

Micellar Rate Effects on Alkaline Hydrolysis of Hydroxamic Acids

Kallol Kumar Ghosh* and Supriya Roy

School of Studies in Chemistry, Pt. Ravishankar Shukla University, Raipur M. P., India

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The kinetics of alkaline hydrolysis of *N*-phenylbenzohydroxamic acid and its para-substituted derivatives ($X-C_6H_4-CO-N(OH)C_6H_5$; $X=H, CH_3, OCH_3, NO_2, F$) have been investigated in the presence and absence of cationic (ethyl hexadecyl)dimethylammonium bromide) and anionic (sodium-1-dodecanesulfonate and lithium dodecyl sulfate) surfactants at 55 °C in 5% (v/v) dioxane–water medium. A catalytic effect was found with cationic surfactants and an inhibitory effect in the presence of anionic surfactants was observed. The rate-surfactant profiles can be analyzed in terms of pseudo-phase and Piskiewicz models.

As part of a program for the synthesis and mechanistic studies of hydrolysis of hydroxamic acids^{1,2)} we have been interested in the study of hydrolysis reaction in micellar environments.^{3,4)} Supramolecular aggregates such as micelles, vesicles, or microemulsions were found to be very attractive reaction media for many kinds of organic reactions. Many results have been published in the literature showing different catalytic and inhibitory effects of micelles in chemical reactions like acid-catalyzed hydrolysis, base catalyzed hydrolysis, and oxidation. Surprisingly much less is known about the micellar catalysis of hydroxamic acid.⁵⁾ The hydrolysis of these substances is of interest because of their relationship to peptides. Moreover, its study could be of biological interest.

Some of the hydroxamic acids have already found application in drug-delivery systems,⁶⁾ in iron-solubilization and transport,⁷⁾ and in DNA cleavage.⁸⁾ The possible surfactant interactions in these systems would improve fundamental understanding. It has been generally accepted that the extent of micellar catalysis of bimolecular reaction is due to the concentration of both reactants in the micelle and promoted by hydrophobic and electrostatic effects between substrate and micelles. The basic reaction that concerns us, i.e., the alkaline hydrolysis of *N*-phenylbenzohydroxamic acid (PBHA) may be depicted as follows (Chart 1): In this work we have kinetically studied the effects of cationic micelles of ethyl (hexadecyl) dimeth-

ylammonium bromide (EHDAB) $[(CH_3)_2(C_2H_5)C_{16}H_{33}N^+]$ Br^- and anionic micelles of sodium 1-dodecanesulfonate (SDOS) $[C_{12}H_{25}SO_3^-Na^+]$ and lithium dodecyl sulfate (LDS) $[(C_{12}H_{25}SO_3^-Li^+)]$ on this reaction. We also examined the effects of various inert ions. Hexadecyltrimethylammonium bromide $[C_{16}H_{33}N^+(CH_3)_3] Br^-$ (HTAB) was also used for comparison.

Both a pseudophase model and Piskiewicz model have been applied to rate data.

Experimental

N-phenylbenzohydroxamic acid and its derivatives were prepared by standard methods.⁹⁾ The surfactants were commercial samples [HTAB (LOBA); EHDAB and LDS (Sigma); SDOS (Fluka)] and used without further purification. NaOH solutions were titrated against potassium phthalic acid. All the solutions were prepared with doubly distilled water. Due to the low solubility of PBHA and its derivatives in water all solutions were prepared in 5% (v/v) dioxane medium. The CMC was measured using a conductimeter (Systronics direct reading conductivity meter-304) from the intersection of two lines on the plots of specific conductivity vs concentration in the reaction system. Values of the cmc of HTAB, EHDAB, LDS, and SDOS are 1.74×10^{-3} , 1.16×10^{-3} , 2.02×10^{-3} , and 4.01×10^{-3} M (1 M = 1 mol dm⁻³).

Reaction kinetics were followed spectrophotometrically with a Systronics UV-vis (Type 108) spectrophotometer, measuring the change in absorbance of iron(III) hydroxamic acid complex at 520 nm due to disappearance of hydroxamic acid.

Results and Discussion

Reaction in Absence of Surfactant. The alkaline hydrolysis of all the hydroxamic acids studied is of the first order in both hydroxamic acid and hydroxide ion.

