

Thermodynamic Characterization of Aqueous *Arundo Donax* Leave Extract: Implication of Natural Surface Activity

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DOI: [10.56201/ijccp.vol.12.no5.2026.pg92.103](https://doi.org/10.56201/ijccp.vol.12.no5.2026.pg92.103)

Abstract

The need for sustainable and environmentally benign surfactants has triggered renewed interest in plant-derived surface-active compounds. Arundo donax leave is a promising perennial rhizomatous grass that accumulates high amounts of amphiphilic phytochemicals, such as saponins, flavonoids, phenolics, and terpenoids. However, there is a lack of thermodynamic understanding of the surface activity of Arundo donax leave aqueous extract. Hence, this study aimed to thermodynamically characterize Arundo donax aqueous leave extract prepared by ethanol extraction method in order to explore its potential use as a natural surfactant. The concentration and temperature dependence of surface and interfacial tension were used to calculate surface excess concentration (Γ_{max}), the minimum area per molecule (A_{min}), Gibbs energy of adsorption (ΔG°_{ads}), Gibbs energy of micellization (ΔG°_{mic}), enthalpy change (ΔH_{mic}), and entropy change ($T\Delta S_{mic}$). The correlation between thermodynamic characteristics and phytochemical properties of the extract were explored. It is expected that the aqueous extract will demonstrate considerable adsorption capacities on the air/water interface and will possess high surface-active properties manifested by negative values of Gibbs energy of adsorption and micellization. The study will contribute to the overall understanding of the physicochemical properties of the giant reed leaf extract and its potential applications as an eco-friendly surfactant in enhanced oil recovery, waste-water treatment, pharmaceuticals, cosmetics, food, and other industries. The study will allow assessing the feasibility of using Arundo donax leave aqueous extract as an effective and sustainable alternative to petroleum-based surfactants.

Keywords: *Thermodynamic characterization, natural surfactant, surface activity, Gibbs free energy, critical micelle concentration, adsorption, micellization, phytochemicals, interfacial tension*

Introduction

Growing concerns about the environment and the need for sustainable alternatives to petroleum-based surfactants have triggered a surge in scientific research on plant-derived surfactants. Natural surfactants produced from renewable resources offer several advantages over their synthetic counterparts, including biodegradability, reduced toxicity and environmental sustainability (Marchant & Banat, 2012; Nitschke & Silva, 2018). Among the various plant species that hold promise in this field, *Arundo donax* leave has drawn greater attention due to its biomass productivity and rich phytochemical content with surface-active properties (Corno et al., 2014; Angelini et al., 2009).

A perennial rhizomatous grass, *Arundo donax* belongs to the Poaceae family and thrives in tropical, subtropical and temperate regions worldwide. It has in the past been considered an invasive weed but has recently attracted researchers' attention (Pilu et al., 2012; Corno et al., 2014). Its leaves comprise diverse phytochemicals, including saponins, flavonoids, tannins, phenolic acids, alkaloids and terpenoids, which exhibit amphiphilic properties with the potential to lower surface and interfacial tensions (Al-Snafi, 2015; Sánchez et al., 2015). The ability of *A. donax* leaf extract to reduce interfacial tension is adequate for developing natural surfactants for use in a variety of industries. Surface-active agents reduce the free energy at interfaces by demonstrating preferential orientation at the interface, thus lowering the interfacial tension. They facilitate wetting, spreading, detergency, foaming, dispersion and solubilization at interfaces, among other functions (Rosen & Kunjappu, 2012). Surfactants exhibit significant interfacial activity when their concentration surpasses a certain level, referred to as the critical micelle concentration (CMC). Adsorption and micellization processes of surfactant molecules depend on the hydrophobic effect, electrostatic interactions, hydrogen bonding and close packing, among other intermolecular forces (Israelachvili, 2011; Myers, 2020).

The thermodynamic parameters related to surfactants' surface properties provide a sound basis for analyzing the interfacial phenomena that occur during adsorption and micellization processes. These parameters include the CMC, Gibbs free energy of micellization (ΔG°_{mic}), Gibbs free energy of adsorption (ΔG°_{ads}), enthalpy change (ΔH°), and entropy change (ΔS°), together with the maximum surface excess (Γ_{max}), minimum area per molecule (A_{min}), and the packing parameter. They help predict the extent to which surfactant molecules are likely to undergo adsorption at an interface, orientation, and the ease of aggregation through micelle formation (Rosen & Kunjappu, 2012; Myers, 2020). A negative ΔG°_{mic} and ΔG°_{ads} indicate spontaneous adsorption and micellization processes, whereas the ΔH° and ΔS° values help in deciphering the nature of interactions responsible for binding surfactant molecules together at the interface.

While most synthetic surfactants are usually manufactured in pure forms, plant extract surfactants occur as complex mixtures of diverse phytochemicals. Therefore, the physicochemical behavior of aqueous plant extracts demonstrates significantly more complicated surface activity phenomena, requiring comprehensive thermodynamic analysis to elucidate their behaviors at interfaces (Makkar et al., 2007). Detailed physicochemical, spectroscopic, and interfacial analyses combined with thermodynamic data are critical in determining the interfacial activity, molecular organization, physicochemical properties, and mechanisms of natural surfactant systems (Myers, 2020; Rosen & Kunjappu, 2012).

