

J. G. Manjunatha *Editor*

Advances in Surfactant Biosensor and Sensor Technologies

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Preface

In the realm of science and technology, where the frontiers of human understanding meet the limitless potential of innovation, there exists a fascinating intersection known as biosensors. These unassuming yet extraordinary devices have revolutionized the way we perceive, interact with, and harness the intricacies of the biological world. This book is an exploration of that intersection a journey into the captivating realm of biosensors and their profound impact on our lives. Biosensors, at their core, are bridges between the biological and the digital, the living and the engineered. They are the translators that convert the language of molecules, cells, and biological processes into measurable signals, unveiling the secrets of the microcosms that surround us. With the advancement of technology, these devices have grown in sophistication, becoming indispensable tools in fields ranging from medicine and environmental monitoring to food safety and beyond. Through these chapters, we will journey from the historical roots that laid the foundation for these innovations to the cutting-edge advancements that push the boundaries of what is possible. We will explore the diverse types of biosensors, each tailored to specific applications, and the ingenious principles that underpin their operation. But beyond the technical intricacies, this book also delves into the stories of the brilliant minds who envisioned and crafted these marvels. It is a celebration of human ingenuity, curiosity, and the relentless pursuit of knowledge that drives us forward. Alongside the science, we will encounter the tales of serendipity, perseverance, and collaboration that have shaped the evolution of biosensors.

Dear reader, whether you come to these pages as a seasoned scientist, an aspiring researcher, a curious student, or simply an enthusiast eager to grasp the pulse of innovation, we welcome you. Just as a biosensor opens a window to the hidden realms of biology, this book opens a gateway to understanding the wonders of biosensing technology. So, with curiosity as your guide and these words as your map, embark on this expedition into the captivating world of biosensors. Your journey begins now.

Madikeri, India

Dr. J. G. Manjunatha

Acknowledgements I gratefully acknowledge my sincere gratitude to my mother Sharadamma Jamballi and Springer publishing staff.

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Chapter 1

Overview of Surfactants, Properties, Types, and Role in Chemistry



Jaswinder Kaur, Roheela Farzeen, Manpreet Singh, Nandita Thakur, Madan Lal, Sanjay Kumar Upadhyaya, Yogesh Kumar Walia, and Kamal Kishore

1.1 Introduction

Surfactants are amphiphilic chemicals, composed of a hydrophilic and a hydrophobic group within the molecule. Normally, the hydrophobic part is a long chain hydrocarbon and hydrophilic part is comprised of ionic or polar groups. They have tendency to lower the surface tension at liquid/air interface and prefer to accumulate at surfaces or interfaces [1]. The capability of these molecules to lower the surface tension makes them better emulsifiers, foaming, and dispersing and wetting agents in every field [2].

The molecular structure of surfactant consists of two parts, one is head which is polar in nature (hydrophilic part), and the second one is a hydrocarbon tail which is generally non-polar in nature (hydrophobic part) as shown in Fig. 1.1.

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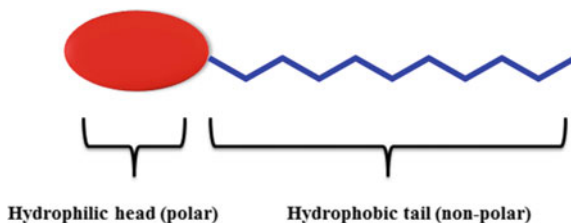
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Fig. 1.1 Structure of surfactant molecule



The hydrophobic part is usually made of alkyl chain comprised of more than eight carbon units and hydrophilic part may be ionic or non-ionic in nature.

1.1.1 Micelle Formation: A Phenomenon

Surfactants exhibit a unique property of self-aggregation due to their amphiphilic nature when dissolved in aqueous or non-aqueous solvents and aggregated molecular form is known as micelle. The minimum concentration required for micellization is referred as critical micelle concentration, CMC [3]. When surfactants are dissolved in an appropriate liquid, they do not form micelles directly. Initially, surfactant molecules accumulate over the air/liquid interface, saturating the interface and lowering the surface tension. After that, the excess of surfactant molecules move to the bulk and form micelles due to hydrophobic interactions [4–6]. The micellization is dynamic in nature, in any micellar or aggregated structures, the surfactant molecules can move constantly in and out of the micelles [7].

The micellization also depends upon the polarity of the solvents used. In aqueous medium, the surfactant molecules form normal micellar structures in which polar heads tend to be towards the aqueous medium side and hydrophobic chains tend towards the inner core of the micelle (Fig. 1.2a). Whereas in non-aqueous solvent medium, the reverse micelles are formed (Fig. 1.2b) in which the hydrophobic chain is missing from the centre and hydrophilic group of surfactants are arranged toward the micelle core. These micellar structures could affect the rate of chemical and enzymatic reactions [8]. Soaps and detergents are the common examples of surfactants. The cleaning action of these surfactants is also well-known. The cleaning action of surfactants in water is well depicted in Fig. 1.3.