Reaction in the Presence of Cationic Micelles. In the presence of EHDAB ($0.54-40 \times 10^{-3}$ M) at fixed concentrations of NaOH (0.001 M) and hydroxamic acid ($2.0-3.0 \times 10^{-3}$ M), the reaction rate increased as the surfactant concentration was increased (Table 1), Figure 1 shows rate constant data for the *p*-substituted PBHA with 0.001 M OH⁻

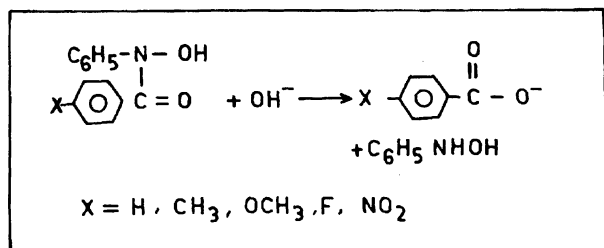
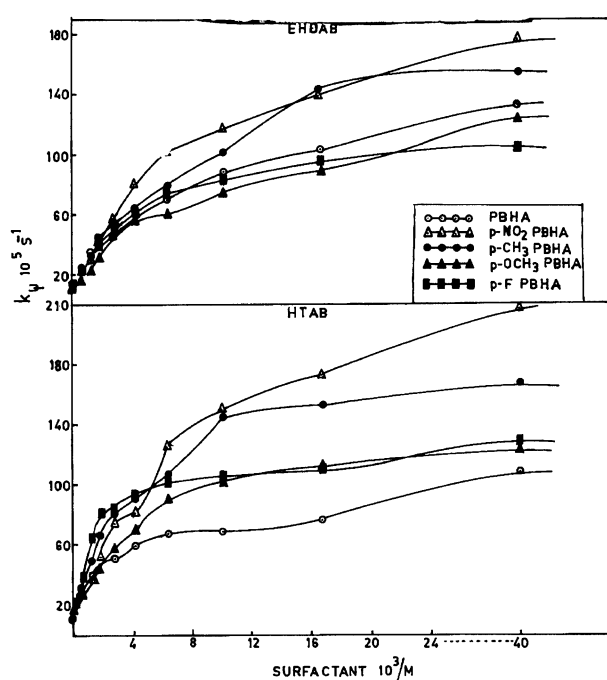


Chart 1.

Table 1. Micellar Hydrolysis of *p*-Substituted *N*-Phenylbenzohydroxamic Acids in (X-C₆H₄-CO-N(OH)C₆H₅) in Cationic Surfactants^{a)}

Surfactant (10 ³ M)	HTAB					EHDAB				
	$(k_{\psi}/10^5 \text{ s}^{-1})$					$(k_{\psi}/10^5 \text{ s}^{-1})$				
	H	OCH ₃	CH ₃	NO ₂	F	-H	-OCH ₃	-CH ₃	-NO ₂	F
0.0	16.3	16.7	12.9	16.8	16.3	16.3	16.7	12.9	16.8	16.3
0.54	28.3	29.7	30.0	27.0	40.1	25.50	19.7	18.8	25.0	22.0
1.37	39.3	39.0	50.0	41.7	62.3	31.0	22.8	36.5	36.0	31.8
1.78	49.6	47.2	67.3	53.2	80.1	44.0	34.2	46.9	40.0	40.2
2.74	50.5	59.1	80.0	76.1	86.0	50.2	48.2	54.8	59.2	51.5
4.11	60.1	76.9	92.5	81.2	93.0	59.2	59.2	66.3	80.1	63.0
6.17	68.3	90.6	107.3	127.1	101.6	70.4	60.1	80.1	102.5	73.5
10.4	69.1	101.6	143.8	151.1	105.0	88.5	73.8	101.1	118.5	83.6
16.8	79.4	111.0	152.1	174.0	110.0	101.0	89.7	142.8	140.6	94.3
40.0	109.0	123.6	169.9	219.7	121.4	131.5	122.9	152.8	176.6	104.1

a) At 55 °C, and with 0.001 M NaOH 5% (v/v) dioxane.

