



## Conductometric Study of the Interaction of Insecticide Profenofos with Cationic and Anionic Surfactants in Aqueous Medium

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### **Authors' contributions**

*This work was carried out in collaboration between all authors. Author IS designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors DDT and AC managed the analyses of the study, revised and corrected the manuscript. Authors EHTB, SS, AM, PAD, MDGS and AT managed the literature searches. All authors read and approved the final manuscript.*

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### **ABSTRACT**

This work aims to find means to improve the solubility of the not very water soluble organic pollutants. For that, we made a study of solubilization of a not very water soluble organophosphorus insecticide, and in weak concentrations in the ionic micellar systems, in the presence of active surface CTAC and SDS. With an aim of better understanding this mechanism of solubilization of insecticide profenofos (PFF). Concentration effect of PFF and temperature on the CMC have been studied by conductometric way and the determination of the thermodynamic parameters of micelization. It was noted in this work that the CMC and the values of the degree of electrolytic dissociation of surfactant used decrease with the effect PFF concentration in solution and according to the temperature of the medium. The results of this study showed on the one hand that the Gibbs

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free enthalpy ( $\Delta G^\circ$ ) of the reactions of association between the PFF and the active surface ones is negative and believes in absolute value. In addition the calculation of the degrees of dissociation showed that the PFF is solubilized in these active surface ionic.

*Keywords: Insecticides; profenofos; surfactants; CTAC; SDS; micellar solubilization.*

## 1. INTRODUCTION

To regularize the production and to increase the agricultural outputs in order to satisfy the nutritional request related to the increase in population, protection against the bio-attackers of the cultures is impossible to circumvent. In this context, the use of the plant health products or pesticides constitutes a powerful lever. They are intended to protect the crop plants against parasitic mushrooms (fungicide), of the devastating insects (insecticide) or to destroy the adventitious ones (herbicide).

However, of many problems involved in the intensive use of these products were observed in the whole world. Those understand the appearance of parasites resistant to the pesticides [1], of the residues of pesticides in the products of bases [2], of the harmful effects on human health [3], of ecological imbalances and the deterioration of qualities of the ground and water [4]. In Senegal, most of the sprayed anti-parasitic compounds are widely used in a large agricultural area, named "Niayes area", where cultures are practiced during all seasons, and where most vegetables consumed in Dakar are harvested. Due to the intensive pesticide use in the Niayes area and their persistence, the environmental matrices (waters, soils and vegetables) are seriously affected by chemical contamination [5-8].

Profenofos (PFF) or the 4-bromo-2-chloro-1-[ethoxy (propylsulfonyl)phosphoryl]oxybenzene is an insecticide organophosphorus largely used in this zone to control the harmful insects with the cultures [9]. It is an insecticide which acts either by blocking the cholinesterase, or by preventing the transmission of the nerve impulse [10]. Because of its strong toxicity and its low solubility in water (28 mg/L), the insecticide presents a potential risk for the pollution of the environment. Consequently, to develop fast, selective, and significant methods for the detection of the product or these residues in the environment samples and to control their accumulation is necessary. Many methods of detection of the PFF were reported in the literature. These methods are mainly based on the

chromatography often associated spectroscopic methods such as for example: liquid chromatography (LC), gas chromatography coupled with the mass spectrometry (GC-MS) [11,12], gas chromatography with potentiometric detection with flame (GC-FPD) [13], and gas chromatography with detection nitrogen phosphate (GC-NPD) [14]. The electrochemical methods [15-18] were the subject of several studies. However, these significant and precise methods take time and require in addition to handling using of the expensive instruments. The conductometric methods have many advantages, in particular the low costs of equipment, the treatment of the samples, the speed, the sensitivity and the performance. Micellar solubilization is a very effective means to improve the degree of solubility of a not very water soluble organic substance. It is for this purpose we have undertaken a study of the reactions of association of insecticide PFF with the surfactants by the conductometric method.

