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# PHYSICAL CHEMICAL STUDIES OF THE AGGREGATION AND CATALYTIC PROPERTIES OF THE SURFACTANT METHYLDODECYLBENZYLTRIMETHYLAMMONIUM CHLORIDE (MDBTACL)

**Lavinel G. Ionescu[a,b] Sílvia Dani[b] and Elizabeth Fátima de Souza[c]**

[a] Departamento de Química Pura, Faculdade de Química, Pontifícia Universidade Católica do Rio Grande do Sul - PUCRS, Porto Alegre, RS, Brazil
[b] Departamento de Química, Centro de Ciências Naturais e Exatas, Universidade Luterana do Brazil - ULBRA, Canoas, RS, Brazil
[c] Instituto de Ciências Biológicas e Química, Pontifícia Universidade Católica de Campinas - PUC-CAMPINAS, Campinas, SP, Brazil

## ABSTRACT

*The micellization of methyldodecylbenzyltrimethylammonium chloride (MDBTACL) in water was studied by means of surface tensiometry. The critical micellar concentration (CMC) was determined at 25°, 32° and 40 °C and thermodynamic properties such as the free energy of micellization ($\Delta G^o_{mic}$), enthalpy ($\Delta H^o_{mic}$) and entropy ($\Delta S^o_{mic}$) of micellization were measured. The CMC at 25 °C was $1.12 \times 10^{-2}$ M and the corresponding values of the thermodynamic parameters were: $\Delta G^o_{mic} = -2.66$ kcal/mol; $\Delta H^o_{mic} = -0.82$ kcal/mol and $\Delta S^o_{mic} = +6.17$ e.u. Micelles of the surfactant MDBTACL were good catalysts for the alkaline hydrolysis of p-nitrophenyl diphenyl phosphate (NPDPP) with a maximum catalytic factor of approximately 60, comparable to that of CTAB. Typical activation parameters measured for 0.012 M surfactant and 0.005 M NaOH were: $E_a = 8.5$ kcal/mol; $\Delta H^{o\neq} = 7.8$ kcal/mol; $\Delta G^{o\neq} = 19.6$ kcal/mol and $\Delta S^{o\neq} = -39.3$ 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 and gave reasonable fits.*

**KEYWORDS:** methyldodecylbenzyltrimethylammonium chloride; micellization; micellar catalysis; phosphate esters

## RESUMO:

*A micelização do cloreto de metildodecilbenziltrimetilamônio (MDBTACL) em água foi estudada por métodos de tensiometria superficial. A concentração micelar crítica (CMC) foi determinada a 25°, 32° e 40 °C e propriedades termodinâmicas tais como a energia livre ($\Delta G^o_{mic}$) de micelização, a entalpia ($\Delta H^o_{mic}$) e a entropia ($\Delta S^o_{mic}$) de micelização foram medidas. A CMC a 25 °C foi $1,25 \times 10^{-2}$ M e os valores correspondentes para os parâmetros termodinâmicos foram os seguintes: $\Delta G^o_{mic} = -2,66$ kcal/mol; $\Delta H^o_{mic} = -0,82$ kcal/mol e $\Delta S^o_{mic} = +6,17$ e.u. Micelas do surfactante MDBTACL foram bons catalisadores para a hidrólise alcalina do p-nitrofenil difenil fosfato, com um fator catalítico máximo de aproximadamente 60, comparável ao do CTAB. Parâmetros de ativação representativos medidos experimentalmente para MDBTACL 0,012 M e 0,005 M NaOH foram: $E_a = 8,5$ kcal/mol; $\Delta H^{o\neq} = 7,8$ kcal/mol; $\Delta G^{o\neq} = 19,6$ kcal/mol e $\Delta S^{o\neq} = -39,3$ e.u. Os resultados cinéticos foram analisados em termos do modelo de pseudofase de troca iônica (PPIE) e mostraram que o modelo é aplicável e satisfatório.*

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# INTRODUCTION

This article reports the results obtained for the study of the micellization of methyldodecylbenzyltrimethylammonium chloride [MDBTACL, $(CH_3)N^+CH_2(C_6H_4)CH_2(CH_2)_9CH(CH_3)_2\ Cl^-$] and its application in the micellar catalyzed hydrolysis of p-nitrophenyl diphenyl phosphate.