Fig. 1. Variation of the pseudo-first rate constant, k_{ψ} , with cationic surfactant concentration with OH⁻ (0.001 M).

in the presence of EHDAB. The addition of surfactant accelerated the hydrolysis rate at concentrations greater than its cmc. Similar trends have been observed in HTAB. Based on analysis of rate surfactant profiles we conclude that a size increase of the head group, i.e. trimethyl to ethyl dimethyl, have no significant effects on the rate constant. Micellar kinetics is governed by electrostatic and hydrophobic interactions between micelles and reactants, transition complexes, and products. The electrostatic surface potential at the micellar surface can attract or repel reaction species, and a strong hydrophobic interaction can bring about the incorporation into micelles even of reagents that bear the same charge as the ionic micelle.

From Table 1 it is clear that the reaction rates increase as the surfactant concentration is increased for all the hydro-

xamic acids. The presence of catalysis is explained as an electrostatic effect, the cationic nature of the surfactant will favor the presence of OH⁻ in the micellar medium, accelerating the hydrolysis of the substrate associated with the micelle. At higher concentrations of surfactants the acceleration effect is slow due to the increase of the concentration of inert Br⁻ anions which compete with OH⁻.

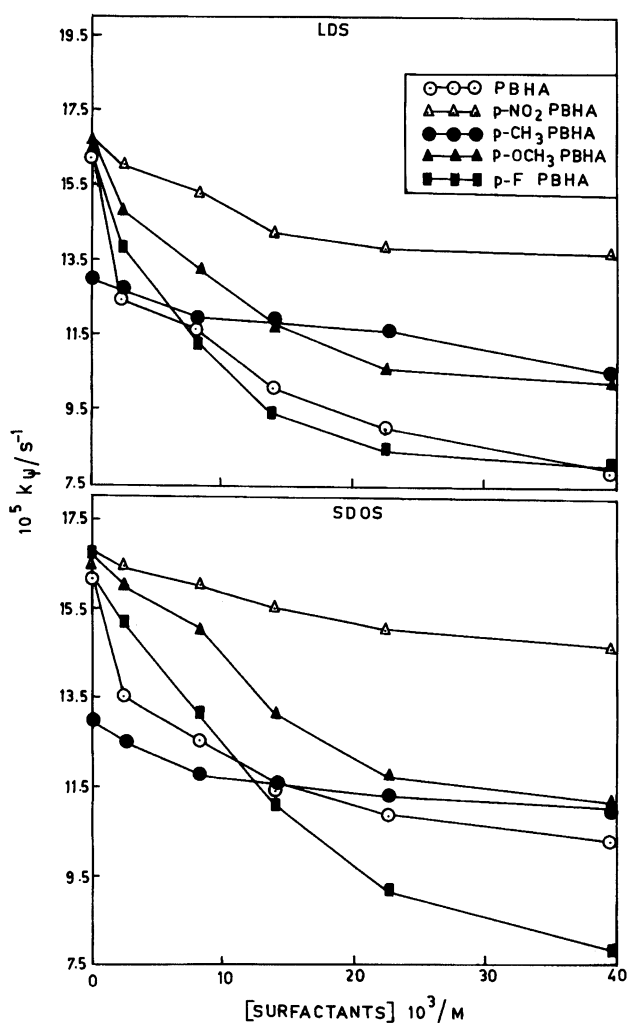
Reactions in the Presence Anionic Micelles. The hydrolysis rate of PBHA and its derivatives is inhibited by anionic micelles of sodium 1-dodecanesulfonate and lithium dodecyl sulfate (Table 2) at constant [OH]. The reaction rate decreased as the surfactant concentration was increased (Fig. 2). The inhibition of the reaction by anionic micelles could be rationalized in terms of proximity effects. In the anionic micelles, the substrates get incorporated hydrophobically and the anionic hydrolyzing agent (OH⁻) repelled electrostatically, thus separating the reactants from one another and resulting in the observed inhibition. A complete understanding of micellar catalysis is not possible, because a number of different interactions are involved including those associated with the head group of the surfactant, different segments of the alkyl chain, and the counter ions. Moreover, the location of compounds in the micellar structure and the degree of penetration of water into the micellar structure have a major influence on the reactivity.

