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Substituent Effect and Activation Parameter Studies in the Oxovanadium (IV) salophen-mediated reactions of Phenylsulfinylacetic Acids by hydrogen peroxide

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Abstract

The oxidative decarboxylation of phenylsulfinylacetic acid and substituted phenylsulfinylacetic acids by hydrogen peroxide catalysed by oxovanadium (IV) salophen complexes has been discussed. The kinetic study for the reactions between PSAAs and H_2O_2 in the presence of four different oxovanadium (IV) salophen complexes was carried out in 100 % acetonitrile medium spectrophotometrically under pseudo first-order conditions with an excess of PSAA concentration over the H_2O_2 and complex concentrations. The substituent effect and linear free energy relationship were studied using several *para*- and *meta*-substituted PSAAs with H_2O_2 in the presence of complexes **I**, **II** and **IV** at 30 °C . Though nonlinear Hammett correlation is observed, the Yukawa–Tsuno plot gave excellent straight lines. These observations are explained on the basis of ground state stabilization of PSAA possessing EDG. The reactions of PSAA and H_2O_2 with various substituted oxovanadium (IV) salophen complexes [**I-IV**] were carried out at three different temperatures *viz.* 30 °C, 35 °C and 40 °C and complex **I** catalysed reactions of different substituted PSAAs with H_2O_2 were also carried out at three different temperatures *viz.* 25 °C, 30 °C and 35 °C. The positive $\Delta^{\ddagger}H$ values suggest that the reactions are endothermic.

Keywords: Oxidative Decarboxylation, Oxovanadium (IV) Salophen Complexes, Hydrogen Peroxide Oxidation, Hammett Correlation, Reaction Kinetics

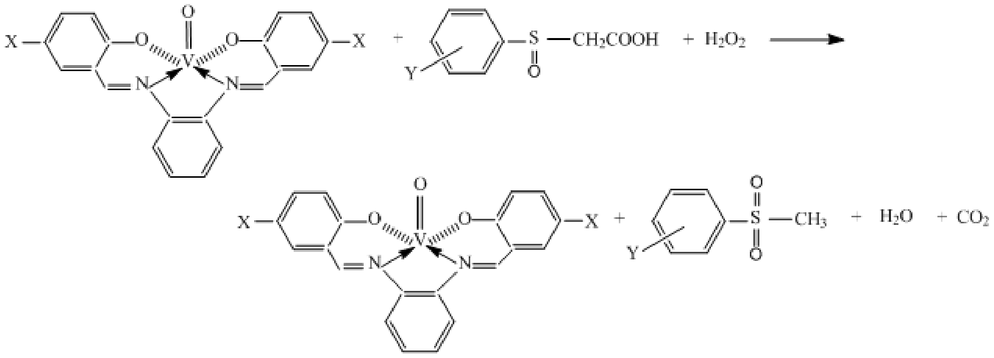
1.Introduction

Schiff base complexes formed between oxovanadium (IV)/dioxovanadium(V) and chelating ligands with hetero atoms are found to be involved in a variety of biochemical processes, such as haloperoxidation [1], phosphorylation [2], nitrogen fixation [3] and in medicinal process like tumor growth inhibition and prophylaxis against carcinogenesis [4]. Metal complexes of salen and salophen ligands have been used in medicinal studies as model for superoxide dismutase [5,6] and to inhibit the growth of AGS gastric cell lines [7]. Certain metal-salophen complexes have been designed for their application as functional materials [8].

The capability of vanadium-Schiff base complexes to form metalloperoxo species which in turn effectively transfer the oxygen atom to reductants with high degree of selectivity, reactivity and specificity [9,10] made them as effective catalysts in the oxidation of sulfides, alcohols, phenols, tertiary amines, epoxidation of olefins and hydroxylation of phenols. Salophen, a tetradentate Schiff base derived from 1,2-phenylenediamine and salicylaldehyde and its derivatives are able to stabilize different metal ions in various oxidation states and control variety of catalytic transformations [11].

Phenylsulfinylacetic acid (PSAA), a sulfoxide containing acid, is an ambidentate ligand and with its three donor atoms acts as a good chelating agent. Some of the phenylsulfinyl compounds have high therapeutic effects and antibacterial activities. They are also used for combating parasite disorders, treating Alzheimer′s disease and enhancing memory activities.

The substituent effects and the activation parameters in the reactions of PSAAs with H_2O_2 and oxovanadium (IV) salophen complexes are explained in this paper. The overall representation of the reaction is shown in Scheme 1.



Complex : **(I)** X = H; **(II)** X = OCH_3; **(III)** X = CH_3; **(IV)** X = Cl

PSAA : Y = H, *p*-Cl, *m*-Cl, *p*-F, *p*-Br, *p*-OMe, *p*-OEt, *p*-Me

Scheme 1. Overall scheme of the reaction

2. Kinetic studies

A double beam BL 222 Elico UV-vis bio-spectrophotometer with an inbuilt thermostat was employed to record the absorption spectra and to follow the kinetics of the reactions. The kinetic study for the oxidative decarboxylation of PSAA and substituted PSAAs by H_2O_2 in the presence of oxovanadium (IV) salophen complexes was carried out in 100 % acetonitrile medium under pseudo first-order conditions with excess of PSAA concentration over H_2O_2 and oxovanadium complex concentrations. The rate of the reaction was measured by following the decay of absorbance of hydroperoxovanadium(V) salophen complexes formed by the reaction between oxovanadium (IV) salophen complex and H_2O_2 with time at the appropriate wavelength.

