Original Research Article

Enhanced solubilisation of insecticide phosalone by using anionic and cationic surfactants in aqueous solution by micellisation

ABSTRACT

The objective of this study is to enhance the solubility of the insecticide phosalone in aqueous solution through micellisation. The cationic cetyltrimethylammonium chloride (CTAC) and the anionic sodium dodecyl sulfate (SDS) surfactants were used. The study employs a conductometric methodology to examine the interactions between the insecticide and the surfactant. The critical micellar concentration (CMC) and degree of counterions dissociation (α) of the surfactants were obtained at varying concentrations (0 to 3 µM) of the insecticide phosalone and temperatures between 301.15 and 321.15 K. In all cases, both parameters (CMC and α) demonstrated a decline in line with the increase in insecticide concentration and temperature. The thermodynamic parameters were calculated using the CMC versus temperature data. A positive value of ΔH° indicates that the micellisation process is endothermic. The negative values of ΔG° as a function of insecticide concentration and temperature demonstrate that the micellisation process is thermodynamically favorable and that the insecticide solubilises in the micelle core. The results of this study demonstrate that it is possible to solubilise a poorly soluble organic molecule by micellisation using conductivity alone, which is a valuable finding.

Keywords: CTAC, SDS, CMC, solubility, surfactants

1. INTRODUCTION

To meet the food needs of a growing global population, there is a significant opportunity for world agricultural production to increase, particularly in developing countries. It is therefore essential to enhance agricultural yields and regulate production by protecting crops from pests. In light of this challenge, the deployment of phytosanitary products, or pesticides, represents an effective strategy for achieving this goal.

These products are designed to safeguard plants from parasitic fungi (fungicides), insect pests (insecticides), or even to eradicate weeds (herbicides) [1]. However, the majority of pesticides found in surface water and groundwater are not particularly soluble, which presents a challenge in their analysis. Moreover, the solubility of pesticides in water often presents obstacles to the implementation of spectroscopic techniques, such as UV absorption and fluorescence [2-4]. This requires the use of organic solvents or other supplementary reagents [5].

With this in mind, we undertook a study of the use of phosalone in the Niayes agricultural zone of Senegal. Phosalone (S-[(6-chloro-2-oxo-1,3-benzoxazol-3(2H)-yl)methyl] O,O-diethylphosphorodithioate) is a organophosphate insecticide used to control a wide range of pests on certain crops, including grapes, apples, peas, potatoes, rice, and cotton [6, 7].

Phosalone inhibits acetylcholine activity, but its low water solubility can result in prolonged persistence in environmental matrices, particularly surface and groundwater [8]. The insecticide has poor solubility in water (3.5 mg L^{-1}) at 298.15 K. The low solubility of the insecticide may result in limited microbiological and photochemical degradation in environmental matrices, which could lead to the persistence of the insecticide in surface and groundwater [9].

Given the low solubility of phosalone insecticide in water, we have initiated a study of micellar solubilisation with the objective of facilitating the solubilisation of phosalone in an aqueous system. This will be achieved by forming mixed micelles with surfactants or by incorporating the insecticide into the interior of micelles.

The formation of micelles is dependent on the concentration of surfactants, which is influenced by the interplay of hydrophilic and hydrophobic interactions [10, 11]. Amphiphilic molecules, such as surfactants, are effective at solubilising feebly soluble organic entities due to their inherent self-assembly properties [10, 11].

The micellisation process in surfactants is a thermodynamic phenomenon that can be studied using a variety of techniques, including isothermal calorimetry, surface tension, UV-visible, fluorimetry and conductometry [12, 13]. In this study, we employ the more straightforward and cost-effective conductometry technique to enhance the solubility of the insecticide phosalone in anionic and cationic aqueous micellar media [14; 15].