Kinetic Salt Effects. The studies on salt effects were made with a constant surfactant and varying salt (NaCl, NaBr, KCl, KBr, NaClO₄) concentration (Table 3). Both in the anionic and cationic micelles, added electrolyte inhibited the reaction by competing with OH⁻ for the micelle. The addition of salts to the reaction mixture usually resulted in the inhibition of micellar catalysis. According to Cordes and Giltler,¹⁰⁾ inhibition of catalysis by added salts could be caused by: (i) Reduction of the micellar charge due to an increase in the number of counterions in the Stern layer. (ii) Exclusion of the substrate from the micelle by competition for the available sites. (iii) Exclusion of hydroxide ions rather than substrate molecules from the micelle. The incorporation of a foreign anion a micelle prevents incorporation of the substrate, and so makes this micelle less effective. With 1-

Table 2. Micellar Hydrolysis of *p*-Substituted *N*-Phenylbenzohydroxamic Acids in (X-C₆H₄-CO-N(OH)C₆H₅) in Anionic Surfactants^{a)}

Surfactant (10 ³ M)	$k_p/10^5 \text{ s}^{-1}$									
	SDOS					LDS				
	H	OCH ₃	CH ₃	NO ₂	F	H	OCH ₃	CH ₃	NO ₂	F
0.0	16.3	16.7	12.9	16.8	16.3	16.3	16.7	12.9	16.8	16.3
2.38	13.5	16.0	12.5	16.4	15.1	12.4	14.8	12.6	16.0	13.8
8.26	12.5	15.0	11.7	16.0	13.0	11.6	13.2	11.9	15.3	11.2
13.9	11.6	13.1	11.5	15.5	11.0	10.1	11.8	11.8	14.2	9.4
22.5	10.5	11.7	11.2	15.0	9.1	9.0	10.6	11.6	13.8	8.4
39.6	10.2	11.1	10.9	14.6	7.1	7.9	10.3	10.4	13.6	8.0

a) At 55 °C, and with 0.001 M NaOH 5% (v/v) dioxane.

Fig. 2. Variation of the pseudo-first rate constant, k_p , with anionic surfactant concentration with OH⁻ (0.001 M).

dodecanesulfonic acid sodium salt the rate decrease in the sequence no salt > NaCl > NaClO₄ > KBr > KCl at 0.01 M and NaClO₄ > NaCl > KCl > KBr at 1.0 M. It is possible to explain the experimental results of salt effects by a pseudophase ion exchange model developed by Bunton¹¹⁾ and Romsted.¹²⁾ The competition between the reactive anion and the inert anion is an expected but interesting mechanism for inhibiting a

Table 3. Salt Effect on the Hydrolysis of *N*-Phenylbenzohydroxamic Acids at 55 °C

Salt M	HTAB ^{a)} ($k_p/10^5 \text{ s}^{-1}$)				SDOS ^{b)} ($k_p/10^5 \text{ s}^{-1}$)			
	NaCl	KCl	KBr	NaClO ₄	NaCl	KCl	KBr	NaClO ₄
0	16.3	16.3	16.3	16.3	16.3	16.3	16.3	16.3
0.01	9.2	10.4	10.6	9.8	10.6	16.0	15.9	14.3
0.02	9.0	9.8	9.7	9.5	—	—	—	—
0.03	—	—	—	—	10.6	15.0	15.5	10.9
0.05	8.4	9.2	9.0	8.9	10.4	14.6	14.2	10.6
0.10	7.7	8.1	8.2	8.5	10.0	13.0	14.0	9.2
0.30	—	—	—	—	9.4	12.4	12.8	9.0
0.50	7.0	7.6	7.9	8.0	9.8	11.9	12.3	8.8
0.70	6.9	7.0	7.3	7.6	8.8	11.8	11.9	8.0
1.00	5.3	6.9	7.2	6.0	8.3	10.8	11.2	7.7

a) HTAB, 1.37×10^{-3} M. b) SDOS, 2.38×10^{-3} M.

reaction catalyzed by micellar media. When the total unreactive counterion concentration is increased the micelle-bound hydroxide concentration decrease due to the ion-exchange equilibrium. However, fluorescence spectroscopy¹³⁾ shows that very hydrophilic anions e.g., OH⁻ and F⁻, are singularly ineffective in displacing Br⁻, although competition between Br⁻ and other moderately hydrophilic anions follows the pseudophase ion-exchange model. In the Poisson-Boltzman model^{14,15)} counter-ions are assumed to compete in two different ways. Addition of counterions reduces the surface potential of micelles and therefore reduces coulombic attraction of counter-ions and repulsion of co-ions. But counterions such as Br⁻, which interact specifically with the head groups, also neutralize their charge and this reduction of the surface charge density further reduces the surface potential.