Due to amphiphilic nature of the surfactants, they have the tendency to get accumulate at liquid/air interface to lower the surface tension of the solvent at lower concentration. On increasing the concentration, these surfactants aggregate tightly to make interface saturated and at higher concentration, surfactants can form micelles in the bulk, i.e., solvent. The relation between the surface tension and surfactant's concentration for micelles formation is depicted in Fig. 1.4.

Self-assembly of surfactant amphiphilic molecules into microscopic aggregates (micelles) in solution is a well-studied phenomenon [9]. The sudden change of a physical characteristic in a very narrow range of concentration is typically used

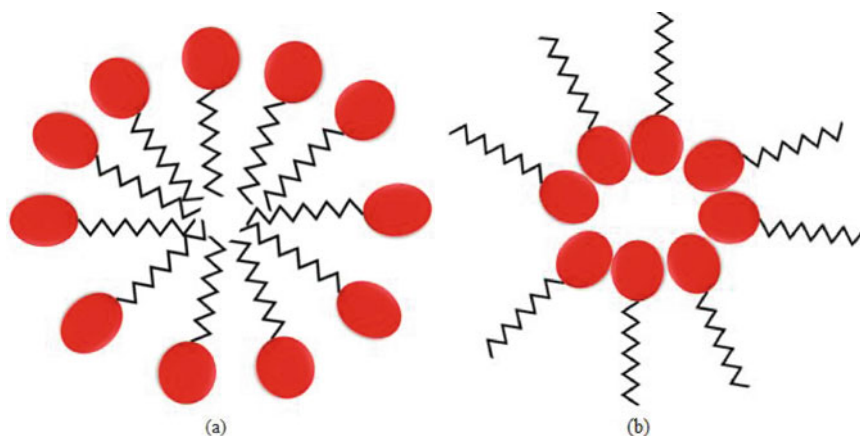


Fig. 1.2 a Normal micelle in aqueous medium and b reverse micelle in non-aqueous medium

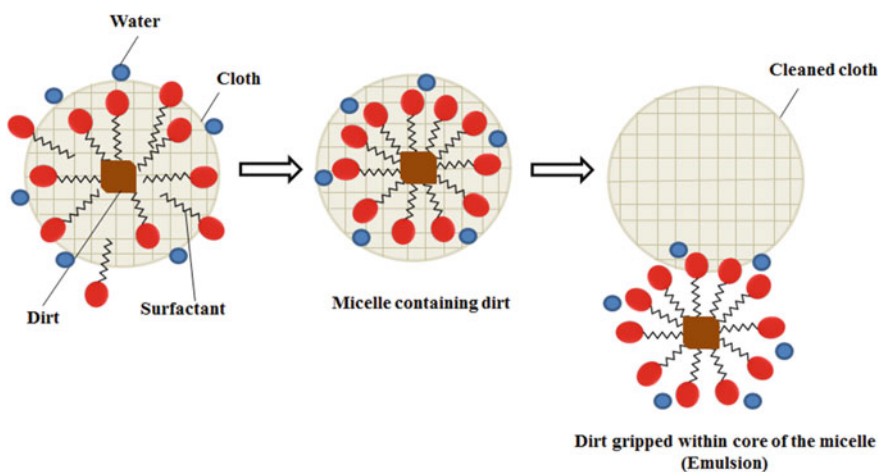


Fig. 1.3 Schematic representation of cleaning action of surfactants

to calculate the critical micellar concentration (CMC) values. The structure of the surfactant, addition of an electrolyte and various organic compounds, solvent used, presence of a second liquid phase and temperature are the variables that influence the value of CMC [4].

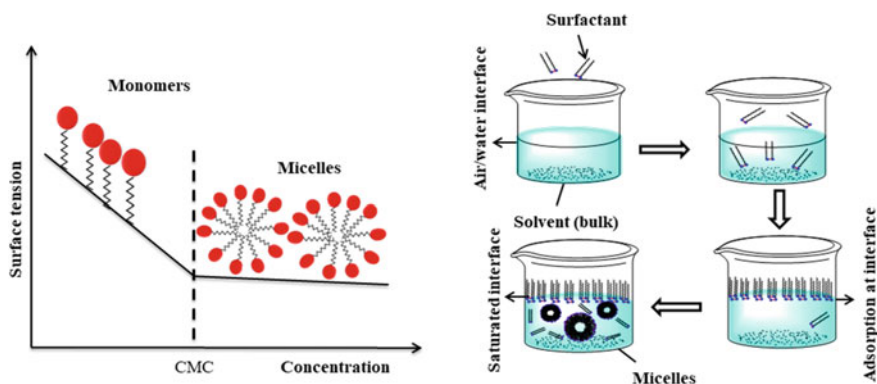


Fig. 1.4 The relationship between the surface tension and concentration forming a micelle

1.2 Properties of Surfactants

Due to the structure and amphiphilic nature, the surfactants have potential to reduce interfacial tension [10]. Some of the important properties of surfactants are discussed below.

