

“Analysing the Energetics of Phase Separation in Non-Ionic Surfactant Brij-30: Investigating the Influence of Head Group Gathering”.

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ABSTRACT:

The main motto of this paper was to study how the hydrophilic surfactant head group gathering affects the phase behavior of a 22% alcoholic solution of a non-ionic surfactant, Polyoxyethylene (4) lauryl ether (Brij-30) and hydroxyl amine mixtures at different concentrations. Our conclusions show that the hydrophilic part of the surfactant suppresses the system's propensity to undergo phase separation, an increased sensitivity due to the presence of hydroxyl amines. The CP decreases with an increasing concentration of mono, di, and Triethanolamine in a 22% alcoholic solution of Brij 30 surfactant. CP increases with concentration in methyl ethanolamine while decreases in diethanolamine with concentration the same trend is observed in the case of methyl diethanolamine and triethanolamine. Generally, ethanolic groups exhibit an increase in Cloud Point (CP) with increased alkyl branching. The amines in ethanolamine show variation in CP by integrating into the head region with proton-rich amine groups at the micelle interface. By utilizing the traditional thermodynamic model for phase separation, we assessed the various corresponding energy parameters.

Amongst all instances, we found that all the parameters fluctuated based on non-ionic surfactant type, its micellar concentration, and the involvement of hydroxyl amine in the aqueous medium. All the experimental findings are consistent because phase separation occurs due to decreased hydration of the surfactant head group induced by elevated temperature.

KEYWORDS: *Hydrophilic head group, Hydroxyl amine, Cloud point, Thermodynamic Model*

INTRODUCTION:

The word surfactants meaning surface-active agents, consists of a diverse group of compounds famous, for their multifaceted attributes, which have gained substantial usefulness across a wide array of industries [1-3].

Their unique molecular formulation comprises hydrophilic (attracted to water) and hydrophobic (repelled by water) segments promoting interactions at the interfaces of various phases, like those between oil and water. The ability to diminish interfacial tension and foster the formation of long-lasting colloidal structures has cemented the essential role of surfactants across a diverse array of applications and production methods.

Surfactants hold significant value across a variety of sectors, encompassing industries, household and personal care products, pharmaceuticals, and agriculture zones. Day today in household cleaning solutions, surfactants play a pivotal role in removing dirt and oils from surfaces, [4] while in personal hygiene items such as shampoos and soaps, they assist in breaking down oils and enhancing foaming properties. Additionally, surfactants play an important role in pharmaceutical applications where they act as an essential component in drug formulations to improve the solubility, stability, and absorption rate of medications [5].

Additionally, beyond their role as emulsifiers and detergents, surfactants are widely used in various industrial operations, like enhanced oil recovery, where they facilitate the extraction of trapped oil from reservoirs by reducing interfacial tension. Moreover, in the agriculture field, surfactants enhance the efficiency of agrochemicals by improving their spreading and wetting abilities on plant surfaces [6].

The versatility of surfactants arises from their classification into four primary groups according to the ionic charge present on them: anionic, cationic, non-ionic, and amphoteric (zwitterion). Each class of surfactants has unique characteristics and functionalities, which make them useful for various specific applications enabling customized applications based on considerations like compatibility, stability, and performance criteria. Despite their extensive utilization and importance in various fields, the understanding of surfactants remains dynamic, spurred by continuous research endeavors directed toward comprehending their molecular relationships, optimizing their efficiency, and discovering new applications. Non-ionic surfactants are synthesized through a reaction between a water-insoluble compound, like alkyl phenol, and ethylene oxide. This reaction produces a molecule with an oil-soluble segment linked to a water-soluble polyoxyethylene chain.

Non-ionic surfactants are considered to be one of the most important classes of surfactants due to their remarkable solubility capacity in both organic and aqueous phases due to hydrogen bonding of solvent and ether oxygen atom within the chain, minimal toxicity,

chemical coexistence [7-8]. Non-ionic surfactants are often studied and utilized for their versatile properties in fields ranging from pharmaceuticals to cosmetics, agriculture, and material science. Besides all cloud point is one of the most important properties of non-ionic surfactants [9] this research paper aims to enhance the collective understanding by exploring a particular facet of surfactant behaviour the alteration of cloud point with additive incorporation. The cloud point marks the temperature where a solution turns cloudy due to surfactant micelles' phase separation, As temperature changes, hydrogen bonding dynamics are altered. Consequently, each non-ionic surfactant molecule reaches a temperature at which the hydrophilic portion cannot adequately hydrate to solubilize the remaining hydrocarbon segment. This critical temperature is termed the 'Cloud Point' [10-12] which serves as a critical factor shaping the stability and effectiveness of surfactant setups. This CP parameter significantly alters the thermodynamic and colloidal characteristics of the system. Notably, the introduction of additives is known to exert a substantial impact on the cloud point, thereby reshaping the system's properties.