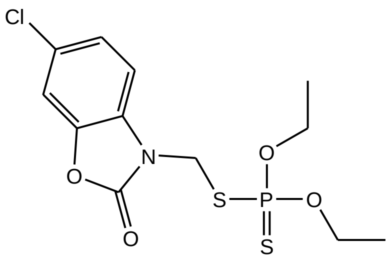

Fig. 1. Chemical structure of phosalone

2. MATERIAL AND METHODS

2.1. Apparatus

This analytical study was conducted using a VWR® pHenomenal® CO 3100L Conductivity/TDS/°C Meter, Bench, which was accompanied by a measuring cell and a temperature sensor. Conductivity Accuracy ±0.5% of measured value, Conductivity Resolution Up to 0.001 µS/cm (depending on probe). The device was calibrated with a 0.01 M KCl solution exhibiting conductivities of 1278 µS cm^{-1} and 1413 µS cm^{-1} at temperatures of 293.15 and 298.15 K, respectively, using the appropriate standard curve. A VELP SCIENTIFICA magnetic stirrer (Healing Magnetic Stirrer) was utilized to facilitate the homogenization of the stock solutions. All weighing operations were conducted using a Sartorius U3600S electronic balance, with a precision of 0.1 mg. A variety of laboratory vessels, including beakers, test tubes, flasks, and micropipettes, were employed for the implementation of the diverse measurements.

2.2. Chemicals

The phosalone (99% pure) is sourced from Sigma-Aldrich, a company based in Germany. The surfactant molecules utilised are cetyltrimethylammonium chloride (CTAC, 98% m/m, in aqueous solution d= 0.968) with the empirical formula $CH_3(CH_2)_{15}N(CH_3)_3Cl$, and sodium dodecyl sulfate (SDS) with the empirical formula $CH_3(CH_2)_{11}SO_4Na$ (98%, d = 1.01) are also procured from Sigma Aldrich. Figure 1 depicts the chemical structure of phosalone, while Table 1 enumerates its physicochemical properties.

3. Solutions preparation

The phosalone solution was prepared in distilled water at a pH = 7, with a concentration of

10^{-3} M. The stock solutions of the SDS and CTAC were prepared at a concentration of 0.1 M in distilled water. All solutions were stored in glass vials. The latter were then placed in a refrigerator, protected from light. The physicochemical properties of phosalone are presented in table 1 [16-18].

Table 1. Physicochemical properties of phosalone, SDS and the CTAC

Physicochemical properties	Phosalone	SDS	CTAC
Chemical formula	$C_{12}H_{15}ClNO_4PS_2$	$C_{12}H_{25}NaO_4S$	$C_{19}H_{42}NCl$
Molecular weight (g mol^{-1})	367.8	288.38	319.5
Solubility (mg L^{-1}) 298.15 K	3.5	10^5	440
Melting point (K)	318.15-321.15	477.15-480.15	505.15-507.15
Relative density 298.15 K	1.4	1.01	0.968
Vapor pressure (Pa) 298.15 K	4.54 10^{-8}	--------	--------
CMC (mM) 298.15 K	--------	8.2	1.3
DL50 (mg L^{-1})	120 (oral, rat)	1288 (oral, rat)	400 (oral, mouse)

2.4. Conductivity measurement procedure

The conductivity measurement procedure was employed to gain insight into the micellar behavior of the surfactant-phosalone mixture in aqueous media. The values of conductivity were recorded with the aid of a digital conductivity meter, which was equipped with a measuring cell and a temperature sensor. The temperature at which the solution is prepared is fixed at a specific value. Subsequently, 0.2 mL of the titrant solution is taken and poured into a beaker containing 150 mL of a solution with a known concentration of the insecticide. This volume is sufficient to immerse the electrode. Subsequently, the beaker is placed on a magnetic stirrer, which may facilitate the heating of the solution. The solution is simultaneously homogenized through stirring, and a reading of the specific conductivity is obtained. The recorded conductivity values of the mixture were plotted against the corresponding surfactant concentrations in order to determine the critical micelle concentration (CMC).

2.5. Determination Method of Thermodynamic Parameters of Micelles

In order to ascertain the degree of spontaneity of the micellar self-aggregation reaction of insecticide with surfactants, we proceeded to determine the thermodynamic parameters of micellisation and the degree of ionisation α of the counterions.