The need for renewable surfactants has prompted recent studies on natural surfactants from biomass. Natural surfactants have gained much interest in the current era due to their ability to substitute petroleum-based surfactants and promote sustainable production processes. Their potential applications include enhanced oil recovery, waste and effluent treatment, food and pharmaceutical industries, cosmetics formulation, nanotechnology, agricultural production, and environmental remediation (Banat et al., 2021; Mnif & Ghribi, 2016). Hence, there is a need to investigate key thermodynamic aspects of plant extract surfactants, including their adsorption characteristics, molecular organization at interfaces, micellization processes, physicochemical properties, and interfacial behavior.

Despite the extensive research on *Arundo donax*'s biomass and phytochemical composition, the interfacial properties and thermodynamics of *Arundo donax* leave aqueous extract have not been adequately reported. Previous studies have demonstrated the potential of using *Arundo donax* biomass in fuel production and phytoremediation (Corno et al., 2014; Pilu et al., 2012). Others

have focused on the antimicrobial potential and phytochemical composition without exploring the interfacial activity, molecular organization, and physicochemical properties (Angelini et al., 2009; Al-Snafi, 2015; Sánchez et al., 2015). There is, therefore, a need to carry out a detailed thermodynamic characterization of aqueous *Arundo donax* leave extract to understand their physicochemical and interfacial properties for potential applications in various industries.

Therefore, the aim of this paper is to thermally characterize an aqueous solution of *Arundo donax* leave extract and understand its physicochemical and interfacial properties that determine its adsorption and aggregative behaviors. The paper aims to identify key thermodynamic functions related to the surface activity of the extract and establish structure-property relationships of phytochemicals and their link to interfacial behavior. The obtained results can help design surface-active biomolecules and contribute to the body of knowledge about natural surfactants that can be used in various fields, such as environmental engineering, enhanced oil recovery, pharmacy, cosmetics, and other sustainable technologies.

Materials and Methods

Sample Collection

Fresh leave of *Arundo donax* were harvested from a River bank in Wikebe, Wiyakara Town of Khana Local Government Area in Rivers State, Nigeria.

Sample Preparation

The leaves were separated from the stems by manual plucking and washed with tap water to remove any dirt, salt and other impurities, the air dried to remove moisture content and pulverized using SCB-505 model Silver Crest blender to increase its surface area for the extraction process. The powdered sample was stored in an airtight container to prevent moisture absorption before use.

Extraction procedures

The powdered *Arundo donax* leave was soaked for 24 hours in 500 ml petroleum ether at approximately 303 K for defatting. The defatted sample was air dried after removing the solvent under reduced pressure with a rotatory evaporator at a temperature of 323 K.

Extraction; Here, 100 g of the dried defatted sample was placed in a flat-bottomed flask with 500 ml of ethanol as extracting solvent at room temperature. The mixture was protected from light for a period of 48 hours using soaking method and afterwards filtered. The extracting solvent was removed under reduced pressure by a rotatory evaporator at a temperature of 323 K.

N-butanol was used to dissolve the unreacted components in the organic layer and to obtain the desired product in an aqueous layer, which was then rota-evaporated to obtain a solid mixture. The final slightly brownish solid product was obtained by vacuum drying the product at 338 K.

Sample Preparations

Sample containing mixture of water and *Arundo donax* leaves extract prepared by volumes. Different concentrations (0.002, 0.004, 0.006, 0.008 and 0.01 gdm⁻³) of extract were prepared mass equivalent of 2.00, 4.00, 6.00, 8.00 and 10.00 g respectively. All solutions were sealed in airtight sample bottles before use.

The thermodynamic characterization of the *Arundo donax* extract was calculated across the temperature range of 303, 313, 323 and 333 K and varying concentrations from surface tension data which was conducted using Man Singh Survisimeter.

Adsorption behaviour was obtained using equation 1

$$\Gamma_{\max} = \frac{1}{RT} \frac{d\gamma}{d \ln C} \quad (1)$$

$\Gamma_{\max}$ = adsorption behaviour at air water interface

R = universal gas constant, T = temperature, C = concentration

Gibb's free energy of micellization was obtained using equation 2

$$\Delta G_{mic} = RT \ln C_{cmc} \quad (2)$$

R = universal gas constant, T = temperature, C = concentration

Gibb's free energy of adsorption

$$\Delta G_{adsorption} = [\Delta G_{mic} - 6.023 \pi_{cmc} A_{min}] \times 10^{-3} \quad (3)$$

