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PHYSICAL CHEMICAL STUDIES OF THE SURFACTANT DILAURYLDIMETHYLAMMONIUM BROMIDE (DLDMAB): AGGREGATION AND CATALYTIC PROPERTIES

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ABSTRACT

The micellization of dilauryldimethylammonium bromide (DLDMAB) in water was studied by using surface tension measurements. The critical micellar concentration (CMC) was determined at 25°, 32° and 40 °C and thermodynamic parameters such as the free energy of micellization (ΔG^o_{mic})*, enthalpy* (ΔH^o_{mic}) *and entropy* (ΔS^o_{mic}) *of micellization were measured. The CMC at 25 °C was* 4.93×10^{-5} *M and the corresponding values of the thermodynamic parameters were:* $\Delta G^o_{mic} = -5.87$ *kcal/mol;* $\Delta H^o_{mic} = -1.12$ *kcal/mol and* $\Delta S^o_{mic} = +16.00$ *e.u. Micelles of the surfactant DLDMAB act as catalysts for the alkaline hydrolysis of p-nitrophenyl diphenyl phosphate (NPDPP) with a maximum catalytic factor of approximately 120 compared to 80 for CTAB. Typical activation parameters measured for* 1.00×10^{-3} *M surfactant and 0.005 M NaOH were:* $E_a = 9.7$ *kcal/mol;* $\Delta H^{o\neq} = 9.1$ *kcal/mol;* $\Delta G^{o\neq} = 19.6$ *kcal/mol and* $\Delta S^{o\neq} = -33.9$ *e.u. The kinetic results were also analyzed in terms of the pseudo-phase ion exchange models (PPIE) and showed that the model is applicable to describe the experimental results.*

KEYWORDS: dilauryldimethylammonium bromide; micellization; micellar catalysis; phosphate esters

RESUMO:

A micelização do brometo de dilaurildimetilamônio (DLDMAB) em água foi estudada por medidas de tensão superficial. A concentração micelar crítica (CMC) foi determinada a 25°, 32° e 40 °C e propriedades termodinâmicas tais como a energia livre (ΔG^o_{mic}) *de micelização, a entalpia* (ΔH^o_{mic}) *e a entropia* (ΔS^o_{mic}) *de micelização foram medidas. A 25 °C CMC foi de* $4{,}93 \times 10^{-5}$ *M e os valores correspondentes para os parâmetros termodinâmicos foram os seguintes:* $\Delta G^o_{mic} = -5{,}87$ *kcal/mol;* $\Delta H^o_{mic} = -1{,}12$ *kcal/mol e* $\Delta S^o_{mic} = +16{,}00$ *e.u. Micelas do surfactante DLDMAB atuam como catalisadoras para a hidrólise alcalina do p-nitrofenil difenil fosfato (NPDPP), com um fator catalítico máximo de aproximadamente 120, comparável ao de 80 do CTAB. Parâmetros de ativação representativos medidos experimentalmente para DLDMAB* $1{,}00 \times 10^{-3}$ *M e NaOH 0,005 M foram:* $E_a = 9{,}7$ *kcal/mol;* $\Delta H^{o\neq} = 9{,}1$ *kcal/mol;* $\Delta G^{o\neq} = 19{,}2$ *kcal/mol e* $\Delta S^{o\neq} = -33{,}9$ *e.u. Os resultados cinéticos foram analisados em termos do modelo de pseudofase de troca iônica (PPIE) e mostraram que o modelo é aplicável para a descrição dos resultados experimentais.*

PALAVRAS CHAVE: brometo de dilaurildimetilamônio, micelização, catálise micelar, ésteres de fosfato

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INTRODUCTION

This article reports the results obtained for the study of the micellization of dilauryldimethylammonium bromide (DLDMAB) and its application in the micellar catalyzed hydrolysis of p-nitrophenyl diphenyl phosphate.

