



Efficient aerobic photooxygenation of aldehydes to carboxylic acids using cobalt(II) phthalocyanine sulfonate as a photosensitizer in organic-water biphasic media

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Abstract

The aerobic oxidation of a variety of aromatic aldehydes to the corresponding carboxylic acids by molecular oxygen in the presence of 4-carboxyl tetraphenylporphyrin (H₂TCPP), methylene blue (MB), cobalt(II) phthalocyanine sulfonate (CoPcS) and FeTCPPCl as water-soluble photosensitizers in organic-water biphasic media at room temperature under either visible light or sunlight is described. The products were obtained with 25–100% conversion and 100% selectivity. This method has a wide range of applicabilities, has a straightforward workup procedure, is chemoselective and proceeds under mild reaction conditions. The resulting products were obtained in good yields in reasonable times.

Introduction

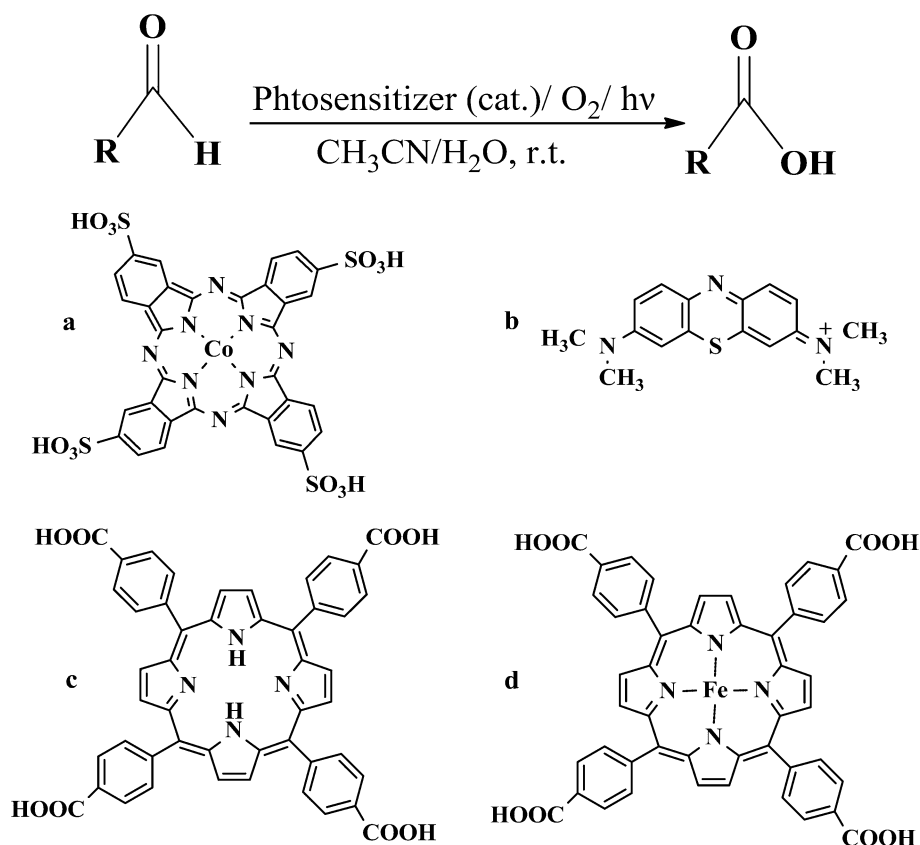
Oxidation is one of the most essential reactions in synthetic organic chemistry, and for this purpose, a very wide variety of oxidants have been developed. The oxidation of aldehydes to carboxylic acids is of abiding interest in synthetic organic chemistry [1–4]. The popular conventional route to carboxylic acids involves the use of Jones reagent [5–10]. This is a stoichiometric reaction using highly acidic conditions which may not be tolerated by acid-sensitive functionalities in the substrate. Furthermore, the production of Cr-based side products may be viewed as a potential environmental hazard. Other efficient reagents that have been reported in the literature to achieve this transformation include oxone [11], calcium hypochlorite [12] and 2-hydroperoxyhexafluoro-2-propanol [13]. Some interesting methodologies involving metal-mediated transformation of aldehydes to the corresponding carboxylic acids have also been recently reported [14–18]. However, the development of catalytic systems for hydrocarbon oxidation by O₂ under mild conditions is still a challenging problem. Iron porphyrins have been used as catalysts for alkane hydroxylation by O₂ while consuming a stoichiometric amount of a reducing agent