3. Absorption spectral studies and Active species

The absorption of parent oxovanadium (IV) salophen complex (**I**) at 242 and 312 nm in CH_3CN corresponds to the ligand centered transition and the absorption at 396 nm corresponds to ligand to metal charge transfer (LMCT) transition. The LMCT absorption maximum is sensitive to the nature of substituent present in the salophen ligand. Introduction of OMe group in the 5,5′-positions of the salophen ligand (**II**) shifts the λ_{max} value from 396 nm to 440 nm (red shift) whereas chloro group at 5,5′- position (**IV**) causes a comparatively less shift from 396 nm to 410 nm. The shift in the λ_{max} towards red region is due to the stabilization of excited state by substituent groups The absorption spectral data for the complexes (**I** to **IV**) and their structures known by IR spectra are consistent with the literature data [12].

The possibility of self-decay of the oxidant, H_2O_2 with complex **I** was measured by plotting absorbance *vs.* time at the λ_{max} of 396 nm in the absence of the PSAA. From this measurement, it is inferred that the rate of self-decay of H_2O_2 is negligible compared to the rate in the presence of PSAA. This clearly shows that the decrease in absorbance with time is due to the reaction between oxovanadium (IV) salophen complex, H_2O_2 and PSAA. A similar observation was noted in the oxidation of sulfides by H_2O_2 using iron (III) salen catalyst. The mixture of PSAA and oxovanadium (IV) salophen complex in the reaction conditions in the absence of H_2O_2 also showed very negligible decrease in absorbance. This clearly shows that neither oxovanadium (IV) salophen complex nor H_2O_2 efficiently oxidizes the PSAA individually, but they do so only in their combination in acetonitrile medium. Representative kinetic runs showing the decrease in absorption with time due to the reaction between hydroperoxo vanadium species and PSAA are shown in Figure.1.

