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Metal-Free Synthesis of Sulfones and Sulfoxides through Aldehyde-Promoted Aerobic Oxidation of Sulfides

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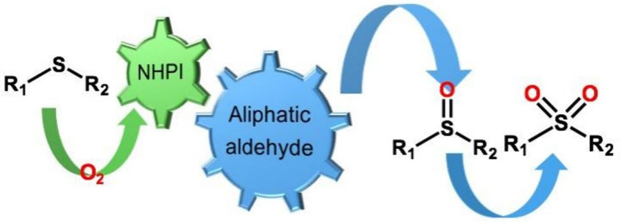
Abstract

Metal-free aerobic oxidation of aryl sulfides to sulfoxides and sulfones has been developed in the presence of aliphatic aldehydes with excellent selectivity and yields. The reaction proceeded under mild conditions with the catalysis of *N*-hydroxyphthalimide (NHPI). Control experiments indicated that the reaction underwent a free radical pathway with the acylperoxyl radicals generated from aldehydes in situ as the key intermediates. The aromatic aldehydes were less efficient in the sulfide oxidation, which might be explained by the fact that the aromatic ring dispersed the electrons of free radicals and thus weakened the attacking ability of peroxy free radicals.

Graphic Abstract



Keywords Sulfide · Aerobic oxidation · *N*-hydroxyphthalimide · Aldehyde · Radical

1 Introduction

Organic sulfones and sulfoxides are important oxygen-containing compounds that widely exist in natural products, pharmaceuticals, and flavors [1–3]. While many organic methodologies have been developed for the synthesis of sulfones and sulfoxide [4–6], the direct and selective oxidation of sulfide to sulfone and sulfoxide remains the most useful and practical method in biology and pharmaceutical industry [7–13]. Therefore, it is enticing and necessary to explore green and mild catalytic system for the controllable oxidation of sulfides, particularly under sustainable metal-free conditions with air or oxygen as the oxidant.

To date, the direct oxidation of organic substrates by the combined use of molecular oxygen and aldehyde has attracted a lot of attention [14–19]. The O_2/aldehyde/*N*-hydroxyphthalimide (NHPI) system has been developed by us and other groups for the efficient epoxidation of alkene (Scheme 1a), C–H oxidation of alkane, and Baeyer–Villiger oxidation of ketone (Scheme 1b) [20–22]. The oxidation of sulfides to sulfoxides and sulfones has also been achieved by the aldehyde–O_2 system. However, much excessive aldehydes (4 ~ 64 equiv.) were required to completely consume the sulfides as well as to provide reasonable yield and selectivity. For example, Venkateshwar Rao et al. [23, 24] reported the co-oxidation of acyclic sulfides with the

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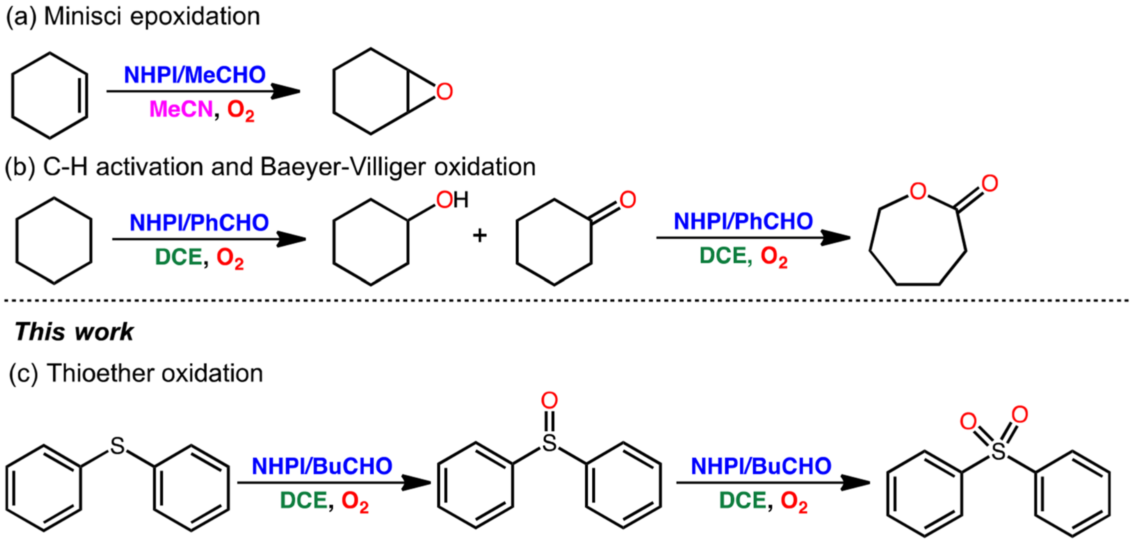