[19]. The oxidation of alkanes to alcohols and ketones has been accomplished at 80 °C under 10 atm pressure of oxygen [20]. A few examples of photochemical photooxidation using O₂ and a metalloporphyrin catalyst have been reported [21, 22]. The photosensitized production of singlet oxygen is relevant to the photooxidation of organic compounds, as well as to DNA damage and photodynamic therapy [23, 24]. Consequently, a variety of photosensitizers have been developed and their photochemical and photophysical properties have been studied extensively [25]. Among such compounds, tetrapyrrolic compounds such as porphyrins and phthalocyanines are promising candidates for photosensitizers due to their special photophysical properties [26–30]. In particular, cobalt phthalocyanine has been widely used as a photosensitizer in photocatalytic reactions [31–34]. In our previous studies, we introduced efficient systems for the aerobic oxidation of alcohols and alkenes in organic solvents under visible light or sunlight [35–38]. The current study develops the photocatalytic oxygenation of aldehydes using air under visible light or sunlight irradiation with water-soluble photosensitizers in organic-water biphasic media at room temperature (Scheme 1). The products were obtained with 25–100% conversion and 100% selectivity.

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Scheme 1 Photooxygenation of aldehydes to carboxylic acids in the acetonitrile–water media with different kinds of water-soluble photosensitizers. **a** Coph(SO₃H)₄, **b** methylene blue, **c** H₂TCPP, **d** FeTCPPCl



Experimental

Materials and methods

Benzaldehyde, 4-methoxybenzaldehyde, 2,3-dimethoxybenzaldehyde, 3,4-dimethoxybenzaldehyde, 3-chlorobenzaldehyde, 4-chlorobenzaldehyde, anthracene, anthracene-9-carbaldehyde, cinnamaldehyde, 2,3,4,5,6-pentafluorobenzaldehyde, iron(II) chloride and solvents were purchased from Fluka or Merck and used without further purification. MB was purchased from Fluka. H₂TCPP, FeTCPPCl and CoPcS were synthesized according to the literature procedures [39–42].

For generation of the carboxylic acids, 5×10^{-4} mol of the required aldehyde and 8×10^{-6} mol of water-soluble photosensitizer were added to acetonitrile (7.5 ml) plus water (7.5 ml) in a test tube. The samples were irradiated under visible light (288 power LED lamps, 1 W, 2.3 V, 59,660 Lux for 150 h at room temperature while bubbling air through the mixture ($20 \text{ cm}^3 \text{ min}^{-1}$). To prevent evaporation of the solvent, the photoreactor was equipped with a strong fan that kept the reaction temperature low and the test tube was closed with parafilm. The stability of the sensitizer was monitored by UV–Vis spectroscopy (Cintra 101) during the reaction, by following the Q band