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, MDBTACL, was motivated by its similarity to cetyltrimethylammonium bromide, CTAB, the main difference being the counter-ion chloride, $Cl^-$.

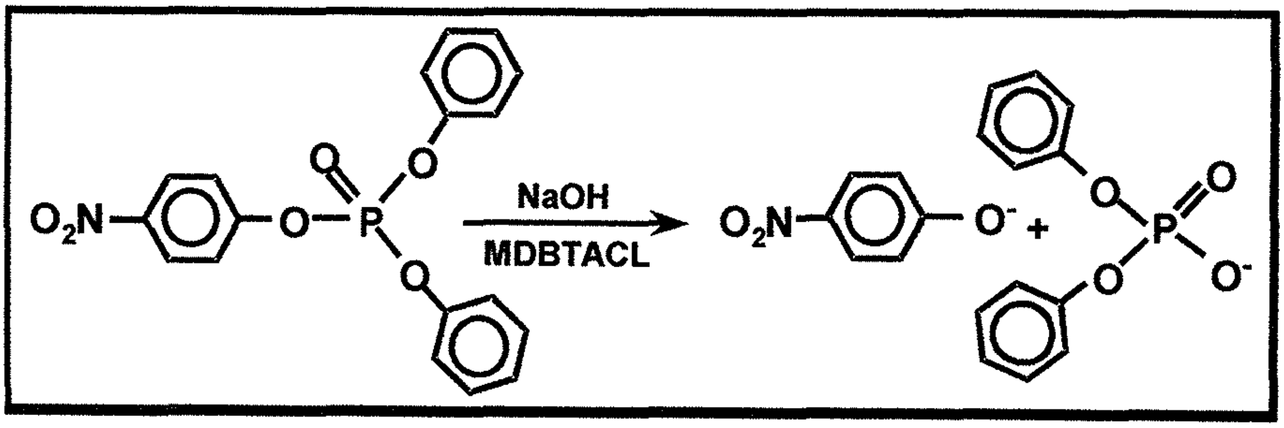
Organic phosphorus compounds, and in particular phosphate esters, are of paramount biological and pharmacological importance and have been widely used as drugs, nerve gases and pesticides [15,16].

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 [17-20].

The micellar catalyzed oxidative cleavage of a carbon-carbon bond in Dicofol(TM) [21] 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 [22,23].

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 [24-28]. In a more recent article we have described the physical chemical studies of the aggregation and catalytic properties of the surfactant cetyldimethylethylammonium bromide (CDEAB) [29].

The present article deals with the study of the hydrolysis of p-nitrophenyl diphenyl phosphate (NPDPP) in the presence of micelles of methyldodecylbenzyltrimethylammonium chloride (MDBTACL) in aqueous solutions containing NaOH, as illustrated by the following scheme:



## EXPERIMENTAL PROCEDURE

**Materials.** The p-nitrophenyl diphenyl phosphate (NPDPP) was prepared using standard methods [30,31]. A sample was also obtained from Prof. Fred Menger, Emory University, Atlanta, Georgia, USA. The surfactant methyldodecylbenzyltrimethylammonium chloride (MDBTACL) 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 MDBTACL. The surface tension of the MDBTACL-$H_2O$ 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 MDBTACL. 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.

The thermodynamic parameters $\Delta G^{o}_{mic}$, $\Delta H^{o}_{mic}$ and $\Delta S^{o}_{mic}$ were determined using standard equations [33,34] 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 $\Delta G^{o}_{mic}$ is about ± 100 cal/mole. On the other hand, $\Delta H^{o}_{mic}$ and $\Delta S^{o}_{mic}$ 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 ($k_\psi$), 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 $k_\psi$ and the hydroxide ion concentration. Activation parameters such as the activation energy ($E_a$), the activation entalphy ($\Delta H^{o\neq}$) and the activation entropy ($\Delta S^{o\neq}$) were determined from experimental $k_\psi$ values measured at three different temperatures using the following equations.

