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Properties of novel cationic surfactants

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Abstract
The most important properties of Novel Cationic Surfactants is micelle formation i.e. to aggregate self-association in the solution to form micelles. These micelles are of three types – Normal micelle, Reverse micelle and mixed micelle. Their aggregation process depends upon the amphiphillic species and the condition in which they dissolved. The surfactant solution in which they dissolve shows a sudden change in their physical as well as chemical properties are called Critical micelle concentration i.e. (CMC) for the formation micelle. There are many factors which affects the critical micelle formation or concentration such as structure of surfactant, presence of various additives in the solution and effect of experimental conditions etc. Due to numerous applications of this system, there is lot of interest towards understanding of mixing behavior by using various techniques.

Keywords: CMC of surfactant solution, Conductivity, Surface tension, Viscosity, Various additives, structure of surfactants, Experimental conditions, Temperatures, PH, Pressure etc

Introduction

One of the most important characteristic properties of the amphiphilic molecules is their capacity to aggregate self association in the solution to forms the micelles. The aggregation process depends on the amphiphilic species and the conditions in which they are dissolved. The small concentration range in which surfactant solutions shows a sudden change in their physical as well as chemical properties is called critical concentration (CMC) for the formation of micelle [1-2]. This term of CMC was defined by Davies and Busy [3] in 1930 as threshold concentration at which micelle first appears in the solution. The properties (Chemical as well as physical) of surfactants vary remarkably above or below the CMC value [4-6]. Below this value the physio-chemical properties of ionic surfactants like Conductance, electromotive for us resembles to those like strong electrolytes where as above this CMC value their Physio-chemical properties changes drastically which indicates the highly co-operative association process i.e. micellation takes place. This can be illustrated by Preston's [1] classical graphs. (Shown in figure: 1.1).
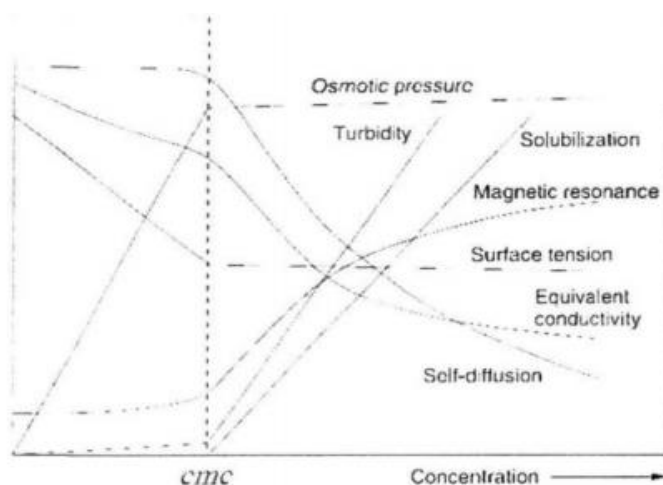

Fig 1: change in the concentration dependence of wide range of physico chemical changes around the critical micelle concentration

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Types of micelles

The exact structure of the micelle is still a controversial fact, but just like the CMC as discussed earlier it is considered to be roughly globular or spherical [7].

There are three types of micelles formed in the surfactant solutions. All these has been described as —

Normal micelle

The structure of normal micelle is considered to be roughly spherical [8-9]. If the hydrocarbon chain is the hydrophobic part of the surfactant molecule then micelle has liquid like hydrocarbon core of radius which is equals to the length of fully extended carbon chain (~ 12-13A). The polar head part gets attracted towards the surrounding water and arranged in regular pattern at the micelle surface which is rough [10]. According to Menger water has the ability to penetrate into the micelle for some certain limit [11], he derived this idea from fluorescence and H-NMR measurements. With the use of partial molar volume determinations. We came to the fact that alkyl chains in the core are more expanded than those in the normal liquid state.

An ionic micelle is formed in polar solvents like water, consists of three regions. Shown in figure 1.2)

(I) The interior or core of the micelle consists of a hydrocarbon chains of ionic surfactant molecule.

(II) There is an aqueous layer surrounding the core, called stem layer which constitutes the inner part of electrical double layer. It contains regularly spaced head groups and 60-90% of the counterions. The head part is hydrated by the no. of water molecules. The methylene groups attached to the head group may be wet. The stem layer and the core together form the kinetic micelle.

