



## Gemini surfactants – A short overview

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Gemini surfactants emerged in the nineties and within a short spell of time they achieved a tremendous recognition in the domain of colloid and surface science. Geminis are basically bis-surfactants with unique surface activity compared to conventional surfactants. The structural features of geminis provide the key towards their uniqueness. In the course of time, geminis have found potential applications in various industries. This short review attempts to provide a very basic understanding about the geminis regarding their structure, surface activity and field of application.

Keywords: Gemini surfactant, HLB, CMC.

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### Introduction

Surfactants are considered as one of the pillars of modern day civilization. From domestic sector to industrial sector, the applications of surfactants are tremendous. Surfactants possess the exquisite properties of lowering the surface tension of liquid at liquid-air boundary or the interfacial tension at the interface of two immiscible liquids like water and oil. A surfactant molecule is basically composed of at least two moieties, one hydrophobic and another hydrophilic, adjoined together in the same structure. Long carbon chain of 8-18 carbon atoms in the form of straight or branched hydrocarbon or fluorocarbon chain usually form the hydrophobic part while a polar or ionic group, generally termed as the 'head group', serves the role of hydrophilic moiety. The hydrophilic-lipophilic balance (HLB), which is a measure of the ratio of the hydrophobic and hydrophilic parts of the amphiphilic surfactants, is accountable for the preferential adsorption of the surfactants in surfaces and interfaces compared to the bulk phase and also for the formation of self-assembly aggregates in solution<sup>1</sup>. The driving force for this preferential adsorption and consequent self-assembly stems from the lowering of free energy of the phase boundary ultimately resulting in lowering of surface and interfacial tension. The variation in surface tension lowering activity of surfactants attributes to the structure of the surfactants which can be effected by modulation of structures of either the head group or the hydrophobic part or both of the two. Reports of

new classes of surfactants always remain at the top of science publications. The synthesis and property elucidation of *bis*-surfactants that is two surfactant moieties in a single structure attracted the prime attention of chemists. Gemini surfactants are the bis-surfactants that had the origin in the late nineties. Gemini surfactants are considered as unique regarding their structure and properties in the domain of surfactants. The term was first coined by the group of Fredric M. Menger in 1991 as Menger and Littau assigned the name gemini to bis-surfactants connected by rigid spacer (i.e. benzene, stilbene)<sup>2</sup>. The initial assignment of the term by Menger was later extended to incorporate all the bis-surfactants with spacers that are not essentially rigid. In this context, gemini surfactants may also be defined as the family of surfactant molecules possessing more than one hydrophobic tail and one hydrophilic head group<sup>3</sup>. However, if the extended or generalized definition of gemini surfactants is to be considered, it must be revoked that bis-surfactants without the constrain of essentially rigid spacer, had already been reported before the actual coining of the term as can be evidenced by the work of Bunton *et al.* in the study of catalytic effect of 'dicationic detergents' on nucleophilic substitution<sup>4</sup>, also by the study of Devinsky *et al.* regarding the surface activity and micelle formation of some new 'bisquaternary ammonium salts'<sup>5</sup> as well as by the report of Okahara *et al.* on the preparation and property elucidation of 'amphipathic compounds with two sulfate groups and two lipophilic alkyl

chains<sup>6</sup>. Gemini surfactants are potentially more surface-active than conventional surfactants and they can also self-assemble at nearly hundred times lower concentration compared to conventional surfactants. The surface activity can also be increased thousandfold<sup>7</sup>. Geminis have found potential application in skin care, construction of high-porosity materials, analytical separations, antibacterial regimens and solubilization processes.