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

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

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

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

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

## RESULTS AND DISCUSSION

Typical experimental results obtained for the surface tension of MDBTACL in water solutions at 25°, 32° and 40°C are shown in Figure 1.

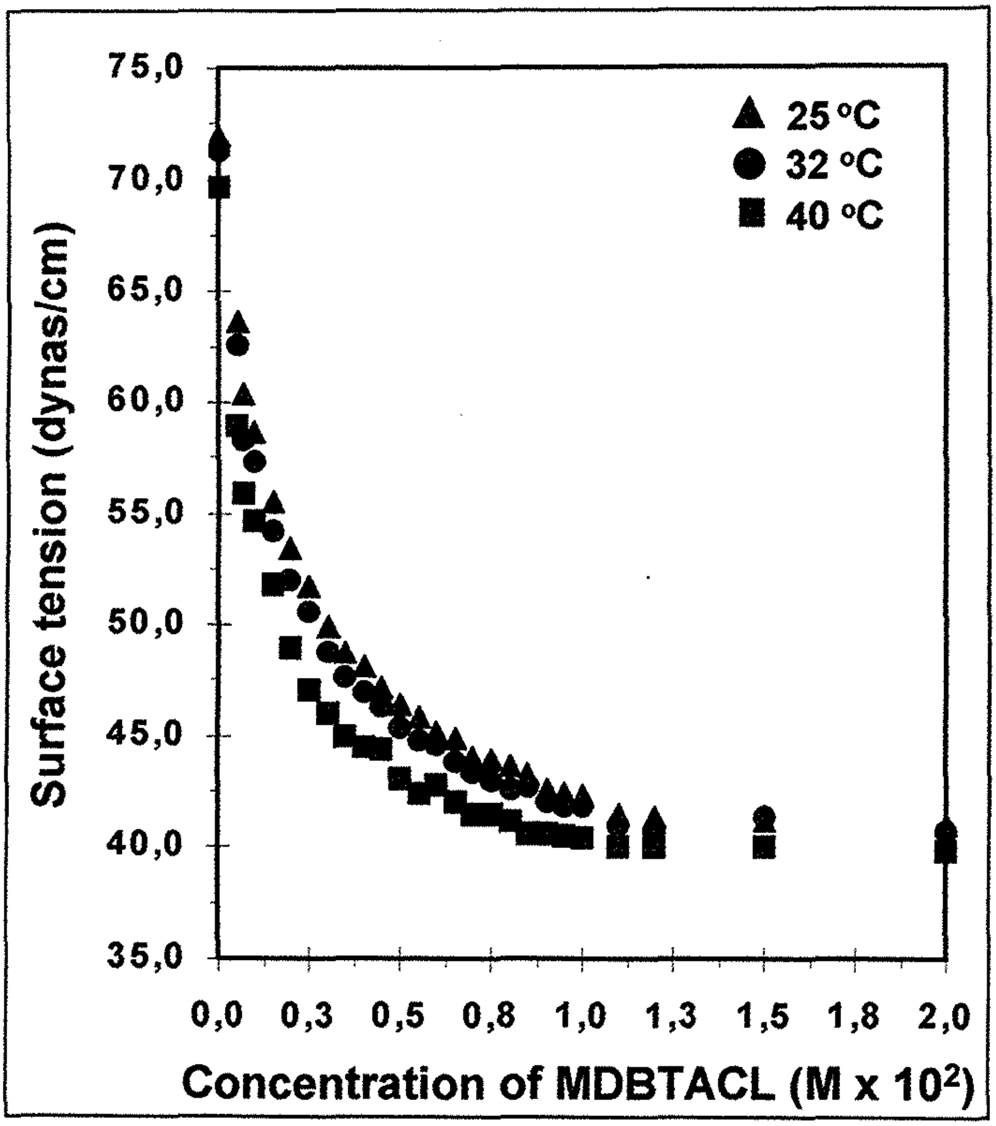


**Figure 1.** Plot of Surface Tension versus Concentration of Methyldodecylbenzyltrimethylammonium Chloride (MDBTACL) in Water at 25°, 32° and 40°C.