(III) The outer layer is a diffused layer which contains the remaining counterions to thus called Gouy – Chapman layer that extends further into the aqueous phase. The thickness of this layer is determined by the effective ionic strength of the solution.

Counterions are strongly bounded by electrical field which is created by head groups and by specific interactions that depends upon head group and type of counterion [12]. There is a model which can be successfully applied to the distributions of counterions i.e. they are assumed to be either 'bound' to the micelle pseudophase or 'free' in the aqueous phase [8, 13-14]. The order of head group and counterion in the interfacial region of ionic micelle is 3-5 M that gives the micelle surface the properties of concentrated salt solutions [15]. Till the solutions as a whole is neutral but both micellar and aqueous pseudo phases carry a net charge as thermal forces provides the counterions to it. For nonionic micellar as the structure is same except the outer region which contains no counterions, but have coils of hydrated polyoxyethylene chains water molecules appears to be trapped on the surface of oxyethylene sites [16].
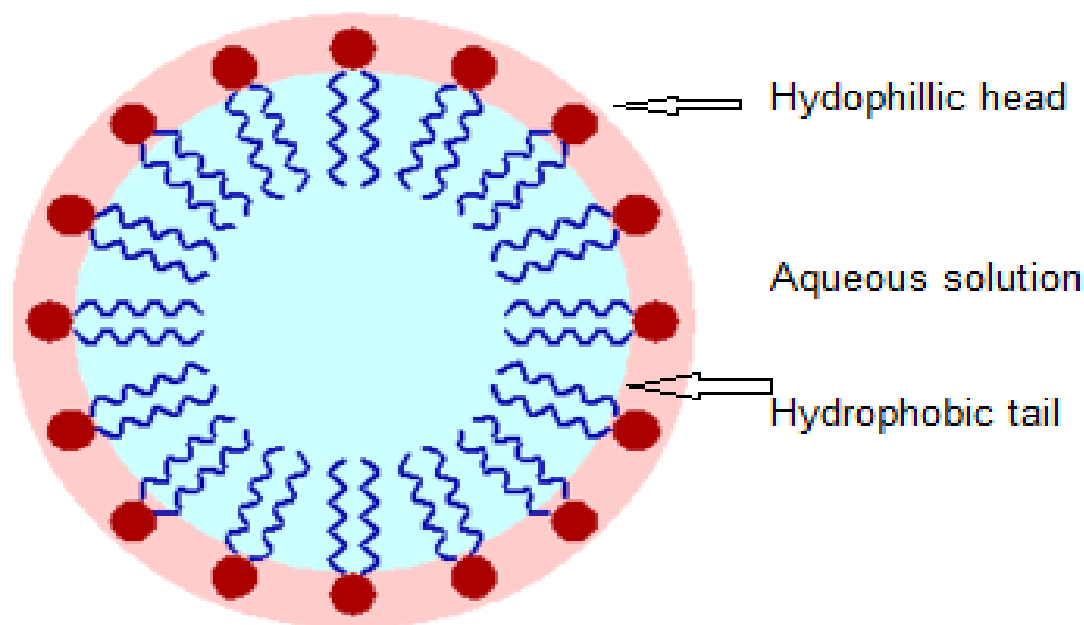

Fig 1.2: Schematic diagram of a micelle of oil in aqueous suspension

Reverse Micelle

When surfactant molecules dissolves in non-polar solvents in the presence of traces of water, then these associates to form the reverse, inverted or reverted micelles (shown in figure 1.3 a). In reversed micelles the head part of surfactant molecules is towards the inside to form a polar core and the hydrocarbon tails are directed towards the bulk solvent to form the outside shell of the micelle [17-20]. The reverse micelles are almost spherical if the concentration of surfactant is very low. Thus, in that case the water molecules occupy the central part of the sphere and forms micro- water pool and these water molecules remains connected with the head groups of reverse micelles forming surfactant molecules. The tails of these surfactants are directed towards bulk non-polar solvent phase. (Shown in figure: 1.3 b).

