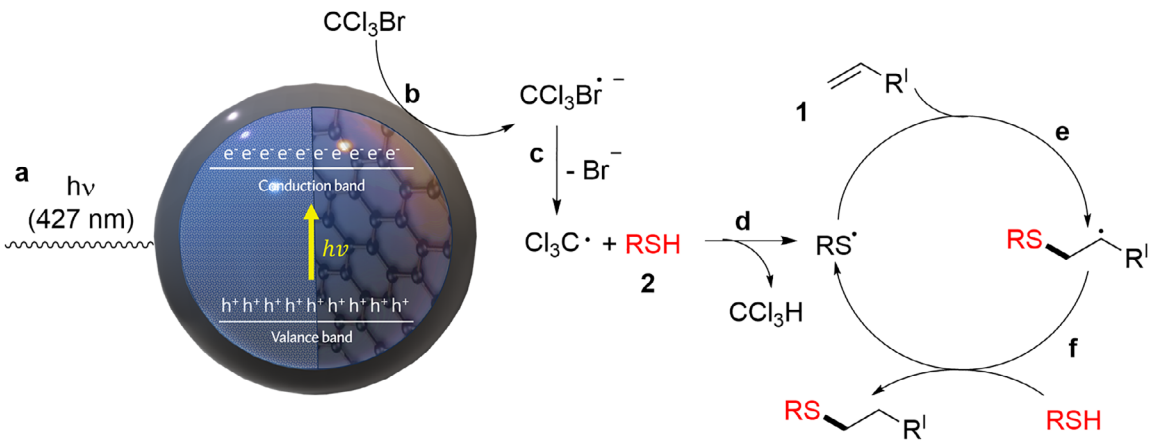


Entry	Conditions of the synthesis of CDs from raspberries	16 %Yield
1	180°C, 6 h	63
2	180°C, 24 h	53
3	180°C, 6 h, *N*-doped[a]	67
4	180°C, 24 h, *N*-doped[a]	62
Entry	**N-CDs precursor**	**16 %Yield**
5	Blueberries	59
6	Blackberries	75
7	Pomegranate	58
8	Wild strawberries ($scCO_2$ extracted)	48

[a]Diethylenetriamine used as an *N*-donor.



SCHEME 3 | Radical clock experiment between *N*-tosylated diene **1n** and dodecanethiol **2a**.



SCHEME 4 | Suggested mechanism for the CDs photoinitiated anti-Markovnikov hydrothiolation of alkenes **1**.

reaction conditions through a series of experiments conducted under varied parameters and refined the synthesis of the CDs based on outcomes obtained from applying these nanomaterials to a model reaction. A library of thioethers was synthesized in good to excellent yields. We evaluated a wide range of substrates and demonstrated the applicability of this protocol to various unsaturated compounds, including those with triple bonds, as well as numerous aliphatic and aromatic thiols. This method offers several advantages over traditional synthetic approaches: it employs mild conditions, uses water as the solvent, and relies on an inexpensive, metal-free catalyst derived from food waste. In contrast to rare-metal complexes, which are rather costly and, in some cases, pose environmental disposal challenges, this approach aligns with circular-economy principles by emphasizing waste valorization.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available in the Supporting Information of this article.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section.

Supporting File: cctc70780-sup-0001-SuppMat.pdf.