### Structure

The uniqueness of the geminis arises from their structure. In the simplest idea, minimum two surfactant moieties are joined in a single structure in the geminis through a spacer as in Fig. 1. The structural feature can also be depicted in a different way as at least two head groups and no less than two hydrophobic chains must be there to be categorized as gemini surfactant. In case of symmetric geminis where two alike surfactant units are joined, the spacer can be positioned in either of two ways. The spacer unit may connect the two head groups or it can connect two hydrophobic groups. Symmetric geminis are also termed as dimeric surfactants. Apart from the symmetric geminis, heterogeminis have also been

charge of the head group, the neutral charge of the molecule is maintained by the presence of organic or inorganic counterions. The nonionic head groups may be composed of polyether and sugar<sup>10</sup> moieties. Some representative structures of the synthesized geminis have been outlined in Figs. 2 to 4<sup>10-12</sup>. Geminis containing three or even greater number of polar groups or hydrophobic tails have also been reported<sup>13-15</sup>. A gemini type surfactant has also been reported where the structure (Fig. 5) is based on the adamantane moiety consisting of four head groups and four hydrocarbon tails with the tails being tetrahedrally set about the adamantane moiety<sup>7</sup>. Fig. 6 depicts a special gemini with positively charged nitrogen in annulene unsaturated ring serving as the head group, here the ring functions the role of spacer to some extent<sup>16</sup>. However, the structure of a gemini is not the proper guide towards required surface activity. Surfactant behavior only emerges out of proper HLB parameter and the manifestation of surface activity can only be estimated through proper experimentation. Presence of long hydrocarbon chain suffice to enhance surface activity but at the same time it also induces greater hydrophobicity leading to scarce solubility which can be overcome by the hydro-

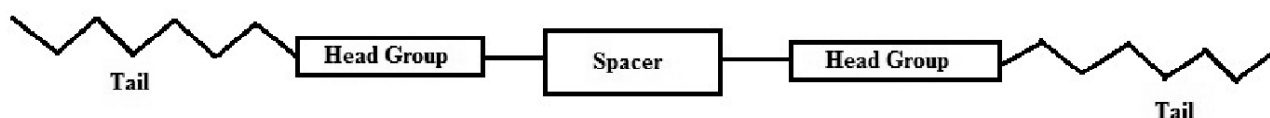


Fig. 1. Schematic diagram of a gemini surfactant.

synthesized corresponding to structures arising out of two alike hydrophobic groups with two dissimilar polar head groups, or two dissimilar hydrophobic groups with two alike polar head groups or in the last variation two dissimilar hydrophobic groups with two dissimilar polar head groups<sup>8,9</sup>. A lot of variations are exemplified in the structures of the spacer unit. The spacer may be rigid structured like stilbene or it may be flexible like methylene chain and regarding the length of the spacer it can be short like 2 methylene groups as well as long with 12 methylene groups<sup>7</sup>. The spacer may be inherently polar as polyether or it can be of non-polar nature like aliphatic or aromatic chains. The polar head group can be either ionic or nonionic. The ionic head group can be positively charged nitrogen in acyclic chain or in saturated and unsaturated rings or it can be negatively charged phosphate, sulfate and carboxylate groups as well. Whatever be the

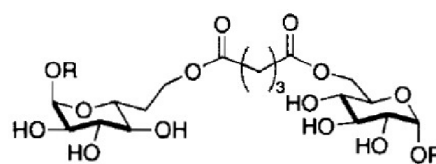


Fig. 2. A symmetric nonionic gemini surfactant<sup>10</sup>.

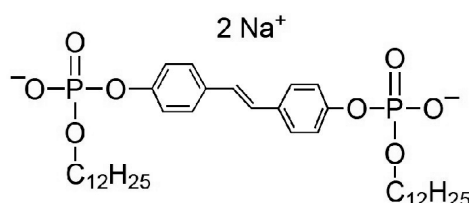


Fig. 3. A symmetric anionic gemini surfactant with rigid hydrophobic spacer<sup>11</sup>.

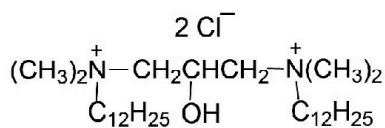


Fig. 4. A symmetric cationic gemini surfactant with flexible hydrophilic spacer<sup>12</sup>.