Understanding the factors governing cloud point variation upon additive introduction is paramount for optimizing formulations in numerous industrial applications [13]. Gaining insight into the determinants of cloud point variation resulting from additive incorporation is crucial for fine-tuning formulations across various industrial sectors [14]. Through systematic experimental study and examination, our motto is to decipher the fundamental mechanisms governing clouding behaviour and offer valuable insights for enhancing the formulation and efficacy of surfactant systems across diverse industrial domains.

The CPE method is vastly used in analytical chemistry for the extraction and preconcentrating of trace analytes from complex matrices like environmental samples, biological fluids, and food samples [15-16]. After cloud point extraction, the surfactant-rich phase, containing the extracted analytes, is separated from the aqueous phase. Subsequently, the analytes can be recovered from the surfactant phase through various techniques such as solvent extraction, centrifugation, or filtration for subsequent analysis [17-19].

The scientific community continues to engage in ongoing debate regarding the underlying mechanism responsible for the formation of the cloud point (CP). The primary factor for the acceptance of the clouding phenomenon is the dehydration from the hydrophilic head gathering of the surfactant species [20-21].

This results in the change of dielectric constant of the solution ultimately affecting the surfactant solvent interaction, consequently altering the CMC (Critical Micelle Concentration). This loss of water from head gathering alters the dielectric constant of the solution, which also affects the interactions between the surfactant and the solvent molecules consequently, changes in critical micelle concentration (CMC) [22-23]. The cloud point (CP) is varied by changing the surfactant concentration and can be regulated through the addition of both organic and

inorganic materials. Several studies have explained that the addition of foreign materials can alter the CP [24-27].

Hydroxyl amines, are compounds characterized by the presence of both hydroxyl (-OH) and amino (-NH₂) functional groups in a single molecule, and can indeed serve as effective co-surfactants in numerous applications. Hydroxyl amines in combination with surfactants can enhance the performance of surfactant systems by improving their stability, solubility, and emulsification properties [28]. Additionally, hydroxyl amines play a vital role in the reduction of interfacial tension and micelle formation, which are crucial for emulsification and dispersion processes. Their unique chemical properties make them valuable additives in industrial formulations.

The Non-ionic Brij surfactants are derived from polyoxyethylene ethers of fatty alcohols, mainly lauryl alcohol. These surfactants are widely used in various industries, including pharmaceuticals, cosmetics, and food processing, due to their excellent emulsifying, dispersing, and solubilizing properties. There are various types of Brij surfactants for example, Brij 30, Brij 35, Brij 52, Brij 58, Brij 72, and Brij 78 classified based on their unique combinations of polyoxyethylene chain length, alkyl chain length, and HLB values.

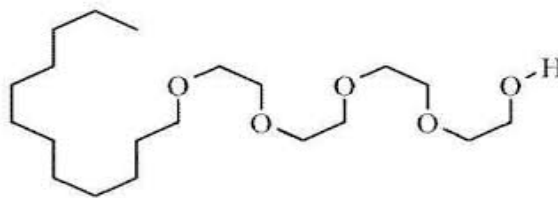
As a non-ionic surfactant, Brij 30 exhibits low toxicity and minimal interference with biological or chemical systems, making it suitable for a wide range of applications, including cloud point determination in various sample matrices. Brij 30 surfactant has maintained good stability over a wide range of temperatures, ensuring consistent performance during cloud point extraction experiments conducted at different temperatures. Brij 30 is also compatible with various organic solvents and aqueous solutions, providing flexibility in the choice of extraction solvents and sample matrices.