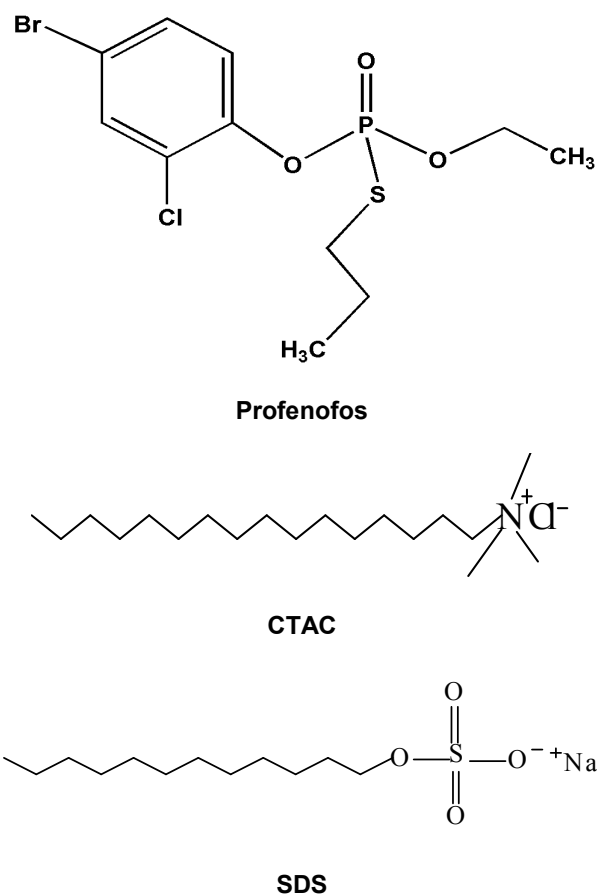
## 2. MATERIALS AND METHODS

### 2.1 Apparatus

A conductometer of the type VWR CO 3100L accompanied by a measuring cell of constant ( $0.84 \text{ cm}^{-1}$ ) and a temperature gauge were used. The apparatus was calibrated with a solution of KCl 0.01M of conductivities 1278 and  $1413 \mu\text{s cm}^{-1}$  to 20 and 25°C. A magnetic stirrer of type VELP SCIENTIFICA (Heating Magnetic Stirrer) is also used. An electronic balance (0.1 mg near) of mark STARTORIUS U3600S made it possible to make the weighing necessary.

### 2.2 Reagents

Profenofos (96.9% m/m) was purchased, cetyltrimethylammonium chloride (CTAC, 25% wt. solution in water,  $d=0.968$ ) and sodium dodecylsulphate (SDS, 98% m/m) were also received from Sigma Aldrich. Distilled water used comes to us from the national laboratory of control of the drugs (Dakar). The chemical structures of profenofos and surfactants were presented in Fig. 1.



**Fig. 1. Chemical structures of profenofos, CTAC and SDS**

### 2.3 Solutions Preparation

Stock solutions of profenofos ( $10^{-3}$  mol/L) and of SDS (0.5 mol/L) were prepared in distilled water. The aqueous solution of CTAC (0.76 mol/L) was used directly. All solutions were protected against light with aluminum foil to avoid any decomposition and stored in a refrigerator.

### 2.4 Methods Measurement

The critical micellar concentration (CMC) the active surface ones were determined by measurements of conductivity. Measurements of the CMC of active surface can be carried out in the direction of the increase or the reduction in the concentration of the surfactant one. A graduated burette is filled of the active surface solution; the temperature of proportioning is fixed at a given value and a volume of 0.2 mL of titrating is versed each time in the beaker containing a known concentration of PFF and

100 mL of distilled water, volume necessary to soak all the part significant of the cell, and placed on a magnetic stirrer also allowing the heating of the solution. The solution is homogenized at the same time by agitation, and finally a reading of specific conductivity is carried out.

### 2.5 Determination of the Thermodynamic Parameters of Micelles

The possibility of formation of the micellar phase on the one hand and the degree of spontaneity of the reaction of micellar association on the other hand, can be given starting from the thermodynamic parameters of micelization. The thermodynamic parameters (Gibbs free energy, enthalpy and entropy of micelization) are given starting from the variation of the CMC with the temperature.

The standard Gibbs free enthalpy ( $\Delta G^\circ$ ) of micelization of surfactant studied, which

monovalent type ionic is having only one chains hydrophobic subject, can be obtained starting from the following relation:

$$\Delta G^0 = (2 - \alpha)RT \ln X_{CMC} \quad (I)$$

With  $\alpha$  the micellar degree of dissociation, T the absolute temperature, R the constant of perfect gases and  $X_{CMC}$  molar fraction to the CMC. The standard enthalpy ( $\Delta H^0$ ) of micelle formation can be obtained starting from the equation of Gibbs-Helmholtz:

$$\Delta H^0 = -T^2 \frac{\partial}{\partial T} \left( \frac{\Delta G^0}{T} \right) \quad (II)$$

By replacing (I) in (II), the enthalpy of micelization can be written as follows:

$$\Delta H^0 = -RT^2(2 - \alpha) \frac{d}{dT} (\ln X_{CMC}) \quad (III)$$

The term  $\frac{d}{dT} (\ln X_{CMC})$  corresponds to the slope of the line of equation  $\ln X_{CMC} = f(T)$ .

