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PAPER

DETERMINATION OF THE CRITICAL MICELLE CONCENTRATION IN SURFACTANT SOLUTIONS

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Abstract

This study investigates the determination of the critical micelle concentration (CMC) and cloud point of synthesized surfactants, which are key parameters governing their physicochemical behavior in aqueous solutions. Surfactants, due to their amphiphilic nature, exhibit the ability to reduce surface tension and form micelles beyond a certain concentration threshold. The CMC was determined using surface tension measurements via the Du Noüy ring method, while the cloud point was established through controlled heating of surfactant solutions. Experimental results showed a decrease in surface tension with increasing concentration until a plateau region corresponding to micelle formation was reached. The synthesized surfactants demonstrated varying CMC and cloud point values, indicating differences in molecular structure and hydrophilic–lipophilic balance (HLB). Among the samples, PAVMS exhibited the highest surface activity, while PAVKM showed favorable characteristics for applications at lower temperatures due to its low CMC and cloud point. The findings highlight the importance of molecular structure in surfactant performance and provide a basis for selecting efficient and environmentally sustainable surfactants for industrial applications.

Key words: Critical micelle concentration (CMC); surfactants; surface tension; micellization; cloud point; amphiphilic compounds; hydrophilic–lipophilic balance (HLB); Du Noüy ring method; colloidal chemistry; interfacial properties.

Introduction

Surfactants are a unique class of chemical compounds characterized by their amphiphilic nature, meaning that each molecule contains both a hydrophobic (nonpolar) fragment and a hydrophilic (polar) fragment. This dual structural feature governs their behavior in aqueous and multiphase

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systems, enabling them to preferentially adsorb at interfaces such as air–water, oil–water, and solid–liquid boundaries. As a result, surfactants play a crucial role in modifying interfacial properties, particularly by reducing surface and interfacial tension, which facilitates processes such as wetting, dispersion, emulsification, and solubilization.

One of the most significant phenomena associated with surfactants in solution is micellization, a self-assembly process driven by the tendency of the system to minimize its free energy. At low concentrations, surfactant molecules are dispersed as individual monomers throughout the solution. However, as their concentration increases, intermolecular interactions become increasingly favorable for aggregation. Beyond a certain threshold, surfactant molecules spontaneously organize into micellar structures. In these aggregates, the hydrophobic "tails" are sequestered in the interior of the micelle, shielded from contact with water, while the hydrophilic "heads" remain oriented outward toward the aqueous phase. This arrangement not only stabilizes the structure but also significantly alters the physicochemical properties of the solution.

A fundamental parameter describing this transition is the critical micelle concentration (CMC). The CMC is defined as the concentration of surfactant in solution at which micelles begin to form in appreciable numbers. Below the CMC, the solution is dominated by monomeric surfactant molecules, and changes in concentration primarily affect interfacial adsorption. Above the CMC, additional surfactant molecules preferentially form micelles rather than accumulating at the interface, resulting in a marked change in the behavior of the system. This transition is typically accompanied by abrupt changes in various physicochemical properties, including surface tension, electrical conductivity, osmotic pressure, and light scattering.

The value of the CMC is strongly influenced by the molecular structure of the surfactant. Key structural factors include the length and branching of the hydrophobic alkyl chain, the nature and size of the hydrophilic head group, and the overall hydrophilic–lipophilic balance (HLB). Generally, an increase in hydrophobic chain length leads to a decrease in the CMC, as stronger hydrophobic interactions promote aggregation. Conversely, highly hydrophilic head groups may increase the CMC due to enhanced solvation in water. The balance between these opposing tendencies determines not only the onset of micellization but also the size, shape, and stability of the resulting micelles.

The determination of the CMC is therefore of central importance in both fundamental and applied research involving surfactants. From a scientific perspective, it provides insight into intermolecular interactions, self-assembly mechanisms, and solution thermodynamics. From a practical standpoint, the CMC defines the minimum concentration required for surfactants to exhibit their functional properties effectively. This has direct implications for formulation design, cost efficiency, and environmental impact, as excessive use of surfactants can lead to unnecessary expense and ecological concerns.

Surfactants are widely utilized across numerous industrial sectors, including petroleum engineering (for enhanced oil recovery and drilling fluids), cosmetics (as cleansing and emulsifying agents), pharmaceuticals (for drug delivery and solubilization), and food technology (for stabilization of emulsions and foams). In each of these applications, the performance of surfactants is closely linked to their ability to form micelles and modify interfacial properties. Consequently, accurate determination and control of the CMC are essential for optimizing product performance and ensuring process efficiency.

In summary, the study of micellization and the determination of the critical micelle concentration provide a fundamental basis for understanding the behavior of surfactants in solution. These concepts bridge molecular-level interactions and macroscopic properties, enabling the rational design and application of surfactant systems in a wide range of scientific and technological fields.

Experimental Methods

Determination of Surface Tension

One of the most common methods for determining the CMC is based on measuring the surface tension of surfactant solutions. In this work, the Du Noüy ring method was employed. The experimental setup consists of a platinum ring connected to a sensitive dynamometer and a vessel containing the test liquid.

The procedure involves measuring the force required to detach the ring from the liquid surface. Initially, measurements are carried out using distilled water as a reference. Subsequently, surfactant solutions of varying concentrations are analyzed under identical conditions. The measured detachment force is directly related to the surface tension of the liquid.

As the concentration of surfactant increases, the surface tension decreases due to adsorption of surfactant molecules at the air–water interface. At concentrations approaching the CMC, this decrease becomes less pronounced, and eventually, the surface tension reaches a nearly constant value. This transition indicates the onset of micelle formation in the bulk solution.

