

Mg and Zn Complexes with Phosphonate Substituted β -Octaphenylporphyrin as Photocatalysts for Oxidation of Sulfides

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Dedicated to the Anniversary of Academician Irina P. Beletskaya

Magnesium 2,3,7,8,12,13,17,18-octa[(4-diethoxyphosphoryl)phenyl]porphyrinate (MgOPPP) was prepared in a good yield by reacting the free-base porphyrin with magnesium acetate in DMF at 130 °C. The resulting complex was used as a catalyst for the selective photocatalytic oxidation of sulfides to sulfoxides by molecular oxygen. Both dialkyl and alkyl aryl sulfides were almost quantitatively transformed into sulfoxides in an acetonitrile/water solvent mixture (5:1, v/v) under irradiation with blue LED (30 W), using a low loading of 0.05 mol% of MgOPPP as a photocatalyst. Comparative studies have shown that ZnOPPP also affords sulfoxides in quantitative yields and allows for shortening of the reaction time.

Keywords: Magnesium porphyrinate, zinc porphyrinate, photooxidation, sulfide, sulfoxide.

Фосфонатзамещенные β -октафенилпорфирилаты Mg и Zn как фотокатализаторы для окисления сульфидов

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2,3,7,8,12,13,17,18-Окта[(4-диэтоксифосфорил)фенил]порфирилат магния (MgOPPP) был получен с хорошим выходом в реакции свободного основания порфирина с ацетатом магния в ДМФА при 130 °C. Полученный комплекс использовали как катализатор для селективного фотоокисления сульфидов в сульфоксиды кислородом. Как диалкил-, так и алкиларилсульфиды дали сульфоксиды с выходами близкими к количественным при проведении реакции при облучении синим светодиодом (30 Вт) в смеси ацетонитрил/вода (5:1, об./об.) при использовании 0.05 мол.% MgOPPP в качестве фотокатализатора. Сравнительные исследования показали, что порфирилат цинка ZnOPPP позволяет сократить время проведения реакции без потери её селективности.

Ключевые слова: Порфирилат магния, порфирилат цинка, фотоокисление, сульфид, сульфоксид.

The past two decades have seen a surge in research aimed at improving photocatalysts for use in organic synthesis, in response to a growing interest in green processes and photoredox catalytic reactions involving electron transfer reactions.^[1-4] One class of photocatalysts that has been extensively studied is porphyrins and their metal complexes.^[5-7] The *meso*-tetraphenylporphyrin scaffold has been

identified long ago as a stable and convenient platform for the development of catalysts for oxidation reactions.^[8,9] However, magnesium complexes of *meso*-tetraarylporphyrins are not easy to handle due to their low chemical stability. Their behavior in photocatalytic processes^[10,11] is still not well-understood despite their high potential in electron transfer processes.^[12] Indeed, specifically magnesium complexes

with porphyrins and related macrocycles are used by plants, algae, and bacteria in photosynthesis, where the primary processes involve the transfer of electrons from these complexes to electron acceptors.^[12,13]

β -Octaarylporphyrins are tetrapyrrolic macrocycles that are of interest as ligands related to many naturally occurring β -substituted porphyrin derivatives.^[14] However, their low solubility in common organic solvents has been a limitation to their practical use. Recently, we have shown that the introduction of diethyl phosphonate groups into the aryl residues can address this problem.^[15] This opens up new possibilities for the development of complexes having practical applications in various fields, such as catalysis, bioimaging, and photodynamic therapy. In a previous study, we developed a convenient synthetic approach to electron-deficient 2,3,7,8,12,13,17,18-octa[4-(diethoxyphosphoryl)phenyl]porphyrin (H_2OPPP) and investigated physical properties and electrochemical behavior of its complexes with redox-inactive metals such as zinc(II) ($ZnOPPP$), copper(II), and nickel(II).^[15] In this work, a magnesium complex $MgOPPP$ (Figure 1) was prepared, and the photocatalytic properties of the Zn and Mg complexes were compared in photocatalyzed sulfide oxygenation.