Here, we choose a 10% solution of Brij-30 in 22% alcohol for our cloud point measurements due to its well-established suitability. This concentration maintains well balance between sensitivity and practicality, which provides an optimal environment for observing the CP phenomenon. In addition to this Brij-30 surfactant readily forms micelles at a temperature conducive to experimental measurements, enhancing the reliability and efficiency of our observations.

2. EXPERIMENTAL

2.1 Materials

The Brij-30 [Polyethylene Glycol Dodecyl Ether] a non-ionic surfactant of 99% purity, was procured from Sisko Research Chemical Laboratory. Analytical grade hydroxyl amines, such as 2-methylaminoethanol, diethanolamine, dimethylamine, triethanolamine, dimethylaminoethanol and N-methyl diethanolamine were purchased from Sigma Aldrich. All solutions used in the experimental procedures were meticulously prepared using double-distilled water. Before experimentation, all the glassware's cleaning process involves a 50% ethanol solution followed by subsequent drying. The molecular framework of Brij-30 is depicted below.



Molecular Weight -230.39

Fig.1 Structural Framework of Brij-30

3. METHEDOLOGY

3.1 Cloud point determination:

The cloud point outlined technique involves, the determination of the cloud point (CP) through direct visual observation of temperature changes, when a micellar surfactant solution changes from clear to cloudy, as described by Kabir-ud din [29-30]. The experimental setup for the determination of clouding, contained the solution in a hard glass tube, to minimize solvent loss by evaporation sealing the open end with a cork. Temperature control is regulated manually, and the observations are made during both heating and cooling cycles to note the formation and clearance of turbidity in the surfactant solution.

The CP is recorded as the temperature at which clouding first occurs, which is typically observed initially at the boundary where the liquid meets the glass tube before progressing to the air-solvent interface. For getting more accuracy in the CP determination the average temperature at which clouds appear and disappear is based on three recorded measurements for each. The clear visibility of the CP is observed by placing a lamp near to the tube.

3.2 Determination of foam ability and foam stability:

The micellar gathering is influenced by the cloud point (CP), was effectively explored through the assessment of foam ability and foam stability, as elucidated in the literature [31]. Foam ability and foam stability give us detailed information on surface tension and viscosity characteristics, which are evaluated by following the methodology outlined by D.O. Shah [32]. 20 cm³ of the surfactant-additive mixture was poured into a 100 cm³ calibrated measuring glass cylinder. The solution underwent ten uniform strokes at a frequency of one stroke per second. Amount of foam generated represented the system's foam ability, while the time taken for the foam volume to halve indicated foam stability. The experiment was repeated three times to ensure accuracy in the obtained data.

4.0 RESULTS AND DISCUSSIONS

4.1 Effect of surfactant concentration:

A versatile non-ionic surfactant Brij-30, demonstrates diverse applications across various fields. Its important points were experimentally determined within aqueous ethanolic solutions across varying concentrations, as detailed in our previous research article [33]. The cloud point (CP) of Brij-30 in an aqueous ethanolic solution displays concentration-dependent variation. Specifically, within a 22% aqueous ethanolic solution, the CP of Brij-30 decreases with increasing surfactant concentration. Elevating the concentration of the pure Brij-30 surfactant prompts a rise in micellar molecular weight, leading to an increased aggregation number within the micelle and heightened hydrophobicity. Upon heating the solutions to a specific temperature, the polyoxyethylene chain undergoes dehydration, inducing a notable reduction in surfactant solubility and consequently lowering the cloud point (CP).

4.2 Cloud Point of Brij-30 & Secondary Hydroxylamine Systems:

The increase in aqueous solubility of poorly soluble or insoluble substances due to the presence of micelles is known as solubilisation. A secondary ethanolamine with dual hydrophilic and hydrophobic features due to its two groups attached to the nitrogen atom introduces a multifaceted interaction within the solution. Its addition results in potential interactions with alcohol and water molecules alongside other solution components. Consequently, secondary ethanolamine's presence can induce modifications in the micellar characteristics, influencing both the size and stability of micelles formed by the surfactant. These alterations in micellar properties and interactions can consequently instigate a shift in the cloud point temperature.

The physicochemical behaviors of mixed micelle systems are significantly influenced by the presence of additives, which exhibit varying behaviors within surfactant systems. In an aqueous alcoholic solution, Brij-30 self-aggregates to reduce solvent interaction, forming different aggregates based on physicochemical properties. Solvophobic interactions play a crucial role in determining clouding interactions. Consequently, numerous studies have investigated the impact of altering solvent properties by incorporating various additives [34-37].