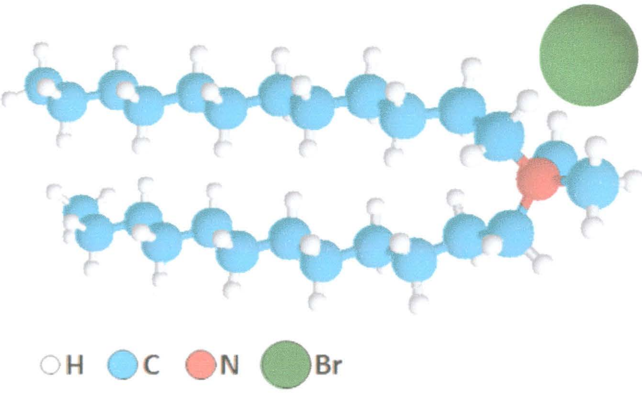


Figure 1. Structural formula of the dilauryldimethylammonium bromide (DLDMAB). **Software ACD/ChemSketch.**

As a part of a systematic study of the micellization process we have studied a variety of surfactants in water, non-aqueous solvents and water solutions containing co-solvents or additives using experimental methods such as surface tensiometry, nuclear magnetic resonance (NMR) and quasi-elastic light scattering (QELS) [1-14]. The study of the surfactant under consideration, DLDMAB, was motivated by its similarity to cetyltrimethylammonium bromide, CTAB, the main difference being the presence of two shorter, C12, carbon tails.

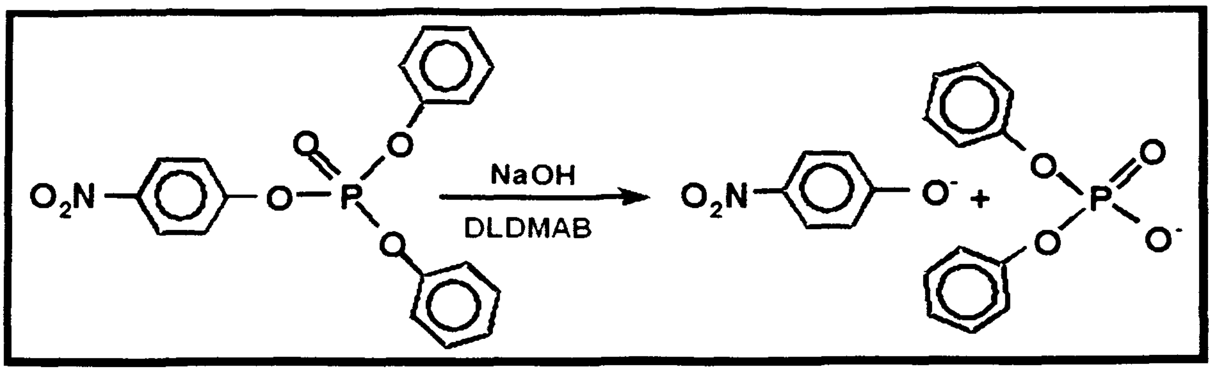
Organic phosphorus compounds, and in particular phosphate esters, are of paramount biological and pharmacological importance and have been widely used as drugs, nerve gases and pesticides [15-17].

In studies described in the literature we have reported the hydrolysis of di- and tri-substituted phosphate esters in the presence of micelles of a variety of surfactants, including some that form functional micelles [18-21].

The micellar catalyzed oxidative cleavage of a carbon-carbon bond in Dicofol(TM) [22] and the micellar catalyzed dehydrochlorination of 1,1,1-trichloro-2,2-bis(*p*-chlorophenyl)ethane (DDT) and some of its derivatives have also been subject of our investigations [23,24].

We have also reported results obtained for the hydrolysis of p-nitrophenyl diphenyl phosphate in aqueous solutions in the presence of micelles of diethyl heptadecyl imidazolinium ethyl sulfate (DEHIES) and CTAB, sodium hydroxide and dimethylsulfoxide (DMSO) and analyzed the effect of internal pressure of the medium, dielectric constant, donor number and polarity of the solvent and the effect of DMSO on micellization [25-29]. In a more recent article we have described the physical chemical studies of the aggregation and catalytic properties of the surfactant cetyldimethylethylammonium bromide (CDEAB) [30].

The present article deals with the study of the hydrolysis of p-nitrophenyl diphenyl phosphate (NPDPP) in the presence of micelles of dilauryldimethylammonium bromide (DLDMAB) in aqueous solutions containing NaOH, as illustrated by the Scheme 1.



Scheme 1. Hydrolysis of p-nitrophenyl diphenyl phosphate (NPDPP) in the presence of micelles of dilauryldimethylammonium bromide (DLDMAB) within aqueous solutions containing sodium hydroxide (NaOH).

EXPERIMENTAL PROCEDURE

Materials. The p-nitrophenyl diphenyl phosphate (NPDPP) was prepared using standard methods [31,32]. A sample was also obtained from Prof. Fred Menger, Emory University, Atlanta, Georgia, USA. The surfactant dilauryldimethylammonium bromide (DLDMAB) was purchased from Chem. Service, West Chester, Pa., USA. The sodium hydroxide was analytical reagent grade and was purchased from Merck Co.