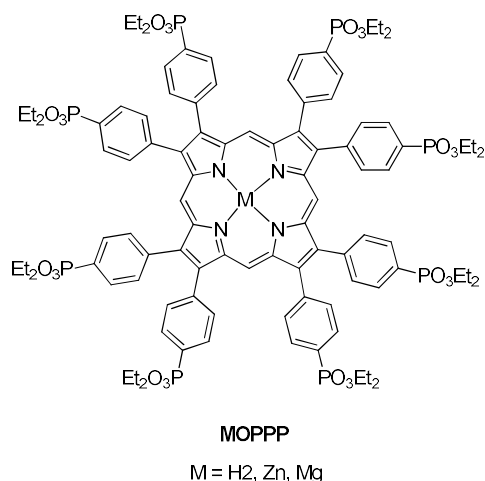


Figure 1. The structures of studied compounds.

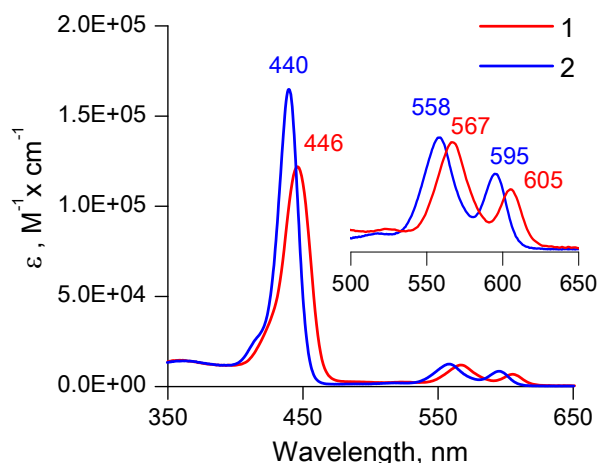


Figure 2. UV-vis spectra of $MgOPPP$ (1) and $ZnOPPP$ (2) in $CHCl_3/MeOH$ solvent mixture (2:1, v/v).

The Mg complex $MgOPPP$ was prepared by adapting published procedures.^[16] First, the free-base porphyrin H_2OPPP was reacted with magnesium acetate tetrahydrate in DMF. When the reaction was performed at 150 °C using 10 equivalents of magnesium salt, the complex was only obtained in a low yield of 15%, likely due to the instability of the phosphonate substituents in the presence of strong Lewis-acidic magnesium ions. However, by decreasing the amount of magnesium acetate to 2 equivalents and conducting the reaction at 130 °C, $MgOPPP$ was obtained in a higher yield of 67% after 10 min of reacting[#]. Attempts to increase the product yield by performing the reaction in a refluxing methanol ($MeOH$) /chloroform ($CHCl_3$) mixture were unsuccessful, as the insertion of magnesium ions was not observed under these mild conditions. The complex was found to be stable in air and could be isolated using column chromatography on silica or neutral alumina using commercial solvents without additional purification. After being dried at 25 °C and 2 mm Hg for 2 hours, the complex exhibited very low solubility in toluene, acetonitrile ($MeCN$), THF, and chlorinated solvents (CH_2Cl_2 and $CHCl_3$), likely indicating the presence of strong intermolecular interactions through coordinative bonding of the oxygen atoms of phosphonate groups to magnesium ions of nearby molecules. Solvents bearing strong Lewis base sites such as dimethylformamide, dimethyl sulfoxide, $MeOH$, or water (as a co-solvent) were found to be more efficient in solubilizing this compound. In solution, $MgOPPP$ displays UV-vis spectrum typical for metalloporphyrins with redox-inactive metal centers (Figure 2) and rather high fluorescence quantum yield even in aqueous media[§].

To investigate the photocatalytic properties of $MgOPPP$, the photooxygenation of sulfides by molecular oxygen was chosen as a model reaction due to its relevance in warfare agent disposal, fuel desulfurization, and organic synthesis.^[17-21] Selective methods for transforming different sulfides to sulfoxides are highly desirable because this reaction is commonly accompanied by side reactions such as overoxidation of sulfides to sulfones and cleavage of S-C and (S)C-H bonds.^[22-24] Porphyrins were already used as catalysts for the selective oxidation of sulfides to sulfoxides^[19,25-31] and sulfones^[25] but most of reported reactions were performed using heterogenized catalysts. It was demonstrated that in the synthesis of sulfoxides, strong oxidants can be replaced by molecular oxygen conducting the reaction under visible-light irradiation in the presence of porphyrins as photocatalysts.^[19,27,28,30]

