

Research Article

Enhanced Solubility of Herbicide Diuron in Aqueous Solution by Micellisation

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Abstract

Surfactants are substances widely used in agricultural sprays to improve the solubility and mobility of pesticides across crops. This study investigates the micellar properties of two ionic surfactants sodium dodecyl sulfate (SDS) and tetramethylammonium tetrafluoroborate (TMAFb) with respect to their ability to solubilize the herbicide diuron in aqueous solution. Conductometric measurements were performed in aqueous media over a temperature range of 298 to 331 K to analyze the micellization behavior and evaluate the efficiency of solubilization. From the conductivity data the critical micelle concentration (CMC), and degree of ionization were obtained at various temperatures. Concentration and temperature effect on the CMC have been studied and the different thermodynamic parameters were evaluated. The critical micellar concentration (CMC) values of surfactants decreased with increasing temperature, indicating enhanced micelle formation under thermal influence. Additionally, the solubility of diuron varied significantly across different surfactant micelles concentration, suggesting that specific interactions occur between the surfactant head groups and the pesticide. The standard Gibbs free energy (ΔG°) for the diuron–surfactant mixtures was attained to be negative throughout the study suggesting spontaneous micellization process. The enthalpy (ΔH°) and entropy (ΔS°) were also evaluated, offering additional insight into the thermodynamic driving forces involved. The obtained thermodynamic parameters showed that $|T\Delta S^\circ|$ is greater than $|\Delta H^\circ|$, suggesting that the micellization process is controlled by entropy.

Keywords

Diuron, Herbicide, Surfactants, Solubilisation, Water

1. Introduction

Pesticides are substances applied to crops to control pests. Their use is of considerable importance in agriculture, as they contribute to increased crop yields. Furthermore, pesticides have helped reduce production disruptions caused by severe pest infestations and have played a key role in combating hunger by protecting both crops in the field and stored food

reserves [1, 2]. However, the large-scale use of pesticides can lead to indirect and harmful effects on the environment. Numerous studies have reported the presence of pesticide residues in food, as well as in surface and groundwater sources [3–5]. During application, a significant portion of the pesticides does not reach the target crops and may persist in the

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environment for many years [6, 7]. Depending on the method of application and prevailing weather conditions, it is estimated that between 25% and 75% of pesticides fail to deposit on the intended treatment areas [8]. Pesticides can enter the atmosphere directly during application, contaminating both the air and precipitation. Numerous studies have shown that this form of pollution is not limited to agricultural areas, as atmospheric conditions such as wind, temperature, and pressure differences can facilitate the transport of pesticide particles to urban environments [9-11]. This atmospheric diffusion can occur over long distances and affect regions far from the original site of application.

This study focuses on the herbicide diuron. Herbicides are a class of pesticides used to eliminate weeds that hinder the healthy growth of crops, and their use is widespread in modern agriculture. Among the various types of herbicides, some offer total weed control, removing all vegetation exposed to the chemical, while others provide selective weed control, targeting specific weed species without significantly harming the cultivated crops [12, 13]. Herbicide degradation occurs when the compound is broken down into smaller molecules ultimately into carbon dioxide and water through photochemical, chemical, or biological processes [14, 15]. Degradation can also occur through adsorption by plants or soil, as well as through leaching into deeper soil layers via drainage [16, 17]. When an herbicide is degraded in the environment, it produces several by-products known as metabolites. Each metabolite has its own chemical properties, including specific levels of toxicity, adsorption capacity, and resistance to further degradation. In many cases, the exact nature and behavior of these metabolites remain largely unknown [7, 17]. Some may contain substances capable of harming terrestrial ecosystems [18].

In Senegal, most sprayed anti-parasitic compounds are widely used in a major agricultural region known as the Niayes Area, where farming is practiced year-round [19]. Diuron, like many other herbicides, is extensively applied in this region to increase crop yields by eliminating weeds. Many of these compounds exhibit significant photochemical instability, especially under the Niayes Area's sunny, warm, and dry summer climate, which accelerates their degradation in the environment [19]. To address concerns related to pollution and toxicity, numerous analytical methods for detecting

diuron have been reported in the literature. These methods primarily rely on chromatography, combined with spectroscopic or electrochemical techniques [20-23]. The direct fluorescence (DF), photochemically induced fluorescence (PIF) and UV-visible methods [19, 24, 25].

