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Effect of Organic Solvents on the Rate of Oxidation of Sulfoxides with Peroxy Acids

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Abstract—The reaction of sulfoxides with peroxy acids in various organic media was studied. The reaction mechanism involves the rapid formation of a sulfoxide–peroxy acid intermediate which decomposes in the second stage to form carboxylic acid and the corresponding sulfone. The second stage is the rate-limiting step. The reaction medium significantly affects the rate of oxidation. The calculated activation parameters of the oxidation process indicate a compensation effect in the investigated reaction. Correlations between the main physicochemical parameters of solvents and the effective rate constants (k) of dimethyl sulfoxide oxidation with peroxy acids were found. Depending on the reaction conditions, the main factors affecting the k values are specific and nonspecific solvation of the reactants and structural factors.

Keywords: sulfoxides, peroxy acids, influence of solvents, correlation equations

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Oxidation of sulfides to sulfoxides with peroxy compounds is widely used in practice [1–3]. In turn, sulfoxides can be oxidized to sulfones. In most cases, hydroxide peroxide is used as oxidant, and sometimes catalytic systems consisting of hydrogen peroxide and an appropriate catalyst are utilized [4, 5]. Peroxy acids have a number of advantages over traditional oxidants in the oxidation of organic sulfides, nitrogen-containing aromatics, and ethylene derivatives [6–8]. The oxidation of sulfoxides with peroxy acids provides higher yields of sulfones. The solvent used as reaction medium affects both the oxidation rate and product yield. Oxidation of sulfoxides with various oxidants such as benzoyl peroxide and hydrogen peroxide was reported [9–11]. The oxidation of sulfoxides with peroxy compounds was shown [12–14] to proceed as a typical electrophilic reaction. The role of solvent in such processes has been poorly studied, though the solvent should be expected to affect both reaction rate and yield.

In this work we used dimethyl sulfoxide, dibutyl sulfoxide, and diphenyl sulfoxide as substrates, and peroxydecanooc, peroxynonanoic, peroxybenzoic, peroxydodecanoic, and peroxypentanoic acids were taken as oxidants. Preliminary kinetic experiments showed the first order of the reaction in each component and overall second-order kinetics. The oxidation process involves two stages. The first stage is fast formation of a sulfoxide–peroxy acid intermediate which decomposes in the second stage to give the corresponding acid and sulfone. The first stage is reversible (K_{eq}), while the second stage is rate-determining (k_{ef}). The solvent affects both stages of the oxidation process.

Table 1 contains the rate constants k for the oxidation of sulfoxides with peroxy acids, which were determined in the initial period of the process. It is seen that neither sulfoxide nor peroxy acid nature affects the reaction rate to an appreciable extent. The apparent energies of activation (E_a) are similar, and they range from 36.0 to 40.2 kJ/mol for the reactions in ethyl acetate. The kinetics of oxidation of dimethyl sulfoxide (DMSO) with peroxydecanoic acid were studied in 11 solvents in the temperature range 298–318 K. The highest reaction rate at 298 K was observed in benzene, whereas the rate constant k in acetic acid was lower by approximately an

Table 1. Effective rate constants for the oxidation of sulfoxides with peroxy acids in ethyl acetate

Sulfoxide	Peroxy acid	$k\times10^3$,[a] L mol^{-1} s^{-1}			E_a,[b] kJ/mol
		298 K	308 K	318 K	
DMSO	Peroxydecanoic acid	29.8	48.0	80.0	39.4
DMSO	Peroxynonanoic acid	29.2	46.6	78.4	39.5
DMSO	Peroxybenzoic acid	28.3	45.6	75.2	39.2
DMSO	Peroxypentanoic acid	29.5	47.5	72.2	36.0
Dibutyl sulfoxide	Peroxydecanoic acid	30.1	48.5	81.6	40,2
Diphenyl sulfoxide	Peroxydecanoic acid	28.0	45.1	76.0	40.1
Diphenyl sulfoxide	Peroxybenzoic acid	26.8	44.2	71.2	39.3
DMSO	Peroxydodecanoic acid	28.3	45.2	75.2	39.4

[a] $\Delta k = \pm0.03k$.