Temperature Effects. A dependency of the reaction rates on temperature was examined for PBHA both in the absence and presence of surfactants. The reaction rates were measured over the temperature range of 55–75 °C, and the surfactants used were HTAB, EHDAB, LDS, and SDOS. The values are given in Table 4. The ΔH^\ddagger and ΔS^\ddagger values increase in the presence of these micelles compared with those in the absence of micelles. The reaction mechanism is not changed by the presence of micelles.

Quantitative Treatments of Micellar Rate Effects. Several models are available for interpreting rate-surfactant

Table 4. Activation Parameters for Alkaline Hydrolysis PBHA in the Various Surfactants^{a)}

Surfactant	ΔH^\ddagger kJ mol ⁻¹	ΔS^\ddagger J mol ⁻¹ K ⁻¹	ΔG^\ddagger kJ mol ⁻¹
None	61.2	-132	100.5
HTAB	70.8	-93.0	98.7
EHDAB	71.5	-93.0	99.0
SDOS	68.8	-110	101.5
LDS	71.5	-93.4	99.03

a) With 0.001 M NaOH, and 0.00178 M surfactant.

profiles, and they often provide excellent qualitative and good quantitative fits to observed rate constants.

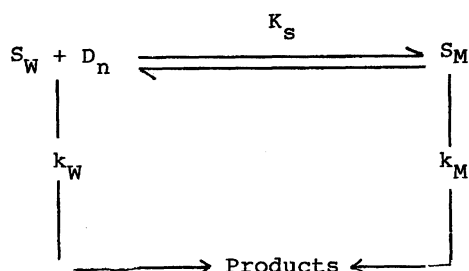
Most quantitative models^{16,17)} treat micelles and water as separate reaction media. The substrate is distributed between the aqueous (W) and micellar (M) pseudophases, with the first-order rate constants being k_W and k_M (Scheme 1). The micellized surfactant is denoted as D_n and its concentration is assumed to be given by $[D]-cmc$, $[D]$ is the total surfactant concentrations and cmc the critical micelle concentration, which is assumed to be concentration of monomeric surfactant. It K_S is the substrate binding constant, the first-order rate constants is given by Eq. 1.

$$k_\psi = \frac{k_W + k_M K_S [D_n]}{1 + K_S [D_n]} \quad (1)$$

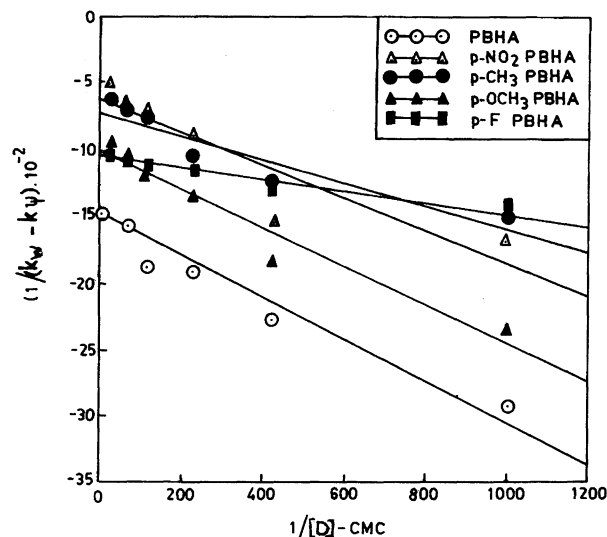
This equation leads to the relationship.

$$\frac{1}{k_W - k_\psi} = \frac{1}{k_W - k_M} + \frac{1}{k_W - k_M} \left(\frac{1}{K_S([D] - cmc)} \right) \quad (2)$$

This equation is sensitive to the value of the cmc, especially for reactions of hydrophobic substrates. A graph of the left hand side of Eq. 2 vs. $1/([D]-cmc)$ is a good linear plot within experimental error. Some representative plots are given in Fig. 3. It is very difficult to measure the volume of the Stern layer or the micellized nucleophile concentration kinetically. Therefore, this paper attempted to evaluate the binding constants (K_S) of reactants with the micelle from the kinetically accessible parameters (Table 4). Table 5 shows the estimated rate constants in the micellar pseudo-phase (k_M) and K_S that best fit the kinetic results for the reaction of hydroxamic acids in micelles. The value of k_M indicates the reactivity in the micellar phase. Comparing k_M with k_W we find that the hydrolysis rate is smaller in micellar media for



Scheme 1.