Surface Tension Measurements. All solutions were prepared volumetrically with deionized double distilled water and contained a series of at least fifteen different concentrations of DLDMAB. The surface tension of the DLDMAB-H_2O solutions was measured at 25°, 32° and 40 °C by means of a Fisher Model 21, Semi-Automatic Tensiometer. Ten milliliters aliquots of the solutions were placed in a Petri dish with a diameter of 6 cm. The temperature of the solutions was brought to the chosen temperature using a water bath and the Petri dish was kept at the desired temperature by placing it in a container through which water was circulated from the constant temperature bath. The tensiometer was set a constant height. The final surface tension of any solution was the average of at least three independent measurements.

The critical micellar concentrations (CMC's) were determined from plots of the surface tension of the solutions versus the concentration or log concentration of DLDMAB. The marked change in the plots was taken as an indication of micelle formation and the inflection point was considered to correspond to the CMC [33].

The thermodynamic parameters ΔG^o_{mic}, ΔH^o_{mic} and ΔS^o_{mic} were determined using standard equations [34,35] derived on the basis of the assumption that the process of micellization involves the formation of a distinct micellar phase at the CMC and that the concentration of monomers in solution is constant, once micelles are formed. The experimental accuracy in the values determined for ΔG^o_{mic} is about ± 100 cal/mole. On the other hand, ΔH^o_{mic} and ΔS^o_{mic} are more approximate since they were calculated on the basis of measurements at three temperatures only.

Kinetic Measurements. The hydrolysis of p-nitrophenyl diphenyl phosphate was studied spectrophotometrically by measuring the rate of appearance of the p-nitrophenoxide anion at 4030 Å with a Varian DMS-80 spectrophotometer equipped with a temperature controlled cell compartment. The reaction was studied at 15°, 25° and 35°C at various concentrations of NaOH and MDBTACL. The pseudo-first order rate constant (k_ψ), in s^{-1}, was determined from linear plots of logarithm of absorbance versus time and the second order rate constants (k_{2m}) in the micellar phase and (k_2^0) in the aqueous phase, in $s^{-1}M^{-1}$, were calculated from k_ψ and the hydroxide ion concentration. Activation parameters such as the activation energy (E_a), the activation enthalpy ($\Delta H^{o\neq}$) and the activation entropy ($\Delta S^{o\neq}$) were determined from experimental k_ψ values measured at three different temperatures using the following equations.

$$\ln k_\psi = \ln A - (E_a/R)(1/T) \quad (1)$$

$$\Delta H^{o\neq} = E_a - RT \quad (2)$$

$$\Delta S^{o\neq} = 4.576\,(\log k_\psi - 10.753 - \log T + E_a/4.576T) \quad (3)$$

$$\Delta G^{o\neq} = \Delta H^{o\neq} + \Delta S^{o\neq} \quad (4)$$

where, R corresponds to the gas constant and T to the absolute temperature.

RESULTS AND DISCUSSION

Representative results obtained for the surface tension of DLDMAB in water solutions at 25°, 32° and 40°C are illustrated in Figure 2.