However, these accurate and reliable methods are often time-consuming and require the use of expensive instruments and skilled handling. In contrast, conductometric methods offer several advantages, including lower equipment costs, simpler sample preparation, faster analysis, high sensitivity, and good overall performance. Micellar solubilisation is an effective approach to enhance the solubility of organic substances that are poorly soluble in water. For this reason, we have undertaken a study of the interaction between the herbicide diuron and surfactants in aqueous solution using conductometric methods.

2. Materials and Methods

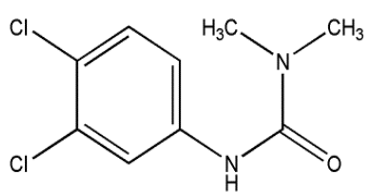
2.1. Apparatus

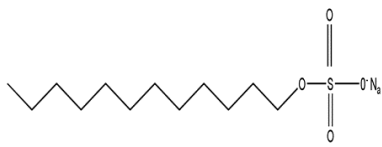
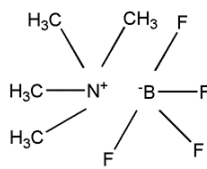
A VWR CO 3100L Conductometer, equipped with a measuring cell of constant 0.84 cm^{-1} and a temperature probe, was used for the conductometric measurements. The apparatus was calibrated using a 0.01 M KCl solution with conductivities of 1278 and 1413 $\mu\text{S}/\text{cm}$ at 20°C and 25°C , respectively. A VELP SCIENTIFICA heating magnetic stirrer was employed for sample mixing. Weighing was performed using a Sartorius U3600S electronic balance with a precision of 0.1 mg.

2.2. Reagents

Diuron (98% w/w) was purchased from Sigma-Aldrich Chemical Company (USA) and was used without purification. Sodium dodecyl sulfate (SDS 98%, m/m) and tetramethyl ammonium tetrafluoroborate (TMATFB $[(\text{CH}_3)_4\text{N}^+, \text{BF}_4^-]$ 97% m/m) were purchased from Sigma Aldrich. Distilled water is used to prepare surfactant stock solutions. The chemical properties of diuron and surfactants are presented in Table 1.

Table 1. Chemical properties of diuron and surfactants [24, 26-28].

Product	Formula	Molar mass (g mol^{-1})	WS (25 °C) (mg L^{-1})	MP (°C)	BP (°C)	Structure
Diuron	$\text{C}_9\text{H}_{10}\text{Cl}_2\text{N}_2\text{O}$	233.095	45	158	180	

Product	Formula	Molar mass (g mol ⁻¹)	WS (25 °C) (mg L ⁻¹)	MP (°C)	BP (°C)	Structure
SDS	CH ₃ (CH ₂) ₁₁ SO ₄ Na	288.38	1000	206	NF	
TMATFB	(CH ₃) ₄ NBF ₄	160.95	NF	NF	NF	

WS = Water solubility; MP = Melting point; BP = Boiling point.

2.3. Solutions Preparation

Stock solutions of diuron (10^{-3} mol·L⁻¹) were freshly prepared in methanol and surfactants (SDS and TMATFB) in distilled water at 0.5 mol·L⁻¹. These solutions were subsequently diluted to obtain the desired working concentrations. All solutions were stored in glass vials wrapped with aluminum foil and kept in a refrigerator, protected from light and heat to prevent decomposition.

2.4. Conductivity Measurements

Conductivities were performed with a digital conductivity meter (A VWR CO 3100 LK) well-appointed with a measuring cell and a temperature sensor. The dosing temperature is set to a specific value, and volumes of 0.2 mL of the titrant solution are taken and then poured each time into a beaker containing 150 mL of a pesticide solution of known concentration using a graduated burette and filled with the surfactant solution. The beaker is placed on a heating magnetic stirrer and the mixture was homogenized magnetically during 10 seconds before we noted the specific conductivity.