Scheme 1 Oxidation promoted by NHPI/RCHO system

aldehyde/O_2 system, where 8–64 equiv. of aldehydes were necessary. Murata et al. [25] found that the aerobic oxidation of dibenzothiophene into sulfone could be achieved under the catalysis of $Co(OAc)_2$ or $CoCl_2$ using 4 equiv. of *n*-octanal. Tada et al. [26] studied the sulfoxidation reaction using a SiO_2-supported Ru complex in the presence of O_2 and aldehydes. Our group [27] developed a recoverable photocatalytic system for the selective oxidation of sulfides to sulfoxides by utilizing O_2/isopropyl aldehyde/mpg-C_3N_4 without transition metals. Herein, we would like to report our consequent study on the mild oxidation of sulfides to sulfoxides and sulfones adopting the O_2/aldehyde/NHPI system in the absence of metal catalysts (Scheme 1c).

2 Experiments

2.1 Materials

Diphenyl sulfide (DPS) (> 99.0%), diphenyl sulfoxide (> 99.0%), diphenyl sulfone (> 99.0%), *n*-butyraldehyde (99.5%), acetaldehyde (98%), propionaldehyde (98%), isobutyraldehyde (99%), valeraldehyde (99%), 3-methyl butanal (> 99.0%) and were purchased from TCI without further purification unless indicated. 1,1,1,3,3,3-Hexafluoro-2-propanol (HFIP) (99%), trifluoroethanol (TFE) (99%), 4-phenylacetaldehyde ($PhCH_2CHO$) (98%), biphenyl (99.5%), benzaldehyde (PhCHO) (98%), 4-nitrobenzaldehyde (98%), NHPI (98%), and 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (98%), were purchased from Energy Chemical without further purification unless indicated. 1,2-Dichloroethane (DCE), dichloromethane (DCM), carbon tetrachloride (CCl_4), acetonitrile (MeCN), toluene and ethyl acetate (EtOAc) were analytical grade and purchased from J & K Scientific Ltd.

2.2 General Methods

The reaction was monitored by taking samples at various intervals using gas chromatography (GC) (Agilent 7820 A) equipped with a DB-35/ZB-35/HP-35 column (30 m × 0.32 mm × 0.25 mm) and a flame ionization detector (FID) for analysis. The conversion and yield were calculated on the basis of the peak area ratio of sulfide, sulfone and sulfoxide against the internal standard, biphenyl.

2.3 General Procedure for Sulfide Oxidation

In a typical reaction, sulfides (2 mmol), *n*-butylaldehyde (6 mmol), NHPI (5 mol%) and DCE (3 g) were placed into a three-necked round bottom flask (10 mL) fitted with an oxygen balloon and a magnetic stir bar. The reaction mixture was stirred at 40 °C for 24–36 h and monitored by GC.

2.4 General EPR Experiments

To a 50 mL three-necked round bottom glass flask equipped with a water-cooled reflux condenser, a magnetic stir bar and an oxygen balloon was added 6 mmol of *n*-butylaldehyde, 0.1 mmol NHPI and 20 mL of DCE. The mixture was stirred at 40 °C for 30 min at atmospheric pressure. The EPR spectra of the reaction solution (Fig. 2a, black) were recorded using a computer-controlled X-band (9.5 GHz) EPR spectrometer (Bruker A300). Then, 2 mmol of DPS was added into the mixture and the mixture was stirred for another 30 min. Thereafter, EPR signals were measured immediately upon addition of 2 mmol of DPS into the reaction system (Fig. 2b, black). The reaction was continued for 30 min before measuring GC (Fig. 2d, black).

To a 50 mL three-necked round bottom glass flask equipped with a water-cooled reflux condenser, a magnetic stir bar and an oxygen balloon was added 2 mmol of DPS, 6 mmol of *n*-butylaldehyde, 0.1 mmol NHPI and 20 mL of DCE. The mixture was stirred at 40 °C for 30 min at atmospheric pressure. The EPR spectra of the reaction solution (Fig. 2c, black) were obtained immediately. *n*-Butylaldehyde was replaced by *t*-butylaldehyde (red) and PhCHO (blue), respectively.