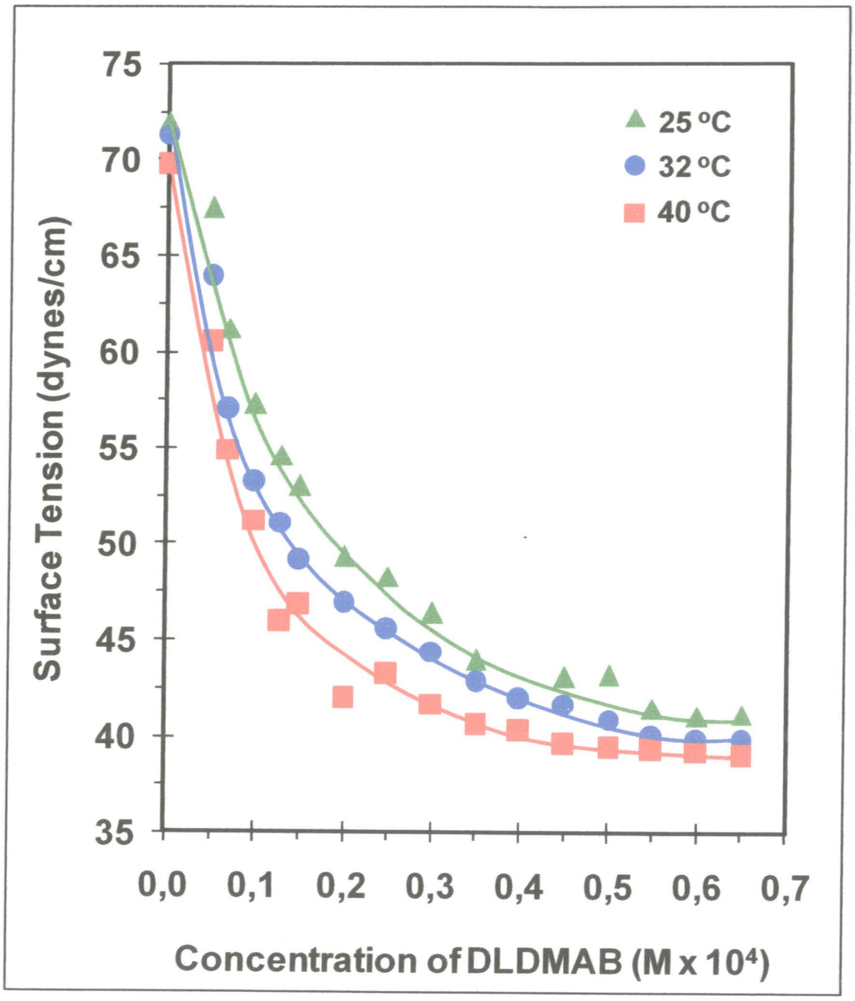


Figure 2. Plot of Surface Tension versus Concentration of Dilauryldimethylammonium Bromide (DLDMAB) in Water at 25°, 32° and 40°C.

All plots of surface tension versus the concentration of DLDMAB exhibited initial marked drops and subsequent leveled off. The inflection point in the given curve was taken as the CMC. At times, plots of surface tension versus the logarithm of the concentration of surfactant gave a better determination for the CMC. Such results are shown in Figure 3 for the same temperatures.

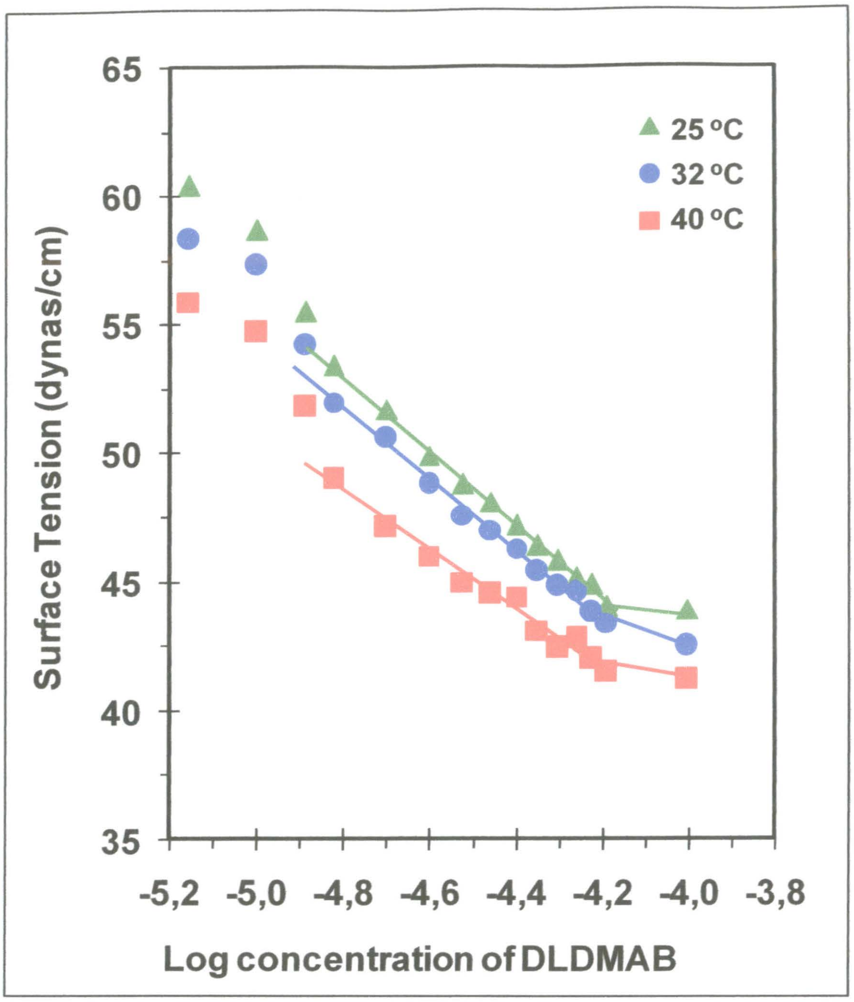


Figure 3. Plot of Surface Tension versus Logarithm of the Concentration of Dilauryldimethylammonium bromide (DLDMAB) in Water at 25° and 32° and 40°C.