2.5. Determination Method of Thermodynamic Parameters of Micelles

The surfactants studied being ionic, of monovalent types (a single hydrophobic chain), the standard free enthalpy of micellisation can be determined by the relationship below: [29, 30].

$$\Delta G^\circ = (2 - \alpha)RT \ln X_{\text{CMC}} \quad (1)$$

Where α is the degree of dissociation, R is gas constant and T is the temperature in Kelvin scale. X_{CMC} is the mole fraction at CMC of the ionic liquids.

$$\Delta H^\circ = -T^2 \frac{d}{dT} \left(\frac{\Delta G^\circ}{T} \right) \quad (2)$$

By replacing equation (1) in equation (2), and if we consider a short temperature interval, we obtain:

$$\Delta H^\circ = -RT^2 (2 - \alpha) \frac{d}{dT} (\ln X_{\text{CMC}}) \quad (3)$$

The values of $\ln X_{\text{CMC}}$ vary linear way as a function of temperature which can be presented by following Eq. (3). The term $\frac{d}{dT} (\ln X_{\text{CMC}})$ corresponds to the slope of the line $\ln X_{\text{CMC}} = f(T)$.

The entropy associated with the micellisation process can be calculated using the following equation:

$$\Delta S^\circ = \frac{1}{T} (\Delta H^\circ - \Delta G^\circ) \quad (4)$$

3. Results and Discussion

3.1. Effect of Diuron on the CMC and α

We followed the evolution of the specific conductivity of a solution containing a fixed and known concentration of diuron as a function of the concentration of ionic surfactants (SDS and TMATFB). Figure 1 present plots of the specific conductivity according to the TMATFB and SDS concentration in water in the absence and the presence of diuron at 298 K. We observe a variation in the specific conductivity as a function of the concentrations of the surfactants. The CMC values were determined by measuring the change in specific conductivity values with the addition of surfactants. This also vary depending on the diuron concentration [31]. Two specific conductivity-dependent regimes were observed below and above the CMC. The corresponding value to the breaking point was taken as the CMC for the system [32]. The increase in specific conductivity can be explained by the increase in the number of

free counterions (BF_4^- or Na^+) in the solution [30]. Degree of ionization of micelles, α can be obtained from the ratio of the slopes above and below the CMC. The values of CMC and α obtained from the specific conductivity plots are listed in Table 2 at different concentration of diuron. Micellar solu-

bilization depends on the type of surfactant, the solute (diuron), and the interactions between the solvent and solute [33, 34]. To better understand the specific influence of surfactants on the conductivity of diuron, we studied the conductivity as a function of diuron concentration in the absence of surfactants.

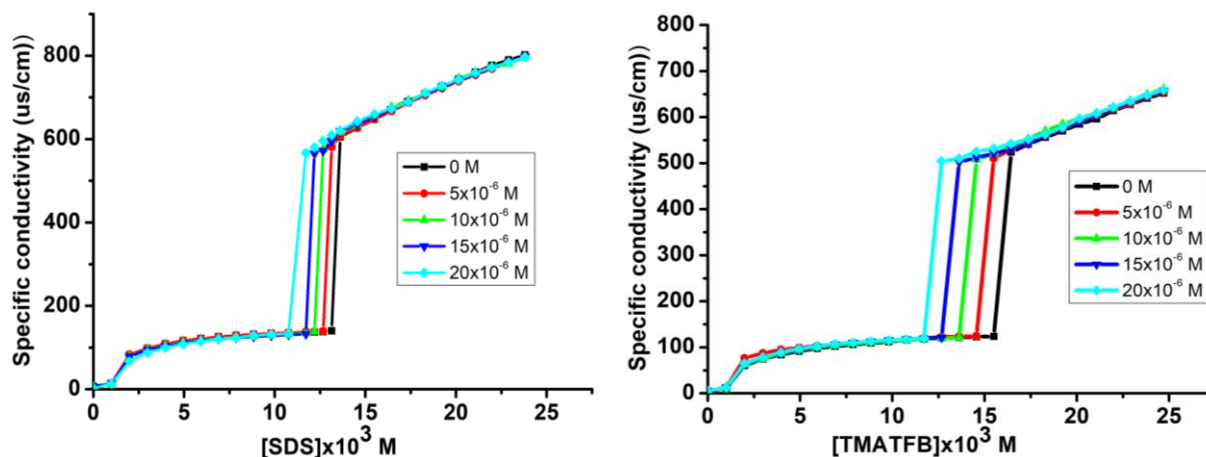


Figure 1. Variation of specific conductivity with TMATFB and SDS concentrations in the absence and presence of diuron at 298 K.