The degree of micellar ionisation α of the counterions in the solution can be determined from the ratio of the slopes observed before and after the critical micelle concentration (CMC) (Raoul Zana). The micellar free energy (ΔG^0) was calculated by solving the following equation: (1) [19, 20].

$$\Delta G^\circ = (2 - \alpha)RT lnXcmc \qquad (1)$$

T = experimental temperature (K), R = universal molar gas constant, X_{CMC} molar fraction of surfactant at the CMC, ΔH°= standard enthalpy.

$$\Delta H^\circ = -T^2 \frac{\partial}{\partial T}\left(\frac{\Delta G^\circ}{T}\right) \qquad (2)$$

By replacing (1) in (2), the enthalpy of micellisation can be written as follows [21]:

$$\Delta H^\circ = -RT^2(2 - \alpha)\frac{\partial}{\partial T}(lnXCMC) \qquad (3)$$

The values of $lnXcmc$ are observed to vary in a linear fashion with temperature, resulting in the formulation of equation (3). $\frac{d}{dT}(lnXCMC)$ corresponds to the slope of the line $lnXCMC = f(T)$. The entropy associated with the micellisation process can be calculated using the following: equation:

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

3. RESULTS AND DISCUSSION

3.1 Effect of phosalone on the critical micellar concentration

The experiment entails monitoring the evolution of the specific conductivity of an initial solution of phosalone with a fixed concentration as a function of the variation in concentration of anionic surfactant SDS or cationic surfactant CTAC, which are added using a micropipette. The effect of phosalone concentration on the CMC and α of the two surfactants was evaluated at various temperatures (301.15, 306.15, 311.15, 316.15, and 321.15 K). For each temperature, the same trend in CMC and α as a function of insecticide concentration was consistently observed. The results obtained at 301.15 K was presented in table 2. It is anticipated that ionic surfactants will undergo complete ionisation in solution, thereby establishing a direct correlation between conductivity and surfactant concentration.

At the outset of the dosage regimen, a slight increase in specific conductivity is observed as a function of surfactant concentration. Subsequently, the surfactant dissolves in water, resulting in the formation of simple monomers. The monomeric surfactant molecules display enhanced mobility in comparison to their aggregated form [9]. In the vicinity of the critical micelle concentration (CMC), the monomers gradually assemble to form approximately spherical micellar aggregates. Upon reaching CMC, a pronounced and precipitous surge in the specific conductivity is observed. Subsequently, the linear enhancement of conductivity values with the increase in surfactant concentration proceeds with a smaller slope following the CMC. This results in a less pronounced increase in specific conductivity. The values of CMC, α, and ΔG° obtained at varying concentrations of phosalone are presented in Table 2.

Table 2. Micellization parameters values as a function of phosalone concentration at 301.15 K

Phosalone	SDS			CTAC		
Concentration x10^6 (M)	CMC [a] (mM)	α (%) [b]	ΔG° [c] (kJ mol^{-1})	CMC [a] (mM)	α (%) [b]	ΔG° [c] (kJ mol^{-1})
0.0	3.101	69.302	-43.289	2.975	78.113	-40.628
0.25	2.975	56.129	-47.955	2.849	76.728	-41.360
0.5	2.597	39.434	-54.629	2.597	70.333	-44.117
1.0	2.344	34.267	-57.246	2.344	49.372	-52.029
1.5	1.832	33.595	-59.554	1.961	43.517	-55.465
2.0	1.445	22.872	-65.511	1.832	33.038	-59.752
3.0	1.311	22.281	-66.587	1.575	32.393	-61.263

[a] Critical micellar concentration ; [b] Degree of dissociation ; [c] Gibbs free enthalpy. M = mol L^{-1}

As illustrated in Table 2, there is a notable decline in both CMC and degree of ionisation as the concentration of phosalone increases. The progressive decrease in CMC values as a function of insecticide concentrations suggests the presence of an interaction between phosalone and surfactants. This phenomenon may be attributed to the solubilisation of the phosalone insecticide in each of the surfactants utilised. In all instances, the absolute value of the free enthalpy of micellisation (ΔG°) is observed to increase with increasing phosalone concentration. In other words, the presence of the insecticide in aqueous solution results in an increased propensity for the formation of the micellar phase, which consequently reduces the CMC. From this, we can infer that phosalone functions as a co-surfactant in the micellisation process [22]. The variation in surfactant CMC as a function of phosalone concentration is illustrated in Fig. 2.