The experimental results obtained for the critical micellar concentration (CMC) are shown in Table I and compared to cetyltrimethylammonium bromide (CTAB). Table II shows the experimental values obtained for the thermodynamic functions, i.e., the standard free energy of micellization, ΔG^o_{mic}, the enthalpy, ΔH^o_{mic}, and the standard entropy of micellization ΔS^o_{mic} at 25°C, again compared to CTAB [7,11,14]. As expected, the difference between the experimental values obtained for the CMC and the thermodynamic properties for the two surfactants is small, the difference being mainly due to the nature of the tails.

Table I. Critical Micellar Concentration (CMC) of Dilauryldimethylammonium Bromide (DLDMAB) in Aqueous Solutions Compared to CTAB [14,29]

Surfactant	**Temperature (°C)**		
	25	**32**	**40**
DLDMAB	4.93×10^{-5} M	5.24×10^{-5} M	5.40×10^{-5} M
CTAB	9.00×10^{-4} M	--	10.00×10^{-4} M

Table II. Some Thermodynamic Properties for the Formation of Micelles of Dilauryldimethylammonium Bromide (DLDMAB) in Water at 25 °C Compared to CTAB [14,29]

Surfactant	**Free Energy of Micellization at 25 °C ΔG^{0}_{mic} (kcal/mole)**	**Enthalpy of Micellization ΔH^{0}_{mic} (kcal/mole)**	**Entropy of Micellization at 25 °C ΔS^{0}_{mic} (e.u.)**
DLDMAB	-5.87	-1.12	+16.00
CTAB	-4.14	-1.03	+10.43

Typical profiles for the pseudo-first order rate constant, k_{ψ}, as a function of the concentration of DLDMAB for the hydrolysis of p-nitrophenyl diphenyl phosphate (NPDPP) at 25°C in aqueous solutions containing NaOH between 0.002 M to 0.005 M are shown in Figure 4 and between 0.0005 and 0.005 M in Figure 5.

The experimental rate profiles obtained in the presence of DLDMAB are characteristic of micellar catalyzed reaction in aqueous solutions with a maximum at 1.0×10^{-3}M. The addition of DLDMAB to the reaction medium causes an increase in the rate of hydrolysis up to a point (the maximum in rate) where there is total incorporation of the substrate in the micellar phase. Subsequent addition of the surfactant causes a decrease in the reaction rate, probably due to the dilution of the reactive counter-ions in the Stern layer of a higher number of micelles. The catalytic factor measured is approximately 120 compared to 80 for CTAB and CDEAB [29]. The higher catalytic factor (k_{ψ}/k_{w}) can probably be explained by the presence of the two hydrophobic tails of the surfactant DLDMAB.

Representative activation parameters determined for the reaction with DLDMAB are shown in Table III and compared to CTAB and CDEAB [30]. As can be seen from the analysis of the results the activation parameters for the three surfactants are comparable and similar and to others measured for micellar catalyzed reactions [30,31].