The impact of various types of secondary amines as additives in a 22% aqueous-ethanolic Brij-30 surfactant solution on the cloud point (CP) was investigated. Our results indicate that alkyl groups increase the cloud point (CP) of Brij-30 surfactant, whereas ethylol groups reduce the CP when secondary hydroxyl amines are used as additives. In general, as the number of ethylol groups in a molecule increases, both its hydrophilicity and solubility in water also increase. Attaching one to three ethylol groups to a nitrogen atom results in a reduction of the Critical Micelle Concentration (CMC). A greater number of ethylol (hydrophilic) groups enhance the molecule's solubility in water, which subsequently lowers both the surface tension and the cloud point (CP).

Solubilization involves enhancing the water solubility of substances that are only slightly soluble or insoluble by using micelles to improve their dispersion in aqueous solutions.

Additives alter micellar organization by modifying the structure of the solvent and how surfactants accumulate within it. The influence of water-soluble compounds is often elucidated by their effect on the water structure. Additives that disrupt the water structure typically raise the cloud point (CP), while those that promote water structure formation generally lower the cloud point [38-42].

The compound with minimal water solubility can penetrate inside the micellar core. This phenomenon demonstrates how the site of solubilisation within the micellar structure affects its behaviour [43]. Fig.1 shows the effect of secondary hydroxylamine's on the CP of the alcoholic Brij-30 surfactant. The results indicate that the cloud point (CP) of a 10% Brij 30 alcoholic solution increases steadily with the concentration of secondary amines in 2-dimethylethanolamine, and dimethylamine, while CP decreases with the concentration in the case of diethanolamine, as shown in Table 1. The increase in CP is attributed to the presence of more alkyl groups. Conversely, an increase in the number of ethanol groups, as seen with ethanolamine, decreases the CP of Brij-30. Typically, as the number of ethylol groups in a molecule increases, its hydrophilicity and water solubility improve. This increase in ethylol groups attached to the nitrogen atom results in a decrease in the critical micelle concentration (CMC). Greater numbers of these hydrophilic ethylol groups enhance water solubility, leading to a reduction in surface tension and critical point (CP).

4.3 Cloud Point of Brij-30 & Tertiary Hydroxylamine Systems:

As like n-amines, ethanolamine's addition influences micelle formation in several ways. First, hydrogen bonds form between the nitrogen atom (with its lone pair of electrons) and water, altering the water structure around the monomer. Second, the hydrophobic alkyl segment of ethanolamine integrates into the micelles, displacing some water molecules at the surface. This displacement reduces the distance between head groups and lowers the charge density of the micellar group. Both effects facilitate the micellization (cloud point formation) of alcoholic Brij-30 in a water-ethanolamine mixture.

The alcoholic Brij-30 surfactant system shows significant physicochemical changes when mixed with a water-tertiary ethanolamine solution due to surfactant-solvent interactions, which improve micellar properties compared to a purely aqueous system. Figure 2 illustrates the impact of tertiary ethanolamine on the cloud point (CP) of alcoholic Brij-30. The data indicate that the CP of 10% alcoholic Brij-30 rises with increasing concentrations of N-methyl diethanolamine and N, N-dimethyl ethanolamine. This phenomenon can be attributed to the enhancement of the water structure through intermolecular hydrogen bonding, facilitated by the amino nitrogen atom, which intensifies the hydrophobic effect and promotes micellization. As a result, an increase in ethanolamine concentration naturally leads to a decrease in the critical micelle concentration (CMC), thereby raising the cloud point (CP). Conversely, the CP decreases with concentration in the case of triethanolamine, as shown in Table 2. An increase in the number of alkyl groups raises the CP, whereas an increase in the number of ethylol groups reduces the CP of alcoholic Brij-30 in the presence of tertiary ethanolamine.