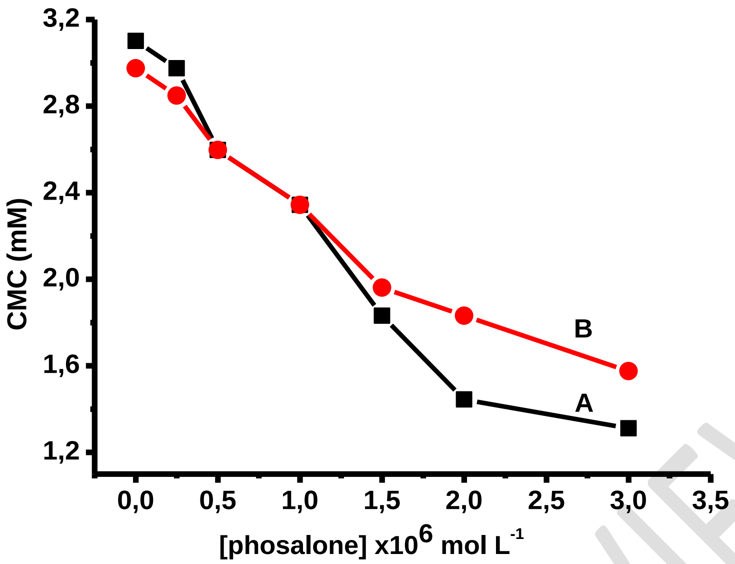

Fig. 2. CMC variation of SDS (A) and CTAC (B) as a function of phosalone concentration at 301.15 K

The plot of the curve of the critical micellar concentration as a function of the concentration of phosalone

(CMC = f ([PSL]) allows us to see a progressive decrease in the CMC for increasing concentrations of the pesticide. However, this decrease is more significant at level of the anionic surfactant SDS [23]. To properly explain this decrease in CMC in the presence of phosalone, it is important to see its chemical structure. Indeed, this molecule of chemical formula $C_{12}H_{15}ClNO_4PS_2$ with a hydrophobic part comprising the benzene ring and polar bonds (C-Cl, C=O, P=O, C-N) shows that it has a very high permanent dipole moment [24]. In solution, it demonstrates a robust capacity to interact with the tails of surfactants. It should be noted, however, that the non-polar sections of the molecule are also capable of interacting with the polar heads of surfactants. This phenomenon allows highly hydrophobic organic compound to make favorable associations for micellar solubilisation [14, 15]. The existence of this dipole moment shows that solubilization takes place at the water/micelle interface. Phosalone increases the action of CTAC and SDS by adsorbing at the interface while at the same time decreasing the surface tension of the micelles. The results obtained are shown in Fig. 3.

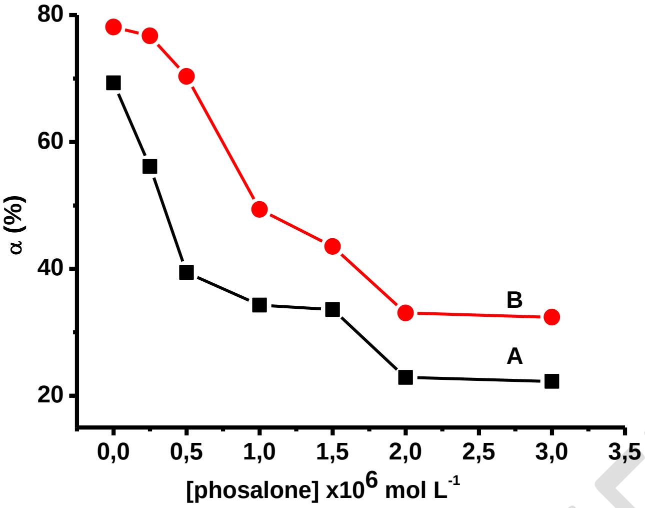

Fig. 3. α variation of SDS (A) and CTAC (B) as a function of phosalone concentration at 301.15 K

In both instances, a reduction of α is evident as the concentration of phosalone increases. The SDS surfactant demonstrates a more pronounced reduction of α, indicative of a substantial solubilisation of phosalone within the core of the micelles. This is attributable to an increase in hydrophobic attraction forces, which results in a reduction of α at the CMC.

3.2. Effect of temperature on CMC

The same assay was conducted, with specific conductivity monitored as a function of surfactant concentration at varying temperatures and at a fixed concentration of phosalone (2.5×10^{-7} M). As indicated in section 2.5, the value of ΔH^0 is calculated using the provided equation 3.