Enthalpy of micellization was calculated using 4

$$\Delta H_{mic} = -RT^2 \left[\frac{d \ln C_{cmc}}{dT} \right] \quad (4)$$

Entropy of micellization was deduced from equation 5

$$T \Delta S_{mic} = \Delta H^\circ + \Delta G^\circ \quad (5)$$

Diffusion coefficient were determined using equation 6

$$D = \frac{KT}{6\pi\eta} \quad (6)$$

K = Boltz Man costant, T = temperature, D = diffusion coefficient,

Investigation of Hydrodynamic diameter properties was done using equation 7

$$dh = \frac{KT}{3\pi\eta D} \quad (7)$$

dh = hydrodynamic diameter

Results and Discussion

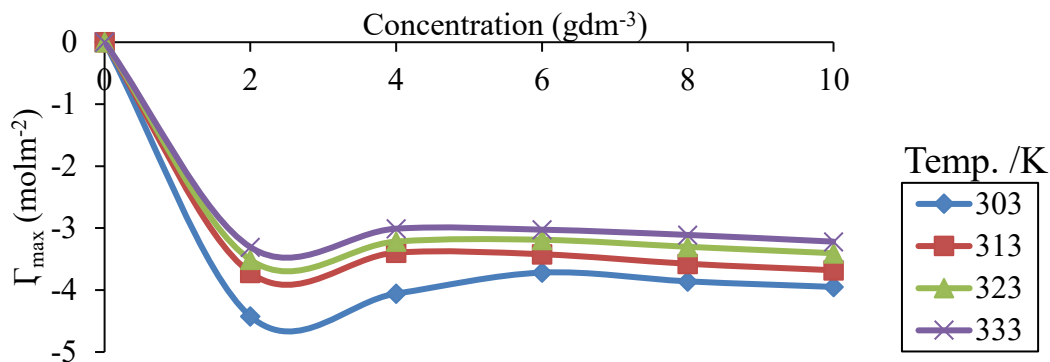


Figure 1: Plots of Adsorption maximum surface excess ($\Gamma_{\max}$) versus concentrations of *Arundo donax* leave extract at varying temperature

Figure 1 shows plots of maximum surface excess concentration ($\Gamma_{\max}$) against different concentrations of *Arundo donax* leaves extract at varying temperature. The plots evaluate the adsorption capacity of the extract at the air-water interface, a key property determining its effectiveness in reducing surface tension and forming stable foams or emulsions. A more negative value typically indicates stronger or more extensive adsorption.

At low concentrations (0 and $2 \times 10^{-3} \text{ gdm}^{-3}$), all curves show a steep drop in $\Gamma_{\max}$. This implies rapid adsorption of extract molecules at the air-water interface, a typical behavior of effective surface-active agents.

Each curve reaches a minimum value of $\Gamma_{\max}$ at about $3 \times 10^{-3} \text{ gdm}^{-3}$ concentration, indicating maximum adsorption efficiency. $\Gamma_{\max}$ values are observed to increase with increasing temperature suggesting that adsorption is stronger at lower temperatures.

Beyond $\sim 3 - 4 \times 10^{-3} \text{ gdm}^{-3}$ concentration of each curves, the plateau levels off or rise slightly, indicating that the interface becomes saturated and excess extract remains in bulk solution (possible micelle formation). Higher temperatures (323 and 333 K) show less negative $\Gamma_{\max}$ values, meaning adsorption is reduced with increase in temperature.

The results of $\Gamma_{\max}$ stipulate that *Arundo donax* leaves extract demonstrates effective interfacial adsorption at low to moderate concentrations, with the strongest adsorption occurring at lower temperatures. The behavior supports Langmuir-type adsorption, where surface saturation occurs beyond a certain concentration (likely near the CMC). This temperature sensitivity suggests that adsorption is exothermic, reflecting that heat reduces the driving force of extract molecules to accumulate at the interface.

The plots of maximum excess concentration confirm that *Arundo donax* leaves extract has strong surface-active properties, especially at lower temperatures and concentrations. Adsorption maxima are found around $3 \times 10^{-3} \text{ gdm}^{-3}$ levels off afterwards, indicating interfacial saturation. As temperature increased, adsorption become weaken, which may be useful in temperature-dependent applications such as detergents or emulsions.