As observed, the specific conductivity of diuron changes only slightly in the absence of surfactants, indicating that surfactants significantly influence conductivity. The pronounced variations in specific conductivity shown in Figure 2 are therefore attributed to the presence of the ionic surfactants TMATFB and SDS. Pesticide solubility in the micelles are cooperatively affected by pesticide hydrophobicity, surfactant head type and micellar structure [35]. The estimated CMC, α , and $\Delta G^0 \alpha$ values of the diuron+surfactants system in aqueous medium are presented in Table 2.

For both surfactants, a decrease in the critical micelle concentration (CMC) was observed with increasing diuron concentration (Figure 2). This decrease was more pronounced for the cationic surfactant (TMATFB). To better understand this trend, it is useful to examine the chemical structure of diuron, which may help explain the reduction in CMC as diuron concentration increases. Due to its molecular structure, Diuron possesses a high dipole moment, enabling strong interactions with the polar head groups of surfactants such as SDS and TMATFB in solution [36, 37]. This interaction facilitates the formation of aggregates with highly hydrophobic organic compounds, promoting micellar solubilisation [38].

Diuron enhances the activity of both TMATFB and SDS by adsorbing at the interface, simultaneously reducing the surface tension of the micelles [39].

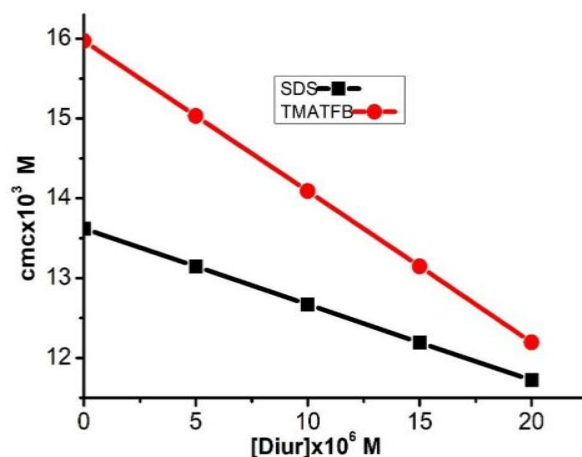


Figure 2. Variation of CMC for TMATFB (A) and SDS (B) as a function of diuron concentration at 298 K.

Table 2. CMC, α and ΔG^0 values as a function of diuron concentration at 298 K.

[Diur] (mM)	SDS			TMATFB		
	CMC ^a (mM)	α^b (%)	ΔG^0 ^c (kJ mol ⁻¹)	CMC ^a (mM)	α^b (%)	ΔG^0 ^c (kJ mol ⁻¹)
0	13.62	0.340	-21.86	15.97	0.359	-20.93

[Diur] (mM)	SDS			TMATFB		
	CMC ^a (mM)	α^b (%)	ΔG° ^c (kJ mol ⁻¹)	CMC ^a (mM)	α^b (%)	ΔG° ^c (kJ mol ⁻¹)
5	13.14	0.290	-22.65	15.03	0.368	-21.44
10	12.67	0.249	-22.65	15.03	0.368	-21.44
15	12.19	0.247	-23.52	13.14	0.471	-20.47
20	11.72	0.232	-23.91	12.19	0.503	-20.94

^a Critical micellar concentration; ^b Degree of dissociation; ^c Gibbs free enthalpy; mM = 10⁻³ mol L⁻¹

However, opposite trends in the variation of the degree of micellar ionization (α) with increasing diuron concentration are observed for SDS and TMATFB (Figure 3). The mechanism of micellar solubilisation of Diuron differs between SDS and TMATFB. In the presence of TMATFB, the increase in α values reflects micellar interactions involving the polar head groups of the surfactants and diuron [40, 41]. Conversely, in SDS, α decreases with increasing diuron concentration, indicating a strengthening of hydrophobic interactions. These results demonstrate that diuron solubilizes within the micelle core [42, 43]. In conclusion, diuron exhibits greater solubilisation in SDS compared to TMATFB. The standard Gibbs free energy change (ΔG°) generally increases with Diuron concentration. In aqueous solution, the presence of diuron significantly promotes micelle formation, thereby reducing the critical micelle concentration (CMC). Diuron effectively acts as a co-surfactant during the micellisation process of both SDS and TMATFB.