Determination of the Cloud Point

Another important characteristic of surfactant solutions is the cloud point (CP), which reflects their thermal behavior and solubility. The cloud point is defined as the temperature at which a clear surfactant solution becomes turbid due to phase separation.

To determine the cloud point, a sample of surfactant (0.5 g, with an accuracy of ±0.01 g) is placed in a conical flask and dissolved in 100 mL of distilled water with thorough mixing. From this solution, 15 mL is transferred into a test tube equipped with a thermometer. The test tube is then placed in a water bath and gradually heated with continuous stirring.

During heating, the appearance of cloudy ring-shaped regions indicates the onset of turbidity. The corresponding temperature is recorded as the cloud point. To ensure accuracy, the temperature should not exceed the expected value by more than 10 °C. Afterward, the solution is slowly cooled while stirring, and the temperature at which clarity is restored is recorded. This procedure is repeated for diluted solutions to obtain consistent and reliable results.

Determination of CMC from Surface Tension Data

The determination of the CMC is based on detecting abrupt changes in the physicochemical properties of surfactant solutions as a function of concentration. These changes arise because micelle formation represents the emergence of a new phase in the system.

In this study, a series of surfactant solutions with different concentrations was prepared by diluting a stock solution (200 mg/dm^3). Ten solutions with a mass of 50 g each were obtained, covering a concentration range that includes the expected CMC region.

Surface tension measurements were conducted at controlled temperatures:

- (20 ± 1) °C for nonionic surfactants
- (20 ± 1) °C or 5 °C above the Krafft point for anionic surfactants

Each solution was placed in a beaker covered with a watch glass to minimize evaporation, and surface tension was measured using a tensiometer.

The experimental data were plotted as surface tension versus the logarithm of surfactant concentration. Typically, the resulting curve consists of two distinct regions:

1. A steep linear decrease at low concentrations (monomer region)
2. A plateau at higher concentrations (micellar region)

The intersection of these two regions corresponds to the CMC.

To improve accuracy, six additional solutions were prepared within the identified concentration range. Their surface tension was monitored over a period of three hours, with measurements taken every 15 minutes. The refined dataset allowed for more precise determination of the CMC.

Results and Discussion

The surface tension curves for synthesized surfactants in the concentration range of 0–2 wt% demonstrate a clear trend of decreasing surface tension with increasing concentration. This behavior is attributed to the progressive adsorption of surfactant molecules at the air–water interface, which reduces intermolecular forces at the surface.

At higher concentrations, the surface tension reaches a constant value, indicating saturation of the interface and the onset of micelle formation in the bulk solution. The equilibrium surface tension values were found to be:

- 38.1 mN/m for PAVMS
- 43.2 mN/m for PAVHS
- 44.1 mN/m for PAVKZh
- 43.1 mN/m for PAVVZh
- 48.2 mN/m for PAVKM

Among these samples, PAVKM exhibits the highest surface tension, indicating the lowest efficiency in reducing interfacial energy. In contrast, PAVMS demonstrates the greatest effectiveness.

The experimental results confirm that the length of the hydrophobic alkyl chain plays a crucial role in determining surfactant performance. Generally, increasing the chain length enhances hydrophobic interactions, leading to more efficient adsorption at the interface and lower surface tension values. However, this trend is also influenced by the hydrophilic–lipophilic balance (HLB). Surfactants with higher HLB values tend to exhibit greater ability to reduce surface tension due to their stronger affinity for the aqueous phase.

CMC and Cloud Point Analysis

The experimentally determined CMC and cloud point values for the synthesized surfactants are summarized below:

Sample	PAVMS	PAVHS	PAVKZh	PAVVZh	PAVKM
CMC_1 (mg/L)	18.7	15.6	17.1	23.5	15.3
CMC_2 (mg/L)	36.7	31.2	36.9	73.2	28.8
Cloud Point (°C)	58–60	57–59	69–71	72–74	54–56

The results reveal significant variation in micelle formation behavior among the samples. The lowest CMC_1 values are observed for PAVKM and PAVHS, indicating that these surfactants readily form micelles even at low concentrations. In contrast, PAVVZh exhibits the highest CMC values, suggesting that higher concentrations are required for micelle formation.

The cloud point data further support these findings. PAVVZh shows the highest cloud point (72–74 °C), indicating that higher temperatures are needed to induce phase separation and micelle formation. This behavior may be attributed to stronger intermolecular interactions or specific structural features of the surfactant.

Conversely, PAVKM has the lowest cloud point (54–56 °C) and relatively low CMC values, indicating good solubility and ease of micelle formation at lower temperatures. This makes it particularly suitable for applications in systems operating under mild thermal conditions.

Practical Implications

The determination of CMC and cloud point provides valuable insight into the performance of surfactants in real-world applications. Surfactants with low CMC values are more efficient, as they require smaller amounts to achieve the desired effect, which is economically advantageous. Additionally, lower cloud points are beneficial for processes conducted at reduced temperatures.

In industrial applications such as enhanced oil recovery, detergency, and cosmetic formulations, selecting surfactants with optimal CMC and cloud point values is essential for achieving high efficiency, stability, and cost-effectiveness. Furthermore, understanding these parameters contributes to the development of environmentally friendly formulations by minimizing the required surfactant concentration.

Conclusion

The study of critical micelle concentration and cloud point is fundamental to the characterization and application of surfactants. The results demonstrate that molecular structure, particularly hydrophobic chain length and hydrophilic–lipophilic balance, plays a decisive role in determining surfactant behavior.

The synthesized surfactants exhibit a wide range of CMC and cloud point values, reflecting their diverse physicochemical properties. Among the studied samples, PAVMS shows the highest surface activity, while PAVKM demonstrates favorable properties for low-temperature applications.

Overall, the findings provide a comprehensive basis for selecting and optimizing surfactants for specific industrial applications, contributing to improved performance and sustainability.

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