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Analytical Tools for Characterization of the Micellar Surfactant System

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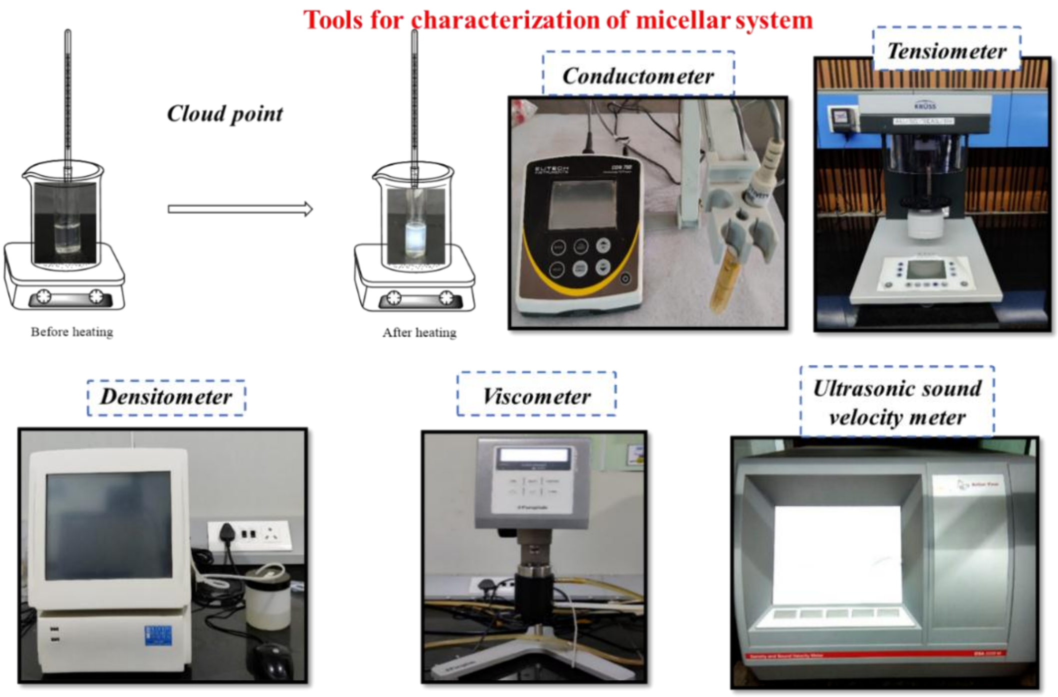
Abstract

A unique class of chemical compounds known as surface active agents (surfactants) possesses hydrophilicity and hydrophobicity. Surfactants are used in every facet of refined activity and have many uses in science and industry. With a wide range of applications in the fields of engineering, chemistry, physics, biology, medicine, and pollution control, surfactant science is a field that is quickly growing. Despite this, surfactants today have enormous potential, importance, and cost-effectiveness. In the present work, a study on the determination of critical micellar concentration (CMC) and characterization of micellar surfactant system by using the local laboratory methods such as cloud point (CP), conductivity, tensiometer, densitometer, viscometry, and ultrasonic sound velocity, is reported.

Keywords: Surfactant, CMC, micelles, cloud point, conductivity

1. Introduction

Surfactants are amphiphilic molecules characterized by hydrophilic and hydrophobic regions that associate dynamically and spontaneously in polar or nonpolar solvents at and above a specific concentration called critical micellar concentration (CMC) [1]. Above this concentration, the surfactant molecules form large molecular aggregates with colloidal dimensions defined as micelles. At concentrations below CMC, the surfactant is maisnly in the form of monomers. CMC depends on surfactant structure (length of the hydrocarbon chain) as well as on the type of solvent, temperature, pH, etc. [2]. Surfactants are used in every facet of refined activity and have many uses in science and industry. With a wide range of applications in the fields of engineering, chemistry, physics, biology, medicine, and pollution control, surfactant science is a field that is quickly growing [3]. Despite this, surfactants today have enormous potential, importance, and cost-effectiveness.



The changes in nature/micellar structure have been characterized using techniques/methods/tools such as CP, conductivity, tensiometer, densitometer, viscometry, and ultrasonic sound velocity.

2. Characterization techniques

a. Cloud point (CP)

CP is observed in nonionic surfactants. Hydrogen bonding causes these surfactants to dissolve in water. The hydrogen bonds are broken down when the temperature rises. Solubility is reduced as a result of this. The hydrophobic part's CP falls as the chain length increases. The sample solution in thin glass vials or 20 ml glass test tubes was heated in a beaker containing distilled water to measure CP. The solution contained in a narrow glass vial that has been immersed in a stirring heating bath. The solution was continually agitated with a magnetic stirrer as the temperature was raised gradually at a rate of 1°C per minute [4-6]. When the solution turned hazy, the temperature at CP was recorded. The image below (Figure 1) depicts a schematic illustration of CP.