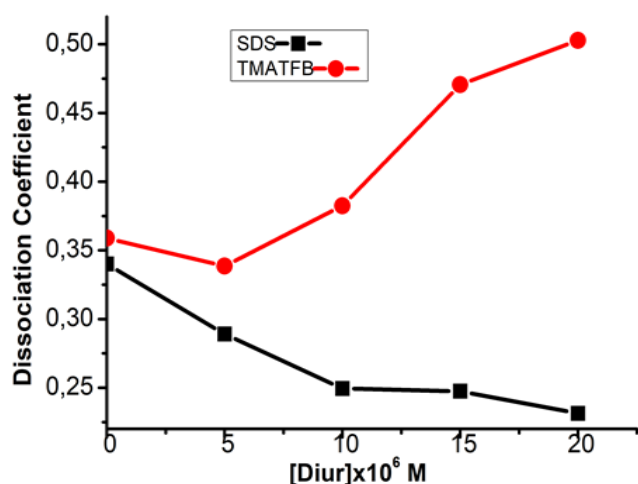


Figure 3. Variation of α for TMATFB (A) and SDS (B) as a function of diuron concentration at 298 K.

3.2. Determination of the Krafft Temperature

The Krafft temperature is the minimum temperature at which micellar aggregates are formed. Using surfactant con-

centrations above the CMC (15.50 mM for SDS and 19.23 mM for TMATFB), we investigated the variation of diuron specific conductivity as a function of temperature (Figure 4). The resulting curves exhibited three distinct regions, from which the Krafft temperature was determined based on changes in slope. The Krafft temperatures were found to be 11 °C for SDS and 22 °C for TMATFB, with an uncertainty of ± 1 °C.

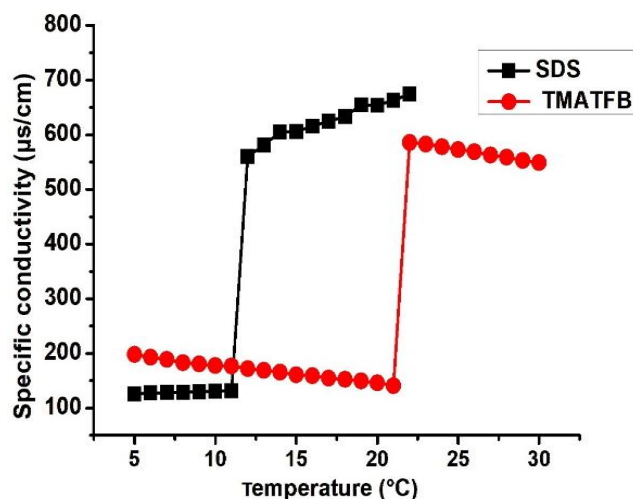


Figure 4. Variation of the specific conductivity of surfactants SDS and TMATFB as a function of temperature.

3.3. Temperature Effect on the CMC and α

Figure 5 shows the variation of specific conductivity as a function of surfactant concentration at 298 K, 303 K, 310 K, 317 K, 324 K, and 331 K, in the presence of 15.10⁻⁶ M diuron. For both surfactants studied, temperature has a significant effect on the solubilisation of diuron in water [44, 45]. The CMC decreases with increasing temperature, enhancing adsorption at the interface and simultaneously reducing the surface tension of the micelles [46-47]. The increase in temperature can also disrupt the structured water molecules surrounding the hydrophobic groups (linear chains of the surfactants), this could lead to increased dehydration of the hy-

drophilic heads, favoring to faster micellisation [48]. In the presence of SDS, the micellar solubilisation of diuron results in co-micellisation, forming mixed micelles containing both SDS and diuron. The decrease in the dissociation coefficient with rising temperature likely reflects stronger hydrophobic interactions. The increase in α with TMATFB indicates that diuron resides between the surfactant's polar head groups

during solubilisation. To verify the actual influence of surfactants on the specific conductivity of diuron at different temperatures, we studied the conductivity in the presence of diuron ($15 \cdot 10^{-6}$ M) without surfactants (Figure 6). No variation in specific conductivity was observed. This shows that temperature has no effect on the variation in the conductivity of the diuron molecule.