A 20 μL of reaction solutions was adopted at certain time and mixed with 5,5-dimethyl-1-pyrroline *N*-oxide (DMPO) solution (0.1 M in phosphate buffer saline) of equivalent volume. After shaking for 3 min, the mixture was injected into a capillary and tested at room temperature in the presence of *n*-butylaldehyde (Fig. 3, black) and *t*-butylaldehyde (Fig. 3, red), respectively.

3 Results and Discussion

3.1 Oxidation of DPS

DPS, 2 mmol, was initially used as a model substrate for study. The results were summarized in Table 1. The aerobic oxidation reaction was first performed with NHPI (5 mol%) and *n*-butyraldehyde (3 equiv.) in MeCN (3 g) at 40 °C. The conversion of DPS was 71% with a 98% selectivity of diphenyl sulfoxide (DPSO) and a 2% selectivity of diphenyl sulfone ($DPSO_2$) (entry 1, Table 1). It was found the reaction medium had a significant effect on the reaction efficiency. Among the solvents of MeCN, toluene, EtOAc, TFE, HFIP, CCl_4, DCM and DCE, DCE was the most effective for the reaction (entries 1–7, 10). Reducing the DCE amount to 1 or 2 g, the yield of $DPSO_2$ decreased to 23% and 34%, respectively (entries 8, 9). The yield of $DPSO_2$ remained unchanged when the DCE amount increased to 4 or 5 g (entries 11, 12). The dual decrease of temperature to 25 °C and reaction time to 6 h resulted in the increase of DPSO (91% yield) but the decrease of $DPSO_2$ (9% yield) (entry 13). Prolonging the reaction time to 24 h, the yield of DPSO decreased to 61% while the yield of $DPSO_2$ increased to 39% (entry 14). When a blank reaction was carried out without NHPI or *n*-butyraldehyde, the reaction hardly proceeded (entry 15). Thus, it is likely that NHPI/*n*-butyraldehyde system had a great impact on the oxidation of sulfide. *n*-Butyraldehyde as the sacrifice was vital to the reaction. Reducing the amount of *n*-butyraldehyde led to a decrease of $DPSO_2$ yield but an increase of DPSO selectivity in the presence or absence of NHPI (entries 16–19).

The effects of different aldehydes on the NHPI-promoted oxidation of DPS were investigated. (Table 2). Among the aldehydes studied, *n*-butyraldehyde and propionaldehyde were found with the most positive effect, leading to the complete oxidation to sulfone $DPSO_2$ at 40 °C within 24 h (entries 1 and 4). Acetaldehyde was less efficient. Only DPSO was detected in the presence of acetaldehyde under the same catalytic conditions with a conversion of 3% for 24 h and 15% for 52 h (entries 2, 3). Branched aliphatic aldehydes, such as isobutyraldehyde and 3-methyl butanal, were more favorable for the formation of DPSO (entries 5, 7) while linear aliphatic aldehyde valeraldehyde offered the $DPSO_2$ as the major product (entry 6). It is interesting to note that aromatic aldehydes were inactive in the sulfide oxidation reaction system (entries 8–11). This result was opposite to the trend in the Baeyer–Villiger oxidation reaction with the O_2/aldehyde/NHPI system [20]. To verify whether the carbonyl group conjugated with an aromatic ring had a negative effect on the reaction, we took $PhCH_2CHO$ as a sacrifice to carry out same the experiment. The results suggested that $PhCH_2CHO$ was effective for sulfoxidation and even better than acetaldehyde (entries 12, 13). This indicated that only aromatic ring directly conjugated with the carbonyl group weakened the activity of aldehyde. Notably, *n*-butyraldehyde is a highly cost-effective aldehyde and the conversion of *n*-butyraldehyde into the corresponding acid is a value increasing process. Considering the economic efficiency, it is very promising to use *n*-butyraldehyde as a candidate of the sacrificial materials in the practical application.