[b] The E_a values were determined with an accuracy of ±5.0 kJ/mol.

Table 2. Effective rate constants for the oxidation of DMSO with peroxydecanoic acid in different solvents at different temperatures

Solvent	$k\times10^3$,[a] L mol^{-1} s^{-1}				
	298 K	303 K	308 K	313 K	318 K
Acetic acid	5.24	7.08	9.70	13.5	16.3
Propan-2-ol	8.46	12.5	17.4	23.0	36.8
Dimethylformamide	18.9	24.4	33.0	43.8	54.6
Acetone	27.2	35.6	45.2	56.0	66.6
Chloroform	27.8	37.2	51.6	68.6	97.8
Ethyl acetate	29.8	37.6	48.0	64.0	80.0
1,2-Dichloroethane	33.8	45.0	62.8	–	107
Nitrobenzene	35.2	48.4	69.4	98.4	133
Carbon tetrachloride	38.4	52.0	68.0	98.4	130
Chlorobenzene	40.4	57.8	78.8	109	152
1,4-Dioxane	46.8	53.2	71.8	94.6	121
Benzene	55.2	68.0	87.6	106	145

[a] $\Delta k = \pm0.03k$.

order of magnitude (Table 2). Our results suggest that the products do not influence the reaction rate.

It should be noted that in our experiments peroxydecanoic acid was not consumed for side reactions. This is very consistent with our previous data [15, 16] according to which thermal decomposition of peroxy acids in organic solvents begins at a considerably higher temperature.

The apparent energies of activation and transition state parameters were calculated from the temperature dependences of the effective oxidation rate constants (Table 3). The apparent energies of activation (*E*) for the oxidation of DMSO in the examined solvents vary from 31.6 to 57.1 kJ/mol and are close to the reported values for the oxidation of nitrogen-containing heterocyclic compounds, epoxidation of olefins, and oxidation of sulfur-containing compounds [9, 17–19]. The mechanism of reactions of these compounds with peroxy acids is similar.

The $\Delta H^{\neq}$ and $\Delta S^{\neq}$ values for the transition state are related to each other, indicating a compensation effect. The linear relation $\Delta H^{\neq} = f(\Delta S^{\neq})$ suggests a complicated character of the solvent influence on the rate of oxidation of DMSO with peroxydecanoic acid. The isokinetic temperature was estimated at 323±5 K, which is slightly beyond the experimental temperature range. It should be noted that the rates of oxidation of sulfides with peroxy acids are significantly higher than the rates of oxidation of sulfoxides. Depending on the conditions, the oxidation rate constant of bis(4-chlorophenyl) sulfide with peroxybenzoic ranges from 0.5 to 1.0 L mol^{-1} s^{-1} [18], whereas the rate constants determined in our experiments varied from 5.24×10^{-3} to 55.2×10^{-3} L mol^{-1} s^{-1} at 298 K (Table 2).

Table 3. Activation parameters of the oxidation of dimethyl sulfoxide with peroxydecanoic acid in different solvents[a]

Solvent	E_a,[b] kJ/mol	$\Delta H^{\neq}$, kJ/mol	$-\Delta S^{\neq}$, J mol^{-1} K^{-1}	$\Delta G^{\neq}$, kJ/mol
Acetic acid	44.9	42.4	146	85.8
Propan-2-ol	53.9	51.4	112	84.6
Dimethylformamide	43.2	40.7	141	82.7
Acetone	36.1	33.6	162	81.8
Chloroform	48.0	45.5	121	81.7
Ethyl acetate	39.4	36.9	150	81.5
1,2-Dichloroethane	46.7	44.2	124	81.2
Nitrobenzene	52.2	49.7	105	81.1
Carbon tetrachloride	44.7	42.2	130	80.9
Chlorobenzene	52.3	49.8	104	80.8
1,4-Dioxane	42.7	40.2	135	80.4
Benzene	37.7	35.2	150	80.0

[a] The $\Delta H^{\neq}$, $\Delta S^{\neq}$, and $\Delta G^{\neq}$ values were calculated for a temperature of 308 K.
[b] The E_a values were determined with an accuracy of ±5.0 kJ/mol.