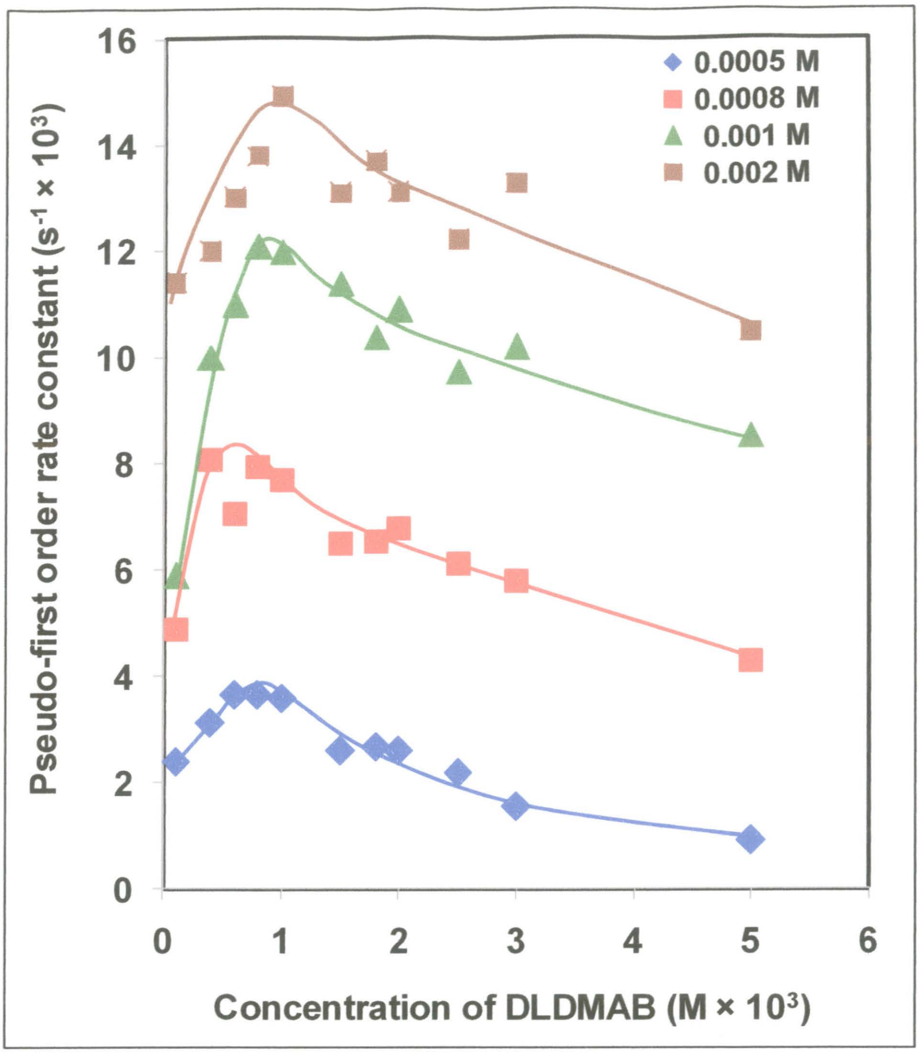


Figure 4. Rate Profiles for the Hydrolysis of p-Nitrophenyl Diphenyl Phosphate in Aqueous Solutions of NaOH (0.0005 – 0.002 M) and Various Concentrations of MDBTACL at 25°C

Table III. Activation Parameters for the Hydrolysis of p-Nitrophenyl Diphenyl Phosphate in Aqueous Solutions of 0.005 M NaOH in the Presence of DLDMAB, CDEAB and CTAB [28-30] at 25 °C.

Surfactant	**Concentration (M)**	**E_a (kcal/mole)**	**$\Delta H^{0\neq}$ (kcal/mole)**	**$\Delta G^{0\neq}$ (kcal/mole)**	**$\Delta S^{0\neq}$ (e.u.)**
No surfactant	--	+ 15.2	+14.6	+21.3	-22.2
DLDMAB	1,0 x 10^{-3}	+ 9.7	+ 9.1	+19.2	-33.9
CDEAB	18 x 10^{-4}	+ 9.0	+ 8.4	+19.2	-36.3
CTAB	15 x 10^{-4}	+ 11.4	+10.8	+18.7	-26.6
CTAB	20 x 10^{-4}	+ 10.5	+ 9.9	+18.8	-29.9