We have chosen this oxidation as a model reaction to study the photocatalytic properties of $MgOPPP$ because we expect to not only propose new efficient photocatalysts for the sulfoxidation reaction, but also gain first sights into the photocatalytic efficiency of porphyrins belonging to the $MOPPP$ series in both electron transfer (ET) and energy transfer (EnT) reactions. The oxidation of sulfides to sulfoxides has been widely studied, and it has been shown to occur through three different mechanisms.^[32-35] The first one involves energy transfer from the excited-state photocatalyst (*PC) to an oxygen molecule (Scheme 1, pathway A),^[36,37] while the other two mechanisms involve electron transfer processes (Scheme 1, pathways B and C).^[38,39,32] Alkyl sulfides are commonly oxidized by the singlet oxygen (1O_2), while aryl sulfides tend to react through both mechanisms in the presence of most organic photocatalysts.^[24,34,40] In con-

trast, diphenylsulfide seems to react only by ET pathways.^[41] We decided to evaluate the performance of our complexes in redox and EnT photocatalytic reactions by studying the sulfoxidation of aryl sulfides and alkyl sulfides, respectively.

Recently, Cibulca *et al.* reported that the photooxygenation of sulfides is very efficient in a mixture of MeCN and water (15%).^[40] This reaction medium is inconvenient for classical *meso*-tetraphenylporphyrins due to their low solubility under these experimental conditions, but it can be successfully used with MOPPP complexes.

Our preliminary experiment was performed using thioanisole as a model substrate and 0.05 mol% of MgOPPP in a MeCN/H₂O solvent mixture under blue LED irradiation ($\lambda = 450$ nm, 30 W)[†]. These conditions were chosen because they have been reported to be optimal for sulfoxidation with Ru^{II} complexes, which are among the most efficient catalysts for this reaction.^[2] The reaction proceeded smoothly in molecular oxygen (balloon, 1 L), and the target sulfoxide was obtained in high yield after 2 h of irradiation.

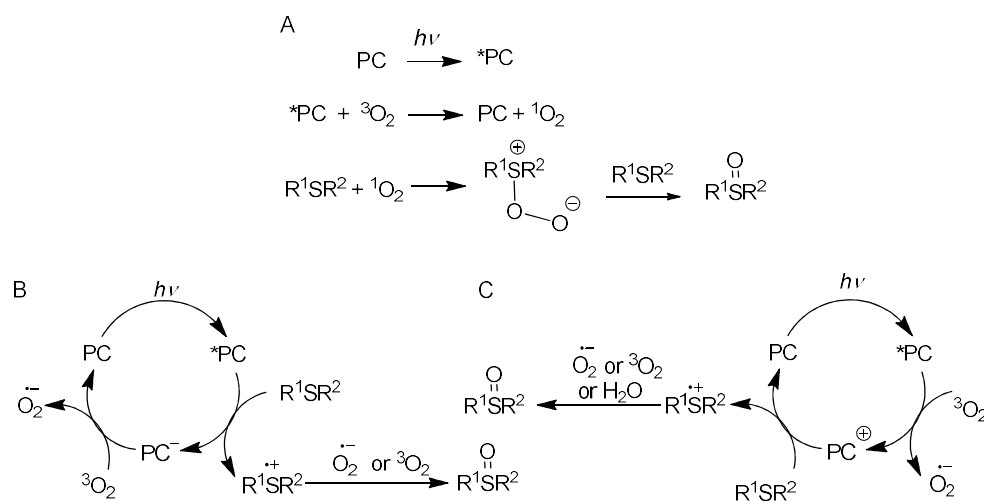
We then investigated the substrate scope and found that all types of aryl methyl sulfides were transformed to sulfoxides chemoselectively and almost quantitatively, although the reaction time varied depending on the sulfide structure (Table 1). Overoxidation was significantly suppressed, and sulfones were observed only as traces (1–2%). Sulfides bearing electron-donating groups were more active than those bearing electron-withdrawing groups (entries 1, 3, 5, and 7). The nitro-substituted derivative, which is known to be resistant in ET reactions,^[40] reacted much more slowly, and its high conversion was obtained only after 2 d of irradiation (entry 9). Bulky *ortho*-bromothioanisole was also selectively photooxidized, although after 1.5 d of irradiation (entry 11). The selectivity of the photooxygenation did not decrease even in the oxidation of benzyl phenyl sulfide, which is known to give numerous side products in the photocatalytic oxygenation (entry 13).^[34] However, diphenyl sulfide, which is unreactive towards singlet oxygen and resistant in ET reactions,^[40] could not be oxidized under these conditions (entry 15). Dibutyl sulfide was even more reactive than aryl methyl sulfides and gave the product in quantitative yield after 30 min of irradiation (entry 17). When the amount of catalyst was decreased by 10 times, this oxygenation reac-

tion was finished in 1 h (entry 18). Based on these experimental data on the reactivity of different types of sulfides, we hypothesized that photooxygenation of sulfides in the presence of MgOPPP primarily proceeds through the EnT mechanism (generation of singlet oxygen, Scheme 1A).