The rate constants for the oxidation of sulfoxides are comparable to those found for the oxidation of olefins with peroxy acids [17]. According to the data of [19], the effective rate constants for the oxidation of olefins with peroxybenzoic acid in various solvents range from 9.0×10^{-3} to 0.3×10^{-3} L mol^{-1} s^{-1}. These values are close to those found by us for the oxidation of DMSO with peroxydecanoic acid. The rates of the reactions of peroxydecanoic acid with α-pinene and Δ^3-carene in various organic solvents also approached the rates of oxidation of DMSO with peroxy acids [17].

We used the known six-parameter correlation equation (1) [16, 20] to find correlations between the oxidation rate constants (k) and transition state parameters, on the one hand, and solvent parameters, on the other.

$$\log k = a_0 + a_1(n^2 - 1)/(n^2 + 2) + a_2(\varepsilon - 1)/(2\varepsilon + 1) + a_3B + a_4E_T + a_5\delta^2 + a_6V_M. \quad (1)$$

Here n and ε are, respectively, refractive index and dielectric constant of a solvent, which characterize its polarizability and polarity that are factors responsible for nonspecific solvation. The Pal'm basicity parameter B [21] and Reichardt electrophilicity parameter E_T [22] characterize acid–base properties of a solvent, i.e., its specific solvation ability. The effect of structural factors is characterized by the Hildebrand parameter δ (δ^2 is proportional to the cohesive energy density of a solvent) and molar volume V_M. The solvent parameters were taken from [23, 24], and the results are presented according to [25].

On the basis of Eq. (1) we obtained six-parameter correlation (2) between the effective rate constants for the oxidation of DMSO at 298 K and solvent parameters. The multiple correlation coefficient R was equal to 0.9780 which corresponds to a good correlation.

$$\log k = 3.0531 + (5.7263\pm1.5554)f(n^2) + (1.6635\pm0.3679)f(\varepsilon) + (0.0016\pm0.0005)B - (0.0016\pm0.0005)E_T - (0.0018\pm0.0007)\delta^2 - (0.0082\pm0.0031)V_M; \quad (2)$$
$$N = 12,\ R = 0.9780,\ S = \pm0.0633,\ F = 75.8515.$$

Here, N is the number of solvents, S is Student's t test, and F is Fisher's test. The pair correlation coefficients (r_i) are 0.5929, –0.4662, –0.4133, –0.9155, –0.5104, and 0.742, respectively. Analysis of Eq. (2) shows that the parameters V_M and δ^2 are insignificant. Successive removal of these parameters slightly reduced the R value to 0.9640 and 0.9542, respectively. As a result, four-parameter correlation (3) was obtained:

$$\log k = 3.0387 + (1.4182\pm1.1635)f(n^2) + (0.9100\pm0.4136)f(\varepsilon) + (0.0002\pm0.0004)B - (0.0584\pm0.0080)E_T; \quad (3)$$
$$N = 12,\ R = 0.9542,\ S = \pm0.0908,\ F = 36.8334.$$

From the kinetic data obtained at 303 K, we constructed correlation (4) with a multiple correlation coefficient of 0.9798:

$$\log k = 3.0299 + (4.8813\pm1.4321)f(n^2) + (1.6387\pm0.3388)f(\varepsilon) + (0.0011\pm0.0005)B - (0.0592\pm0.0085)E_T - (0.0014\pm0.0006)\delta^2 - (0.0064\pm0.0029)V_M; \quad (4)$$
$$N = 12,\ R = 0.9798,\ S = \pm0.0582,\ F = 19.9728.$$

The pair correlation coefficients (r_i) for Eq. (4) are 0.6307, –0.4321, –0.4570, –0.9101, –0.5059, and 0.7740, respectively.