The standard entropy ( $\Delta S^0$ ) related to the process of micelization is obtained by the following equation:

$$\Delta S^0 = \frac{1}{T} (\Delta H^0 - \Delta G^0) \quad (IV)$$

## 2.6 Determination of the Micellar Ionization Level $\alpha$

The micellar ionization level  $\alpha$  is defined as being the fraction dissociated of the polar heads on the surface of micelles. This parameter is given starting from conductometric measurements by using the method of Raoul Zana who proposes to calculate  $\alpha$  by using the report/ratio of the slopes after and before the CMC [20].

## 3. RESULTS AND DISCUSSION

### 3.1 Effect of PFF Concentration on the Critical Micellar Concentration (CMC) and the Degree of Dissociation ( $\alpha$ )

The experiment consists in following the evolution of the specific conductivity of an initial solution of PFF of fixed concentration, according to surfactants concentration additions. It is a direct proportioning of a solution of PFF concentration known with solutions the active surface ones with various concentrations. The active surface ionic ones (CTAC and SDS) were used to study the micellar solubilization of the PFF in aqueous medium. Fig. 2 shows the variation of specific conductivity according to the concentration of active surface in the absence and in the presence of PFF at the temperature of 302 K.

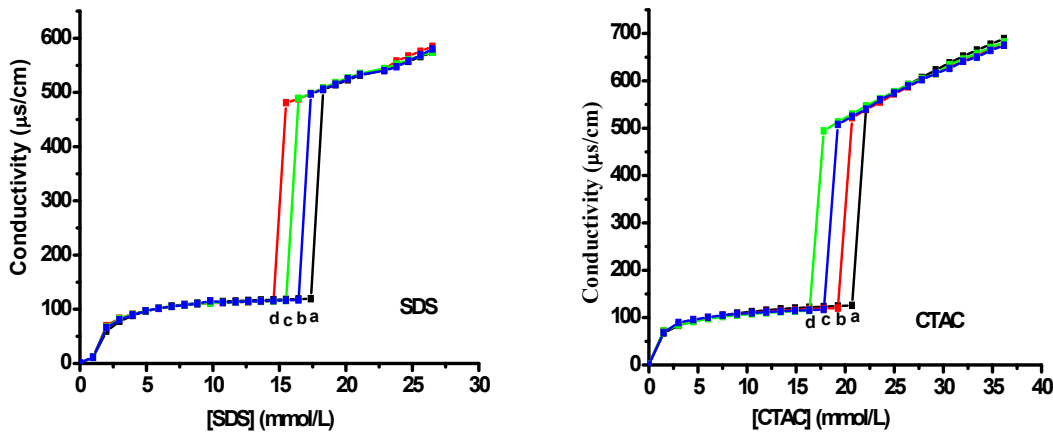


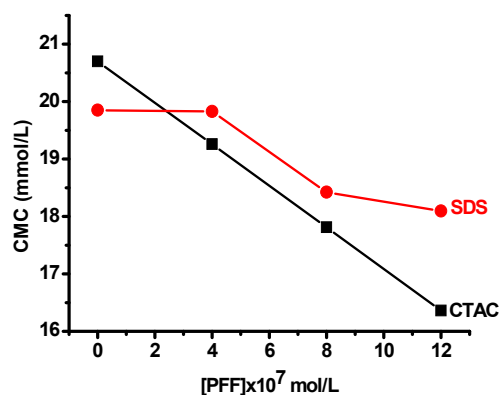
Fig. 2. Variation of specific conductivity according to the concentration of the CTAC and the SDS in absence and in the presence of PFF (a: 0; b:  $4 \times 10^{-7}$ ; c:  $8 \times 10^{-7}$  and d:  $12 \times 10^{-7}$  mol/L) to 302 K