All plots of surface tension versus the concentration of MDBTACL 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 2 for the same temperatures.

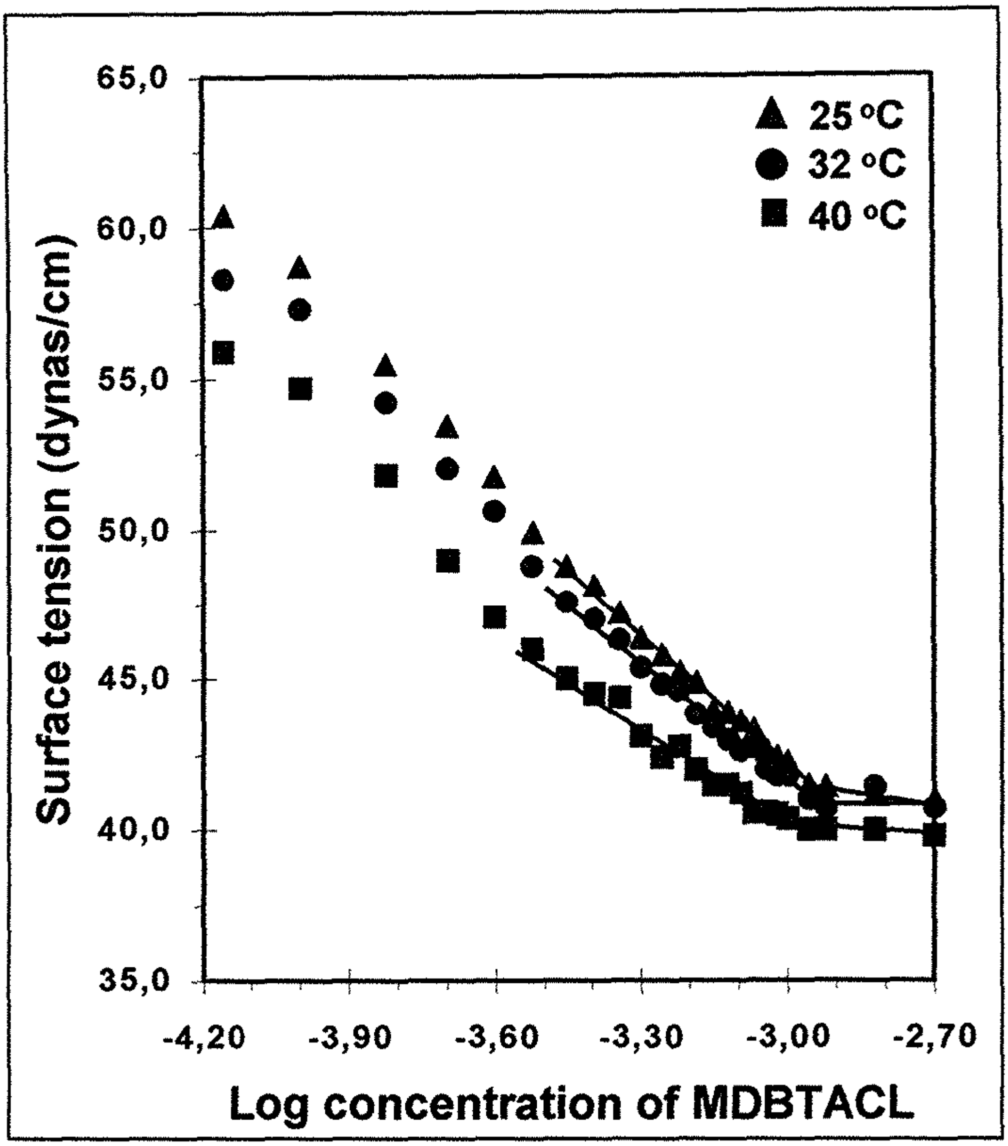


**Figure 2.** Plot of Surface Tension versus Logarithm of the Concentration of Methyldodecylbenzyltrimethylammonium Chloride (MDBTACL) in Water at 25° and 32° and 40°C.