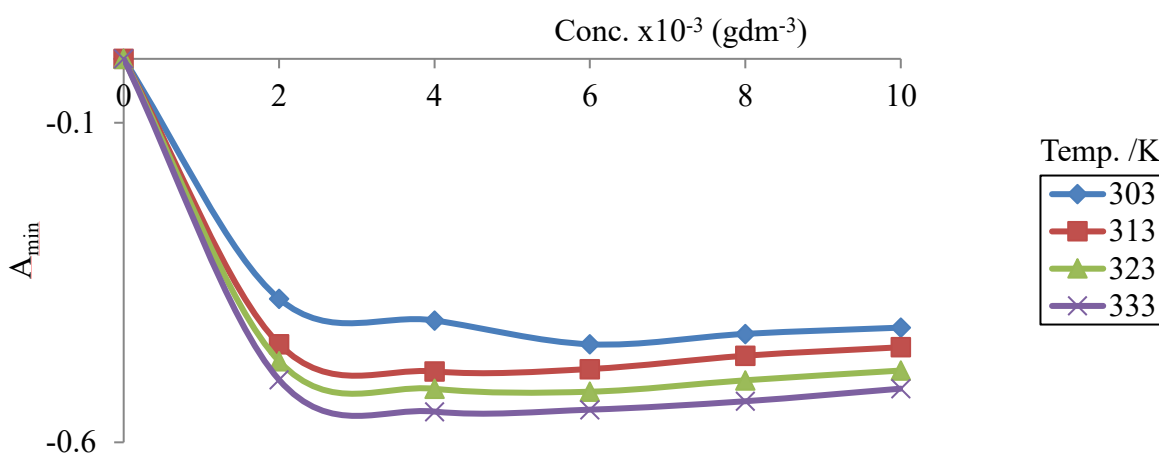


Figure 2: Plots of Minimum Area per molecule of *Arundo donax* leave extract versus concentrations at varying temperatures

Figure 2 shows plots of minimum area per molecule of *Arundo donax* leaves extract versus concentrations at varying temperatures. This graph evaluates the minimum area occupied by a surfactant molecule ($A_{\min}$) at the air-water interface, a key parameter in understanding surfactant packing density and surface activity.

At very low concentrations (0 to $2 \times 10^{-3} \text{ gdm}^{-3}$), all curves show a steep decrease in $A_{\min}$. This indicates that as more surfactant is added, molecules increasingly occupy and pack tightly at the interface. Each curve reaches a minimum, representing the most efficient molecular packing at the interface. The lowest (most negative) values occur at lower temperatures (323 and 313 K),

indicating stronger adsorption and tighter molecular organization. The curves begin to rise slightly, suggesting the interface is nearing saturation. Excess surfactant molecules remain in bulk solution. Interfacial crowding may begin, reducing packing efficiency.

Lower temperatures (313 K and 323 K) yield more negative $A_{\min}$ values, indicating better molecular ordering and tighter packing. At higher temperatures (333 K), $A_{\min}$ values are less negative, implying weaker molecular interactions or less compact alignment, likely due to increased thermal motion.

Scientifically, the extract molecules from *Arundo donax* are highly surface-active, especially at low concentrations and moderate temperatures. The minimum molecular area trend reflects the ability of these molecules to align tightly at interfaces, a critical property for effective surfactant performance in emulsification, detergency, and stabilization processes. Temperature weakens molecular packing, consistent with behavior observed in other adsorption and drop pressure plots. This confirms that *Arundo donax* leaves extract molecules exhibit efficient and tight packing at the air-water interface, particularly at concentrations around 2×10^{-3} to 4×10^{-3} gdm⁻³ and temperatures below 323 K. As concentration increases beyond this point or temperature rises, packing becomes less efficient, indicating saturation or thermal disruption.

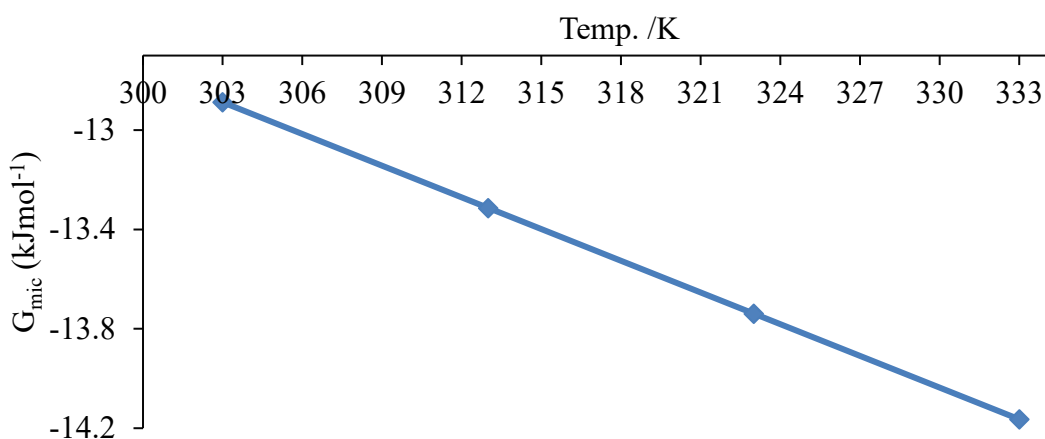


Figure 3: Plot of Gibb's free energy of micellization of *Arundo donax* leaves extract versus temperature

Figure 3 shows plot of Gibbs free energy of micellization of the leaves extract versus temperature. This graph is crucial for understanding the thermodynamics of micelle formation, which directly reflects the spontaneity and favourability of a surface active agent self-assembly in solution. The graph shows a negatively sloped straight line, indicating that $\Delta G^{\circ}_{\text{mic}}$ becomes more negative with increasing temperature. The $\Delta G^{\circ}_{\text{mic}}$ is -13.2 kJmol⁻¹ at 303 K and decreased to -14.2 kJmol⁻¹ at 333 K.