The micellar solubilization of the PFF has the same behavior in the both active surface of surfactants. One observes an increase in the specific conductivity of the active surface ones which is explained by an increase in the number of free counter ions in the solution, ( $\text{Cl}^-$  and  $\text{Na}^+$  are the counter ions of the active surface of CTAC and SDS respectively). Before the CMC, the small quantity of active surface is not enough sufficient to form micelles. The surfactant one dissolves in water to give simple monomers. At the CMC, there is a sharp variation of conductivity. It is in the vicinity of the CMC that the monomers are assembled gradually in micellar aggregates of roughly spherical form. One then notices a brutal increase in the specific conductivity of the active surface ones, which increases proportionally, with the number of formed micelles. After the CMC, the micelle formation continues and the presence of monomer also increases making increase conductivity but in a less significant way. The values of the CMC,  $\alpha$  and  $\Delta G^\circ$  obtained with various concentrations of PFF are presented in Table 1.

The analysis of the results (Table 2) shows a reduction in the CMC and degree of dissociation according to the concentration of the PFF, which explains the solubilization of insecticide in the both micellar mediums used. We notice also an increase in the Gibbs free enthalpy of micelization ( $\Delta G^\circ$ ) in absolute value with the increase in the concentration of the PFF for the both active surface ones used. That is explained by the presence of the PFF in aqueous solution of the active surface ones which facilitates and supports the formation of the micellar phase and entrained the reduction in the CMC. The curves of CMC of the both active surface ones vs. PFF concentration are represented on Fig. 3.

We observe a progressive reduction in the CMC according to the increase in the concentration of the PFF for the both active surface ones used. However, this reduction is more significant in the

case of CTAC active surface. It is thus significant to study the chemical structure of the PFF for well explaining the reduction in the CMC according to its concentration. The PFF is a molecule having an aromatic nucleus (hydrophobic part) and comprising several polar heads (C-Br, C-Cl, P=O) what shows that the molecule of PFF has one permanent dipole moment very high [21]. The molecule can thus be regarded as an aqueous solution able to strongly interact with the polar heads of the both active surface ones. This phenomenon allows the strongly hydrophobic organic compounds a behavior favorable for micellar solubilization [22]. The PFF increases the action of the CTAC and the SDS while being adsorbed at interface or the heart of micelles and decreases at the same time the surface tension of micelles. For more information, it will be thus necessary to study the variation of the degree of electrolytic dissociation ( $\alpha$ ) according to the PFF concentration. Fig. 4 shows the evolution of  $\alpha$ , according to the PFF concentration.



**Fig. 3. Variation of the CMC of the CTAC and the SDS according to the PFF concentration to 302 K**

In both cases, we observe a reduction of  $\alpha$  according to the increase in the PFF concentration.

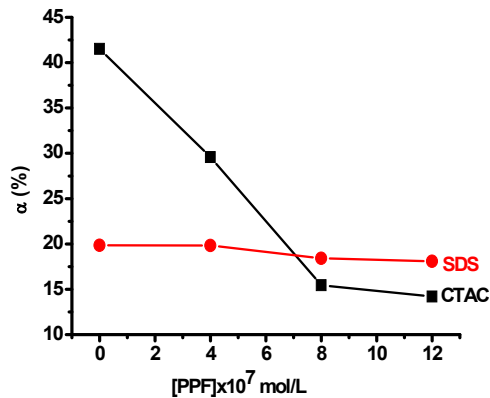
**Table 1. Values of the CMC,  $\alpha$ ,  $\Delta G^\circ$ , according to the PFF concentration**

[PFF] (mol/L)	CTAC-PFF			SDS-PFF		
	CMC <sup>a</sup> (mmol/L)	$\alpha$ (%) <sup>b</sup>	$\Delta G^\circ$ (kJmol <sup>-1</sup> ) <sup>c</sup>	CMC <sup>a</sup>	$\alpha$ (%) <sup>b</sup>	$\Delta G^\circ$ (kJmol <sup>-1</sup> ) <sup>c</sup>
0	20.70	41.52	-14.34	17.37	19.85	-15.40
4x10 <sup>-7</sup>	19.26	29.58	-15.71	15.50	19.82	-15.71
8x10 <sup>-7</sup>	17.81	15.45	-17.45	15.30	18.42	-15.89
12x10 <sup>-7</sup>	16.36	14.20	-17.91	14.56	18.09	-16.15

<sup>a</sup> Critical micellar concentration

<sup>b</sup> Degree of dissociation

<sup>c</sup> Gibbs free enthalpy



**Fig. 4. Variation of the degree of electrolytic dissociation ( $\alpha$ ) of the CTAC and the SDS according to the PPF concentration to 302 K**

The active surface CTAC, presents a remarkable reduction in  $\alpha$  that confirms clearly that the PPF is solubilized with heart of micelles by increasing the hydrophobic attraction forces which causes the decrease of  $\alpha$  to the CMC.