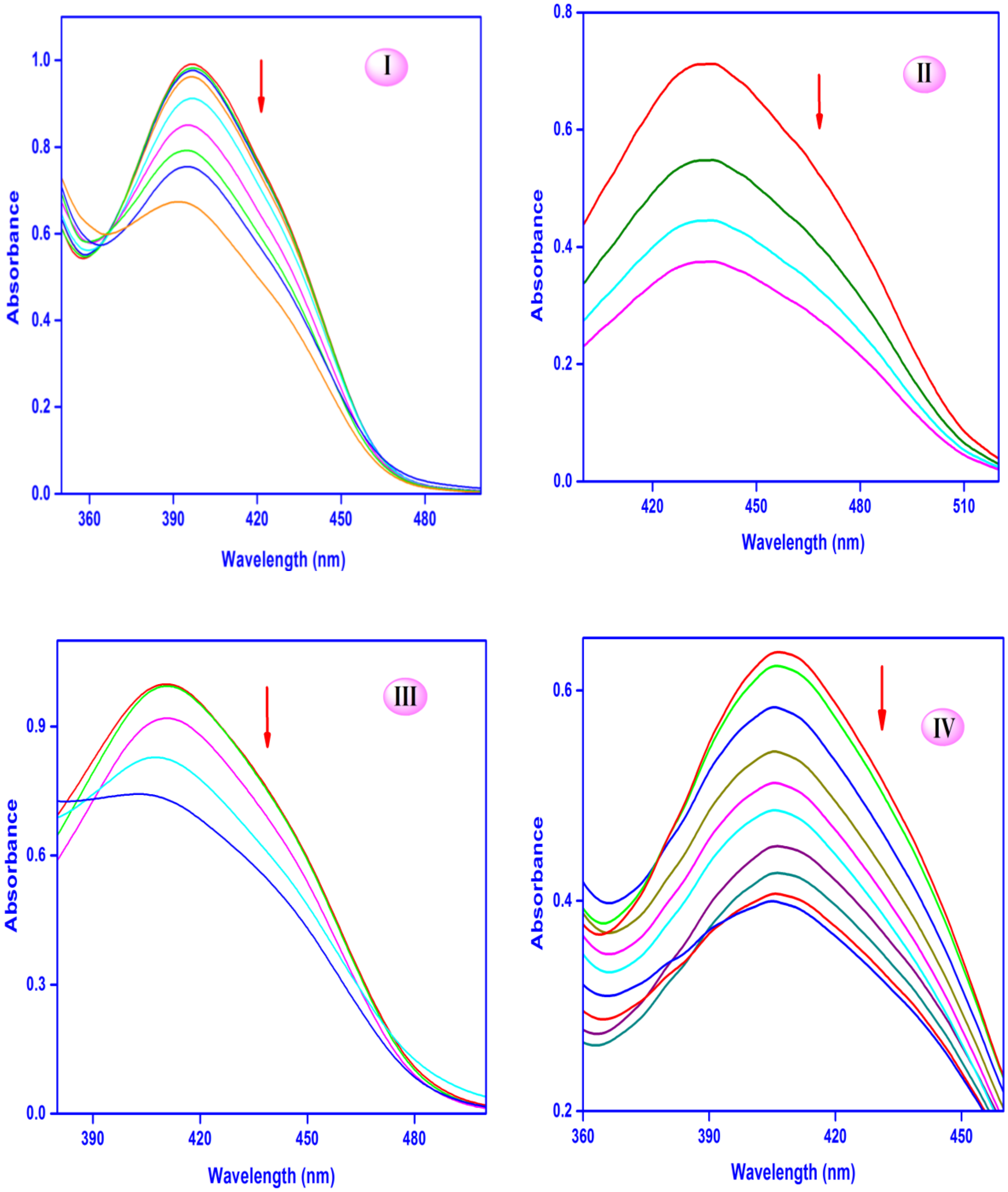

I
Absorbance
1.0
0.8
0.6
0.4
0.2
0.0
360
390
420
450
480
Wavelength (nm)
II
Absorbance
0.8
0.6
0.4
0.2
0.0
420
450
480
510
Wavelength (nm)
III
Absorbance
0.9
0.6
0.3
0.0
390
420
450
480
Wavelength (nm)
IV
Absorbance
0.6
0.4
0.2
360
390
420
450
Wavelength (nm)

Figure 1. Decay of absorbance of hydroperoxovanadium(V) salophen complexes with time. [PSAA] = 5.0 × 10^{-2} M; [H_2O_2] = 7.0 × 10^{-3} M; [**I**] = [**II**] = [**III**] = [**IV**] = 1.0 × 10^{-4} M.

The absorption maximum of oxovanadium(IV) salophen (I) at 396 nm is slightly blue-shifted by the addition of H2O2, reaching 393 nm. In addition, it is interesting to note that addition of H_2O_2 to the complex at higher concentrations shows an increase in intensity of a new broad absorption peak around 610 nm after a time interval, followed by a decrease in absorbance during the course of time. These observations demonstrate the formation of a new intermediate vanadium species in the reaction mixture.

4. Substituent effects and linear free energy relationships

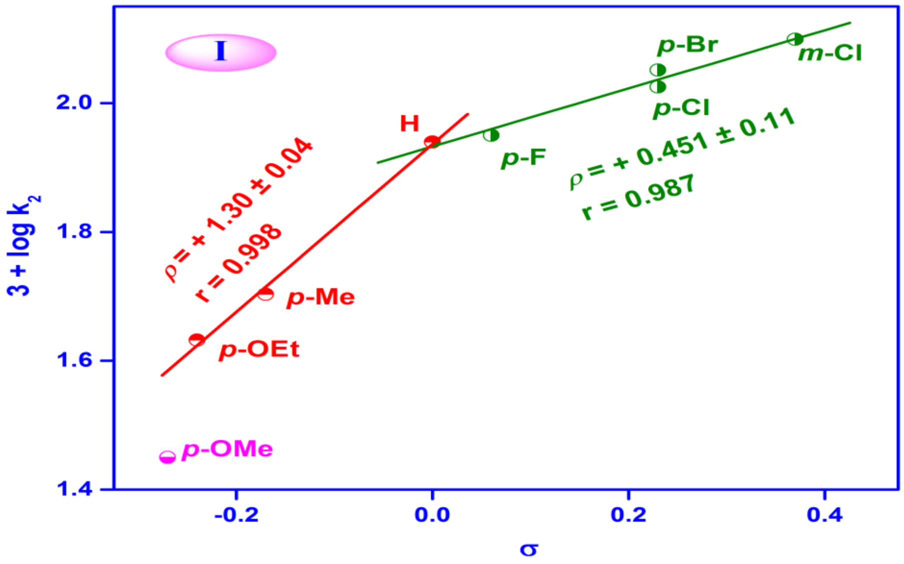
Substituent effect and linear free energy relationship were studied using several *para*- and *meta*-substituted PSAAs with H_2O_2 in the presence of complexes **I**, **II** and **IV** at 30 °C and also at three different temperatures with complex (**I**) to understand the nature of the transition state, the rate-determining step and the extent of charge transfer. The observed kinetic data in Table 1 show that the reaction is sensitive to the change of substituents both in the phenyl ring of PSAA and the salophen moiety of the complex. It is observed that electron-donating groups (EDG) in PSAA retard the rate of reaction, while electron-withdrawing groups (EWG) accelerate the rate. The analysis of kinetic data and the plot of log k_2 *vs.* Hammett's substituent constants (σ) shows a nonlinear behavior. The Hammett plots (Figure 2) exhibit a distinct curvature, with two intersecting linear regions. The data in Table 1 and Figure 2 reveal that the reaction is fairly susceptible to electron-donating substituents, with positive ρ values (+0.853 to +1.40), and less susceptible to electron-withdrawing substituents, as indicated by rather small positive ρ values (+0.180 to +0.451).