3.2 Substrate Scope of Sulfone and Sulfoxide

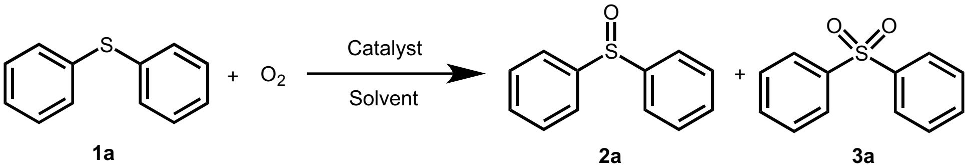
With the optimal reaction conditions in hand, the scope of sulfide oxidation was investigated. A plenty of substituted sulfides were tested using the standardized reaction conditions. The results were summarized in Table 3. Both aliphatic and aromatic sulfides were efficiently converted into sulfones with nearly quantitative yield in 24–36 h.

3.3 Mechanistic Studies

The time-course experiments of the diphenyl sulfide oxidation at different temperatures were conducted with the results shown in Fig. 1.

The results at 40 °C indicated that formation of the oxidation products increased with time (Fig. 1a). With the conversion of diphenyl sulfide, the selectivity of sulfoxide gradually decreased, while the selectivity of sulfone gradually increased. Almost all of the diphenyl sulfide was consumed after 2.5 h with similar selectivity of sulfoxide and sulfone. After 24 h, all the sulfoxide and aldehyde were converted to sulfone and acid, respectively. The reaction was much more slowly at 25 °C (Fig. 1b). After 24 h of the reaction, the final

Table 1 Optimization of conditions



Entry	Solvent (g)	Temp. (°C)	Time (h)	Conv.[a]		Select.[a]	
				Aldehyde (%)	1a (%)	2a (%)	3a (%)
1	MeCN (3)	40	24	42	71	98	2
2	Toluene (3)	40	24	76	94	94	4
3	EtOAc (3)	40	24	81	100	27	70
4	TFE (3)	40	24	20	12	98	0
5	HFIP (3)	40	24	Trace	Trace	N.D	N.D
6	CCl_4 (3)	40	24	26	17	52	6
7	DCM (3)	40	24	66	100	73	27
8	DCE (1)	40	24	62	100	77	23
9	DCE (2)	40	24	74	100	66	34
10	DCE (3)	40	24	100	100	0	100
11	DCE (4)	40	24	100	100	0	100
12	DCE (5)	40	24	100	100	0	100
13	DCE (3)	25	6	55	100	91	9
14	DCE (3)	25	24	76	100	61	39
15[b]	DCE (3)	25	24	Trace	Trace	N.D	N.D
16[c]	DCE (3)	40	24	100	100	73	25
17[d]	DCE (3)	40	24	100	66	91	9
18[e]	DCE (3)	40	24	30	59	95	4
19[f]	DCE (3)	40	24	27	18	99	1

Conditions: diphenyl sulfide (1a) (2 mmol), NHPI (5 mol%), *n*-butyraldehyde (3 equiv.), solvent, O_2 balloon

[a] Determined by GC chromatography

[b] Without NHPI and *n*-butyraldehyde

[c] *n*-butyraldehyde (2 equiv.)

[d] *n*-butyraldehyde (1 equiv.)

[e] Without NHPI, *n*-butyraldehyde (2 equiv.)

[f] Without NHPI, *n*-butyraldehyde (1 equiv.)

yields of sulfoxide and sulfone were 61% and 39%, respectively. And the conversion of aldehyde was 76%.

3.3.1 Control Experiments

To give some insight into the reaction mechanism of the oxidation procedure, several control experiments were conducted. The addition of 10 mol% of the generally used radical scavenger, TEMPO, nearly completely suppressed the model reaction, which supported the free radical reaction pathway [8, 28] (Scheme 2a). To track the oxygen source, the model reaction was conducted under an $^{18}O_2$ atmosphere. All the products were the corresponding ^{18}O-labeled oxidative products 2a and 3a. And the conversion of reactant was 100% with the selectivities of corresponding ^{18}O-labeled oxidative products in 52% and 48%, respectively (Scheme 2b). This indicated that the oxygen atom of the products originated from molecular oxygen. Under standard conditions, DPSO could be transformed into $DPSO_2$ in > 99% yield, suggesting that the generation of sulfones underwent the sulfoxide intermediate (Scheme 2c).