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, $\Delta G^{o}_{mic}$, the enthalpy, $\Delta H^{o}_{mic}$, and the standard entropy of micellization $\Delta S^{o}_{mic}$ 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 very large, the difference being mainly due to the counter-ion that changes from $Cl^-$ to $Br^-$.

**Table I.** Critical Micellar Concentration (CMC) of Methyldodecylbenzyltrimethylammonium Chloride (MDBTACL) in Aqueous Solutions Compared to CTAB [14,29]

| **Surfactant** | **Temperature (°C)** | | |
|---|---|---|---|
| | **25** | **32** | **40** |
| **MDBTACL** | $1.12 \times 10^{-2}$ M | $1.15 \times 10^{-2}$ M | $1.15 \times 10^{-2}$ M |
| **CTAB** | $9.00 \times 10^{-4}$ M | -- | $10.0 \times 10^{-4}$ M |

**Table II.** Some Thermodynamic Properties for the Formation of Micelles of Methyldodecylbenzyltrimethylammonium Chloride (MDBTACL) in Water at 25 °C Compared to CTAB [14,29]

| **Surfactant** | **Free Energy of Micellization at 25 °C $\Delta G^{o}_{mic}$ (kcal/mole)** | **Enthalpy of Micellization $\Delta H^{o}_{mic}$ (kcal/mole)** | **Entropy of Micellization at 25 °C $\Delta S^{o}_{mic}$ (e.u.)** |
|---|---|---|---|
| MDBTACL | -2.66 | -0.82 | +6.17 |
| CTAB | -4.14 | -1.03 | +10.43 |

Typical profiles for the pseudo-first order rate constant, $k_\psi$, as a function of the concentration of MDBTACL for the hydrolysis of p-nitrophenyl diphenyl phosphate (NPDPP) at 25°C in aqueous solutions containing NaOH between 0.003 M to 0.010 M are shown in Figure 3.

The experimental rate profiles obtained are characteristic of micellar catalyzed reaction in aqueous solutions with a maximum at $1.0 \times 10^{-2}$ M MDBTACL, considerably higher than the concentrations observed for surfactants having $Br^-$ as the counter-ion. The addition of MDBTACL 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 60 compared to 80 for CTAB and CDEAB [29].

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

| **Surfactant** | **Concentration (M)** | **$E_a$ (kcal/mole)** | **$\Delta H^{o\neq}$ (kcal/mole)** | **$\Delta G^{o\neq}$ (kcal/mole)** | **$\Delta S^{o\neq}$ (e.u.)** |
|---|---|---|---|---|---|
| -- | -- | + 15.2 | +14.6 | +21.3 | -22.2 |
| MDBTACL | $12 \times 10^{-1}$ | + 8.6 | + 7.9 | +19.6 | -39.3 |
| CDEAB | $18 \times 10^{-4}$ | + 9.0 | + 8.4 | +19.2 | -36.3 |
| CTAB | $15 \times 10^{-4}$ | + 11.4 | +10.8 | +18.7 | -26.6 |
| CTAB | $20 \times 10^{-4}$ | + 10.5 | + 9.9 | +18.8 | -29.9 |

Representative activation parameters determined for the reaction with MDBTACL are shown in Table III and compared to CTAB and CDEAB [29]. 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 [29,30].