Next, comparative studies of MgOPPP and ZnOPPP complexes were performed under the same experimental conditions. As shown in Table 1, ZnOPPP was more efficient and gave the products in 30 min (entries 2, 4, 6, 8, 12, 14, 19, and 20) with the exception of methyl 4-nitrophenyl sulfide (entry 10) and diphenyl sulfide (entry 16). In particular, the oxidation of bulky methyl *ortho*-bromophenyl sulfide was completed in 30 min. It is also worth noting that the oxygenation of diphenyl sulfide was slow, but it was observed (entry 16), likely indicating that ZnOPPP can be efficient in the reactions proceeding through both EnT and ET mechanisms (Scheme). As in the case of MgOPPP, dibutyl sulfide was successfully oxidized in the presence of only 0.005 mol% of photocatalyst and the product was obtained after of only 30 min of reacting (entry 20).

It is worth noting that UV–vis studies of reaction mixtures before and after reaction completion have demonstrated that photobleaching of both porphyrin complexes is also observed in these mixtures.

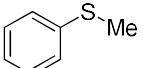
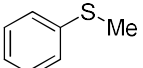
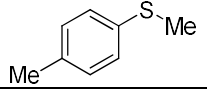
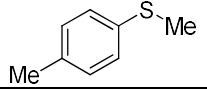
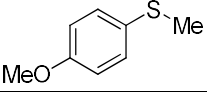
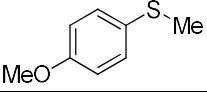
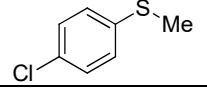
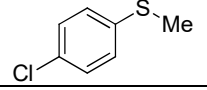
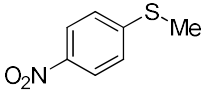
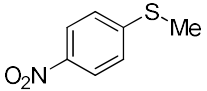
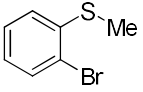
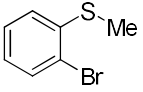
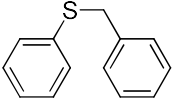
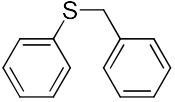
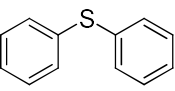
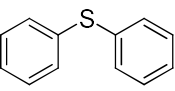
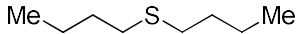
To summarize, this study aimed to develop more efficient and effective photocatalysts for organic synthesis, with a specific focus on β -octaarylporphyrins as potential candidates. The findings revealed that complexes MgOPPP and ZnOPPP proved to be efficient photocatalysts in the photooxygenation of various sulfides by molecular oxygen. Both porphyrins demonstrated excellent selectivity towards the oxidation of various sulfides to sulfoxides, with minimal over-oxidation to sulfones. However, the reaction rates were higher in the case of the zinc complex. These new photocatalysts are among the most efficient promoters reported in the literature for this reaction. While further theoretical and experimental investigations are required to better understand the value of β -octaarylporphyrins in photocatalysis, this research provides valuable preliminary data and demonstrates the potential of these porphyrins as photocatalysts. To the best of our knowledge, this is the first comparative study of Mg and Zn porphyrinates in photocatalysis.



Scheme 1. Possible mechanisms of sulfide photooxygenation proceeding through singlet oxygen generation (A) or electron transfer (B and C).