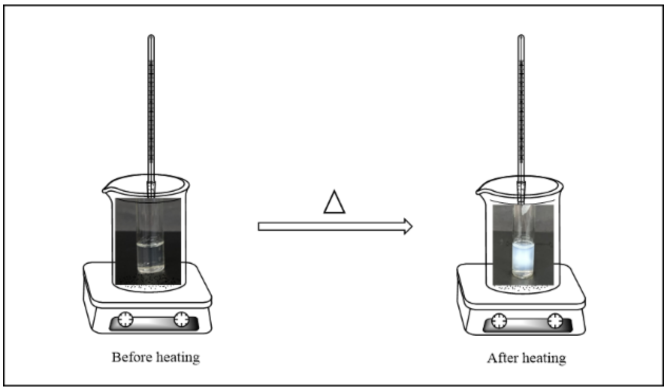


Figure 1: Schematic illustration of cloud point

b. Conductivity

The conductivity meter measured the conductance with a cell constant close to 1.0. The equipment was calibrated using a solution of known concentration of KCl. The glass cell with two platinum electrodes was immersed in a corning glass tube carrying the sample solution, which was immersed in a thermostated water bath controlled by an electronic circuit [7,8]. Conductivity is one of the electrochemical estimations in which the free electrons of ions are estimated. It estimates how much current goes through the specific region. With the help of a conductometer, the specific conductivity of the solution can also be determined. The surfactant systems' CMC values are determined from the intersection of two concentration lines on a conductivity plot while measuring electrical conductivity. The figure below depicts the instrumental Figure 2.

Figure 2: Conductometer instrument

c. Tensiometer

A tensiometer is a device used to gauge a liquid's surface tension. Surface tension can be measured using a variety of techniques.

Drop Weight Method is a traditional method. A series of droplets of the liquid are formed at the tip of a tube in this approach. Allowing these drips to fall into a container. The weight of a single drop is computed using the weight of the liquid collected and the number of drops created. The drop weight w is related to surface tension by the modified Tate’s law.

$$\mathbf{W = 2\pi r \Upsilon f}$$

where r is the radius of the tip, and f is the correction factor.

Table 1: Measurements of surface tension

Method	Suitability for pure liquids	Suitability for solutions
Drop weight	Very suitable when atmospheric contamination suspected	Poor when ageing effect suspected
The Du Nouy ring	Satisfactory	Not suitable
The Wilhelmy plate	Very quick and easy to operate; susceptible to atmospheric contamination	Accuracy good; suitable when ageing occurs
Capillary height	Very satisfactory	Not suitable if the contact angle different from zero
Maximum bubble pressure	Somewhat challenging to operate successfully	Poor when ageing effect suspected
Sessile drop profile	Very satisfactory	Very suitable when ageing occurs
Pendant drop profile	Very satisfactory but limited experimentally	Very suitable when ageing occurs
Spinning drop	Suitable for very low interfacial tension	Very convenient to study the effect of ageing

The tensiometer principle is “The property of the surface of a liquid that allows it to resist an external force due to the cohesive nature of its molecules.” The Wilhelmy plate (Platinum plate) method detects the surfaced tension using a force tensiometer [9,10]. A platinum plate that is immersed in a liquid is used in tensiometers. The Wilhelmy plate method governs the liquid's surface tension by carefully measuring the force required to separate the ring from the liquid. Ludwig Wilhelmy, a German chemist, is honoured with the Wilhelmy plate method's name [11]. A thin plate is employed to determine the equilibrium surface or interfacial tension between two fluids, such as air and liquid. This technique involves positioning the plate at right angles to the interface and measuring the resulting force. In each experiment, 30 ml of the solution is used in a beaker of 50 ml, and a platinum plate is placed on top of the solution beaker. The instrument determines Surface tension measurements automatically while maintaining a gap of around 3 mm between the immersed plate and the liquid's surface. The tensiometer is calibrated using deionized water with surface tension values of 72 ±0.1 mN/m at 27 °C. Pure water is used to clean the platinum plate, which is then placed on a burner. The test plate was attached to the microbalance, and the liquid reservoir on the mobile platform could rise or fall to submerge or remove the solid plate [12].