3.3.2 EPR Evidence

A series of EPR experiments were conducted to reveal the radicals involved in the reaction. As benzoylperoxyl radical

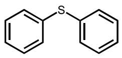
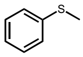
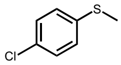
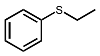
Table 2 Effect of different aldehydes in the oxidation of diphenyl sulfide

Entry	Aldehyde	Temp. (°C)	Time (h)	Conv.[a] (%)	Select.[a]	
					2a (%)	3a (%)
1	*n*-Butyraldehyde	40	24	100	0	> 99
2	Acetaldehyde	40	24	3	> 99	0
3	Acetaldehyde	40	52	15	> 99	0
4	Propionaldehyde	40	24	100	0	> 99
5	Isobutyraldehyde	40	24	99	88	10
6	Valeraldehyde	40	24	100	4	96
7	3-Methyl butanal	40	24	100	56	44
8	Benzaldehyde	40	24	Trace	N.D	N.D
9	4-Chlorbenzaldehyde	40	24	Trace	N.D	N.D
10	4-Nitrobenzaldehyde	40	24	Trace	N.D	N.D
11	4-Hydroxybenzaldehyde	40	24	Trace	N.D	N.D
12	Phenylacetaldehyde	40	24	85	> 99	0
13	Phenylacetaldehyde	40	48	100	87	13

Conditions: diphenyl sulfide (1a) (2 mmol), NHPI (5 mol%), aldehyde (3 equiv.), Solvent, O_2 balloon

[a] Determined by GC chromatography

Table 3 Oxidation of organic sulfides

Entry	Substrates	Time	Conv. (%)	Select. (%) Sulfone
1		24	100	> 99
2		24	100	> 99
3		36	100	> 99
4		36	100	> 99

could oxidize NHPI to PINO, there was a strong radical signal from PINO (A = 4.72 G, g = 2.0069) when combining PhCHO and NHPI. Without DPS, the signal intensity of PINO under the reaction conditions of different aldehydes was in the order of PhCHO > *n*-butyraldehyde > isobutyraldehyde (Fig. 2a). After adding DPS into the solution, the PINO signal immediately vanished (Fig. 2b). By the preheating method, the conversion of DPS at *t* = 30 min with *n*-butyraldehyde, isobutyraldehyde and PhCHO was 45%, 31% and 41%, respectively (Fig. 2d). This result was different from that obtained from the typical procedure used for sulfide oxidation, where PhCHO was not effective at all. Such phenomenon suggested that the oxidation of aromatic aldehyde was hampered by sulfides while preheating aromatic aldehyde for some time could initiate the aromatic acyl radicals, which can be used for promoting the oxidation of sulfide. When sulfide was added at the beginning of the reaction, the PINO signal could not be detected by EPR after the reactant was heated and stirred for 30 min (Fig. 2c).