Reverse micelles are being studied deeply at the primary stage because of its useful property of micro reactors for chemical and biochemical reactions [21]. In the recent years, there is a significant growth in the field of reverse micelle due to the assumption or a hope to find that proteins and other biopolymers, even bacterial cell may be soluble in the reverse micelle system. In fact, it gives rise to extension of interest to new domains like bio catalysis and chemical biotechnology.
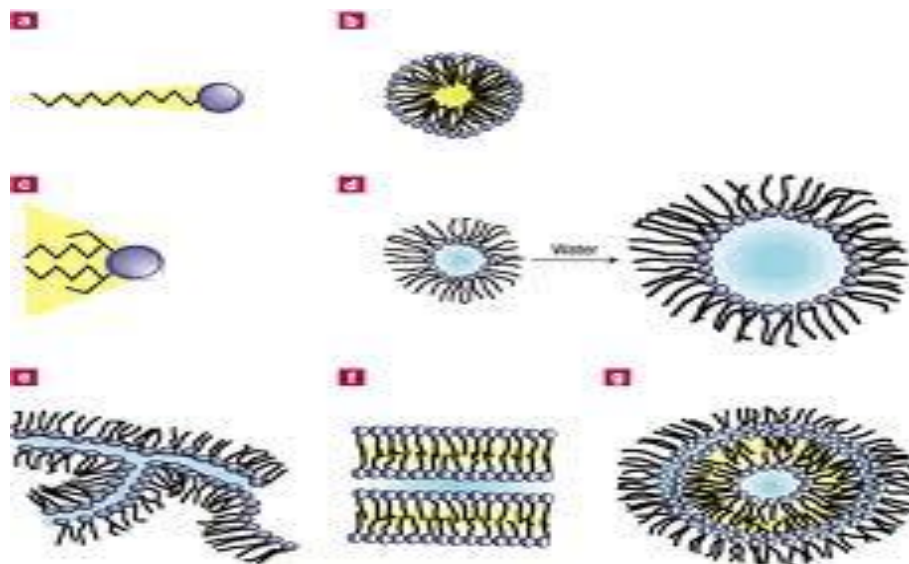
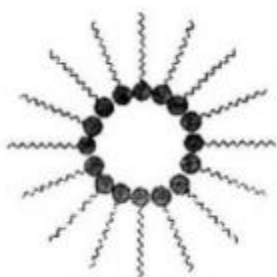**Fig 1.3 a:** Formation of reverse micelles in surfactants

Fig 1.3 b: A two dimensional schematic representation of reverse micelle

Mixed Micelle mixing or two or more surfactants in a solution which results into the formation of mixed micelle (shown in figure 1.4). In generally used language mixed micelle means a micelle which is made up of amphiphiles capable of forming micelle. Thus, mixed micellization is a special case of solubilization. The physio chemical properties of mixed micelle are a little different and have better properties (improved) from pure micelle that has individual components.
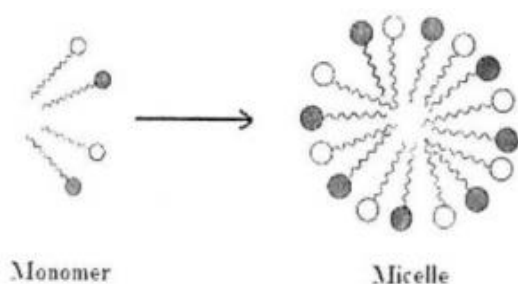

Fig 1.4: Schematic representation of formation of mixed micelle by the monomers

In terms of its application, they are of great importance in various processes like biological, technological, pharmaceutical and medicinal formulations, in advanced oil recovery process for the purpose of solubilization, suspension, dispersion etc [22]. Due to numerous applications of these systems, there is a lot of interest towards the understanding of mixing behavior by using various techniques like conductivity, surface tension, viscosity, NMR, calorimetry, potentiometry, fluorimetry and density, SANS etc [23-37].