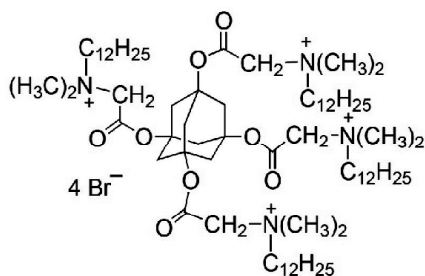


Fig. 5. A gemini type surfactant with four head groups and four hydrophobic tails<sup>7</sup>.

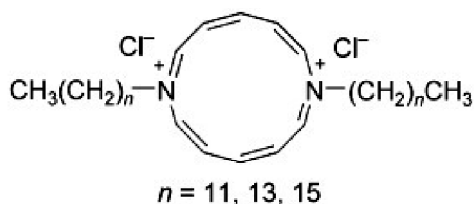


Fig. 6. Annulene gemini surfactant<sup>16</sup>.

philic head group and presence of hydrophilic groups in the spacer unit<sup>17</sup>. So optimization of HLB parameter is crucial for required surface activity along with a balance in solubility. Therefore the initially synthesized structure is improvised with introduction of hydrophobic or hydrophilic groups in the different regions of the geminis.

### Surface activity

In case of oil in water mixture, the presence of surfactants in the system prompts the surfactants molecules to arrange themselves at the interface with the hydrophobic hydrocarbon part being directed towards the oil phase and the hydrophilic head groups dragging towards aqueous phase. This is the equilibrium situation of lower free energy for the surfactants rather than its complete solution in either phase. So, the surfactants are strongly adsorbed at the interface forming an orientated monomolecular layer – the phenom-

enon that is actually considered as surface activity. Surface activity is rendered as a dynamic phenomenon because the final state of a surface or interface evolves out of a balance between the tendency of adsorption and the tendency of complete mixing due to the thermal motion of the molecules<sup>18</sup>. Now greater surface activity actually refers to greater accumulation of the surfactant molecules at the interface rather than either of the bulk phases and this continues with increase of concentration of the surfactants in the bulk phase only up to a certain concentration limit. Once this concentration limit is reached the surfactant molecules form aggregates by mechanism of self assembly with the hydrophobic portions pointing towards interior core while the hydrophilic head groups directing towards the aqueous phase. These aggregates are termed micelles and the process is referred to as micellization. The fairly well defined concentration above which micelle formation becomes appreciable is termed the critical micellar concentration (CMC). So, micellization can be conceived as an alternative mechanism to adsorption by which the interfacial energy of a surfactant solution might decrease. Consequently it can be concluded that greater the surface activity the lower will be the value of CMC. In this regard, CMC serves as the guide towards estimation of the surface activity of a surfactant. Experimental determination of CMC can be carried out by measuring any micelle-influenced physical property as a function of concentration<sup>18</sup>. Quite a few physical properties, such as turbidity, osmotic pressure, electrical conductance and surface tension are very much sensitive to CMC and therefore surface tension, electrical conductivity and dye solubilisation measurements are mostly performed for determination of CMC. Spectroscopic methods have also been utilized to measure the CMC values of geminis<sup>19</sup>. Geminis possess considerable low values of CMC in comparison with conventional surfactants of equivalent chain length as revealed in Table 1 (names of conventional surfactants have been put on bold in Table 1)<sup>7</sup>. The table also reveals the fact that polarity of short spacers (2-8 atoms) does not influence the CMC values, but the influence is prominent in case of long hydrocarbon spacer of 16 methylene groups which decreases the CMC value roughly ten-fold relative to a short spacer of 3-8 methylene groups. The greater hydrophobicity incurred by the long hydrocarbon spacer unit actually lowers the solubility of the monomer leading to aggregate formation. It is also found that the CMC