Exclusion of insignificant parameters B, δ^2, V_M, and $f(n)$ gave two-parameter correlation (5) with $R = 0.9545$:

$$\log k = 3.5948 + (1.2007\pm0.3748)f(\varepsilon) - (0.0620\pm0.0065)E_T; \quad (5)$$
$$N = 12,\ R = 0.9545,\ S = \pm0.0868,\ F = 38.6580.$$

Likewise, the effective rate constants measured at 308 K were approximated by six-parameter correlation (6) with a multiple correlation coefficient R of 0.9758, which indicates a good correlation.

$$\log k = 2.9813 + (5.0421\pm1.5441)f(n^2) + (1.5969\pm0.3653)f(\varepsilon) + (0.0010\pm0.0005)B - (0.0569\pm0.0092)E_T - (0.0013\pm0.0007)\delta^2 - (0.0062\pm0.0031)V_M; \quad (6)$$
$$N = 12,\ R = 0.9758,\ S = 0.0628,\ F = 16.6142.$$

The pair correlation coefficients (r_i) are 0.6607, –0.4148, –0.4698, –0.8968, –0.4853, and 0.7805, respectively.

Analysis of Eq. (6) showed that the solvent parameters B, δ^2, and V_M insignificantly affect the oxidation of DMSO. After exclusion of these parameters, we arrived at three-parameter correlation (7):

$$\log k = 2.9583 + (1.8267\pm0.8705)f(n^2) + (1.0478\pm0.3528)f(\varepsilon) - (0.0535\pm0.0070)E_T; \quad (7)$$
$$N = 12,\ R = 0.9614,\ S = \pm0.0791,\ F = 45.9270.$$

Six-parameter correlation (8) obtained for the effective rate constants of DMSO oxidation at 313 K was characterized by R = 0.9842, which corresponds to a good correlation:

$$\log k = 2.7883 + (4.8335\pm1.2367)f(n^2) + (1.3905\pm0.2935)f(\varepsilon) + (0.0009\pm0.0004)B - (0.0513\pm0.0074)E_T - (0.0012\pm0.0005)\delta^2 - (0.0040\pm0.0025)V_M; \quad (8)$$
$$N = 12,\ R = 0.9842,\ S = \pm0.0503,\ F = 112.2226.$$

The pair correlation coefficients (r_i) are 0.6899, –0.4237, –0.4874, –0.9061, –0.4875, and 0.8156, respectively. The parameters B, δ^2, V_M, and $f(n)$ almost do not affect the DMSO oxidation process at 313 K. Exclusion of these parameters from correlation gave two-parameter Eq. (9) with a multiple correlation coefficient R of 0.9532.

$$\log k = 3.7918 + (1.2036\pm0.3708)f(\varepsilon) - (0.0606\pm0.0065)E_T; \quad (9)$$
$$N = 12,\ R = 0.9532,\ S = \pm0.8532,\ F = 44.7247.$$

From the kinetic data for the oxidation of DMSO at 318 K we obtained correlation (10) with R = 0.9712:

$$\log k = 2.9568 + (3.5706\pm1.6915)f(n^2) + (1.1538\pm0.4001)f(\varepsilon) + (0.0003\pm0.0006)B - (0.0509\pm0.0101)E_T - (0.0004\pm0.0007)\delta^2 - (0.0019\pm0.0034)V_M; \quad (10)$$
$$N = 12,\ R = 0.9712,\ S = \pm0.0688,\ F = 61.0359.$$