CP of 22% alcoholic solution of Brij-30 with secondary ethanolamine's as additives			
Conc. of amines (mol kg ⁻¹) x 10 ²	Cloud Point (°C)		
	Diethanolamine	Dimethylamine	2-Methylethanolamine
0.1	54.8	52.4	57.2
0.2	52.6	53.8	59.8
0.4	52.2	54.2	61.8
0.6	49.8	55.8	64.2
0.8	45.8	56.8	66.8
1.0	41.2	58.2	68.8

Table 1: Cloud point values of a 22% alcoholic Brij-30 solution in the presence of secondary ethanolamine at varying concentrations.

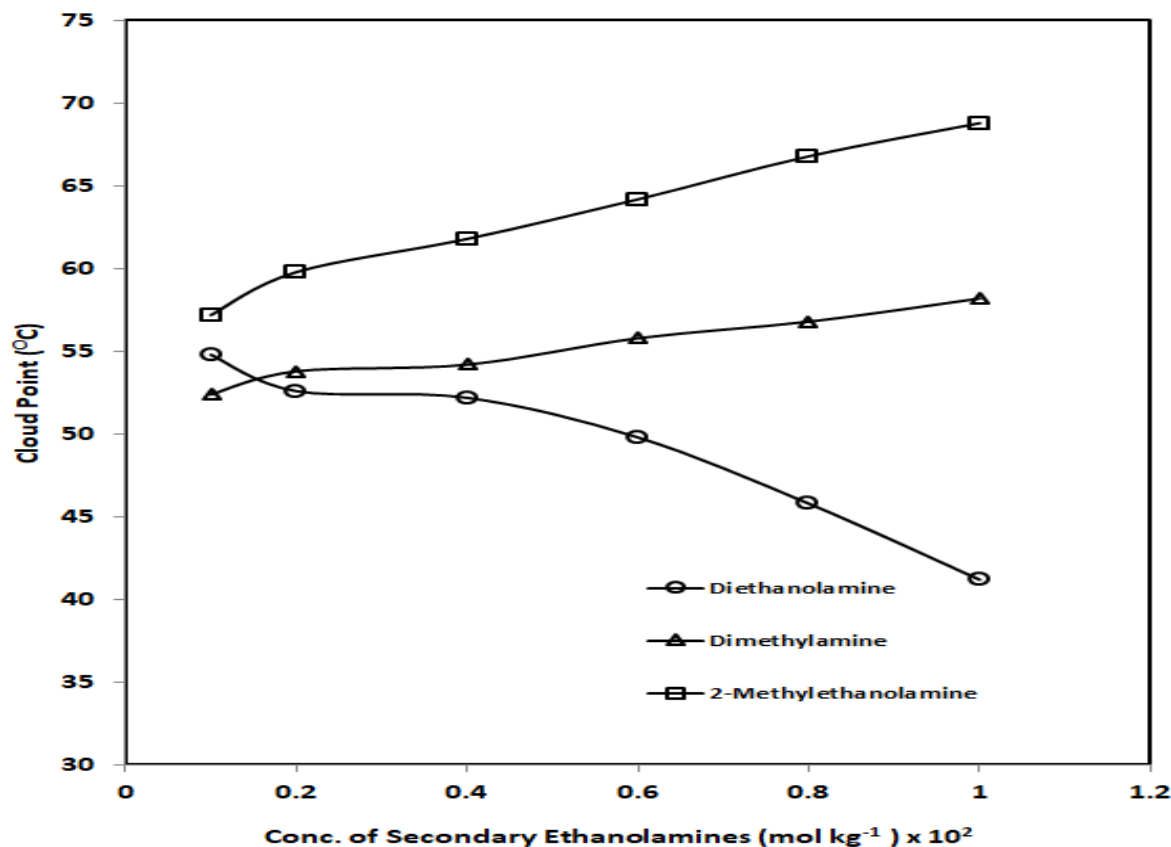


Figure 1: Variation in the cloud point of Brij-30 surfactant in a 22% aqueous ethanolic medium with varying concentrations of secondary hydroxylamine

Table 2: Cloud point values of a 22% alcoholic Brij-30 solution in the presence of tertiary ethanolamine at varying concentrations.