**Table 1.** CMC values of representative conventional (1-4) and gemini surfactants<sup>7</sup>

Surfactant	CMC (mM)
1. C <sub>12</sub> H <sub>25</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>3</sub> Br <sup>-</sup> ( <b>DOTAB</b> )	16
2. C <sub>12</sub> H <sub>25</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>3</sub> Cl <sup>-</sup> ( <b>DOTAC</b> )	22
3. C <sub>16</sub> H <sub>33</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>3</sub> Br <sup>-</sup> ( <b>CTAB</b> )	1
4. C <sub>12</sub> H <sub>25</sub> OSO <sub>3</sub> <sup>-</sup> Na <sup>+</sup> ( <b>SDS</b> )	8
5. C <sub>12</sub> H <sub>25</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -(CH <sub>2</sub> ) <sub>n</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>12</sub> H <sub>25</sub> 2Br <sup>-</sup> (n = 3–8)	1
6. C <sub>12</sub> H <sub>25</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -(CH <sub>2</sub> ) <sub>16</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>12</sub> H <sub>25</sub> 2Br <sup>-</sup>	0.12
7. C <sub>16</sub> H <sub>33</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -(CH <sub>2</sub> ) <sub>2</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>16</sub> H <sub>33</sub> 2Br <sup>-</sup>	0.003
8. C <sub>8</sub> H <sub>17</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -(CH <sub>2</sub> ) <sub>3</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>8</sub> H <sub>17</sub> 2Br <sup>-</sup>	55
9. C <sub>12</sub> H <sub>25</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -(CH <sub>2</sub> ) <sub>2</sub> -O-(CH <sub>2</sub> ) <sub>2</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>12</sub> H <sub>25</sub> 2Cl <sup>-</sup>	0.5
10. C <sub>16</sub> H <sub>33</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -(CH <sub>2</sub> ) <sub>5</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>16</sub> H <sub>33</sub> 2Br <sup>-</sup>	0.009
11. C <sub>16</sub> H <sub>33</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -(CH <sub>2</sub> ) <sub>2</sub> -O-(CH <sub>2</sub> ) <sub>2</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>16</sub> H <sub>33</sub> 2Br <sup>-</sup>	0.004
12. C <sub>16</sub> H <sub>33</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -CH <sub>2</sub> -(CH <sub>2</sub> -O-CH <sub>2</sub> ) <sub>3</sub> -CH <sub>2</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>16</sub> H <sub>33</sub> 2Br <sup>-</sup>	0.02
13. C <sub>12</sub> H <sub>25</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -CH <sub>2</sub> -CH(OH)-CH <sub>2</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>12</sub> H <sub>25</sub> 2Br <sup>-</sup>	0.8
14. C <sub>12</sub> H <sub>25</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -CH <sub>2</sub> -C <sub>6</sub> H <sub>4</sub> -CH <sub>2</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>12</sub> H <sub>25</sub> 2Br <sup>-</sup>	0.03
15. C <sub>12</sub> H <sub>25</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -CH <sub>2</sub> -CH(OH)-CH(OH)-CH <sub>2</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>12</sub> H <sub>25</sub> 2Br <sup>-</sup>	0.7
16. C <sub>12</sub> H <sub>25</sub> N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -CH <sub>2</sub> -CH(OH)-CH <sub>2</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> -CH <sub>2</sub> -CH(OH)-CH <sub>2</sub> -N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> C <sub>12</sub> H <sub>25</sub> 3Cl <sup>-</sup>	0.5
17. C <sub>12</sub> H <sub>25</sub> OPO <sub>2</sub> <sup>-</sup> -O-(CH <sub>2</sub> ) <sub>6</sub> -OPO <sub>2</sub> <sup>-</sup> -OC <sub>12</sub> H <sub>25</sub> 2Na <sup>+</sup>	0.4
18. C <sub>10</sub> H <sub>21</sub> O-CH <sub>2</sub> -CH(OSO <sub>3</sub> <sup>-</sup> )-CH <sub>2</sub> -O-(CH <sub>2</sub> ) <sub>2</sub> -O-CH <sub>2</sub> -CH(OSO <sub>3</sub> <sup>-</sup> )-CH <sub>2</sub> -OC <sub>10</sub> H <sub>21</sub> 2Na <sup>+</sup>	0.01