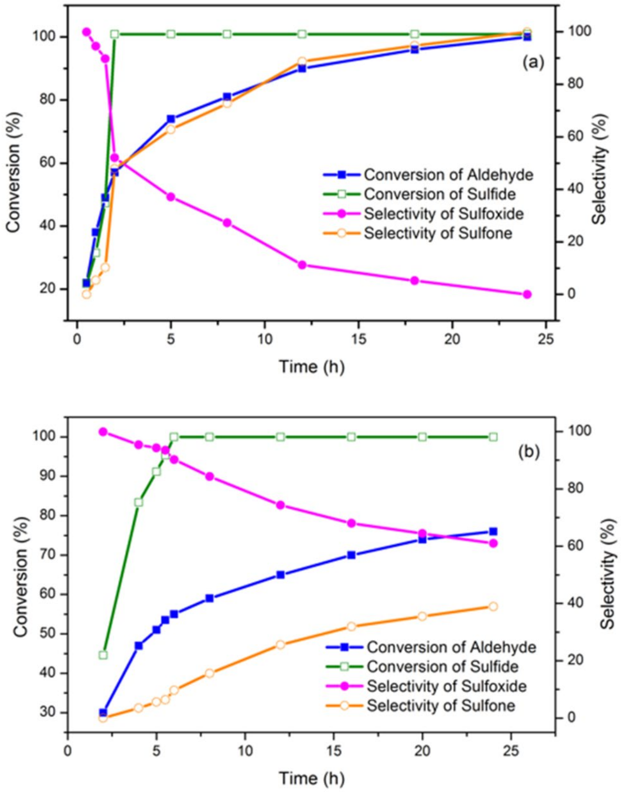
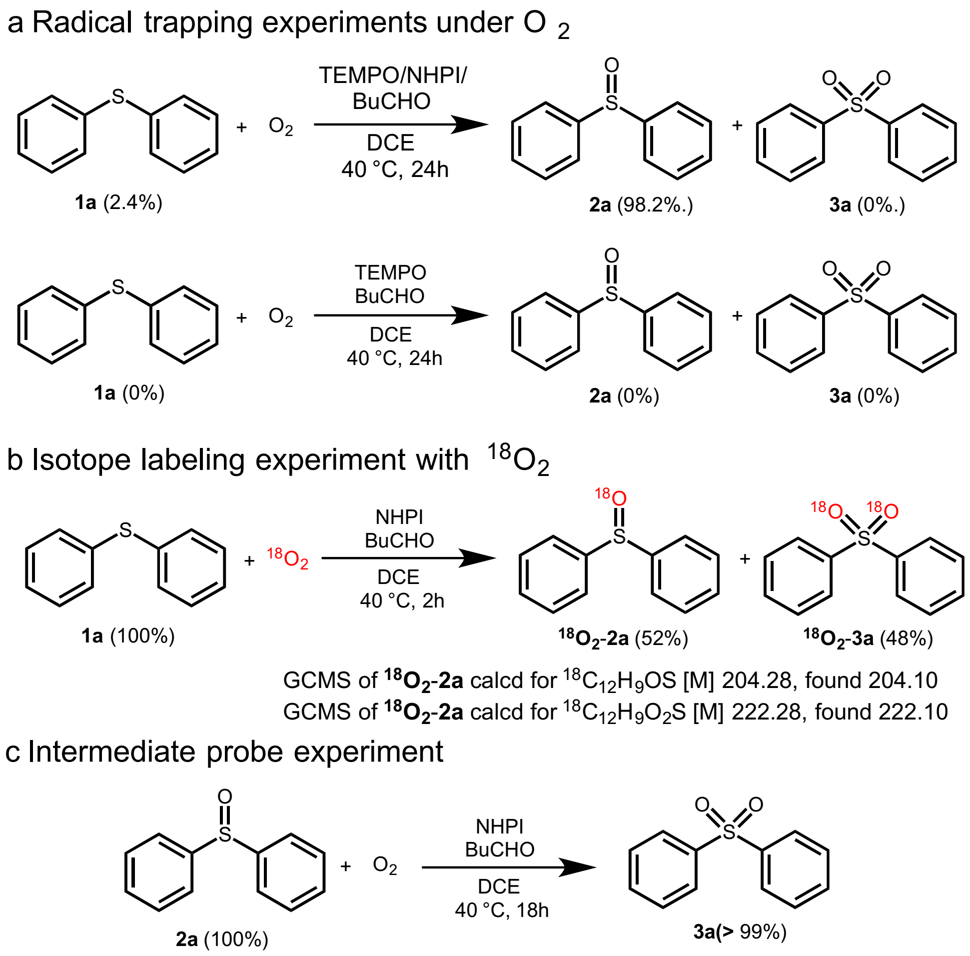


Fig. 1 Time-course of the diphenyl sulfide oxidation with NHPI in DCE. **a** 40 °C; **b** 25 °C

To gain more direct evidences of the existence of radical intermediates, the EPR experiments with the assistance of spin trap technique using DMPO as the spin trap agent were conducted [29]. As shown in Fig. 3, a clear signal

Scheme 2 Control experiments



of DMPOX [g = 2.007, a_N = 6.8 G, a_H = 3.9 G (2H)] that was oxidized by the peroxy radical in the presence of *n*-butyraldehyde was observed. Meanwhile, a weak triplet signal (g = 2.005, a_N = 14.4 G, $a_{H\gamma}$ = 1.5 G) was found when the addition of DMPO was performed in the presence of *t*-butyraldehyde (Fig. 3, red line).

We speculated that this radical species may be the double adducts of DMPO and isobutyryl radical. It was inferred that the linear fatty aldehyde was more active than the branched.

Based on the above experimental results as well as the previous literature, a possible radical chain mechanism pathway was proposed in Fig. 4. First of all, the aldehyde was initiated by the PINO hydrogen abstraction to form an acyl radical (I), which readily reacted with O_2 to produce an acylperoxyl radical (II). The acylperoxyl radical (II) formed in situ acylperoxyacid (III) that attacked the sulfide with NHPI to generate the adduct intermediate (IV). Through the rearrangement, the intermediate (IV) converted to intermediate (V) which undergo O–O bond dissociation to produce the sulfoxide and acylacid. And the sulfoxide would be further oxidized to the sulfone with oxygen.