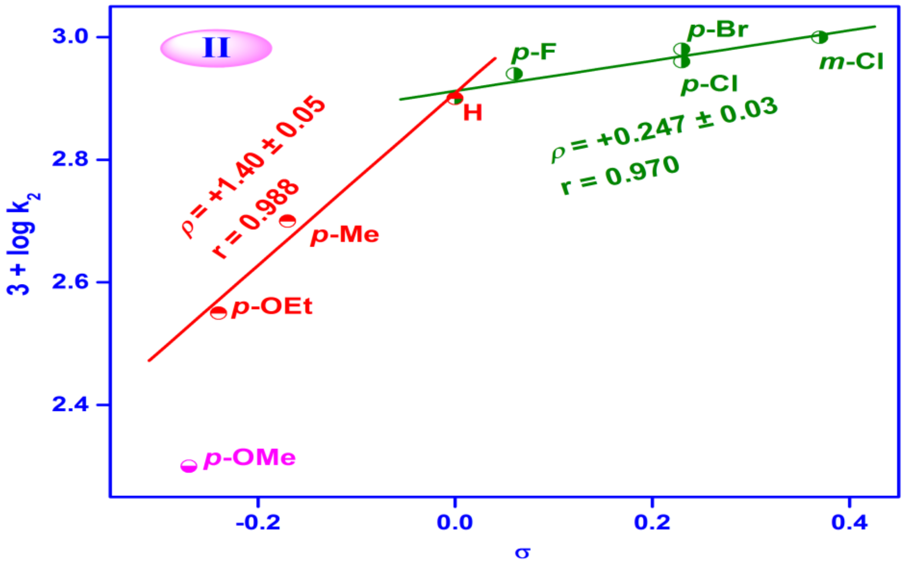
Table 1. Second-order rate constants (k_2), thermodynamic and Hammett parameters for the reactions of PSAAs with H_2O_2 catalyzed by oxovanadium(IV) salophen complexes.

Y	I			$\Delta^{\ddagger}H$ ($kJmol^{-1}$)	$-\Delta^{\ddagger}S$ ($JK^{-1}mol^{-1}$)	II	IV	ρ	r
	$10^2 k_2$ ($M^{-1}s^{-1}$)					$10^2 k_2$ ($M^{-1}s^{-1}$)			
	25 °C	30 °C	35 °C			30 °C	30 °C		
p-Cl	7.98 ± 0.15	10.6 ± 0.23	12.9 ± 0.29	33.7 ± 3.1	153 ± 6.8	91.2 ± 0.31	1.79 ± 0.08	-1.74 ± 0.09	0.999
m-Cl	8.98 ± 0.48	12.6 ± 0.31	15.8 ± 0.47	40.9 ± 2.8	128 ± 8.1	99.9 ± 0.50	1.89 ± 0.12	-1.72 ± 0.14	0.999
p-F	7.46 ± 0.39	8.91 ± 0.35	11.5 ± 0.25	29.7 ± 1.5	167 ± 7.3	87.1 ± 0.44	1.71 ± 0.03	-1.76 ± 0.10	0.998
p-Br	8.20 ± 0.18	11.3 ± 0.41	13.4 ± 0.34	35.0 ± 1.2	148 ± 5.3	95.5 ± 0.40	1.81 ± 0.07	-1.72 ± 0.11	0.999
H	7.28 ± 0.19	8.70 ± 0.16	10.6 ± 0.12	25.8 ± 1.7	180 ± 12	79.4 ± 0.10	1.60 ± 0.09	-1.70 ± 0.10	0.999
p-Me	4.79 ± 0.11	5.05 ± 0.09	7.50 ± 0.22	30.6 ± 2.8	168 ± 9.2	50.1 ± 0.21	1.12 ± 0.03	-1.66 ± 0.12	0.997
p-OMe	1.40 ± 0.06	2.82 ± 0.02	3.55 ± 0.11	68.4 ± 2.1	50.2 ± 8.5	20.0 ± 0.35	0.591 ± 0.06	-1.53 ± 0.08	0.999
p-OEt	3.89 ± 0.09	4.29 ± 0.10	6.64 ± 0.24	37.2 ± 3.0	148 ± 7.8	35.5 ± 1.3	0.901 ± 0.09	-1.60 ± 0.06	0.999

ρ^{+}_{EWG}	0.239 ± 0.03	0.451 ± 0.11	0.446 ± 0.03	-	-	0.247 ± 0.03	0.180 ± 0.02	-	-
r	0.982	0.987	0.986	-	-	0.970	0.985	-	-
ρ^{+}_{EDG}	1.12 ± 0.07	1.30 ± 0.04	0.853 ± 0.07	-	-	1.40 ± 0.05	1.01 ± 0.04	-	-
r	0.998	0.998	0.999	-	-	0.988	0.995	-	-

[PSAA] = 5.0×10^{-2} M; [H_2O_2] = 5.0×10^{-3} M; [**I**] = [**II**] = [**IV**] = 1.0×10^{-4} M; solvent = 100 % CH_3CN.