CP of 22% alcoholic solution of Brij-30 with tertiary ethanolamine's as additives			
Conc. of amines (mol kg ⁻¹) × 10 ²	Cloud Point (°C)		
	Triethanolamine	N-Methyl diethanolamine	N, N-dimethyl ethanolamine
0.1	62.4	64.6	54.2
0.2	61.2	62.8	55.8
0.4	60.4	61.2	63.4
0.6	59.4	60.4	63.8
0.8	58.2	59.6	67.8
1.0	56.8	57.2	68.4

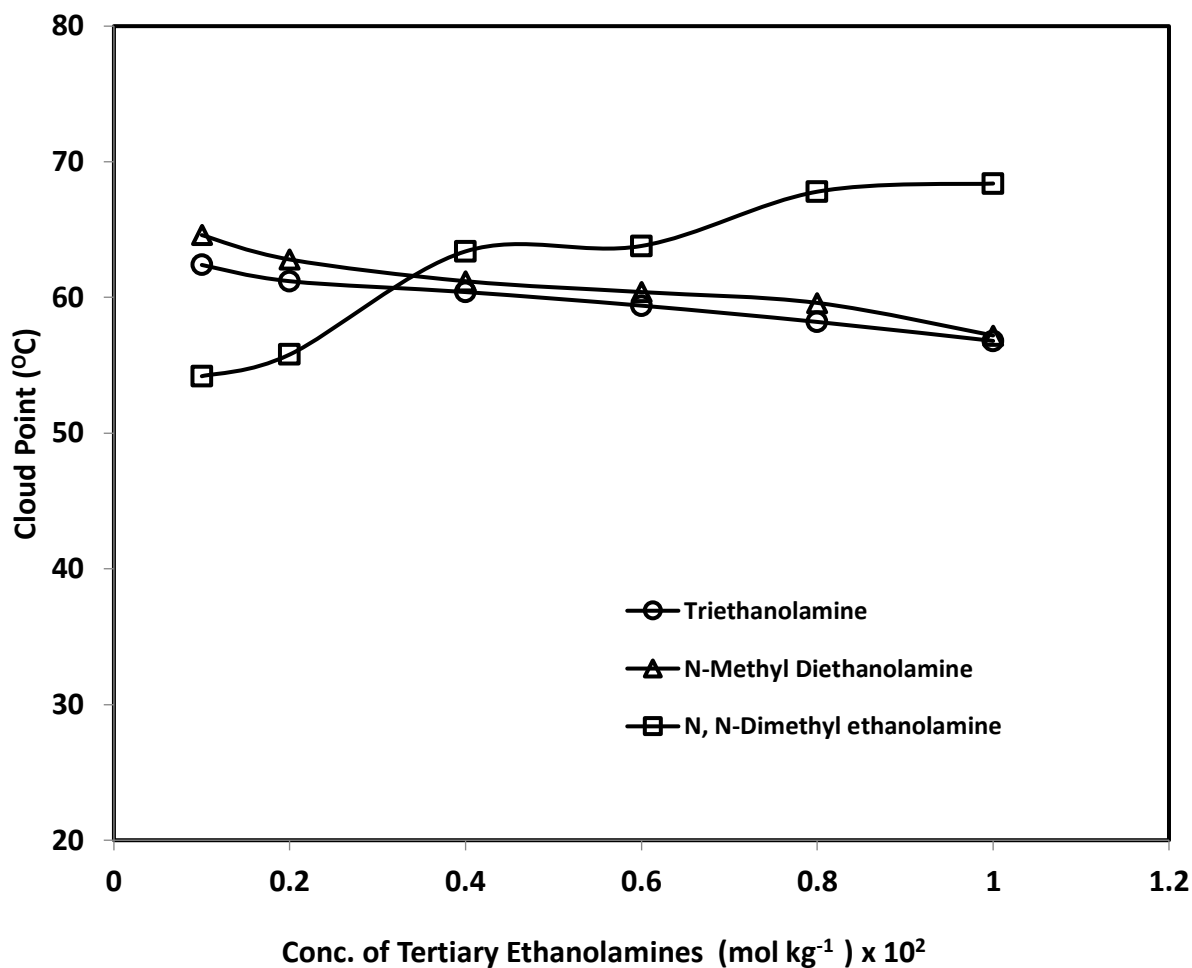


Figure 2: Variation in the cloud point of Brij-30 surfactant in a 22% aqueous ethanolic medium with varying concentrations of tertiary hydroxylamines.