### 3.2 Effect of Temperature on the Critical Micellar Concentration (CMC) and the Degree of Dissociation ( $\alpha$ ) in the Presence of PPF

We used the same proportioning carried out previously by way conductometric while following the evolution of specific conductivity according to the concentration of active surface to various temperatures and in the presence of  $4 \times 10^{-7}$  mol/L of PPF. Fig. 5 present the evolution of conductivity specific according to the concentrations of CTAC and SDS.

One notes an evolution of specific conductivity here representing an increase in the ions in the solution. When the critical micellar concentration is reached, a jump appears and the variation of conductivity becomes less intense after the CMC.

The thermodynamic parameters  $\Delta G^\circ$ ,  $\Delta H^\circ$  and  $\Delta S^\circ$  were calculated respectively starting from equations I, III, and IV.

The representation of  $\ln X_{CMC} = f(T)$ , enables us to obtain the term

$\frac{d}{dT}(\ln X_{CMC})$  of the equation III which is nothing other than the line slope obtained (Fig. 6).

The Tables 2 and 3 present the values of the CMC,  $\Delta G^\circ$ ,  $\Delta H^\circ$  and  $\Delta S^\circ$  of the association reactions surfactant-PPF at various temperatures.

By analyzing the data of Tables 2 and 3, we note a strong influence of the temperature on the CMC. In both cases, one observes a reduction in the CMC when the temperature increases. This increase in the temperature leads a dehydration of the absorbent part (polar heads) of active surface supporting the micelization. It can also cause the rupture of the water molecules around the hydrophobic groups (linear chains of active surface), which results in a faster micelization [20]. Furthermore, the negative values  $\Delta G^\circ$  show that the process of micelization is spontaneous. The Gibbs free enthalpy decreases when the temperature increases, consequently the micelization of the PPF in the active surface ones used will be favored at higher temperature. However, these negative values of  $\Delta G^\circ$  prove that the reactions of association of the PPF with the active surface ones used are spontaneous in other words thermodynamically favorable. Values of  $\Delta H^\circ > 0$  were obtained representing an endothermic process. According to laws' of thermodynamics, for a process to take place, it is necessary that variation of Gibbs free enthalpy ( $\Delta G^\circ$ ) which depends on enthalpy factor ( $\Delta H^\circ$ ) and entropic ( $T\Delta S^\circ$ ) is to say negative. The enthalpy is an energy characteristic, while the entropy is a characteristic of induced disorder during a process. According to equation IV, if the variation of enthalpy of a process is positive, its entropy must also increase.  $|T\Delta S^\circ|$  being higher than  $|\Delta H^\circ|$  shows that the reaction of association between the PPF and the active surface ones (CTAC and SDS) is entropic i.e. the increase in enthalpy during the micelization is compensated by the increase in the disorder of the system. This increase in entropy is due to the aggregation of the hydrophobic chains and the release of counter ions. What also shows that the micelization between the active surface ones and the PPF is controlled by the aggregation of the active surface ones, as it is also recognized that the micelization is directed by the entropic term [19,23].