Factors affecting critical micelle formation/concentration
Since during the micelle formation the properties of the solution of amphiphiles under goes change drastically, hence there is a great effort to be made to consider the various factors that determine the concentration at which micelle formation occurs and becomes stable, especially in the aqueous solution. The factors that effects the CMC in aqueous micellar solutions are –
1) Structure of surfactants
2) Presence of various additives in the solution
3) Effect of experimental conditions

1) Structure of surfactants
In aqueous medium, the CMC decreases strongly as the hydrophobic nature of the surfactant increases. The reduction results to a good approximation by using empirical equation.

$$\text{Log cmc} = A - Bn_c$$

Where n_c is the number of carbon in the alkyl chain. When the number of carbon atoms in a straight chain of hydrophobic tail exceeds 16, then the CMC values remain unchanged with increase in the chain length due to coiling of this long chain in water.
As a general rule, for ionic surfactants the CMC is halved when the length of the straight hydrocarbon chain is increased by one methylene group, while in case of nonionic surfactants, the addition of one methylene group causes the CMC to decrease by approximately 1/3 of its original value [38].
Since there is a lot of difference between ionic and nonionic but still the effects of the head group are moderate. Cations have slightly higher value of CMC than anions. In the polyoxy ethylenated nonionic surfactants, the CMC values decrease with decrease in the number of oxyethylene units in the polyoxyethylene chain as its difference made the surfactant more hydrophobic. Zwitter ionic surfactants have slightly smaller CMC value than ionics having same number

of carbon atoms in the hydrophobic groups. In quaternary cationics pyridinium compounds have smaller cmc than the corresponding tri methyl ammonium compounds. This may be due to greater ease of Arrangement of planer pyridinium as compared to tetrahedral trimethyl ammonium group into micelle. In the series of compounds like dodecyl trialkyl ammonium bromides $C_{12}H_{25}N^+ (R)_3 Br^-$,the CMC decreases with increase in R atoms/groups due to increases hydrophobic character.

The valency of the counterion is also important. Due to the presence of larger non-polar part in case of organic counterions, the CMC value is higher as that in inorganic counterions. Muller et al. [39-42] had also reported that of the CF_3 group is attached or act as the substitute of terminal CH_3 group then the value of CMC becomes almost double due to the unfavorable interaction between hydro carbon and the fluorocarbon groups. This was further confirmed by Gerry et al. [43] by observing the decrease in the aggregation number. A phenyl group is almost equivalent to the three and a half methylene group in the effect on CMC value if introduced into a straight hydrocarbon chain.

(II) Presence of various additives in solutions
Effect of electrolytes

If the solution has higher ionic strength then the force of electrostatic repulsion between the head groups of the ionic micelle gets reduced which easily forms the micelle at lower concentration. The presence of various electrolytes in an aqueous solution changes the CMC which effects more strongly for anionic and cationic surfactants then for zwitter ionic surfactants and even more effect for zwitter ionic rather than non-ionic surfactants. The following empirical equation is used to relate the CMC with the electrolyte concentration [44].

$$Log\ CMC = - alog C_i + b$$

Where 'a' and 'b' are constants used for a particular ionic group and 'c' donates the total counterion concentration. This equation doesn't holds good for nonionic and zwitterion surfactants and their effect can be explained by the following equation [45].

$$Log\ CMC = - KC_S + Constant\ (C_S < 1)$$

Where K is a constant for a particular surfactant, electrolyte and the temperature and C_S represents the concentration of electrolytes in moles per liter.

The intra micellar and inter micellar both the interaction are affected by the presence of electrolytes in aqueous surfactant solution. If there is a decrease in the CMC by the presence of electrolytes because of reduced repulsion b/w the electrostatic head groups in the micelles that enhance the easy formation of micelle at lower concentration. There are several attempts made by different persons to check the salt effect on micelle formation in the light of Hoffmeister (lyotropic) series [46-47]. This series plays an important role for the wider range of biological and physicochemical phenomena. A latest study of Moulik and coworkers [48] shows that in case of anionic surfactants, the order of effectiveness in reducing the cmc decreases in the order $Mg^{2+} > Cs^+ > K^+ > NH_4^+ > Li^+$. They had taken two cmc value for a given cationic surfactant in the presence of anions like salicylate $(C_7H_5O_3)$, benzoate $(C_7H_5O_2)$ for a given non-ionic surfactant the effect of anions on the cmc

follows the order as- $F^- > Cl^- > SO_4^{2-} > Br^- > PO_4^{3-} > C_3H_5O(COO)^{3-}_3 > I^- > SCN^-$ and the effect of cations is in the order of $K^+ > Na^+ > Rb^+ > Li^+ > Ca^{2+} > Al^{3+}$ [49].