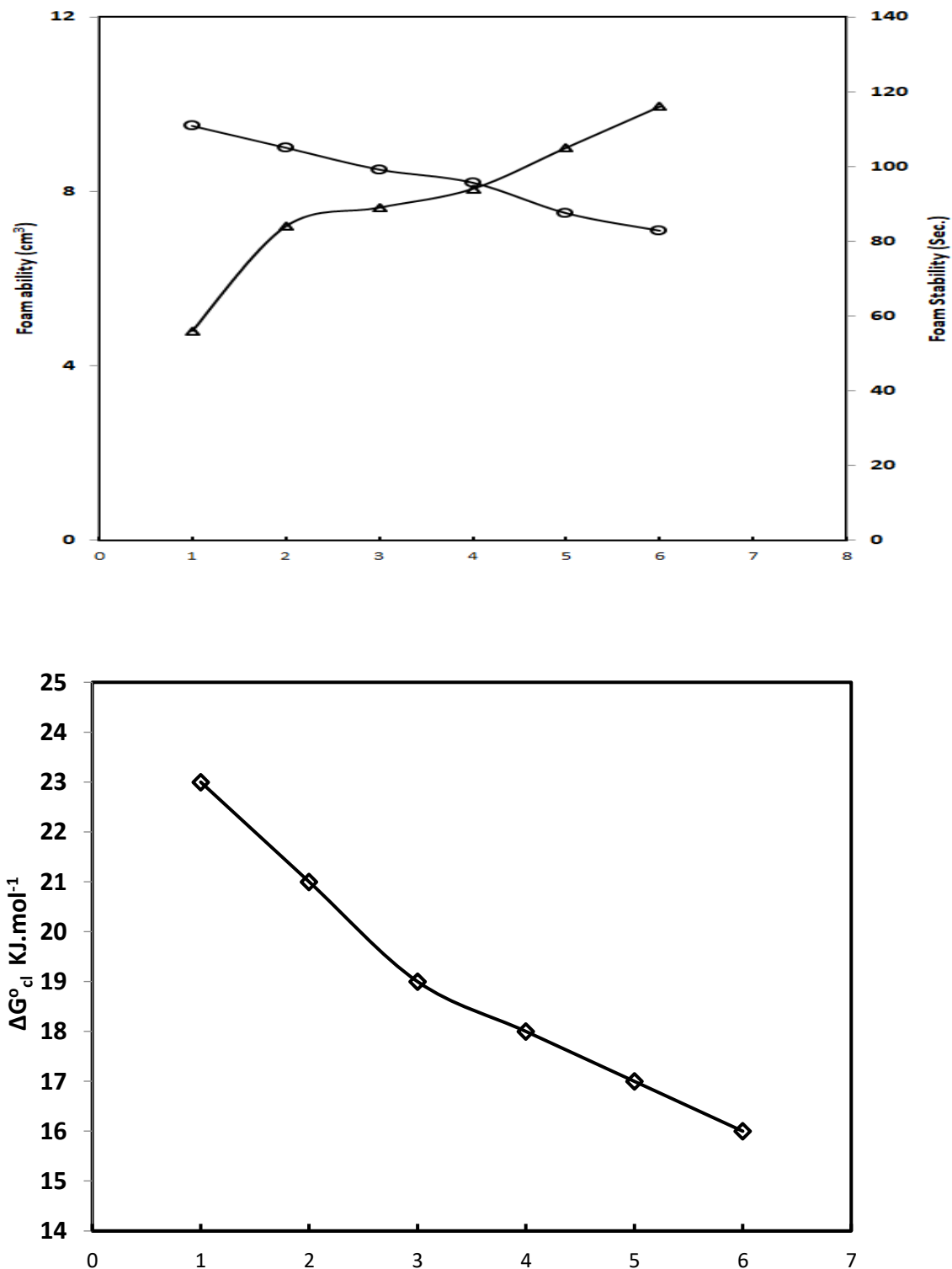


Figure 3: Variation in foam ability, foam stability and Gibbs free energy for of Brij-30 surfactant in a 22% aqueous ethanolic solution.