Fig. 3. Plots of $1/(k_W - k_\psi)$ vs. $1/[D]-cmc$ for the alkaline hydrolysis of para substituted PBHA in HTAB.

anionic surfactants and greater in micellar media for cationic surfactants than in the aqueous phase.

Piszkiewicz Co-Operativity Index in Micellar Hydrolysis. Binding of additional substrate to enzymes may increase or decrease the observed reaction rate, by analogy to the cooperativity between the surfactant molecules, and the substrate may account for micellar catalysis. For such a reaction Piszkiewicz¹⁸⁾ proposed a cooperativity model and Eq. 3 arrived at from such a model is

$$\log \frac{(k_\psi - k_W)}{(k_M - k_\psi)} = n \log [D] - \log K_D \quad (3)$$

In this equation K_D is the dissociation constant of the micelle back to its free components, and $[D]$ is the total concentration of surfactant. From the data in Table 6 $\log [(k_\psi - k_W)/(k_M - k_\psi)]$ versus $\log [D]$ plots have been drawn. Fairly linear correlations are observed (Fig. 4) and the values of the co-operativity index, n and $\log [D]_{50}$ derived there from are

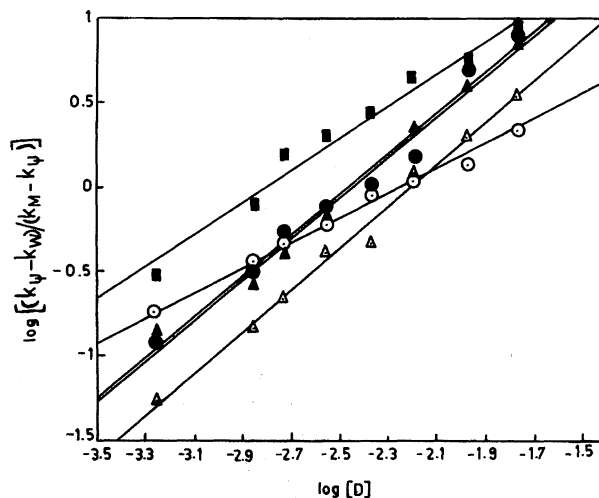
Fig. 4. Plots of $\log [(k_\psi - k_W)/(k_M - k_\psi)]$ vs. $\log [D]$ for the alkaline hydrolysis of para substituted PBHA in HTAB.

Table 5. Summary of Pseudophase Model^{a)}

X.C ₆ H ₄ - CON(OH) C ₆ H ₅	HTAB			EHDAB			SDOS			LDS		
	k_M	K_s	Corr. coeff. points	k_M	K_s	Corr. coeff. points	k_M	K_s	Corr. coeff. points	k_M	K_s	Corr. coeff. points
-H	0.85×10^{-3}	928.4	0.923	1.23×10^{-3}	221.9	0.995	9.58×10^{-5}	209.0	0.999	7.36×10^{-5}	254.8	0.996
-NO ₂	2.50×10^{-3}	166.4	0.990	1.81×10^{-3}	208.5	0.993	15.4×10^{-5}	1530.8	0.999	12.8×10^{-5}	142.3	0.998
-CH ₃	1.76×10^{-3}	378.7	0.982	1.71×10^{-3}	164.1	0.984	11.3×10^{-5}	20.7	0.980	11.5×10^{-5}	524.2	0.970
-OCH ₃	1.18×10^{-3}	708.1	0.996	1.36×10^{-3}	109.1	0.989	2.15×10^{-5}	1471.4	0.990	9.15×10^{-5}	200.2	0.999
-F	1.45×10^{-3}	865.6	0.949	1.10×10^{-3}	302.5	0.994	2.83×10^{-5}	52.3	0.999	6.72×10^{-5}	238.1	0.998

a) k_W : -H = 16.36×10^{-5} , -NO₂ = 16.80×10^{-5} , -CH₃ = 12.95×10^{-5} , -OCH₃ = 16.70×10^{-5} , -F = 18.30×10^{-5} .