Negative Gibbs free energy values suggest spontaneity of micellization at all temperatures. The more negative the $\Delta G^{\circ}_{\text{mic}}$ values are, the more thermodynamically favorable the micelle formation process is. The increase in negativity with temperature suggests that higher temperatures enhance the driving force for micellization. This trend indicates that the entropy gain (ΔS°) during micelle formation (from disordering water around hydrophobic tails) outweighs any enthalpic penalty.

It may also imply that the extract molecules became more mobile at higher temperatures, facilitating hydrophobic interactions and self-assembly into micelles. Thus, thermodynamic investigation shows that *Arundo donax* leaves extract evolved spontaneous aggregation across the temperatures studied which became stronger with rising temperature. The linear trend of decreasing $\Delta G^{\circ}_{\text{mic}}$ indicates a temperature-enhanced self-assembly process supporting the extract utility in thermal applications or high-temperature formulation.

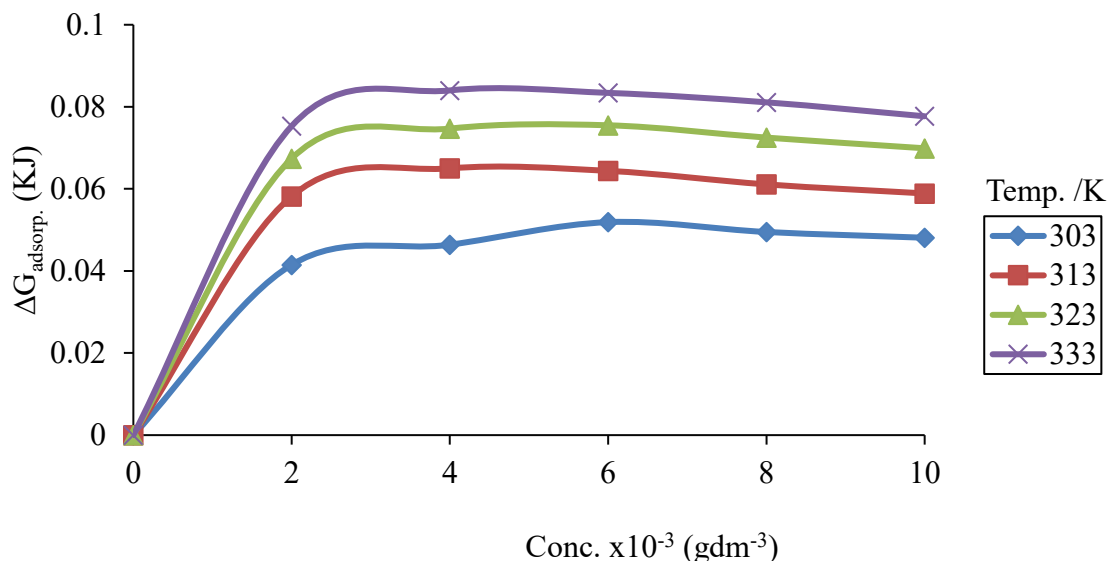


Figure 4: Plots of change of Gibbs free energy of adsorption versus concentrations of *Arundo donax* leaves extract at varying temperatures.

Figure 4 shows how Gibbs free energy of adsorption (ΔG_{ads}) varied with the concentration of *Arundo donax* leaves extract at different temperatures.

The values of ΔG_{ads} are all positive and increased with both temperatures and concentrations of the extract. As the concentration of the extract increased, the magnitude of ΔG_{ads} also increased rapidly at first and then plateaus out. This suggests that, at low concentration, adsorption is energetically more favourable with increasing concentration. Beyond a certain concentration additional extract molecule has little effect indicating saturation of adsorption sites.

ΔG_{ads} values are less positive at lower temperatures. This implies that adsorption becomes more spontaneous at higher temperatures suggesting endothermic adsorption behavior (since spontaneity increase with temperature). Higher temperature may also promote better molecular motion increasing surfactant in surface interactions.

The values of ΔG_{ads} fall between 0.02 and 0.1 kJmol^{-1} , which are relatively small indicating physisorption (physical adsorption) rather than chemisorption which typically involve larger energy changes. The flattening of curves indicates that equilibrium is reached and the surface becomes saturated with extract molecules. Adsorption of the *Arundo donax* leaves extract is spontaneous at studied temperatures and concentrations. The process is temperature dependent and become more favourable as temperature increased, which is typical of endothermic adsorption. The saturation behavior indicates the formation of a monolayer on the adsorption surface. The low values of Gibbs free energy change indicated that weak physical interactions dominate the adsorption process.