The representative curve of $lnXCMC = f(T)$ was used to obtain the term $\frac{d}{dT}(lnXCMC)$ of equation 3, which is the slope (Fig. 4). Subsequently, the value of ΔS^0 was determined using equation 4, following the calculation of ΔG^0 using equation 1.

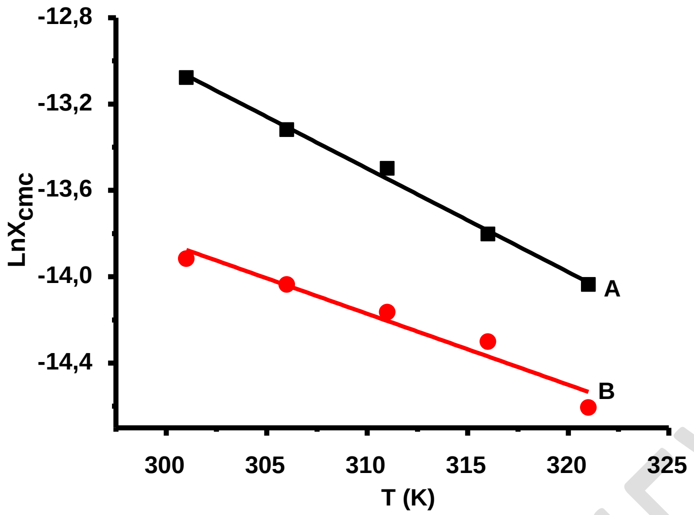

Fig. 4. Representation of lnX_{CMC} as a function of temperature, SDS (A) and CTAC (B)

A review of the data presented in Table 3 reveals a significant correlation between temperature and CMC. It was observed that the concentration of CMC decreased with an increase in temperature. This results in the dehydration of the hydrophilic component (polar heads) of the surfactants, which favours micellisation. Moreover, the interaction of water molecules with hydrophobic groups (linear surfactant chains) may facilitate the process of micellisation by increasing the solubility of the pesticide [14, 15]. Similarly, Thiaré et al. observed this phenomenon when analysing the insecticide fenvalerate in an inverse micellar medium using photoinduced fluorescence [25].

3.3. Thermodynamic parameters

The thermodynamic parameters associated with the association reaction of surfactants with phosalone were calculated using equations (1), (2) and (3). The values of ΔG^0, ΔH^0 and $-T\Delta S^0$ are presented in Table 3.

The negative values of free enthalpy (ΔG°) obtained indicate that the micellisation process is thermodynamically favourable [14, 15, 25]. These values demonstrate a clear decrease with increasing temperature, which indicates that the solubility of phosalone increases with temperature.

Furthermore, the ΔH° values are positive in all cases and increase with temperature, indicating that the process is endothermic and that water molecules near the hydrophobic part of the surfactants are undergoing degradation. This demonstrates that heat is transferred from the surrounding environment to the micellar system, which is the underlying cause of the observed increase in the system's enthalpy. It is described as an energetically ascending process. [14, 15].

In accordance with the principles of thermodynamics, if a process is endothermic, the variation in free enthalpy (ΔG°) must be negative. This is contingent upon the enthalpy factor (ΔH°) and entropy factor (TΔS°) exhibiting a positive and negative variation, respectively, with |TΔS°| being greater than ΔH°.

This is evidenced by the positive entropy variation observed in our study, which increases with temperature. The results demonstrate that the micellisation of the insecticide phosalone with SDS and CTAC is spontaneous.

Table 3. Micellisation parameters (CMC, X_{CMC} and α) and thermodynamic parameters values at various temperatures

Surfactant	T (K)	CMC (mM)	α (%)	ΔG^0 (kJ mol^{-1}) [d]	ΔH^0 (kJ mol^{-1}) [e]	ΔS^0 (kJ mol^{-1}) [e]
SDS	301.15	3.35	30.84	-48.32	45.59	0.312
	306.15	2.97	34.78	-49.71	49.74	0.325
	311.15	2.72	38.48	-50.91	54.83	0.340
	316.15	2.34	38.54	-53.75	59.38	0.358
	321.15	2.09	40.02	-59.48	62.50	0.380
CTAC	301.15	2.22	32.99	-58.16	37.56	0.318
	306.15	2.09	36.42	-58.41	39.51	0.320
	311.15	1.96	40.18	-58.53	42.23	0.324
	316.15	1.83	44.07	-58.59	45.06	0.328
	321.15	1.57	47.92	-59.28	46.97	0.331