intensity of CoPcS. At the end of the reaction, the solvent was removed under vacuum, and the residue was separated by column chromatography (silica gel, n-hexane/EtOAc, 13:1) to give the corresponding carboxylic acid products. The identities of the products given in Table 3 were confirmed from their melting points and ¹H NMR spectra (NMR (BRUKER DRX-300 AVANCE spectrometer). Benzoic acid (1): colorless crystals, mp 120–123 °C. ¹H NMR (300 MHz, CDCl₃) δ 8.17 (s, 2H), 7.60 (s, 3H) ppm, OH not observed. Cinnamic acid (2): colorless crystals, mp 132–135 °C. ¹H NMR (300.13 MHz, CDCl₃) δ 10.80 (brs, 1H, –COOH), 7.78 (d, *J* = 12.05 Hz, 1H), 7.42–7.59 (m, 5H), 6.46 (d, *J* = 12.05 Hz, 1H) ppm. 4-methoxybenzoic acid (3, 4): colorless crystals, mp 182–185 °C. ¹H NMR (300.13 MHz, CDCl₃) δ 11.85 (brs, 1H, –COOH), 8.07 (d, *J* = 8.3 Hz, 2H), 6.95 (d, *J* = 8.3 Hz, 2H), 3.89 (s, 3H) ppm. 2,3,4,5,6-pentafluorobenzoic acid (5): colorless crystals, mp 100–103 °C. ¹H NMR (300.13 MHz, CDCl₃) H of aldehyde (δ 10.01 (1H, s) ppm.) not observed. 3-chlorobenzoic acid (6): colorless crystals, mp 154–157 °C. ¹H NMR. (300.13 MHz, CDCl₃) δ 11.30 (brs, 1H, –COOH), 8.11 (s, 1H), 8.01 (d, *J* = 7.6 Hz, 1H), 7.59 (d, *J* = 7.8 Hz, 1H), 7.42 (t, *J* = 7.8 Hz, 1H) ppm. Anthracene-9-carboxylic acid (7): colorless crystals, mp 214–217 °C ¹H NMR (300.13 MHz, CDCl₃) δ 11.53 (s, 1H, –COOH), 8.97 (d, *J* = 9.0 Hz, 2H), 8.7 (s, 1H), 8.05 (d, *J* = 7.6 Hz, 2H), 7.68

(m, 2H), 7.53(m, 2H). 4-chlorobenzoic acid (8): colorless crystals, mp 237–240 °C. $^1\text{H NMR}$ (300.13 MHz, CDCl_3) δ 8.02 (d, $J=7.9$ Hz, 2H), 7.43 (d, $J=8.2$ Hz, 2H) ppm, OH not observed. 3,4-dimethoxybenzoic acid (9): colorless crystals, mp 179–182 °C. $^1\text{H NMR}$ (300.13 MHz, CDCl_3) δ 7.77 (d, $J=8.3$ Hz, 1H), 7.61 (s, 1H), 6.92 (d, $J=8.4$ Hz, 1H), 3.96 (s, 3H), 3.95 (s, 3H) ppm, OH not observed. 2,3-dimethoxybenzoic acid (10): colorless crystals, mp 121–124 °C. $^1\text{H NMR}$ (300.13 MHz, CDCl_3) δ 7.70 (d, $J=6.9$ Hz, 1H), 7.09–7.22 (m, 2H), 3.98 (s, 3H), 3.91(s, 3H) ppm, OH not observed.

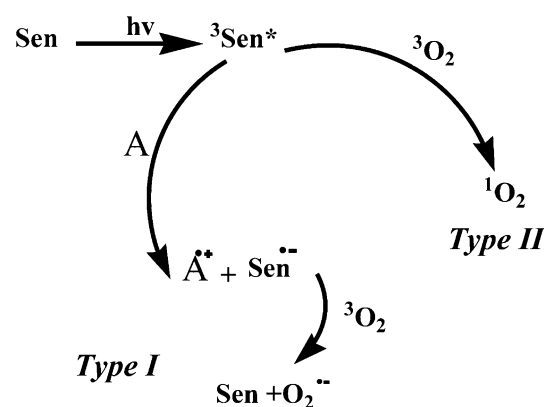
Results and discussion

The photooxygenation of 4-methoxybenzaldehyde was chosen as a model reaction in order to evaluate the different factors involved in the catalytic reaction. Table 1 gives the yields of 4-methoxybenzoic acid with various photosensitizers. The yield of 4-methoxybenzoic acid was $\text{CoPcS} > \text{MB} > \text{FeTCPP} > \text{H}_2\text{TCPP}$ for the same reaction time (Table 1, entries 1–4). It is worth noting that after 150 h of photooxygenation of 4-methoxybenzaldehyde in the presence of MB, FeTCPP and H_2TCPP , the color of the solution had completely disappeared. Although the amount of singlet oxygen that was produced in the presence of CoPcS was low, because of lower degradation of CoPcS compared to MB, FeTCPP and H_2TCPP , it could be used to convert aldehydes to carboxylic acids over a longer period of time. It is important to note that the oxidation of substrate ceased in the absence of photosensitizer, light or air (Table 1 entries 5–7).