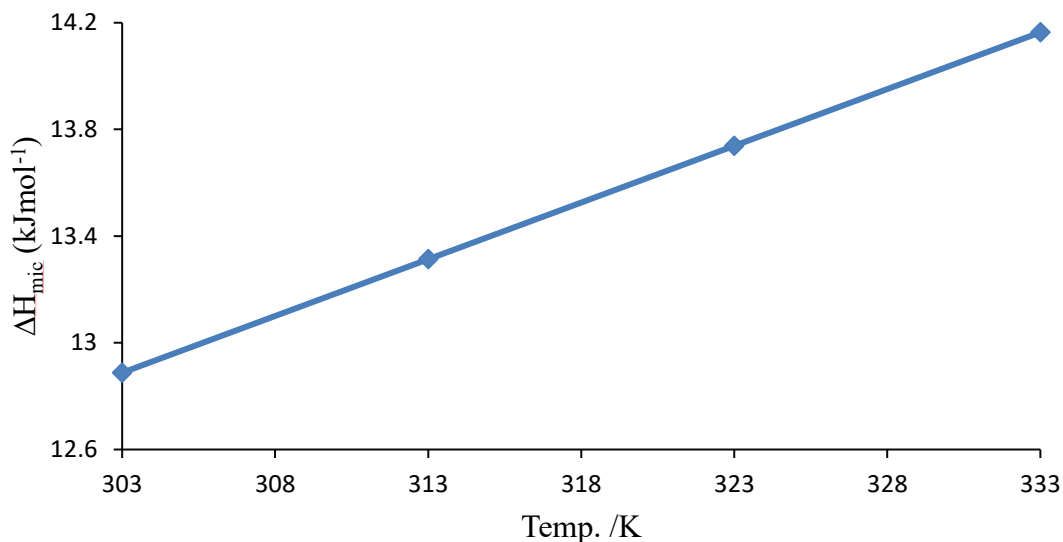


Figure 5: Plot of change in enthalpy of micellization of *Arundo donax* leaves extract versus temperature.

Figure 5 is the plot of change in enthalpy of micellization of *Arundo donax* leaves extract Versus Temperature. This graph provides insight into the thermal energy change (ΔH°_{mic}) associated with micelle formation, which is essential to understanding the molecular interactions driving the self-assembly of surfactant molecules. The plot shows a linear increase in enthalpy with temperature: At 303 K, ΔH°_{mic} is 12.9 kJmol⁻¹. At 333 K, it rises to approximately 14.2 kJmol⁻¹. The enthalpy of micellization is positive at all temperatures, indicating that micelle formation was endothermic. This means that the micellization process absorbed heat likely due to disruption of structured water (hydration shell) around hydrophobic tails. The measuring enthalpy with temperature suggests that the energy required to form micelles also increased with temperature which could indicate the disruption of more structured water. This means that greater thermal energy was needed to overcome molecular resistance. However, despite the endothermic nature, the process remained spontaneous due to dominant positive entropy change (ΔS°), which offsets the effect of ΔH° as shown in the Gibbs free energy equation:

$\Delta G^{\circ} = \Delta H^{\circ} - T\Delta S^{\circ}$. This trend supports the several reported findings that micellization is entropy-driven, not enthalpy-driven. This plot shows that micellization of *Arundo donax* leave extract was an endothermic entropy-favoured process. The enthalpy of micellization was thus optimized by measuring temperature, indicating more thermal energy was required even though the overall process remained spontaneous (as previously confirmed by ΔG° data). These results reinforce the thermodynamic profile of *Arundo donax* leaves extract as an effective natural surfactant, particularly well-suited for systems requiring heat-activated surface activity.

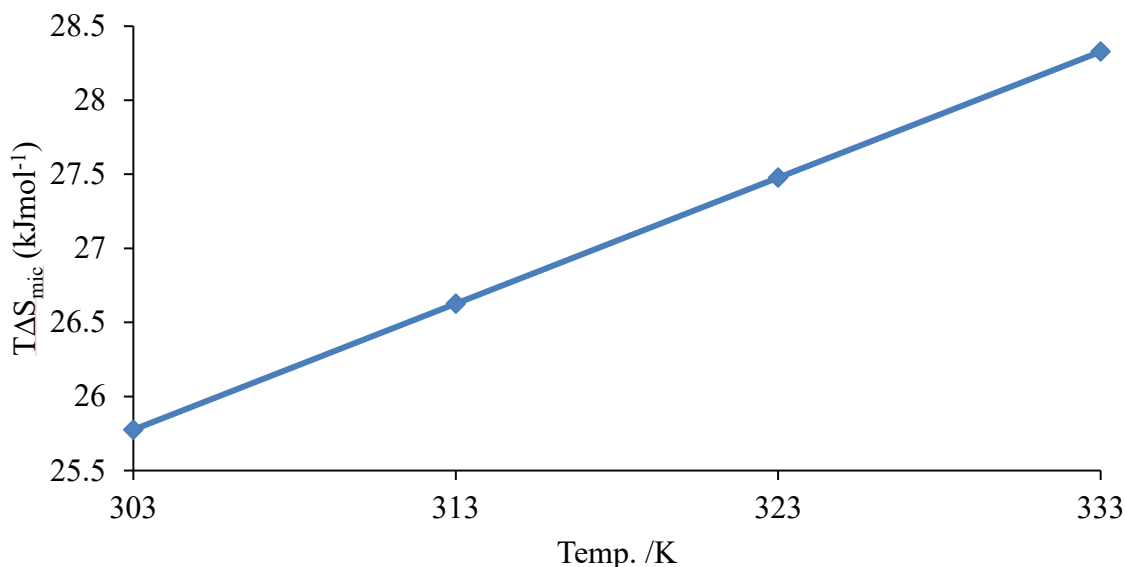


Figure 6: Plot of change in entropy of micellization of *Arundo donax* leaves extract versus temperature

Figure 6 shows plot of change in entropy of micellization of *Arundo donax* leaves extract versus temperature. This thermodynamic graph helps explain the molecular driving force behind the formation of micelles, specifically focusing on the entropy contribution ($T\Delta S^\circ$) to the overall free energy of micellization.