Table 6. Summary of Piskiewicz Model

X.C ₆ H ₄ - CON(OH) C ₆ H ₅	HTAB			EHDAB			SDOS			LDS					
	<i>n</i>	log[D]/50	<i>K</i> _D	Corr. coeff.	No. of points	<i>n</i>	log[D]/50	<i>K</i> _D	Corr. coeff.	No. of points	<i>n</i>	log[D]/50	<i>K</i> _D	Corr. coeff.	No. of points
-H	0.675	-2.56	29.8×10 ⁻³	0.976	7	0.999	-2.20	6.46×10 ⁻³	0.995	6	1.596	-1.40	2.9×10 ⁻⁴	0.998	3
-NO ₂	1.264	-2.20	1.6×10 ⁻³	0.994	7	1.296	-2.38	1.40×10 ⁻³	0.994	6	2.05	-1.40	9.94×10 ⁻⁵	0.997	3
-CH ₃	1.288	-2.38	6.5×10 ⁻⁴	0.994	6	1.032	-2.20	5.51×10 ⁻³	0.999	5	2.95	-1.40	1.81×10 ⁻⁶	0.998	3
-OCH ₃	1.333	-2.38	5.8×10 ⁻⁴	0.999	7	0.911	-2.20	12.7×10 ⁻³	0.970	6	1.377	-1.40	8.64×10 ⁻⁴	0.992	4
-F	0.759	-2.86	5.2×10 ⁻³	0.996	6	1.366	-2.56	5.17×10 ⁻⁴	0.998	7	2.13	-1.64	6.18×10 ⁻⁵	0.995	3

Table 7. The Hammett Reaction Constant (ρ) for the Alkaline Hydrolysis of PBHA in 5% (v/v) Dioxane^{a)}

M/10 ³	0.0			0.54			1.37			2.88			10.4			22.9			40.0		
	ρ	r	SD	ρ	r	SD	ρ	r	SD	ρ	r	SD	ρ	r	SD	ρ	r	SD	ρ	r	SD
Surfactants	0.016	0.999	0.00138	0.420	0.963	0.0281	0.579	0.966	0.0370	—	—	—	0.213	0.999	0.903	—	—	—	0.257	0.944	0.068
HTAB	0.016	0.999	0.00138	0.0095	0.985	0.0130	0.4565	0.995	0.0118	—	—	—	0.190	0.983	0.0197	—	—	—	0.1537	0.999	0.0083
EHDAB	0.016	0.990	0.00138	—	—	—	—	—	—	0.119	0.993	0.0106	—	—	—	—	—	—	0.146	0.969	0.016
SDOS	0.016	0.990	0.00138	—	—	—	—	—	—	0.124	0.990	0.0151	—	—	—	—	—	—	0.206	0.913	0.051
LDS	0.016	0.990	0.00138	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—

a) r = Correlation coefficient. SD = Standard deviation.

summarised in Table 6. The slope of this plot ' n ' functionally denotes the index of cooperativity in micelle formation. A values of n greater than unity indicate positive cooperativity i.e. the binding of the first molecule of a substance, substrate molecule, makes it easier for subsequent molecules to bind. From the intercept, K_D has been evaluated without recourse to the cmc value, which is a variable parameter.

Substituent Effects. Not many reports of substituent effect and Hammett correlations in micellar environments are available.¹⁹ We examined the rate effects of substitution of NO_2 , CH_3 , OCH_3 , and F groups at the para position of the benzoyl group in *N*-phenylbenzohydroxamic acid. Differences in ρ for reactions in water and at micellar surfaces are more informative than raw data in analyzing medium effects of micelles on reactivity. The Hammett plot for the hydrolysis reaction in HTAB, EHDAB, LDS, and SDOS are fairly linear (Figure not shown). In all cases the magnitude of ρ (Table 7) is greater for micellar catalysed hydrolysis. This is typical for micellar catalysed reactions. The hydrolysis of hydroxamic acids generally involves an attack of the hydroxide ion on the carbonyl carbon atom, resulting in a negatively charged tetrahedral intermediate. Now micellar stabilization is felt by the whole transition state which carries a negative charge. However, stabilization should be greatest where the charge is most concentrated, i.e. in the OH^- , and should decrease with charge dispersal as we move to the transition state. Thus, stabilization decreases with new bond formation, and by the Hammond postulates, the transition state becomes more product-like than for the reaction in water. This is reflected in the large values for micellar reaction than for the non-micellar reaction.

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