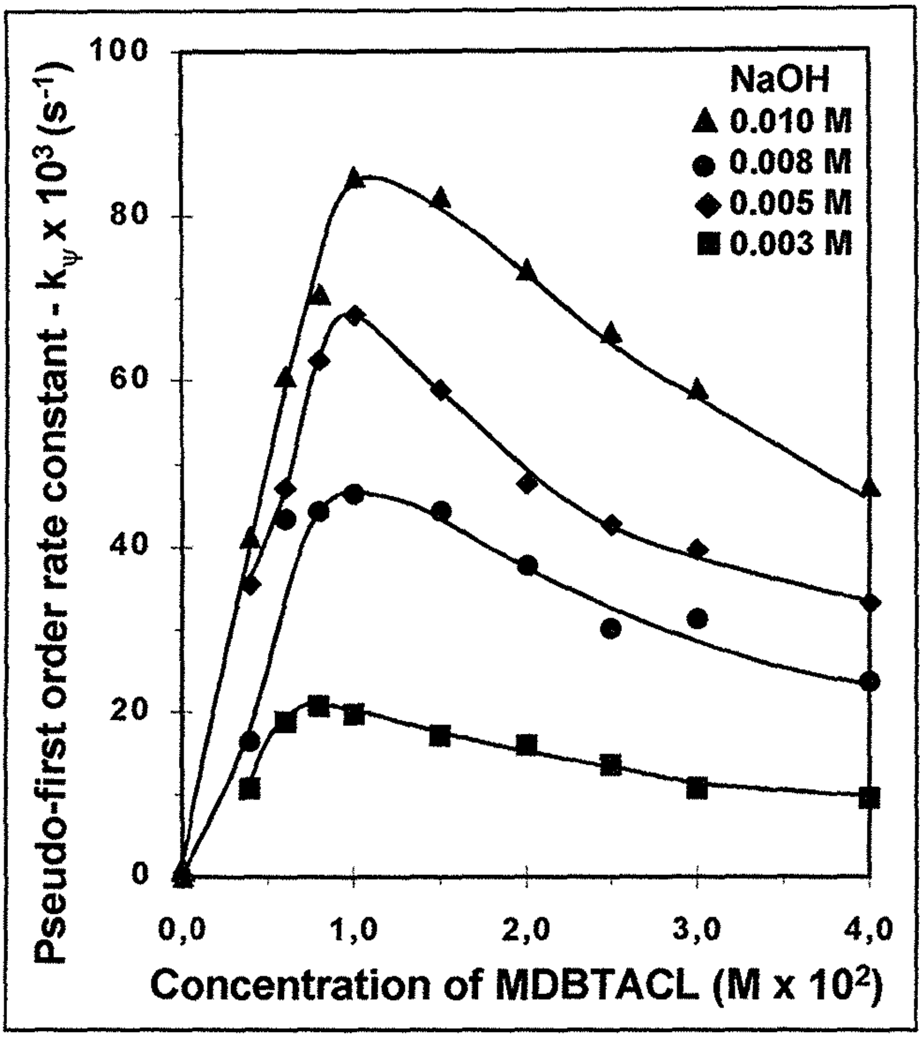


**Figure 3.** Rate Profiles for the Hydrolysis of p-Nitrophenyl Diphenyl Phosphate in Aqueous Solutions of NaOH and Various Concentrations of MDBTACL at 25°C

Most of the models proposed for micellar catalysis [33-39] 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 MDBTACL 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 chloride 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[40] reduces to Equation 5, that gives the theoretical dependence of the pseudo-first order constant, $k_\psi$, as a function of the total hydroxide ion concentration

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

where, $C_D$ is the concentration of micellized surfactant, V is the molar volume of the reactive region at the micelar surface, $k_\psi$ 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, $K_{OH/Cl}$ is the ion exchange constant, $K_s$ is the binding constant for the substrate, $(Cl)_m$ is the concentration of $Cl^-$ in micellar phase, $(Cl)_w$ is the concentration of $Cl^-$ 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 MDBTACL the expression for $k_\psi$ can be reduced to a simpler form given by Equation (6):

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

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

$$A_1 = C_D + CMC + K_{OH/Cl}\,(OH)_T + (1 - \alpha)\, C_D\, K_{OH/Cl} \qquad (7)$$

$$(OH)_m = \frac{(-A_1) + [(A_1)^2 + 4\,(1 - K_{OH/Cl})\,(OH)_T\, K_{OH/Cl}\,(1 - \alpha)\, C\,]^{0.5}}{2\,(1 - K_{OH/Cl})} \qquad (8)$$

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

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

where CMC is the critical micellar concentration, $\alpha$ is the degree of ionization of the micelle and $(OH)_m$ is the concentration of $OH^-$ in the micellar phase.

We have calculated the theoretical values of $k_\psi$ for the reaction discussed above using $\overline{V} = 0.35$ L/mol[43]; $K_{OH/Cl} = 0.10$[44]; $\alpha = 0.40$[45,46] and various concentrations of MDBTACL and NaOH.