Hence, the photosensitizer, light and O_2 are all essential for oxidation of aldehydes.

According to the literature, there are two major pathways for photooxygenation in the presence of photosensitizers, designated as Type I and Type II (Scheme 2) [45]. For investigation of the Type I mechanism, we performed our reactions in the presence H_2O_2 , OH^\cdot and O_2^- . In the presence of these reactive oxygen species, the yield of the reaction was significantly decreased (Table 1, entries 8–10).

Singlet oxygen generation (Type II) and its subsequent reaction with the substrate is the principal mechanism under our reaction conditions, since a very characteristic reaction of singlet oxygen is the [4+2] cycloaddition to conjugated cyclic dienes and polycyclic aromatic hydrocarbons such as



Scheme 2 Alternative mechanisms for the production of reactive oxygen species in the presence of photosensitizers (sen). A is oxidizable substrate

Table 1 Photooxygenation of 4-methoxybenzaldehyde under different reaction conditions^a

Entry	Condition	Conversion (%)	Quantum yield for $^1\text{O}_2$ generation (Φ_Δ)
1	Light + air + CoPcS	100	0.3 [43]
2	Light + air + MB	28	0.49 [25]
3 ^b	Light + air + FeTCPP	23	
4 ^b	Light + air + H_2TCPP	8	0.76 [25]
5	Light + air	Trace	
6	Air + CoPcS	Trace	
7	Light + CoPcS + nitrogen	Trace	
8 ^c	OH^\cdot	Trace	
9 ^d	O_2^-	Trace	
10 ^e	H_2O_2	Trace	
11	Light + air + NaN_3^- + CoPcS	Trace	

^a 8×10^{-6} mol photosensitizer, 5×10^{-4} mol 4-methoxybenzaldehyde, air (1Atm) and 288 power LED lamps, 1 W, 2.3 V (59,660 LUX) in 1:1 $\text{CH}_3\text{CN}/\text{H}_2\text{O}$

^bphotosensitizer was dissolved in $\text{KCl}-\text{NaOH}$ buffer (0.2 M, $\text{pH}=12.0$)

^c OH^\cdot produced from photolysis of 2.5×10^{-3} mol 30% H_2O_2 using light from a high-pressure 500 W mercury lamp ($\lambda=200-280$ nm)

^d O_2^- was prepared by dissolving K_2O in dried DMSO [44]

^e4 mmol 30% H_2O_2 dissolved in the reaction solution

anthracene [46–49]. Hence, singlet oxygen generation by CoPcS is evidenced by chemical trapping of $^1\text{O}_2$ with anthracene. The UV–Vis spectra of anthracene as function of time under photoirradiation with CoPcS as a photosensitizer are displayed in Fig. 1. A decrease in intensity of the absorption band of anthracene ($\lambda_{\text{max}} = 375 \text{ nm}$) was observed over time. This response can be attributed to anthracene-9,10-endoperoxide formation (see Fig. 1). Moreover, the oxidation reaction did not occur under dark conditions, confirming that the anthracene oxidation is mediated by singlet oxygen under visible irradiation.

Furthermore, in the presence of N^{3-} , which is a well-known singlet oxygen scavenger [50], conversion was inhibited (Table 1, entry 11). The photolysis was also studied in different solvent mixtures with variable water-to-acetonitrile ratios. Optimum conversion was observed in 1:1 acetonitrile/water (Table 2). The lifetime of singlet oxygen in acetonitrile is $65 \mu\text{s}$ and $38 \mu\text{s}$ in ethanol [51–53], which corresponds with the results in Table 2, entries 1 and 4, again providing evidence for generation of singlet oxygen in the reaction media.

High turnover number (TON) for the photooxygenation of aldehydes was observed with CoPcS under visible light (Table 3 entries 1–10). It is clear that this reaction will also proceed under solar radiation because the spectral distribution curve of the lamp is similar to that of sunlight (Table 3 entry 4).