I
3 + log k2
σ
2.0
1.8
1.6
1.4
-0.2
0.0
0.2
0.4
ρ = + 1.30 ± 0.04
r = 0.998
H
p-Me
p-OEt
p-OMe
p-F
p-Br
p-Cl
m-Cl
ρ = + 0.451 ± 0.11
r = 0.987


II
3 + log k2
σ
3.0
2.8
2.6
2.4
-0.2
0.0
0.2
0.4
ρ = +1.40 ± 0.05
r = 0.988
H
p-Me
p-OEt
p-OMe
p-F
p-Br
p-Cl
m-Cl
ρ = +0.247 ± 0.03
r = 0.970

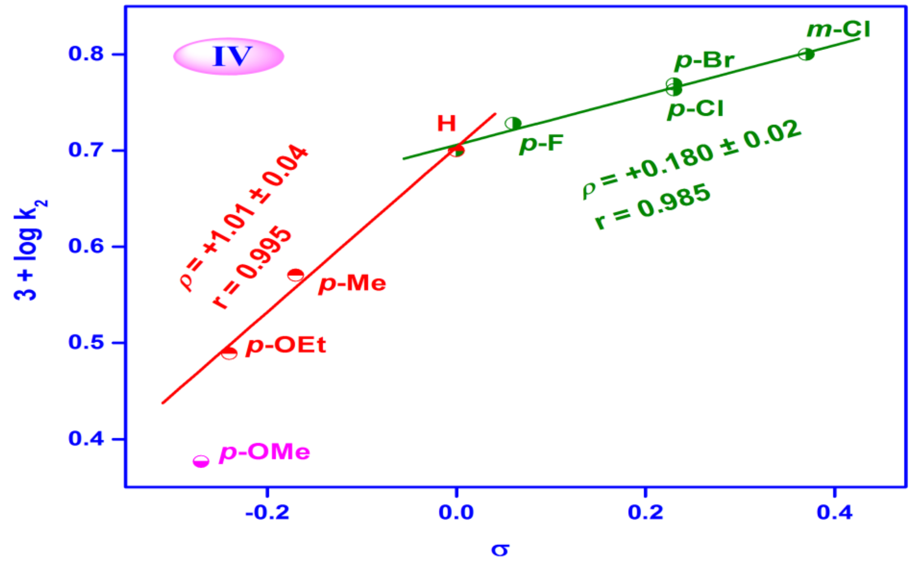


Figure 2. Hammett plots. [PSAA] = 5.0 × 10^{-2} M; [H_2O_2] = 5.0 × 10^{-3} M; [**I**] = [**II**] = [**IV**] = 1.0 × 10^{-4} M; solvent = 100 % CH_3CN; temp = 30 °C.

Similar trend of substituent effect and downward Hammett plot is already reported in the alkaline hydrolysis of *o*-arylthionobenzoates [13], pyridinolysis of 2,4-dinitrophenyl X-substituted benzoates [14] and in the reactions of anionic nucleophiles, OH^-, CN^- and N_3^- with aryl benzoates [15]. In all these cases, the Hammett parameter changes from a large positive ρ value to a small one as the substituent changes from an EDG to a strong EWG and the observed nonlinearity is explained on the basis of ground state stabilization of the substrates possessing EDG. Another interesting observation noted in the Hammett correlation is the anomalous behavior of methoxy substituent showing a significant positive deviation from the rest of the substituents.

The substituent effect in the present study is quite interesting and different from the substituent effects observed with other vanadium complexes. The rate constants for the oxidation of methyl aryl sulfides with hydrogen peroxide catalysed by dioxovanadium(V) in 1:1 CH_3CN:H_2O follow a Hammett correlation, with ρ = - 0.83 . Vanadium catalysed sulfoxidation by Jeong et al. [16] shows a nonlinear Hammett plot with a positive ρ value for electron donating substituents and a negative ρ value for electron withdrawing substituents. van de Velde et al. [17] studied the oxidation of PhSMe by oxovanadium

(IV) and (V) and showed that EWG lowers the rate of oxidation and EDG slightly increases the rate of oxidation. Karunakaran et al. [18] have shown a high negative ρ value of -3.64 during Hammett correlation in the oxidation of phenylthioacetic acids by vanadium(V). In the oxovanadium (IV)-salen ion catalysed H_2O_2 oxidation of tertiary amines to N-oxides, the observed ρ value is found to be in the range of -0.6 to -1.5.

The effect of substituents in the 5,5′-positions of the salophen ligand of oxovanadium (IV) salophen complex on the rate of PSAA oxidation is also investigated. Kinetic data reveal that the reaction rate is very much enhanced by the electron donating methoxy group and strongly retarded by electron withdrawing chloro group when they are introduced into 5,5′-positions. The Hammett plots obtained by plotting log k_2 *vs.* 2σ of substituted complexes with different PSAAs show a single straight line for both electron donating and electron withdrawing substituents with high negative ρ value ranging between -1.53 and -1.76 (Figure 3.). A similar observation has been reported in oxovanadium (IV) salen catalysed H_2O_2 oxidation of tertiary amines to N-oxides . Rayati et al. [19] have also shown the presence of electron-donating substituents on the aromatic ring as well as the imine bond of salen effectively improves the catalytic activity. From the Table 1, it has been observed that the value of reaction constant (ρ) is almost constant for all PSAAs, implying that the extent of transmission of electronic effect is same in each case.