The pair correlation coefficients (r_i) are as follows: 0.7162, –0.4110, –0.5130, –0.8898, –0.4595, and 0.8282, respectively. Here, the terms B, δ^2, and V_M are insignificant. Their exclusion from the correlation gave Eq. (11) with a slightly lower multiple correlation coefficient, R = 0.9697.

$$\log k = 2.9026 + (2.5839\pm0.7761)f(n^2) + (0.9713\pm0.3145)f(\varepsilon) - (0.0502\pm0.0062)E_T; \quad (11)$$
$$N = 12,\ R = 0.9697,\ S = \pm0.0705,\ F = 58.0793.$$

We also tried to find a correlation between the energy of activation (E_a) and solvent parameters. Six-parameter correlation (12) was characterized by a low multiple correlation coefficient, R = 0.8563. The latter was improved to 0.9489 by excluding the data for dioxane, and further exclusion of the data for chlorobenzene gave Eq. (12) with R = 0.9883.

$$\log E_a = 1.3311 - (2.1575\pm0.2419)f(n^2) - (0.1516\pm0.0517f(\varepsilon) - (0.0013\pm0.0001)B + (0.0056\pm0.0013)E_T + (0.0012\pm0.0001)\delta^2 + (0.0039\pm0.0004)V_M; \quad (12)$$
$$N = 10,\ R = 0.9883,\ S = 0.8940,\ F = 7.2413.$$

The pair correlation coefficients (r_i) are 0.2867, 0.3133, –0.1030, 0.3655, 0.4260, and 0.0426, respectively. Successive exclusion of insignificant parameters δ^2 and E_T reduced the R value to 0.9799 and 0.9663, respectively, for four-parameter Eq. (13):

$$\log E_a = 1.6309 - (2.4855\pm0.3837)f(n^2) - (0.0015\pm0.0002)B + (0.0014\pm0.0001)\delta^2 + (0.0028\pm0.0006)V_M; \quad (13)$$
$$N = 10,\ R = 0.9663,\ S = 0.0148,\ F = 262.8374.$$

The six-parameter correlation between the solvent parameters and $\Delta H^{\neq}$ was poor (R = 0.8561). The correlation was improved by excluding the data obtained in dioxane and chlorobenzene (R = 0.9898), which led to Eq. (14):

$$\log \Delta H^{\neq} = 1.2876 - (2.2896\pm0.2566)f(n) - (0.1615\pm0.0548)f(\varepsilon) - (0.0014\pm0.0001)B + (0.0060\pm0.0013)E_T + (0.0013\pm0.0001)\delta^2 + (0.0042\pm0.0004)V_M; \quad (14)$$
$$N = 10,\ R = 0.9898,\ S = 0.087,\ F = 791.6524.$$

The pair correlation coefficients (r_i) are 0.2869, 0.3133, –0.1040, 0.3651, 0.4253, and 0.0423, respectively.

Analysis of Eq. (14) showed that the parameters $f(\varepsilon)$ and E_T do not affect the $\Delta H^{\neq}$ value. Exclusion of the corresponding terms gave four-parameter correlation (15):

$$\log \Delta H^{\neq} = 1.6048 - (2.6355\pm0.4068)f(n^2) - (0.0015\pm0.0002)B + (0.0015\pm0.0001)\delta^2 + (0.0030\pm0.0006)V_M; \quad (15)$$
$$N = 10,\ R = 0.9663,\ S = 0.0158,\ F = 247.9917.$$

A poor correlation (R = 0.8481) was obtained between the entropy of activation $\Delta S^{\neq}$ and six solvent parameters.