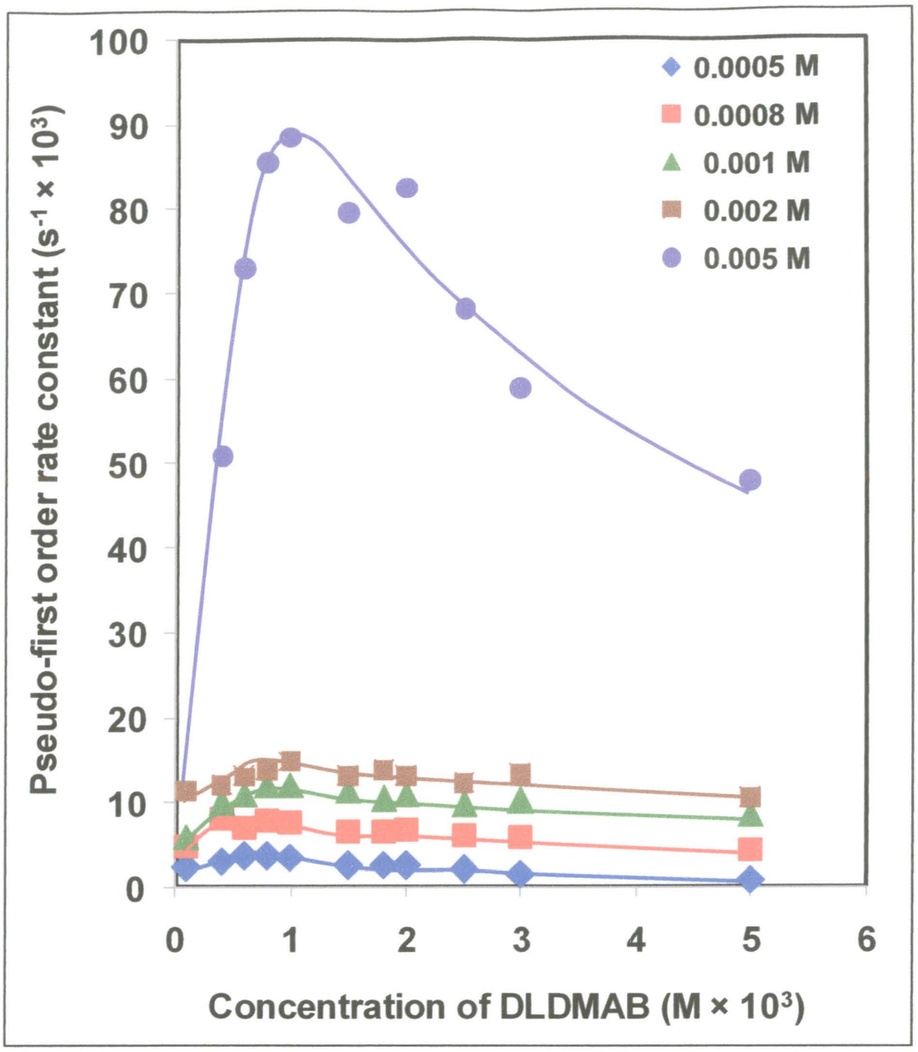


Figure 5. Rate Profiles for the Hydrolysis of p-Nitrophenyl Diphenyl Phosphate in Aqueous Solutions of NaOH (0.0005 – 0.005 M) and Various Concentrations of MDBTACL at 25°C

Most of the models proposed for micellar catalysis [34-55] consider the partition coefficient for the substrate between the micellar and aqueous phase and the distribution of the reagents between the two phases. The hydrolysis of NPDPP with hydroxide ion in the presence of DLDMAB may be considered a bimolecular reaction of OH^- ion and the substrate. Since the concentration of OH^- in the micellar phase is dependent on the concentration of both bromide ions and surfactant, a quantitative treatment of the reaction rate must consider ion exchange on or near the micellar surface. For the reaction under consideration, the model proposed by Quina and Chaimovich [41] reduces to Equation 5, which gives the theoretical dependence of the pseudo-first order constant, k_ψ, as a function of the total hydroxide ion concentration:

$$k_{\psi} = \frac{\{(k_{2m}/V)K_s K_{OH/Br}[(Br)_m/(Br)_w] + k_2^0\}(OH)_T}{(1 + K_s C_D)[1 + K_{OH/Br}(Br)_m(Br)_w]} \qquad (5)$$

where, C_D is the concentration of micellized surfactant, V is the molar volume of the reactive region at the micellar surface, k_ψ is the pseudo-first order rate constant, k_{2m} is the second order rate constant in the micellar phase, k_2^0 is the second order constant in the aqueous phase, $K_{OH/Br}$ is the ion exchange constant, K_s is the binding constant for the substrate, $(Br)_m$ is the concentration of Br^- in micellar phase, $(Br)_w$ is the concentration of Br^- in aqueous phase, $(OH)_T$ is the total concentration of hydroxide ions and V is the molar volume of surfactant.