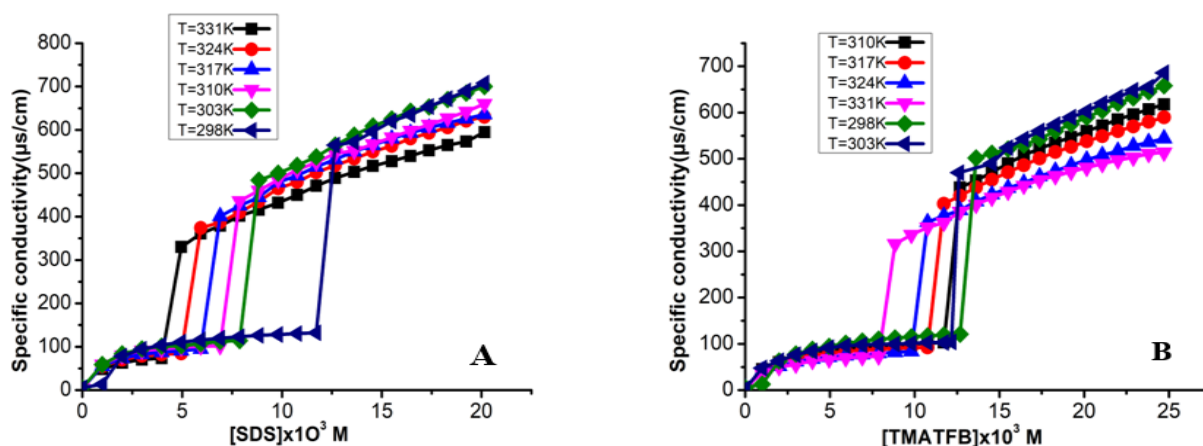


Figure 5. Specific conductivity of SDS (A) and TMATFB (B) in the presence of 15×10^{-6} M diuron as a function of surfactant concentration at temperatures of 298, 303, 310, 317, 324, and 331 K.

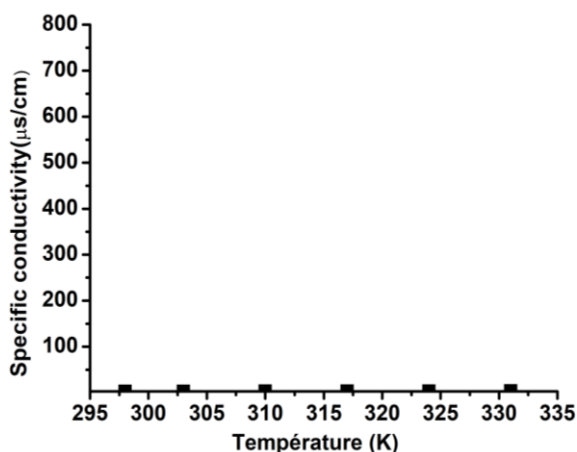


Figure 6. Variation of specific conductivity of diuron (15×10^{-6} M) without surfactants at different temperatures: 298 K, 303 K, 310 K, 317 K, 324 K, and 331 K.

3.4. Thermodynamic Parameters

The thermodynamic parameters ΔG° , ΔH° , and ΔS° were calculated with the help equations (1), (2) et (3). The slope $d(\ln X_{CMC})/dT$ was derived from the plot of $\ln X_{CMC}$ versus temperature (Figure 7). As shown, $\ln X_{CMC}$ decreases with increasing temperature. Furthermore, the negative values of ΔG° confirm that the micellisation process is spontaneous and thermodynamically favorable (Table 3). The Gibbs free en-

ergy decreases as the temperature increases; consequently, the micellisation of diuron will be favored at higher temperatures. The values of $\Delta H^\circ > 0$ indicate an endothermic process. $|T\Delta S^\circ| > |\Delta H^\circ|$ shows that the association of diuron with TMATFB and SDS is entropy-driven, with the enthalpy increase during micellisation compensated by the system's increased disorder. This increase in entropy arises from the aggregation of hydrophobic chains and the release of counter-ions. It can also be stated that the micellization of surfactants with diuron is governed by the aggregation of the latter. Moreover, it is well established that micellisation is driven by the entropic contribution [49].