Since the $\Delta S^{\neq}$ values are negative, the sign was not taken into account. The correlation quality was improved to $R = 0.9860$ after exclusion of the data for dioxane and chlorobenzene.

$$\log \Delta S^{\neq} = 2.4473 + (1.9418 \pm 0.3026) f(n^2) - (0.0090 \pm 0.0648) f(\varepsilon) - (0.0013 \pm 0.0001) B - (0.0030 \pm 0.0016) E_\mathrm{T} - (0.0012 \pm 0.0001) \delta^2 - (0.0041 \pm 0.0005) V_\mathrm{M}; \quad (16)$$

$$N = 10,\ R = 0.9860,\ S = 0.0102,\ F = 606.9405.$$

The pair correlation coefficients (r_i) for Eq. (16) are –0.5129, –0.2434, 0.2647, –0.1021, –0.3034, and –0.3134, respectively. By excluding insignificant terms, $f(\varepsilon)$ and E_T, we obtained four-parameter correlation (17) with a slightly low multiple correlation coefficient (R = 0.9788).

$$\log \Delta S^{\neq} = 2.2639 + (2.2415 \pm 0.3262) f(n^2) + (0.0014 \pm 0.0001) B + (0.0014 \pm 0.0001) \delta^2 + (0.0037 \pm 0.0005) V_\mathrm{M}; \quad (17)$$

$$N = 10,\ R = 0.9788,\ S = 0.0126,\ F = 388.7232.$$

The Gibbs energy of the transition state ($\Delta G^{\neq}$) was approximated by six-parameter Eq. (18) with $R = 0.9758$ and the following pair correlation coefficients (r_i): –0.5955, 0.4769, 0.4196, 0.9173, 0.5176, –0.7444.

$$\log \Delta G^{\neq} = 1.8668 - (0.1698 \pm 0.0485) f(n) - (0.0468 \pm 0.0115) f(\varepsilon) - (0.0000 \pm 0.0000) B + (0.0018 \pm 0.0003) E_\mathrm{T} - (0.0001 \pm 0.0000) \delta^2 - (0.0002 \pm 0.0001) V_\mathrm{M}; \quad (18)$$

$$N = 12,\ R = 0.9860,\ S = 0.0020,\ F = 16.5837.$$

The terms V_M, δ^2, and B almost do not affect the $\Delta G^{\neq}$ value. Exclusion of these terms gave three-parameter correlation (19) with a lightly lower value of R (0.9517);

$$\log \Delta G^{\neq} = 1.8627 - (0.0333 \pm 0.0304) f(n^2) - (0.0266 \pm 0.0123) f(\varepsilon) + (0.0017 \pm 0.0002) E_\mathrm{T}; \quad (19)$$

$$N = 12,\ R = 0.9517,\ S = 0.0028,\ F = 1177.1582.$$

Thus, the main factors affecting the oxidation of dimethyl sulfoxide with peroxydecanoic acid in different solvents are specific and nonspecific solvation of both reactants and structural factors of the reaction medium.

EXPERIMENTAL

Peroxy acids were synthesized by reacting the corresponding carboxylic acid with 60% hydrogen peroxide in concentrated sulfuric acid according to the procedure described in [26] and were purified by repeated recrystallization. Samples of peroxy acids containing no less than 99.0% were used in kinetic experiments. The major impurity was the corresponding carboxylic acid. Peroxybenzoic acid was prepared from benzoyl peroxide as reported in [27]. All solvents used and sulfoxides (DMSO, dibutyl sulfoxide, and diphenyl sulfoxide) were purified as described in [28, 29] and distilled under argon. Dimethyl sulfoxide was additionally purified by recrystallization under argon.

The kinetics of oxidation of sulfoxides with peroxy acids were studied in a glass reactor under argon in the temperature range from 298 to 318 K. A reactor was charged with a solution of peroxy acid with a required concentration and placed in a thermostat where a required temperature was maintained with an accuracy of ±0.05 K. A solution of the corresponding sulfoxide in the same solvent was quickly added, assuming this moment as reaction onset. Samples were withdrawn from the mixture at definite time intervals, and the concentration of unreacted peroxy acid was determined by iodometric titration according to [30]. In all cases, the error in the determination of effective rate constants did not exceed ±3%.

CONFLICT OF INTEREST

The authors declare the absence of conflict of interest.

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