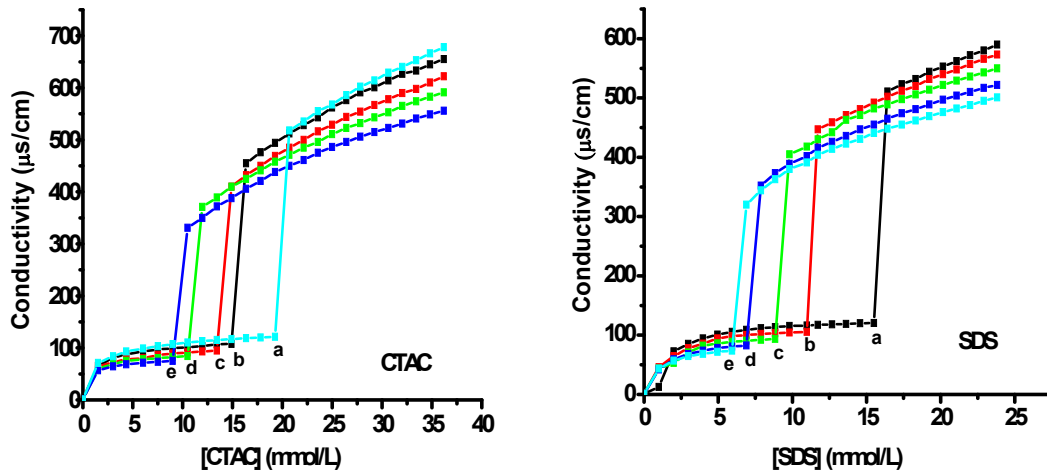


Fig. 5. Evolution of the specific conductivity of the CTAC and SDS in the presence of  $4 \times 10^{-7}$  mmol/L of PFF at various temperatures: 302 K (a), 309 K (b), 316 K (c), 323 K (d) and 330 K (e)

Table 2. Values of the CMC,  $\Delta G^0$ ,  $\Delta H^0$  and  $\Delta S^0$  according to the temperature

T (K)	CMC (mmol/L)	$\Delta G^0$ (kJmol <sup>-1</sup> )	$\Delta H^0$ (kJmol <sup>-1</sup> ) <sup>d</sup>	$\Delta S^0$ (kJmol <sup>-1</sup> ) <sup>e</sup>
302	19.26	-15.73	34.51	0.166
309	14.90	-18.47	38.5	0.185
316	13.44	-19.15	40.06	0.187
323	10.49	-20.76	41.81	0.194
330	9.01	-21.10	41.92	0.191

<sup>d</sup> Variation of enthalpy

<sup>e</sup> Variation of entropy

Table 3. Values of the CMC,  $\Delta G^0$ ,  $\Delta H^0$  and  $\Delta S^0$  according to the temperature

T (K)	CMC (mmol/L)	$\Delta G^0$ (kJmol <sup>-1</sup> )	$\Delta H^0$ (kJmol <sup>-1</sup> )	$\Delta S^0$ (kJmol <sup>-1</sup> )
302	15.50	-16.34	48.32	0.214
309	10.98	-17.94	49.48	0.218
316	8.84	-18.67	49.80	0.217
323	6.90	-18.93	48.70	0.210
330	5.93	-19.49	49.39	0.209

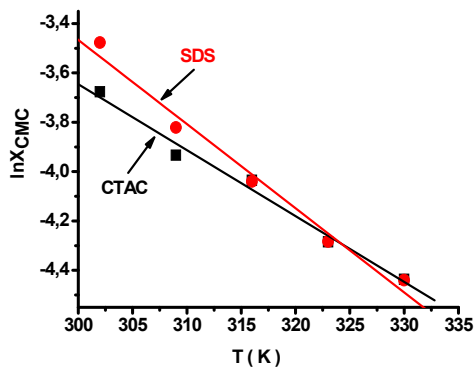


Fig. 6. Representation of  $\ln X_{CMC}$  as a function of temperature

#### 4. CONCLUSION

We studied the micellar properties (CMC and  $\alpha$ ) of active surface of CTAC and SDS to carry out the micellar solubilization of the PFF in aqueous medium. The application of active surface of CTAC and SDS in the micellar solubilization of insecticide PFF carried out by conductometric measurement at the temperature 302 K occurs spontaneously. The results obtained show a reduction in the CMC according to the temperature and of the PFF concentration of the PFF. That shows simply that the temperature and the PFF concentration foster the micelization of active surface used. Other share, the values of

the degree dissociation  $\alpha$  and standard free enthalpy ( $\Delta G^0$ ) of micellar formation, showed that the PFF, is solubilized in the micellar phase in consequence of a strong interaction. This solubilization of insecticide occurs in the heart of micelles. The study of the micellar properties of active surface is an effective means for the solubilization of organic pollutants slightly soluble in water.

For the next of the work, we envisage an application of profenofos in the environmental matrices (waters, fruits, vegetables) by the conductometric method used. The analytical method employed has proved to be simple enough to be applied to routine analysis because of the need for low cost equipment. The low concentrations of profenofos used show that the sensitivity of the method is quite satisfactory to determine the insecticide profenofos in the environmental matrices.

### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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