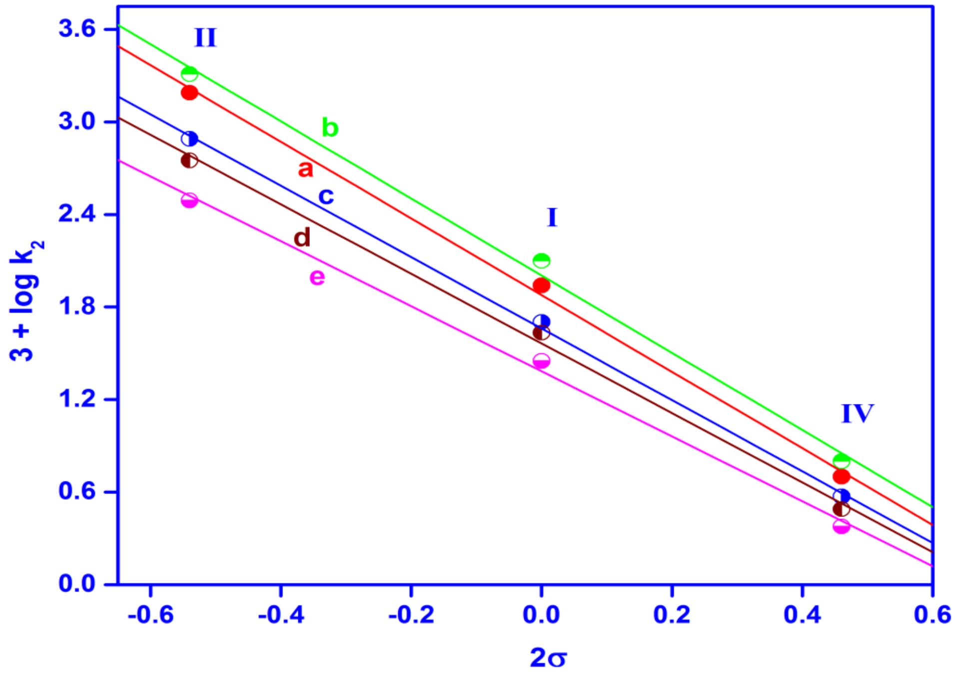


Figure 3. Hammett plots for substituted salophen complexes with PSAAs: a = H; b = *m*-Cl; c = *p*-Me; d = *p*-OEt; e = *p*-OMe.

5. Temperature dependence and activation parameters

The reactions of PSAA and H_2O_2 with various substituted oxovanadium (IV) salophen complexes [**I-IV**] were carried out at three different temperatures *viz.* 30 °C, 35 °C and 40 °C and complex **I** catalysed reactions of different substituted PSAAs with H_2O_2 were also carried out at three different temperatures *viz.* 25 °C, 30 °C and 35 °C. It has been noted that the temperature has a positive effect on the rate only in a limited range of temperatures. The reaction rate is too slow to be measured at low temperatures while at high temperatures the rate decreases. The observed low reactivity at low temperatures may be due to the poor generation of the active peroxo species and at high temperatures beyond a particular temperature it may be due to the decomposition of the peroxo species. The thermodynamic parameters, $\Delta^{‡}H$ and $\Delta^{‡}S$ evaluated from the slope and intercept of the linear Eyring's plots for the substituted complexes and substituted PSAAs (Figure 4) are shown in Table 1 and Table 2. The entropy of activation is found to be high negative while the enthalpy of activation has relatively small positive value ranging from 24.3 to 68.4 $kJmol^{-1}$. The positive $\Delta^{\ddagger}H$ values suggest that the reactions are endothermic in nature.