Effect of Organic additives: Organic additives also effects the CMC of aqueous micellar solutions even in the small amount also some of those materials are present as impurities or byproducts of the amphiphiles due to which the properties of amphiphiles may gets affected so, it is of great importance to have the knowledge of effect of organic materials on the cmc of amphiphiles for both theoretical and practical knowledge/purposes. The major factor for the decrease in CMC is the reduction of free energy of the micelle due to dilution of surface charged density on micelle. They effect the cmc either by entiring in to the micellar region or by modifying solvent micelle or solvent monomer interactions. Some additives like urea shows the increases in the cmc of ionic [50-51] and non-ionic surfactants [52-53] with the additives of urea in case of fluorocarbon there will be a decrease in the cmc value [54] whereas with addition of alcohol the cmc of surfactants increases as well as decreases [55-57]. Decrease in the cmc is observed with increase in the carbon number of linear alcohols in non-aqueous dimethyl form amide [58] with the addition of sugars decreases the cmc value of the system [59-60]. For an ionic surfactants solution, with the different concentration of acetamide it shows decrease [61] as well as increase [62] in the cmc. Since amines are more surface active than alcohols at air water interface [63], hence N- alkylamines (butyl to decyl) are found to be solubilized in micellar phase leaving the amine group on the surface of micelles [64]. These solubilized amine are reported to form mixed micelles with ionic surfactants [65-67].

(III) Effect of Experiments Conditions
(A) Effect of Temperature: The cmc value of amphiphiles in a aqueous medium is affected by the temperature in such a way that it firstly decreases with increase in temperature then increase with increase in temperature. Due to increase the hydration of hydrophilic group decreases with temperature that the favors micellization. However, with increase in temperature disruption of structure occurs of water surrounding the hydrophobic group that opposes the micellization. The magnitude of these two opposing effects thus determines the value of cmc either decreases or increases over a particular temperature range, micelles of many poly oxyethylene non-ionic surfactants increases fastly in size with increase in temperature [68-74] where as the micelle size of ionic surfactant decreases with increase in temperature [75]. By the graph, it is observed that cmc-temperature curve is appeared at around 25 C for ionics [76] and at 50 C for nonionic [77]. For bivalent metal alkyl sulphates, the cmc appears to be practically independent of the temperature [78].

(b) Effect of PH: When amphiphile group has ionic groups such as $-NH_2, -(CH_3)_2\ N \rightarrow O$ and $-COOH$ groups, the degree of dissociation of polar group dependent PH value [79]. Generally, the cmc value is higher at PH value where group is charged C low ph for $-NH_2-(CH_3)_2\ N \rightarrow O$, high ph for $-COOH$) and lower when uncharged. Some zwitter ionic surfactants became cationic at low ph, the change is observed by rapid increase in cmc [80] or modest rise [81] that depends on structure and hydrophilicity of zwitterionic form.

(c) *Effect of pressure*: The effect of pressure on micelle for motion of non-ionic surfactants [82-86] has been studied. Up to the pressure of 1000 atm cmc increases and decreases with further increase in pressure. When surfactant molecules are present in more expanded form in micelle than when present as monomer in solution, so initially pressure compress the micelle and mitigate against the increased freedom of monomer in micelle, thus increases cmc. It may be due to increases in dielectric constant of water, that make less electric work necessary to bring monomer into a micelle. For non-ionic amphiphiles the cmc value increase and than level off with increase in pressure. Nishikido and co-workers [84] studied the effect of pressure on micelle size in aqueous. Sol. Of $NaDS$ and $C_{12}E_6$.

(d) *Effect of solvent medium:* The cmc of surfactants increase with decrease in length of hydrophobic chain but the change is smaller for water [87] than ethylene glycol. For polyoxythylenated nonionic solutions in benzene and carbon tetrachloride, cmc value decreases with rest in length of this group at constant hydrophobic chain length. The cmc of benzene for alkyl ammonium carboxylates increase with increase in length of alkyl chain of cation; in ccl4, there is no significant change in the value of cmc with these structure changes. The cmc is lower in D_2O than H_2O for different amphiphiles [88-89]. The hydrophobic body are expected to be stronger in D_2O than H_2O. Thus, micelle in D_2O is stronger than H_2O.