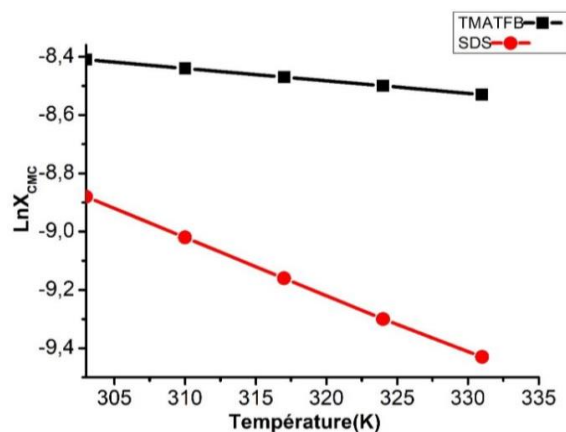


Figure 7. Representation of $\ln X_{CMC}$ of surfactants (SDS, TMATFB) as a function of temperature.

Table 3. Micellisation and thermodynamic parameters values of diuron at different temperatures.

Surfactants	T (K)	cmc $\times 10^3$ M	X _{CMC}	α (%)	ΔG° (KJ mol ⁻¹)	ΔH° (KJ mol ⁻¹)	$T\Delta S^\circ$ (KJ mol ⁻¹)
SDS	303	8.36	0.91 10^{-4}	0.759	-27.88	17.98	41.22
	310	7.34	0.84 10^{-4}	0.725	-29.52	19.32	42.86
	317	6.42	0.80 10^{-4}	0.533	-35.31	23.25	50.50
	324	5.44	0.74 10^{-4}	0.358	-41.20	27.18	57.60
	331	4.46	0.71 10^{-4}	0.323	-43.50	28.98	59.78
TMATFB	303	12.69	2.29 10^{-4}	0.260	-23.149	5.25	38.49
	310	12.19	2.41 10^{-4}	0.318	-23.512	5.53	39.31
	317	11.24	2.09 10^{-4}	0.303	-24.627	5.80	39.58
	324	10.30	2.07 10^{-4}	0.336	-25.082	6.27	41.22
	331	8.36	2.02 10^{-4}	0.395	-25.623	6.56	41.49

France).

4. Conclusion

In this study, the interaction of diuron with surfactants such as SDS and TMATFB was investigated using conductometric measurements at different temperatures. The presence of diuron in aqueous surfactant solutions led to a decrease in the critical micellar concentration (CMC), with this effect becoming more pronounced at higher pesticide concentrations and temperatures. Furthermore, the Gibbs free energy (ΔG°) values of the diuron-surfactant system in aqueous solution were all negative, indicating that micellar aggregation is spontaneous. The positive enthalpy (ΔH°) values suggest that micellisation is an endothermic process. Additionally, the positive entropy (ΔS°) values observed at higher temperatures imply that micellisation of the diuron-surfactant mixture is predominantly driven by entropy. The results clearly demonstrate that micellar solubilization is an effective means for enhancing the solubility of poorly water-soluble pesticides. This work is a key element of our strategy to reduce the presence and persistence of these pesticides in natural waters.

Abbreviations

CMC Critical Micellar Concentration

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Author Contributions

Souleymane Sambou: Writing – original draft, Investigation, Conceptualization

Ibrahima Sarr: Data curation, Resources

Nehou Diouf: Formal Analysis

Coumba Faye: Visualization, Data curation

Abdou Khadre Cisse: Methodology, Formal Analysis

Boubacar Sidibe: Visualization

Aly Cisse: Data curation

Keba Diongue: Data curation, Methodology

El Hadji Tombe Bodian: Writing – original draft, Methodology, Supervision, Validation

Diene Diegane Thiare: Writing – original draft, Validation, Supervision, Conceptualization.

Atanasse Coly: Validation, Supervision

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Data Availability Statement

No data was used.

Conflicts of Interest

The authors declare no conflicts of interest.

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