Table 1. Photooxidation of sulfides by molecular oxygen in the presence of MOPPP (M = Mg, Zn)^{a,b}.

$$\text{R}_1\text{-S-R}_2 \xrightarrow[\text{O}_2, \text{CH}_3\text{CN}/\text{H}_2\text{O (5:1), 450 nm}]{0.05 \text{ mol\% MOPPP}} \text{R}_1\text{-S(=O)-R}_2 + \text{R}_1\text{-S(=O)}_2\text{-R}_2$$

Entry	Sulfide	Catalyst	Time (h)	Conversion ^c (%)	Yield ^c (%)	
					Sulfoxide	Sulfone
1		MgOPPP	2	100	99	1
2		ZnOPPP	0.5	100	99	1
3		MgOPPP	2	100	99	1
4		ZnOPPP	0.5	100	99	1
5		MgOPPP	0.5	73		
6		ZnOPPP	0.5	100	98	2
7		MgOPPP	2.5	97	96	1
8		ZnOPPP	0.5	100	99	1
9		MgOPPP	1	18		
			24	66		
			48	91	91	0
10		ZnOPPP	1	16		
			24	58		
			48	86	86	0
11		MgOPPP	0.5	1		
			36	100	100	0
12		ZnOPPP	0.5	100	100	0
13		MgOPPP	0.5	44		2
			4	100	98	2
14		ZnOPPP	0.5	100	98	2
			48	0		
15		ZnOPPP	2	12		
16			24	36		
			48	40	39	1
17		MgOPPP	0.5	100	100	0
18		MgOPPP ^d	0.5	66		
			1	100	100	0
19		ZnOPPP	0.5	100	100	0
20		ZnOPPP ^d	0.5	100	100	0

^aReaction conditions: 0.5 mmol of sulfide, 0.05 mol% of catalyst were stirred in MeCN/H₂O mixture (5:1, v/v) under molecular oxygen (balloon, 1 L) and irradiation (blue LED (450 nm), 30 W).

^bSelected ¹H NMR spectra (CDCl₃) of sulfoxides, δ_{H} ppm: *Methyl phenyl sulfoxide*^[40] (400 MHz): 2.72 (s, 3H, CH₃), 7.50–7.58 (m, 3H, Ar), 7.63–7.72 (m, 2H, Ar). *4-Methoxyphenyl methyl sulfoxide*^[40] (300 MHz): 2.70 (s, 3H, CH₃), 3.82 (s, 3H, OCH₃), 7.04–7.10 (m, 2H, Ar), 7.59–7.68 (m, 2H, Ar). *Methyl 4-nitrophenyl sulfoxide*^[42] (300 MHz): 2.81 (s, 3H, CH₃), 7.79–7.88 (m, 2H, Ar), 8.37–8.44 (m, 2H, Ar). *Diphenyl sulfoxide*^[43] (300 MHz): 7.29–7.43 (m, 6H, Ar), 7.56 (m, 4H, Ar). *Dibutyl sulfoxide*^[40] (300 MHz): 0.96 (t, *J* = 7.3 Hz, 3H, CH₃), 1.36–1.61 (m, 2H, CH₂), 1.65–1.83 (m, 2H, CH₂), 2.60–2.78 (m, 2H, CH₂).

^cConversion and selectivity were determined by ¹H NMR analysis of reaction mixtures using biphenyl as an internal standard. NMR yields of sulfoxides and sulfones were calculated using these data.

^dThe reaction was performed using 0.005 mol% of catalyst.

Acknowledgements. This work was supported by Russian Science Foundation, Grant No. 21-73-00020.

Notes and References

[#] *Magnesium 2,3,7,8,12,13,17,18-[octa(4-(diethoxyphosphoryl)phenyl)]porphyrinate (MgOPPP)*. A solution of porphyrin H₂OPPP (20.0 mg, 0.01 mmol) in DMF (9 mL) was heated to

130 °C under Ar. To this solution, magnesium acetate tetrahydrate (20.0 mg, 0.13 mmol) and sodium acetate (20.0 mg, 0.24 mmol) in DMF (1 mL) were added by a syringe. The reaction mixture was stirred at 130 °C and monitored for completing by UV-vis spectroscopy. After 10 min of reacting and cooling to room temperature, 40 mL of chloroform was added and reaction mixture was washed by water (5×50 mL). The organic phase was evaporated to dryness under reduced