Figure 2 shows a plot of 4-methoxybenzoic acid formation versus time in oxygenated CH_3CN under visible light irradiation in the presence of CoPcS. The Q band of CoPcS was followed at 660 nm by UV–Vis spectroscopy. The plot

Table 2 Effect of solvent on 4-methoxybenzaldehyde photooxygenation^a

Entry	Solvent	Conversion (%)
1 ^b	($\text{H}_2\text{O}:\text{CH}_3\text{CN}$) 1:1	100
2 ^c	($\text{H}_2\text{O}:\text{CH}_3\text{CN}$) 2:1	80.1
3 ^d	($\text{H}_2\text{O}:\text{CH}_3\text{CN}$) 1:2	68.5
4 ^e	($\text{H}_2\text{O}:\text{EtOH}$) 1:1	76.0
5	H_2O	Trace

^a 8×10^{-6} mol CoPcS, 5×10^{-4} mol 4-methoxybenzaldehyde, air (1Atm) and 288 power LED lamps, 1 W, 2.3 V (59,660 LUX)

^b7.5 ml CH_3CN and 7.5 ml H_2O

^c5 ml CH_3CN and 10 H_2O

^d10 ml CH_3CN and 5 ml H_2O

^e7.5 ml EtOH and 7.5 ml H_2O

shows that sensitizer bleaching for CoPcS was almost complete after 150 h under our reaction conditions. After loss of the Q band of CoPcS, the oxidation reaction ceased, and the yield of product remained constant. Therefore, the reaction time was chosen as 150 h in these studies.

A plausible mechanism for this reaction is shown in Scheme 3. The catalyst is activated by visible light to give singlet oxygen. The singlet oxygen then undergoes insertion into the C–H bond of the aldehyde 1 to form a peracid 3, via the acyl and hydroperoxyl radicals as intermediates [61]. In the final stage of reaction, a second aldehyde molecule reacts with the peracid 3 to generate the adduct 4. Compound 4 then decomposes by a Baeyer–Villiger type rearrangement to the final product 5 [61].

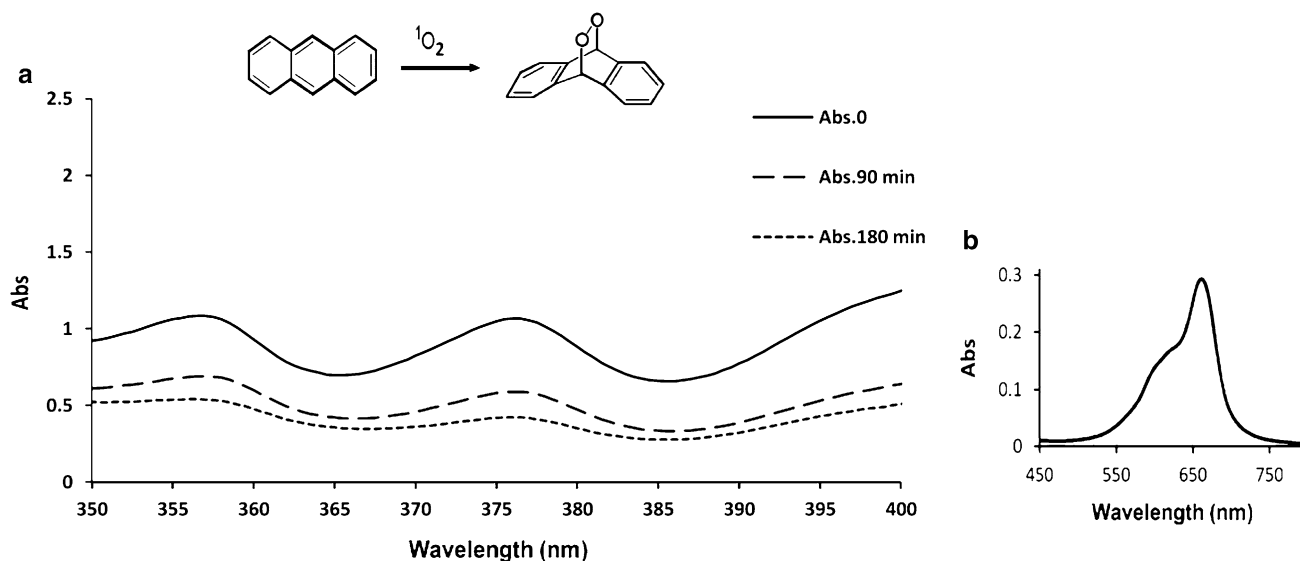
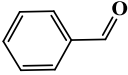
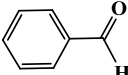
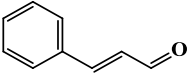
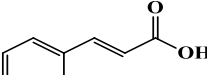
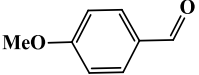
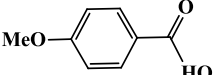
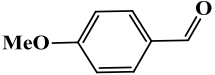
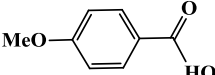
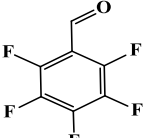
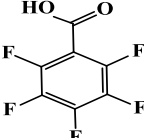
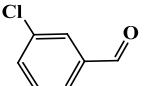
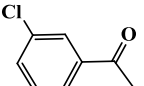
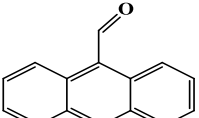
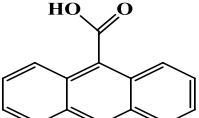
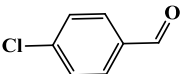
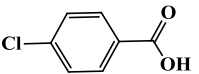
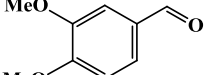
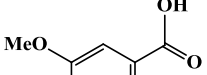
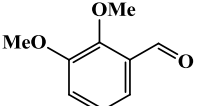
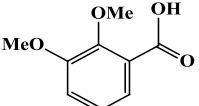