4 Conclusions

In conclusion, we have developed a facile and green methodology for the selective synthesis of sulfones and sulfoxide by aerobic oxidation of sulfide in the presence of NHPI and *n*-butyraldehyde. The simplicity in operation, mild conditions, applicability make this procedure highly attractive. Moreover, *n*-butyraldehyde is very cost effective and the conversion into the corresponding acid is also a value increasing process. Therefore, it is promising to use *n*-butyraldehyde as a potential sacrificial material for practical applications in organic synthesis and chemical industry.

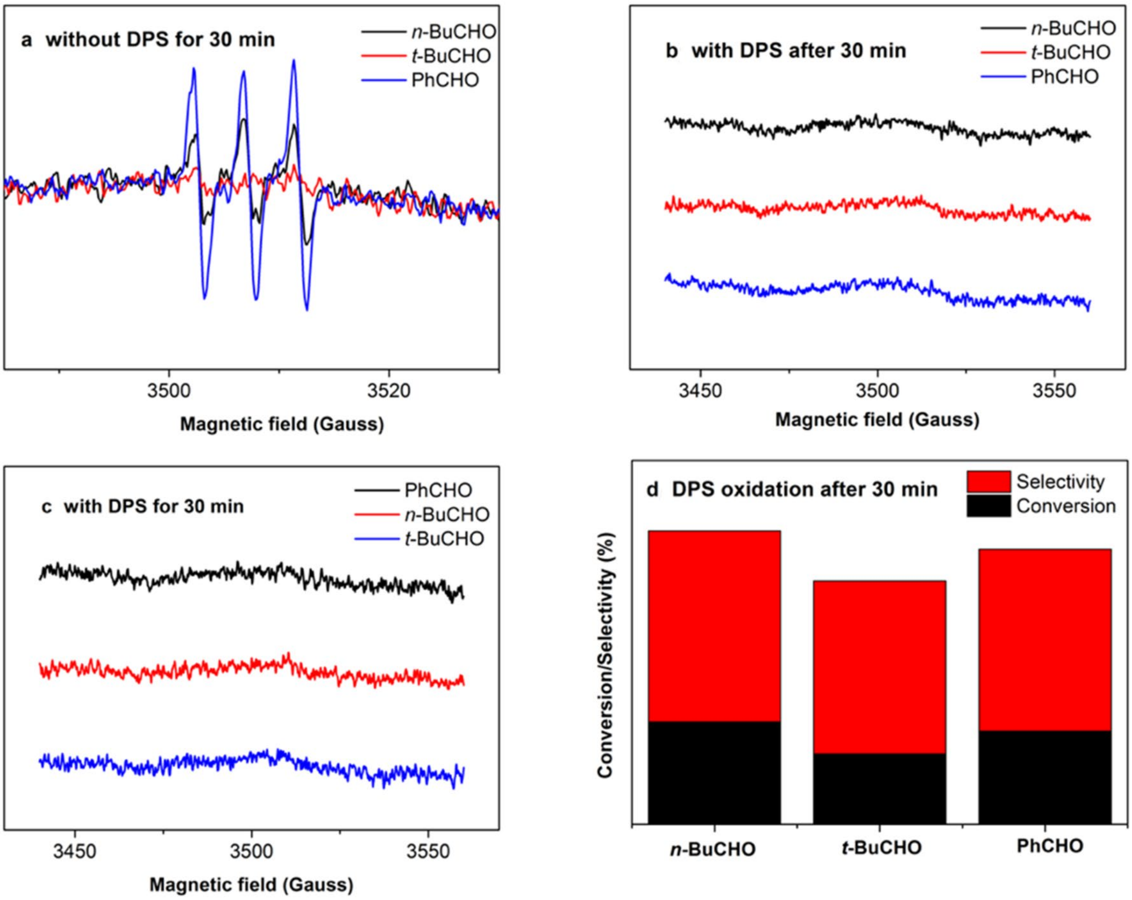


Fig. 2 EPR spectra of the mixture of NHPI and *n*-BuCHO (black), *t*-BuCHO (red) and PhCHO (blue): **a** in the absence of DPS for 30 min; **b** adding DPS after 30 min; **c** in the presence of DPS for 30 min. **d** DPS oxidation results after (**b**) for 30 min