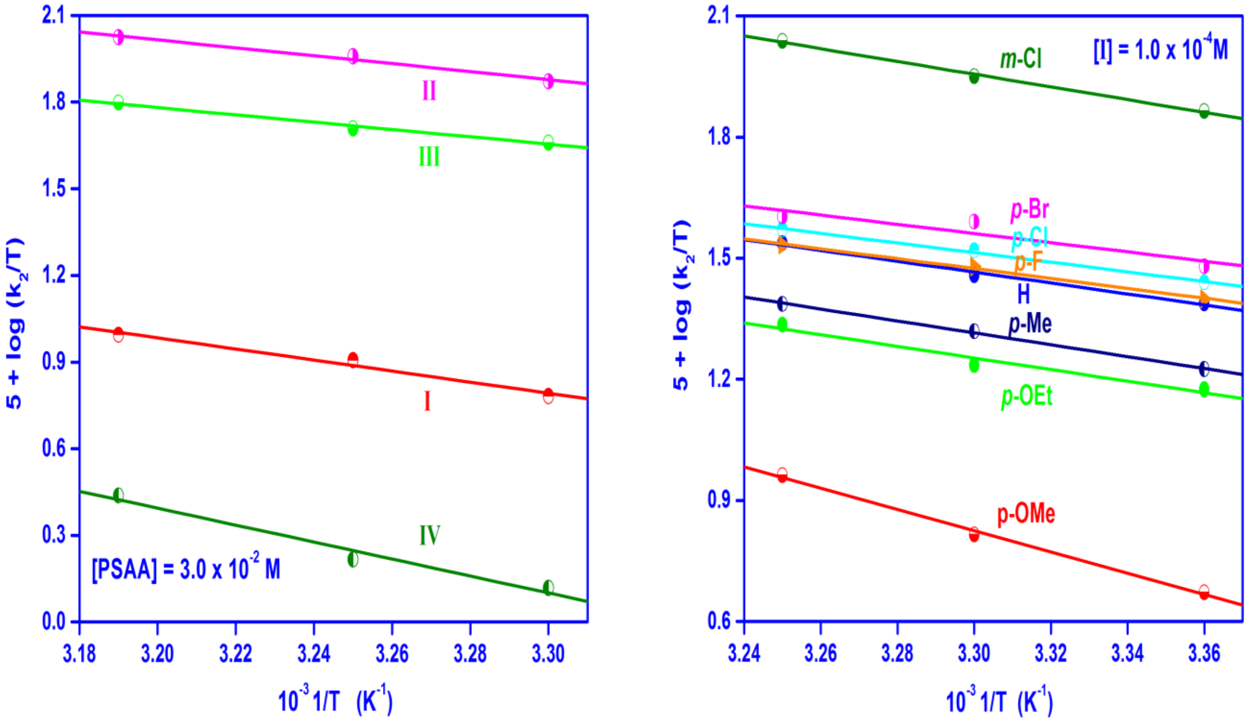


Figure 4. Eyring′s plots for the reactions of PSAAs and H_2O_2 with **I-IV.**

Table 2. Second-order rate constants and activation parameters for the reactions of PSAA and H_2O_2 with **I-IV**.

complex	10^2 k_2 ($M^{-1}s^{-1}$)			$\Delta^{\ddagger}H$	$-\Delta^{\ddagger}S$
	30 °C	35 °C	40 °C	($kJmol^{-1}$)	($JK^{-1}mol^{-1}$)
I	1.83 ± 0.67	2.48 ± 0.14	3.09 ± 0.11	36.6 ± 1.5	157 ± 8.1
II	22.5 ± 1.7	28.0 ± 0.33	33.1 ± 0.20	26.4 ± 2.6	170 ± 3.2
III	13.8 ± 1.0	15.8 ± 0.16	19.7 ± 1.2	24.3 ± 7.9	181 ± 6.5
IV	0.397 ± 0.33	0.507± 0.12	0.857 ± 0.10	53.1 ± 4.1	115 ± 7.6

[PSAA] = 3.0×10^{-2} M; [H_2O_2] = 3.0×10^{-3} M; [**1-1V**] = 1.5×10^{-4} M; solvent = 100 % CH_3CN.

The isokinetic relationship between the enthalpy and entropy of activation is given by the following equation

$$\Delta^{\ddagger}H = \Delta^{\ddagger}H_o + \beta\, \Delta^{\ddagger}S$$

The isokinetic temperature, β is the temperature at which all the substituents in a given series of reactions have the same reactivity which can be obtained from the slope of the linear plot of $\Delta^{\ddagger}H$ *vs.* $\Delta^{\ddagger}S$. In the present study, the correlation between $\Delta^{\ddagger}H$ and $\Delta^{\ddagger}S$ for various substituted complexes and PSAAs (Figure 5) are found to be linear.

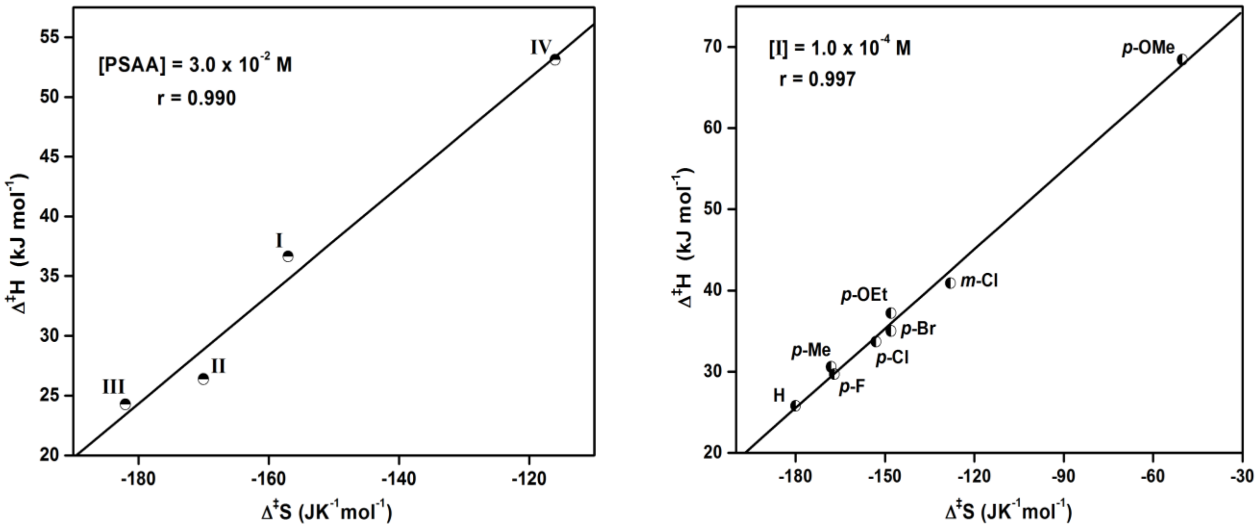


Figure 5. Plots of $\Delta^{\ddagger}H$ vs $\Delta^{\ddagger}S$ for the reactions of PSAAs and H_2O_2 with **I-IV**.