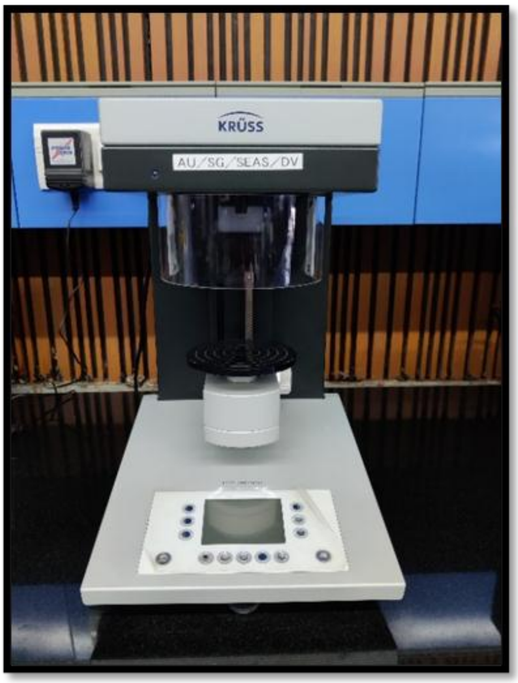


Figure 3: Tnsiometer instrument

d. Densitometer

The density was measured using an analytical digital vibrating glass U-tube densitometer. The device used to test the mixture's density has an accuracy of 0.05 kg/m3. Dry air was delivered using a suction tube packed with silica balls, and doubledistilled water was pumped into the density meter using a syringe. The atmosphere was dried by heating silica balls regularly. Deionized and twice-distilled water was used for standard measurements at each temperature. After calibration, distilled water was used to clean the U-tube densitometer, and acetone and air were used to dry it. The study's density data had an average of at least five times what was previously stated. The tube was cleaned with water and dried with acetone before each measurement. During measurements, the air pump was always turned off to prevent vibrational anomalies [13,14].

Figure 4: Densitometer

e. Viscometer

Viscosity is frequently referred to as a fluid's thickness. The informal thickness notion defines viscosity as the deformation resistance evaluated at a particular rate. Newton seconds per square meter, or Ns / m^2, pascal seconds (Pas), is the unit of viscosity according to international unit systems (SI). The viscosity of a fluid gauges its internal friction or flow resistance. There are two ways to determine how viscous the given solution is: the classical and instrumental methods. The Ostwald and Ubbelhode are the two types of viscometers used in the traditional approach. A viable way to characterize a polymer is to measure its viscosity in solution at low concentrations. This can be accomplished by using a "Ubbelhode viscometer" to measure the time, t, that a polymer solution flows through a capillary and comparing it to the time, t_0, that the fluid flows through the capillary. The viscosity is equal to the efflux times the mass of the liquid. Since diluted liquids are about the same density as pure solvents, we can figure out how viscous they are, η_{rel}, as $\eta_{rel} = t/t_0$.

Fungilab Viscolead Advance Viscometer is a specialized instrument used in viscosity measurement techniques. It measures kinematic viscosity as well as dynamitic viscosity.

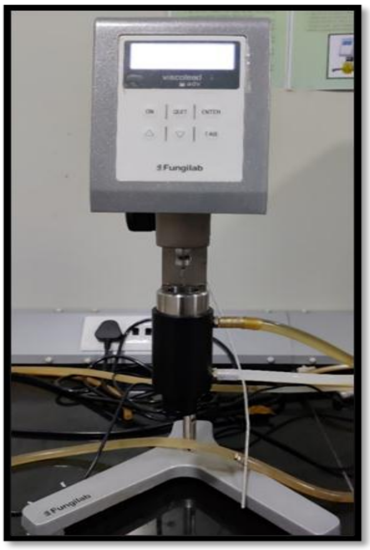


Figure 5: Fungilab's viscometer

f. Ultrasonic sound velocity

There has been considerable success with ultrasound in various research fields, including biology, geology, biochemistry, polymer industry, geography, medicine, engineering, etc. [15]. An analysis of molecular interactions has been carried out using ultrasonic sound velocity measurements between binary and ternary liquid mixtures using ultrasonic sound velocity measurements. There have been many studies to utilize this technique for a variety of solutions, including polymer, other electrolytes, and non-electrolytes.

Figure 1B.7: Ultrasonic sound velocity

3. Conclusions

This article represents a summary of a few experimental techniques/methods used for the characterization of micellar surfactant systems. These methods are easy to understand and available in most research institutes to measure various parameters involved in these techniques that help optimize the micellar system.

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