3.4. Entropy-enthalpy compensation

The enthalpy-entropy compensation can be obtained by utilising the linear relationship between ΔH^0 and ΔS^0. As illustrated in Equation 4, when a process exhibits a positive enthalpy change, it is also accompanied by an increase in entropy ($\Delta S^0 > 0$). The fact that |TΔS°| is greater than ΔH^0 indicates that the association reaction between phosalone and surfactants (SDS and CTAC) is partially controlled by the entropy term. This demonstrates that the rise in enthalpy during micellisation is counterbalanced by the growth in disorder within the system. This expansion in entropy may be attributed to the aggregation of the hydrophobic component of the micelles, as well as the release of counterions [11]. A representative plot of ΔH^0 versus ΔS^0 is illustrated in Fig. 5.

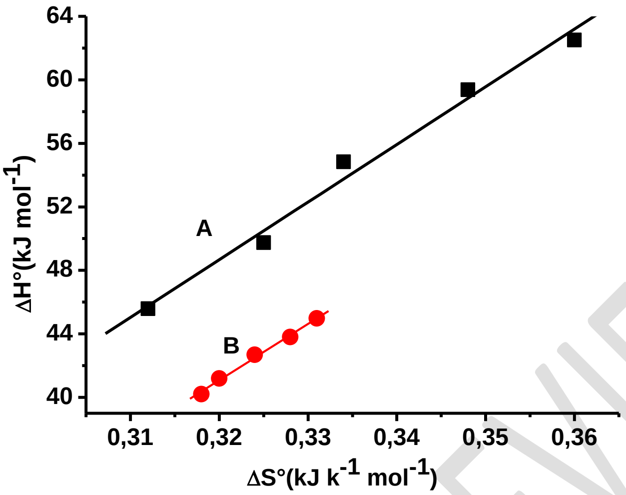

Fig. 5. The enthalpy-entropy compensation of phosalone in SDS (A) and CTAC (B)

The ΔH^0 plot according to ΔS^0 shows a linear curve, thus showing a compensation between enthalpy and entropy. A linear correlation between ΔH^0 and ΔS^0 with r^2 value from 0.987 to 0.994 was observed. ΔH^0 and T_C connote the enthalpy and compensation temperature. The values of ΔH^0 and T_C illustrates the solute-solvent interaction. The T_C values of studied systems lie in the range of 186-199 K. The ΔH^0 values were found to be negative in the SDS and CTAC and show an important participation of the hydrophobic part in the micellization and in the stability of the micelle formed. The higher value of ΔH^0 was found in SDS. Table 4 shows the values of ΔH^0 and T_C.

Table 4. Compensation temperature (Tc) and compensation enthalpy of surfactants in the presence of 2.5×10^{-7} M in the temperature range studied

Medium	Concentration (mol L^{-1})	ΔH^0 (KJ mol^{-1})	Tc (K)
SDS	$2.5\ 10^{-7}$	-67.63	186.15
CTAC	$2.5\ 10^{-7}$	-72.47	199.15

4. CONCLUSION

In order to study the solubilisation process of some organic compounds and their interaction with surfactant solutions, we used the conductometric measurement method to solubilise the pesticide phosalone in SDS and CTAC. A study of the micellar properties of ionic surfactants used in the micellar solubilisation of phosalone was also carried out. These properties are the CMC and α. The application of these two surfactant molecules (CTAC and SDS) in the micellar solubilisation of the insecticide was carried out by measuring the conductivity at different temperature. The CMC values of ionic surfactant in aqueous medium decrease linearly with the concentration of phosalone and the temperature. The results obtained from these studies showed that the temperature and the concentration of phosalone favor the formation of the micellar phase. The negative magnitudes of ΔG^o in each case reveal the spontaneous micellisation, while the positive values of ΔH^0 testify to the existence of an endothermic process. The analysis of the results based on the calculation of the thermodynamic parameters showed that phosalone solubilises in the micellar phase. In both cases, the solubilisation occurs at the water-micelle interface.

Consent to Publish

All authors approved the final manuscript as submitted and agree to be accountable for all aspects of the work.

Disclaimer (Artificial intelligence)

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc.) and text-to-image generators have been used during the writing or editing of this manuscript.

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