values of anionic gemini surfactants are to some extent less compared to their cationic counterparts<sup>7</sup>. The enthalpies of micellization,  $\Delta H_{mic}$  are exothermic quantities but conversely entropies of micellization  $\Delta S_{mic}$  are negative as aggregation is formed. So the spontaneity of the process depends on the balance between them and the spontaneity have been found to decrease with increase in the length of the spacer and interestingly the CMC values also decrease with increase in the length of the spacer<sup>20</sup>. Regarding the role of flexibility of the spacer unit, a general trend is observed that the geminis that have flexible hydrophilic spacers show a greater affinity to aggregate in regard to geminis that have rigid hydrophobic spacers. Hydrophilic short spacers in geminis actually help in closer packing of the hydrophobic groups at the water-air interface compared to conventional surfactants and is the key towards enhanced surface activity of the geminis. The more compact packing of the hydrophilic groups of gemini surfactants also lead to a more cohesive and stable interfacial film<sup>3</sup>. The theoretical modeling of ionic and nonionic geminis both with hydrophobic and hydrophilic spacers have been done using Monte Carlo simulations and it serves as a guide regarding micellar shape arising out of aggregation of the geminis<sup>21</sup>. The geminis with short hydrophobic spacers

mainly form nonspherical micelles while with long hydrophobic spacers rod like micelles are formed. The bending stiffness of the hydrophobic spacer is liable to increase the CMC. In case of hydrophilic spacer the CMC is decreased and generally leads to spherical micelles. However, it is found that whatever be the characteristic of the spacer, the morphologies of the micelles formed out of the ionic and nonionic geminis are very much alike and quite insignificant to the spacer characteristics.

### Application

The ST of water (72 mN m<sup>-1</sup> at 25°C) is generally decreased in the range of 30–40 at the CMC of a surfactant. Gemini surfactants are the better candidates for the job as they are required in much lesser amount than the conventional ones. The field of application of geminis is really very broad<sup>22,23</sup>. The main shares of application are found in cosmetics and skin-care sector<sup>24</sup> and in the field of oil-recovery and storage tank cleaning<sup>25,26</sup>. The other important sectors where geminis are highly employed include textile industry for softening of fabric<sup>27</sup>, as additives for paints and coatings<sup>28</sup> as well as in leather technology<sup>29</sup>, in medicinal and pharmaceutical sectors<sup>30</sup> etc.

Some remarkable application of geminis have also come in light. Gemini surfactants have been successfully applied as synthetic vectors for gene transfection<sup>31,32</sup>. Long-chained gemini surfactants have also been employed for semipermanent wall coatings in capillary electrophoresis of proteins<sup>33,34</sup>. The geminis can also be utilized as corrosion inhibitor of mild steel in acidic condition<sup>35</sup>. Gemini surfactants can function as excellent solubilization agents<sup>36</sup>. Organic pollutants in the category of polycyclic aromatic hydrocarbons (PAHs) like anthracene, naphthalene, fluorene or pyrene as well as organic dyes like Quinizarin, Sudan I, Orange OT can be efficiently removed from water solution by means of gemini surfactants<sup>37-40</sup>. Gemini surfactants also possess the potential to disperse hydrogels into supramolecular, three-dimensional micellar-hybridized network<sup>41</sup>. These are merely a small reflection of the entire field where geminis are being increasingly incorporated to achieve greater functionality and better performance.

## Conclusions

The gemini surfactants emerged with great future potential and they really made the doorway towards very meaningful applications in different domains. Different types of geminis and gemini based systems were developed in the course of time. They are employed in various fields of modern day science as well. In fields of supramolecular catalysis, nanoscale synthesis, targeted drug delivery etc., the incorporation of geminis have proved to be useful. Biocompatibility is a serious concern for any non-biosurfactant and integration of biodegradable units into geminis also remains the choice for better biodegradability. Near future will surely witness more improvised geminis with greater biocompatibility and enhanced surface activity for multidimensional application.

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