Counterion Binding Constant
In an ionic surfactant micelle formation. There are two types of opposing forces: (i) Transfer of hydro-carbon chain from bulk phase prose to micellar phase. As it increasing the aggregation. (ii) Electro-static repulsion between the some charged head groups. Which decreases the process of aggregation? Counterions are bounded to micelle surface which cause the screening effect to second type of forces of interaction. [90] So, it's binding process in an important factor in the process of micellization [91]. The counterion binding value is affected by shapes of ionic micelles. Due to the presence of effective dielectric charge on ionic micelles, electric potential is developed at micelles surface. The surface potential value controls different processes which occurs near the micelle solution interface. The value of counterion binding constant gives the information about the number of counterion dissociate in the micellar solution.

Aggregation Number it is the average number of monomers that makes the micelle and are 30-200, in water appr. The micellar aggregation number gives information about micelle size and shape formed by amphiphiles in the solution. Which is important in calculating the stability of investigated system and the relation of molecular structure of amphiphiles with their properties. To minimize the hydrocarbon water contact in solution surfactant monomers assembles itself into micelles. Due to this reason, the lower limit of number of surfactants monomers which forms a micelle is denoted by minimum number that come together to shield each other from contact with water. In ionic surfactants electrostatic repulsion between ionic head groups at the micellar surface increases the opposing force but in case of non-ionic surfactants steric effect as well as performance for hydration of head group opposes micelle formation. It is affected by many factors like nature of amphiphiles, temperature, [92-95] type and concentration of added electrolyte, [92, 96-98] organic additives [97-100] etc. In aqueous medium as the dissimilarity between amphiphilic

and solvent increases. Aggregation number also increases. So, aggregation number appears to increase with increase in hydro-phobic character of amphiphile with increase in temperature aggregation number decreases in aqueous medium of ions but for nonionic surfactants it increases [101-104]. Micellar aggregation number decreases continuously with increase in pressure for non-ionic surfactants. Although, the number of ionic surfactants passes through a minimum at surround 1000 atmosphere. Aggregation number of ionic micelles increases [105-108] by adding electrolytes. There are many methods have been used to find micellar aggregation number like DLS (Dynamic Light Scattering), SANS (Small angle neutron scattering), SSFQ (Study state fluorescence quenching), TRFQ (Tins resolved fluorescence quenching) [109-115].

Molecular Shape the shape of the micelle formed plays on important role in determining various properties of surfactant solution like viscosity, its capacity to solubilize water in soluble materials and its cloud point. The theory of micellar structure based on geometry of different micellar shapes & space occupied by hydrophilic & hydrophobic groups of amphiphile. Which was developed by Israelachbili, Mitchell & Nimhan [4, 116]. The volume occupied by hydrophobic groups in micelle solution, L_C – the length of hydrophobic group, a_o – is area of cross section occupied by hydrophilic group as used to calculate packing parameter (R_p), which determines the shape of micelle.

$$R_p = v / a_o L_c$$

The optional cross section area per amphiphile molecule is observed experimental by using X-ray diffraction of bilayer system whereas the volume and length of hydrocarbon tail is calculated by Tanford [117] equations.

$$V = (27.4 + 26.9\ n)\ A^3$$
$$L_c = (1.5 + 1.26n)\ A$$

(N is the number of carbon atoms in the hydrocarbon chain). The R_p depends on the chemical structure of surfactants monomer and on the surface repulsion between head groups with the formation of "spherical micelles". The aggregation of surfactant molecules in aqueous media is strongly co-operative and starts with $R_p<1/3$. The size of spherical micelles does not change with surfactant concentration and micelle is mono-dispersed. The examples of these surfactants which form spherical micelles are SDS [118] & DTAB (Dodecyltrimethyl Ammonium Bromide) [119-120] When the volume of Rp, $1/3<R_p<1/2$ no spherical micelles like cylindrical on ellipsoidal are formed. The size & aggregation number of cylindrical micelles are affected by surfactant concentration [121-122] & usually polydisperse.

When the volume of packing parameters is ½ >, $R_p < 1$, Bilayers and Vesicles are formed. Their volume is 2 times to that of micelles forming substances having two changes expecting to form bi-layer^s [123-124]. It can accommodate a large no. of amphiphilic molecules without change in available surface area per amphiphile. When volume of hydrocarbon part is large as compared to head group area having R_p is greater than reverse micelles are formed.

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