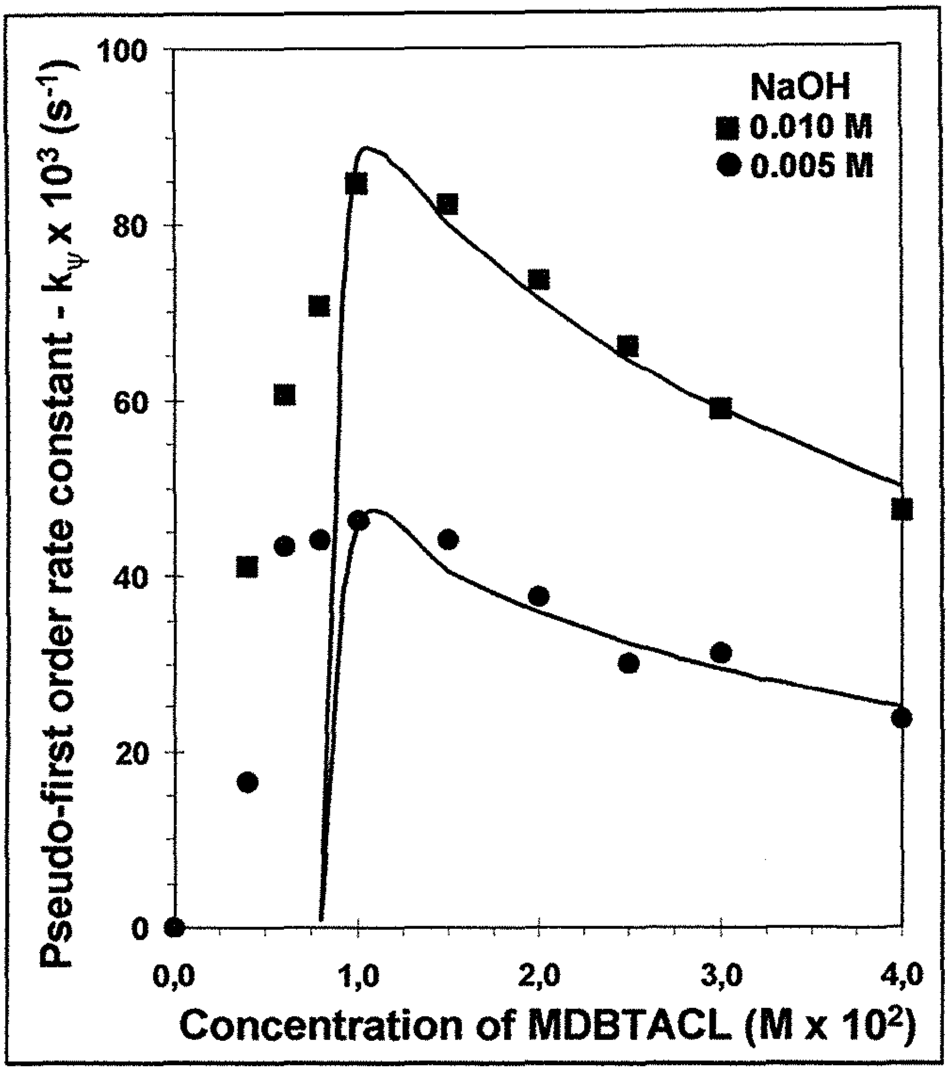


**Figure 4.** Experimental and Theoretical (——) $k_\psi$ Values for the Hydrolysis of p-Nitrophenyl Diphenyl Phosphate in Aqueous Solutions of NaOH and Various Concentrations of MDBTACL at 25°C.

The values for $k_\psi$ were calculated using different values for $k_{2m}$ ranging from 0.60 $M^{-1}s^{-1}$ and 1.20 $M^{-1}s^{-1}$. The best experimental fits were obtained for $k_{2m}$ = 0.87 $M^{-1}s^{-1}$ and are illustrated in Figure 4. As can be seen, the pseudo-phase ion exchange model gives a reasonable agreement for this micellar catalyzed reaction. At low concentrations of MDBTACL (bellow the CMC), the presence of the highly hydrophobic solute induces the formation of kinetically active premicelles.

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