Fig. 1 **a** UV–visible spectra for the photooxygenation of anthracene ($\lambda_{\text{max}} = 375 \text{ nm}$) in the presence of CoPcS as a photosensitizer using simulated solar light (288 power LED lamps, 1 W, 2.3 V (59,660

Lux)) under 1Atm of bubbling air in acetonitrile. **b** UV–Vis spectra of CoPcS ($\lambda_{\text{max}} = 660 \text{ nm}$) before and after 180 min photooxygenation

Table 3 Oxidation of aldehydes to carboxylic acids in the presence of CoPcS^a

Entry	Aldehyde	Carboxylic acid	Yield (%)	CoPcS (mol)	TON ^b	TOF ^c (h ⁻¹)	mp (°C)	Lit. mp (°C)
1			100	7×10^{-6}	71.4	0.72	120–123	121–123 [54]
2			100	6×10^{-6}	83.3	0.55	132–135	132–134 [55]
3			100	8×10^{-6}	62.5	0.42	182–185	178–180 [54]
4 ^d			83	1×10^{-5}	41.5	0.28	182–185	178–180 [54]
5			100	7×10^{-6}	71.4	0.48	100–103	102–104 [56]
6			100	7×10^{-6}	71.4	0.48	154–157	155 [57]
7			100	8×10^{-6}	62.5	0.42	214–217	217 [58]
8			75.5	7×10^{-6}	53.9	0.36	237–240	241 [59]
9			44.4	7×10^{-6}	31.7	0.21	179–182	179–182 [54]
10			25.4	5×10^{-6}	25.4	0.17	121–124	122 [60]

^a 5×10^{-4} mol aldehyde, CoPcS, 7.5 ml CH₃CN and 7.5 ml H₂O, air (1Atm), 288 power LED lamps, 1 W, 2.3 V (59,660 LUX)

^bTurnover number of catalyst

^cTurnover frequency of catalyst

^dSunlight (91,000 LUX)

Conclusion

In conclusion, we have developed a new approach for the aerobic oxidation of aldehydes to the corresponding carboxylic acids using water-soluble photosensitizers

in the presence of white light or sunlight and O₂ in an organic–water biphasic media under very mild conditions. CoPcS was identified as the optimal catalyst for the oxidation reaction. This procedure is very simple and works efficiently at room temperature, giving good to excellent yields for a range of substrates.