pressure. The residue was purified by column chromatography on silica gel using a mixture $\text{CHCl}_3/\text{MeOH}$ (90:10, v/v) as an eluent. Complex MgOPPP was obtained as a violet solid. Yield: 67% (13.6 mg). ^1H NMR (300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD}$ (2:1, v/v)) δ_{H} ppm: 1.47 (t, $J = 7.2$ Hz, 48H, CH_3), 4.25–4.31 (m, 32H, CH_2), 7.98–8.16 (m, 32H, o -, m - H_{Ar}), 10.23 (br.s, 4H, $meso$ -H); ^{31}P NMR (121 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD}$ (2:1, v/v)) δ_{P} ppm: 19.20; ^{13}C NMR (75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD}$ (2:1, v/v)) δ_{C} ppm: 16.1 (d, $J_{\text{CP}} = 6.0$ Hz, 16C, CH_3), 62.8 (d, $J_{\text{CP}} = 5.5$ Hz, 16C, CH_2), 104.5 (4C, $meso$ -C), 126.5 (d, $J_{\text{CP}} = 191.2$ Hz, 8C, i - C_{Ar}), 131.6 (d, $J_{\text{CP}} = 10.3$ Hz, 16C, m - C_{Ar}), 132.6 (d, $J_{\text{CP}} = 15.1$ Hz, 16C, o - C_{Ar}), 140.7 (d, $J_{\text{CP}} = 2.6$ Hz, 8C, p - C_{Ar}); 142.2 (8C, β -C), 147.0 (8C, α -C). FT-IR (neat) ν cm^{-1} : 3368 m, 2919 w, 2858 w, 1719 w, 1634 w, 1605 m, 1562 m, 1538 w, 1438 m, 1419 w, 1224 m, 1143 w, 1129 m, 1046 s, 1019 s, 991 s, 971 m, 847 w, 778 m, 661 w, 535 s. UV-vis ($\text{CHCl}_3/\text{MeOH}$ (2:1, v/v)) λ nm ($\log \epsilon$ ($\text{M}^{-1} \text{cm}^{-1}$)): 357 (4.14), 446 (5.06), 567 (3.97), 605 (3.66). HRMS (ESI): m/z calcd. for $\text{C}_{100}\text{H}_{119}\text{MgN}_4\text{O}_{24}\text{P}_8$ [$\text{M}+3\text{H}$] $^{3+}$ 677.1996, found 677.1983; calcd. for $\text{C}_{100}\text{H}_{118}\text{MgN}_4\text{NaO}_{24}\text{P}_8$ [$\text{M}+2\text{H}+\text{Na}$] $^{3+}$ 684.5269, found 684.5256; calcd. for $\text{C}_{100}\text{H}_{117}\text{MgN}_4\text{Na}_2\text{O}_{24}\text{P}_8$ [$\text{M}+\text{H}+2\text{Na}$] $^{3+}$ 691.8545, found 691.8529; calcd. for $\text{C}_{100}\text{H}_{118}\text{MgN}_4\text{O}_{24}\text{P}_8$ [$\text{M}+2\text{H}$] $^{2+}$ 1015.2960, found 1015.2938; calcd. for $\text{C}_{100}\text{H}_{117}\text{MgN}_4\text{NaO}_{24}\text{P}_8$ [$\text{M}+\text{H}+\text{Na}$] $^{2+}$ 1026.2876, found 1026.2848.

[§] Fluorescence quantum yields (Φ_{F}) were measured at 323 K by a relative method using ZnTPP ($\Phi_{\text{F}} = 3.3\%$ in acetonitrile) as a standard ($\lambda_{\text{ex}} = 550$ nm). They were found to be equal to 14% for MgOPPP and 9% for ZnOPPP in MeCN/water solvent mixture (5:1, v/v).

[¶] A glass vial equipped with a magnetic stir bar was fulfilled 3 times with molecular oxygen using vacuum- O_2 cycles. This vial was charged with 0.5 mmol of sulfide (see Table) and calculated amount of standard solution of the photocatalyst. Then, a mixture of MeCN/ H_2O (10:1, v/v) was added to obtain a solution of reagents in 3 mL of the solvent mixture. The reaction was irradiated with blue LED (450 nm) under O_2 (balloon, 1 L) in PhotoRedOxTM Box (HepatoChem, USA) photoreactor. The reaction was periodically monitored by ^1H NMR spectroscopy. When the reaction was complete, the mixture was diluted with 7 mL of water and extracted with methylene chloride (3 \times 5 mL). The combined extracts were dried over sodium sulfate and evaporated under reduced pressure at room temperature. The yield and purity of the products were determined by ^1H NMR using biphenyl as an internal standard. The oxidation of aryl sulfides was performed in the presence of 0.05 mol% of photocatalysts as shown in Table. Dialkyl sulfides were also oxidized using 0.005 mol% of photocatalysts.

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