4.4 Thermodynamics of Cloud Formation:

The chemical behaviour of substances is fundamentally influenced by energetic factors. Thermodynamic principles help explain the creation of spontaneous micelles. For non-ionic surfactants, phase separation during clouding is linked to specific thermodynamic entities. These thermodynamic entities, including change in Gibbs free energy (ΔG^0_{cl}), change in standard enthalpy (ΔH^0_{cl}), and change in entropy (ΔS^0_{cl}), for phase separation were evaluated using the solubility term at the CP temperature. This assessment was carried out through the phase separation model, employing the following equations:

$$\Delta G^0_{cl} = -RT \ln X_s \dots\dots\dots (1)$$

$$\Delta H^0_{cl} = RT^2 d \ln X_s / dT \dots\dots\dots (2)$$

$$\Delta S^0_{cl} = \Delta H^0_{cl} - \Delta G^0_{cl} / T \dots\dots\dots (3)$$

In this context, 'cl' refers to clouding, and X_s represents mole fraction of the solute in the surfactant mixture. T is the cloud point (CP) temperature measured in Kelvin. The ΔH^0_{cl} at phase separation is determined from the slope of the curve in the plot of $\ln(X_s)$ versus $1/T$. The thermodynamic values for various additive mixed systems have been documented in the following table No.3.

Table 3: Thermodynamic entities at clouding for 22% alcoholic solution of Brij-30 (10% w/v) in the presence of alkyl hydroxyl amines.

(a) 22% alcoholic solution of Brij-30 (w/v) + Diethanolamine			
Concentration of amines (M)	ΔG^0_{cl} kJmol ⁻¹	$-\Delta H^0_{cl}$ kJmol ⁻¹	$-\Delta S^0_{cl}$ Jmol ⁻¹ K ⁻¹
0.1	23.252	35.54	179.34
0.2	21.220		174.26
0.4	19.292		168.61
0.6	18.086		166.12
0.8	17.105		165.13
1.0	16.267		164.99
(b) 22% alcoholic solution of Brij-30 (w/v) + Dimethylamine.			39.61
0.1	20.791	185.62	
0.2	18.998	179.90	
0.4	17.152	173.47	
0.6	16.117	169.48	
0.8	15.379	166.52	
1.0	14.833	164.38	
(c) 22% alcoholic solution of Brij-30 (w/v) + 2-methylethanolamine			
0.1	23.229	34.51	174.86
0.2	21.494		168.28
0.4	19.695		161.90
0.6	18.701		157.80
0.8	18.034		154.63
1.0	17.507		152.18

CONCLUSION:

The nonionic surfactant Polyoxyethylene (4) lauryl ether undergoes a clouding phenomenon in the presence of hydroxyl amines as additives. The CP change in the presence of hydroxylamine indicates that water-dissolving hydroxylamines interact favorably with Polyoxyethylene (4) lauryl ether transformed the water structure in to expanded form results change in CP. The CP of 10% aqueous alcoholic solution of Brij-30 in the attendance of the secondary and tertiary ethanolamine is varies with the concentration of hydroxylamine's. The CP steadily increases with the concentration of secondary amines such as 2-dimethylethanolamine and dimethylamine, while the CP decreases with the concentration of diethanolamine, the increase in CP is due to the presence of more alkyl groups. While CP decreases with an increase in the number of ethanoyl groups, as seen with ethanolamine, generally, the hydrophilicity and solubility in water of a molecule increase with the number of ethylol groups it contains. Higher the number of ethylol groups nitrogen atom that, causes a reduction in the CMC. More the (hydrophilic) groups present, the higher is the solubility in water, which lowers both the surface tension and the CP. Cloud temperature increases with the concentration of Methyl diethanolamine and Dimethyl ethanolamine due to the strengthening of the water structure through hydrogen bonding, facilitated by the nitrogen atom, which enhances the hydrophobic effect and promotes micellization. Consequently, a natural decrease in the critical micelle concentration (CMC) is observed with an increase in ethanolamine concentration, leading to a higher CP. while the CP decreases with the concentration of triethanolamine. The negative values of ΔH^0_{cl} and ΔS^0_{cl} indicate that the phase change is an enthalpy-favored process. The decrease in ΔG^0_{cl} values with increasing ethanolamine concentration suggests that the clouding phenomenon becomes more spontaneous as the concentration of amines increases.

The negative ΔH^0_{cl} values are primarily due to the hydration of the hydrophilic head region, which is more dominant than the disruption of water molecules in the hydrophobic alkyl chain of the surfactant. Additionally, the negative ΔH^0_{cl} values indicate that dispersive forces play a significant role in molecular association.

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