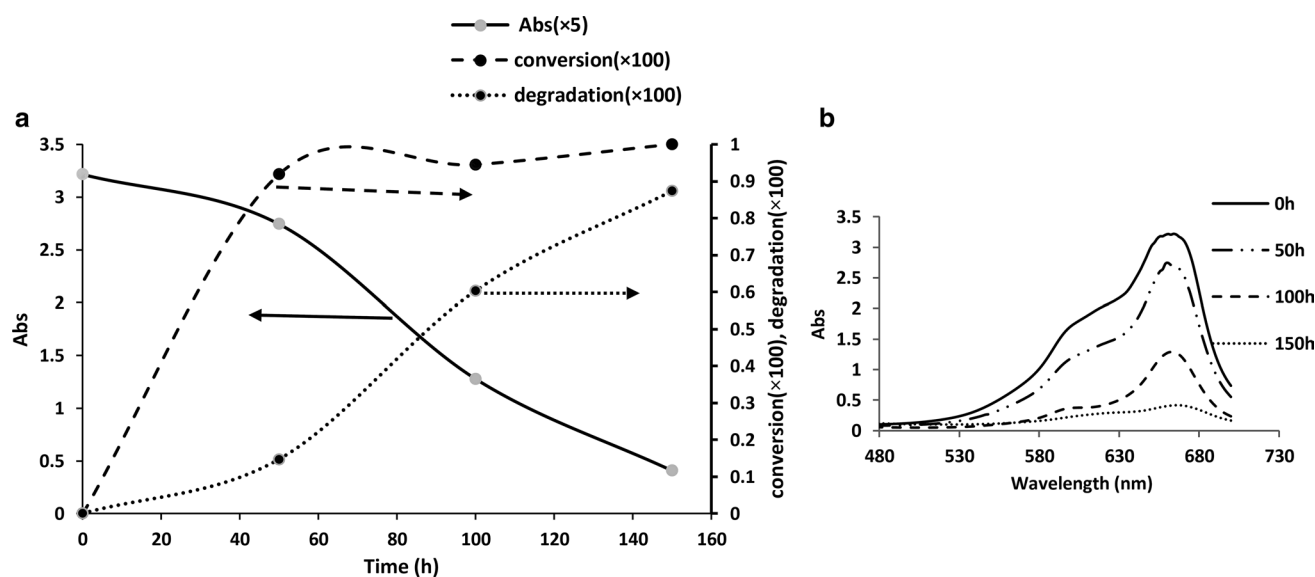
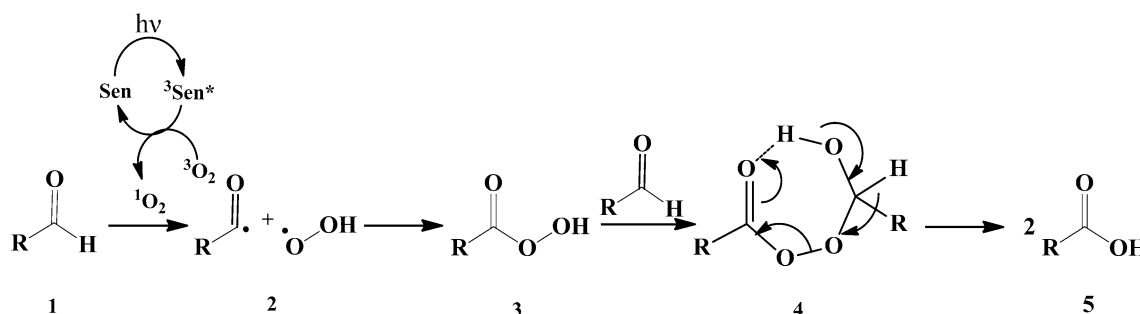


Fig. 2 a Conversion versus time for 4-methoxybenzaldehyde, degradation percent and absorption of CoPcS in 4-methoxybenzaldehyde photooxygenation. b UV–Vis spectra of CoPcS ($\lambda_{\text{max}} = 660$ nm) over time



Scheme 3 Proposed mechanism for photooxygenation of aldehydes by molecular oxygen in the presence of singlet oxygen in acetonitrile/water biphasic media

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