6. Discussion

The isokinetic temperature calculated from the linear $\Delta^{\ddagger}H$ vs. $\Delta^{\ddagger}S$ plot is found to be 326 K for substituted PSAAs which is above the experimental temperature. This indicates the validity of the Hammett equation to the title reaction. The linear isokinetic relation between $\Delta^{\ddagger}H$ and $\Delta^{\ddagger}S$ reveals that all complexes and PSAAs follow the same mechanism.

Interpretation of nonlinear Hammett plot

The existence of distinct curvature with two intersecting linear portions in a nonlinear Hammett plot has been traditionally interpreted as a change in the reaction mechanism or a change in RDS within a given mechanism [20]. However, in the present study, the apparent downward curvature in the Hammett plots has been ascribed to the stabilization of the ground state of PSAA through resonance interaction on changing the substituents from electron withdrawing to electron donating. This argument is supported by the significant negative deviation observed in the Hammett plot as the substituent becomes a stronger EDG. The resonance structures as a result of interaction between the electron donating substituent and the thionyl functionality are represented in Figure 6.

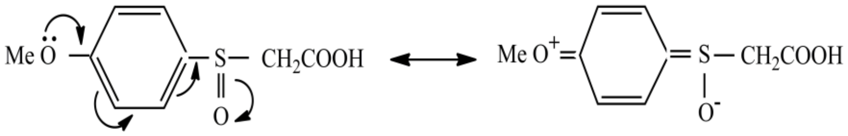


Figure 6. Ground state stabilization of PSAA.

The presence of such resonance structures in organic compounds with electron donating group stabilizes the ground state (GS) and causes a decrease in reactivity. Accordingly, one can expect a large ρ value for EDG compared with EWG. In fact the ρ value decreases considerably from 1.30 for the reactions with EDG to 0.451 for those with EWG. Thus the deviation from Hammett correlation is taken as evidence for the ground state stabilization of the substrate by EDG. The observed nonlinear Hammett plots in the aminolysis reactions of substituted phenyl-2-methoxy benzoates [21], 4-pyridyl substituted benzoates [22] and 2-pyridyl substituted benzoates [23] have been interpreted by the ground state stabilization of the substrates. Neuvonen et al. have explained the enhanced reactivity of esters containing EWG by decrease in the resonance stabilization of the GS of esters.

To examine and ascertain the validity of the above argument, the rate data have been treated with the Yukawa-Tsuno equation [24,25].

$$\log (k_X / k_H) = \rho [\sigma^o + r (\sigma^+ - \sigma^o)]$$

The term (σ^+- σ^o) is the resonance substituent constant, while the 'r' value is a parameter characteristic of a reaction representing the extent of resonance contribution. Interestingly the Yukawa-Tsuno plots (Figure 7) exhibit a good linearity for the reactions under study and the 'r' values obtained (0.877–0.920) suggest that the resonance interaction is relatively significant. The observed linear Yukawa-Tsuno plots not only confirm a common mechanism for all PSAAs but also prove that the ground state stabilization of PSAAs through a resonance interaction is the cause for the non-linearity in the Hammett plots. Um and coworkers [26] have reported such type of linear Yukawa-Tsuno plots for the nonlinear Hammett plots and explained on the basis of the ground state stabilization of the substrate.

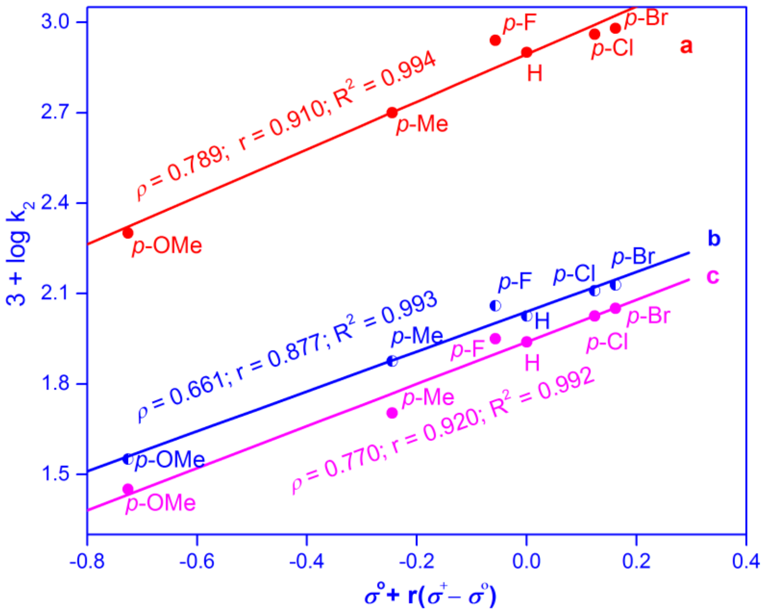


Figure 7. Yukawa-Tsuno plots for the reactions of PSAAs with H_2O_2 and complex.
a = **II** at 30 °C; b = **I** at 35 °C; c = **I** at 30 °C.

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