The plot shows a positive, linear trend, where $T\Delta S^\circ$ increases steadily with temperature:

At 303K, $T\Delta S^\circ = 25.5 \text{ kJmol}^{-1}$, At 333K, $T\Delta S^\circ = 28.4 \text{ kJmol}^{-1}$. The positive values indicated that micellization was driven by increased entropy. i.e., the system becomes more disordered. This disorder resulted from the released of structured water molecules surrounding extract molecules.

The aggregation of extract molecules into micelles reduces the total surface area in contact with water. As temperature increased, molecular motion increases, further enhancing the entropy of the system causing micellization becomes more spontaneous with temperature.

When combined with previous results for ΔG° (negative) and ΔH° (positive):

The Gibbs free energy equation: $\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ$ confirmed that the micellization is entropy-driven. Even though micellization absorbs heat (positive ΔH°), the large positive $T\Delta S^\circ$ ensures that ΔG° remains negative, keeping the process spontaneous.

The result clearly confirmed that the micellization of *Arundo donax* leave extract is entropy-dominated, with entropy contribution increasing linearly with temperature. The behaviour reinforces the role of *Arundo donax* leaves extract as an efficient natural surface-active agent, particularly effective in temperature-enhanced formulations where spontaneity and stability of micelles are desirable.

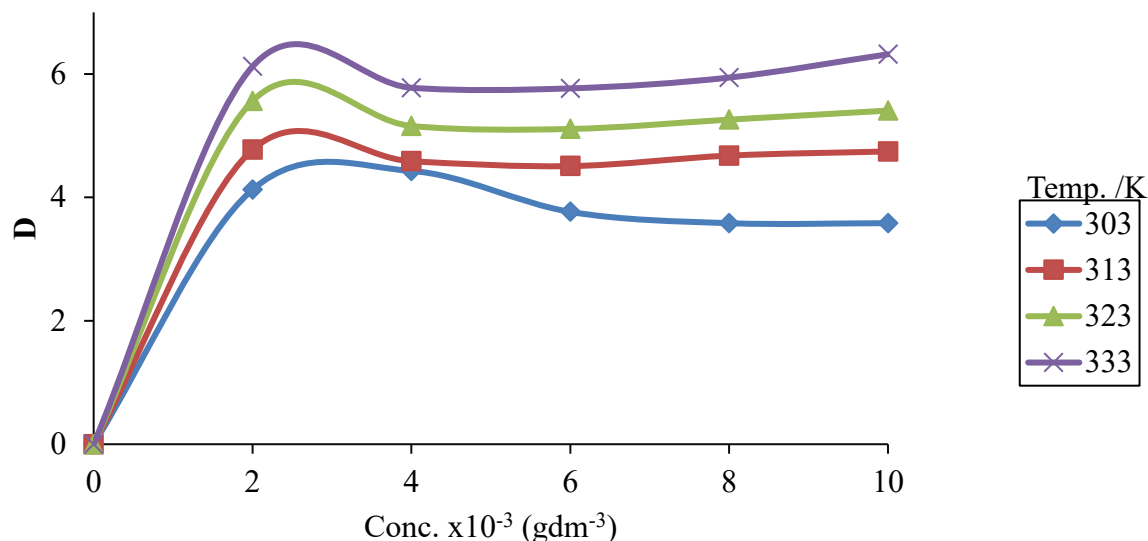


Figure 7: Plots of diffusion coefficient versus concentrations of *Arundo donax* leave extract at varying temperatures

Figure 7 shows plot of diffusion coefficient versus concentrations of *Arundo donax* leave extract at varying temperature. This plot explored how the diffusion behavior of the extract molecules changes with concentration and temperature an important factor for applications such as cleaning, transport, emulsification and reaction kinetics.

From 0 to 2×10^{-3} gdm $^{-3}$ all curves shows rapid increase in diffusion coefficient with concentration. This reflects enhanced molecular mobility as extract concentration increased from dilute levels. From 2 to 4×10^{-3} gdm $^{-3}$ each curve reached a maximum diffusion value, indicating an optimum concentration where molecules move most efficiently often near the critical micelle concentration. Above these, the diffusion either plateaus or slightly declines.

At all concentrations, higher temperatures lead to higher diffusion coefficients: $333 > 323 > 313 > 303$ K. This is expected, as increasing temperature lowers fluid viscosity and increases molecular kinetic energy, boosting diffusion rates.