With substrates such as p-nitrophenyl diphenyl phosphate that are very insoluble in water and are solubilized by DLDMAB the expression for k_ψ can be reduced to a simpler form given by Equation (6):

$$k_\psi = \frac{k_{2m}}{C_D V}(OH)_T \frac{K_{OH/Br}[(Br)_m/(Br)_w]}{[1 + K_{OH/Br}(Br)_m(Br)_w]} \qquad (6)$$

The concentration of Br^- in the micellar and aqueous phases can be obtained using the following equations [38-42]:

$$A_I = C_D + CMC + K_{OH/Br}\,(OH)_T + (1 - \alpha)\,C_D\,K_{OH/Br} \qquad (7)$$

$$(OH)_m = \frac{(-A_1) + \left[(A_1)^2 + 4(1 - K_{OH/Br})(OH)_T K_{OH/Br}(1 - \alpha)c\right]^{0.5}}{2(1 - K_{OH/Br})} \qquad (8)$$

$$(Br)_m = (1 - \alpha)\,C_D - (OH)_m \qquad (9)$$

$$(Br)_w = \alpha\,C_D + CMC + (OH)_m \qquad (10)$$

where CMC is the critical micellar concentration, α is the degree of ionization of the micelle and $(OH)_m$ is the concentration of OH^- in the micellar phase. For systems exhibiting saturation behavior, further analysis of the results using the PPIE treatment as modified to account for HO^-/Br^- exchange allows the evaluation of substrate binding constants, K_S, and micellar rate constants, k_{2m}.[49]

We have calculated the theoretical values of k_ψ for the reaction discussed above using $\overline{V} = 0.37$ L/mol [44]; $K_{OH/Br} = 0.08$ [45]; $\alpha = 0.20$ [46,47] and various concentrations of DLDMAB and NaOH.

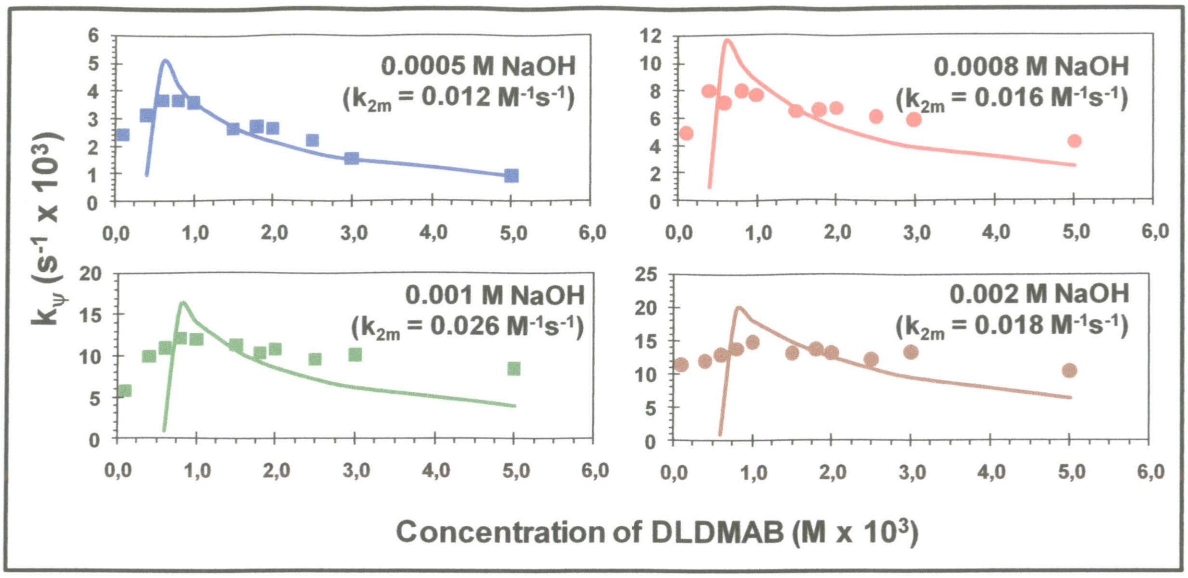


Figure 6. Experimental and Theoretical (—) k_ψ Values for the Hydrolysis of p-Nitrophenyl Diphenyl Phosphate in Aqueous Solutions of NaOH and Various Concentrations of DLDMAB at 25°C.

The values for k_ψ were calculated using different values for k_{2m} ranging from 0.012 $M^{-1}s^{-1}$ and 0.026 $M^{-1}s^{-1}$ and are illustrated in Figure 6. As can be seen, the pseudo-phase ion exchange model gives a reasonable agreement for this micellar catalyzed reaction. At low concentrations of DLDMAB (bellow the CMC), the presence of the highly hydrophobic solute probably induces the formation of kinetically active premicelles.

ACKNOWLEDGMENTS

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