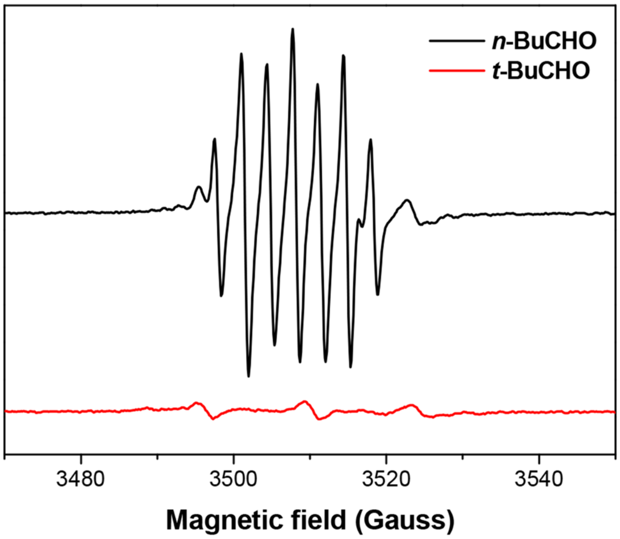
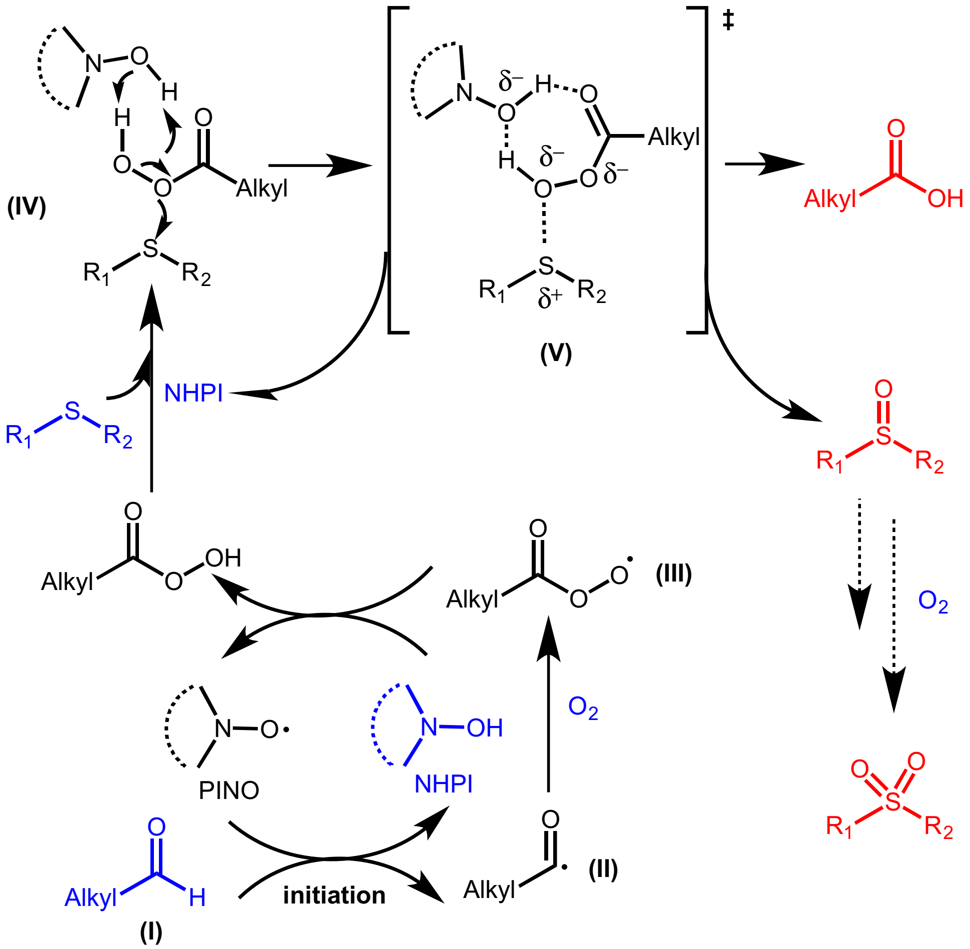


Fig. 3 EPR spectrum of the DMPOX species (black) and the DMPO-double adduct (red)

Fig. 4 Proposed mechanism



Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s10562-021-03706-5.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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