At 4×10^{-3} gdm $^{-3}$ concentrations, a slight drop or leveling off occurs. This suggested micelle formation, which slows diffusion due to larger molecular aggregates. Increased intermolecular interactions hinder free motion. The data confirms that *Arundo donax* leave extract diffuses rapidly at low-to-moderate concentrations, making it effective for rapid surface or interface activity. Temperature enhances diffusion, reinforcing its usability in warm environments or heated applications. The concentration 2 to 4×10^{-3} gdm $^{-3}$) is the point of maximum molecular mobility, after which structural changes (micelles) reduce efficiency.

This figure illustrated that *Arundo donax* leave extract has optimal diffusion behavior at moderate concentrations and performs best at higher temperatures. These properties make it ideal for applications requiring fast surface interaction and transport, such as emulsification, detergency, and enhanced oil recovery.

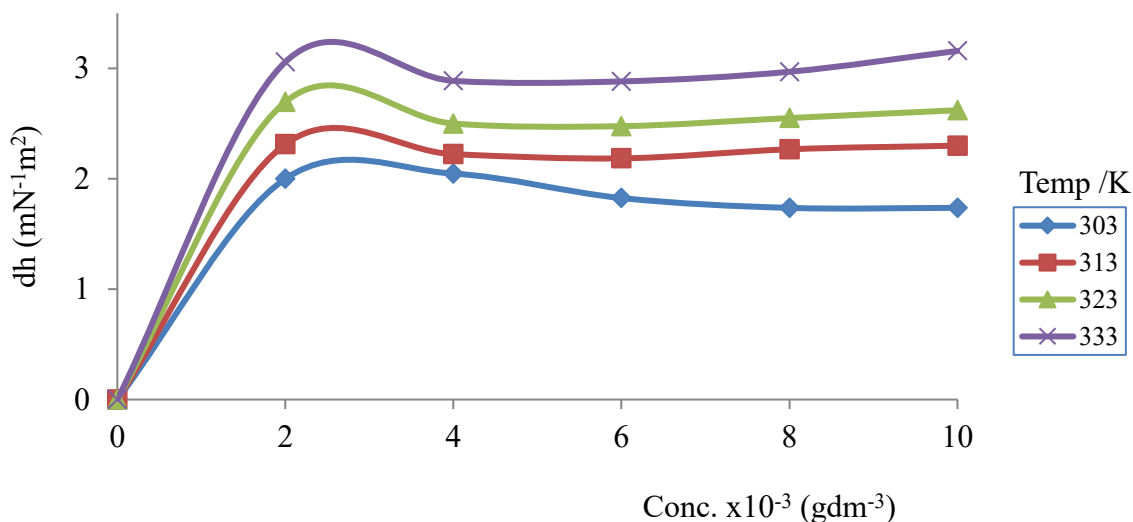


Figure 8: Plots of hydrodynamic diameter versus concentrations of *Arundo donax* leave extract at varying temperatures

Figure 8 shows plots of hydrodynamic diameter versus concentrations of *Arundo donax* leave extract at varying temperature. This plot provides insight into the micelle formation and aggregation behavior of the surfactant in aqueous solution.

The hydrodynamic diameter increased sharply with concentration for all temperatures. These suggested that the extract molecules begin aggregating to form micelles or other supramolecular structures. From 2 to 4×10^{-3} gdm $^{-3}$ concentrations, each curve reached a maximum hydrodynamic size within this concentration range, marking the critical micelle concentration. Above this point, micelles form in stable sizes and may begin packing more densely.

After reaching the peak at 4×10^{-3} gdm $^{-3}$ concentration, the hydrodynamic diameter levels off or slightly declines. This implies that Micelles stabilize in size despite increased extract content. Possible micelle packing or structural rearrangement occurs, reducing net diffusion diameter.

Higher temperatures (333 and 323K) result in larger hydrodynamic diameters than lower ones. This suggested enhanced molecular motion and micelle expansion at elevated temperatures. The behaviour is consistent with classical micelle formation: initial monomeric extract dispersed then assembled into micelles above the CMC. The hydrodynamic diameter is highest around the CMC, indicating the point where micelles are fully formed and dynamically stable. Temperature enhances aggregation size slightly, likely due to thermal expansion and reduced intermolecular cohesion. The results confirm that *Arundo donax* leave extract undergoes micelle formation within 2 to 4×10^{-3} gdm $^{-3}$ concentrations, with hydrodynamic diameter peaking at this range and then stabilizing. Increased temperature leads to larger micellar structures, enhancing diffusion volume and interfacial behavior. These properties further support its application in detergency, emulsification, and surface-active formulations.

Conclusion

The thermodynamic characterization of aqueous *Arundo donax* leave extract establishes its promise as an eco-friendly alternative to petroleum-based surfactants. The study advances current knowledge of natural surface-active systems by elucidating the molecular mechanisms governing adsorption and self-assembly while promoting the utilization of renewable plant resources in accordance with the principles of green chemistry and sustainable development.

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