1.2.1 Kraft Temperature and Cloud Point

Above specific temperature, a sudden increase in the solubility of surfactant in given solvent is known as kraft temperature [11]. At kraft temperature, the solubility of surfactant becomes equal to the CMC. For applications and uses of surfactants, kraft temperature is a very crucial parameter. Micellization process could occur at temperatures above the kraft point. Kraft points are frequently absent for non-ionic surfactants. Instead, non-ionic surfactants become less soluble as temperature of surfactant solution is elevated and start to lose their ability to operate as surface-active agents above certain temperature, known as cloud point. At cloud point, there is tendency of surfactant solution to separate phase into one extra dilute solution and one highly concentrated micellar solution of homogenous micellar solution [12]. Large and flat micelles are formed by the branching of surfactant therefore by branching, cloud point would be decreased [13]. Kraft temperature is determined visually to be the point at which the solution system is fully clarified and necessary to dissolve the hydrated solid surfactant completely [14]. The solubility of the surfactant for micellization does not take place beneath the kraft point. Enormous number of surfactants from micelles can be scattered above the kraft point and solubility of surfactants increases considerably. Rich phase of micelles of surfactants that are swollen segregate above the cloud point and a noticeable increase in turbidity is observed [15].

1.2.2 Adsorption

Adsorption, the surface phenomenon in which partitioning of the adsorbate species between the interface and the bulk, can take place if the surfactant favours the interface over the bulk solution energetically [16]. The assemblage at surfaces and interfaces by the surfactants to enhance solids dispersion stably in various media has been broadly used. Due to the ambivalence nature of surfactants, i.e., the hydrophobic or tail group which is comprised of long hydrocarbon chains and the hydrophilic or polar head groups, their well-defined characteristics raised [17]. By the hydrophobic/hydrophilic interaction, where surface of single wall nanotubes bundle adsorbs by the hydrophobic tail of molecule of surfactants and on the other hand, termination of water is related with the hydrophilic head of surfactants [18]. Adsorption at the interfaces of the molecules of surfactants is because of the hydrophobic effect though at small concentration of the surfactants [15].

1.2.3 Foaming

Considerable amount of energy input is needed to form bubbles in liquids and mainly in water to get the better of high surface tension and increased surface energy at the revealed gas interface. With a tendency to settle with time excepting that these foams are stabilized kinetically or mechanically by the additional processes and are assembled costly and carried on toughly [19]. The system of a liquid/fluid in which gas is distributed is known as foams. By the collection of bubbles of gas isolated from liquid films, these foams are formed [20]. The primary mechanism influencing the foaming capabilities of surfactant solutions can be characterized as surfactant adsorption at the air/liquid interface. The surfactants produced foams that became more stable as the hydrophobic chain lengthened [21]. Both the potential of a liquid to foam and the stability of the ensuing dispersion are caused by these surface-active macromolecules, which adsorb at the gas/liquid interfaces [22].

1.2.4 Emulsification

Due to the amphiphilic nature of the surfactants, these are used as excellent emulsifying agents for the preparation of water in oil or oil in water types of emulsions. Emulsions are useful for food products, personal care and cosmetics formulations. The purpose of many cosmetic products is to add oily ingredients to the skin and hair. Because of their irritating characteristics and concentrated nature, these components are frequently ineffective when utilized directly. As a result, surfactant-based emulsions are essential in the formulation of cosmetics. Surfactants or emulsifiers are used to help mix and balance the mixes [23].

1.2.5 Wettability

A solid surface becomes wet when a drop of liquid is placed on the surface or a finite contact angle is formed at the surface and complete wetting occurs. Partial wetting is defined as the situation in which contact angle is between 0° and 90° . On the other hand, if the surface does not get wet by liquid, then the contact angle is larger than 90° and the situation is known as non-wetting [24]. The different wetting situations on the solid surface of liquid molecules are depicted in Fig. 1.5.

When the inclination of the surfactant, which is to be adsorbed is in such a manner that hydrophobic moiety point off from the surface, there would result a decrease in water wetting and increase in oil-wetting along the surface. Moreover, if the group having polar head orientation away from the surface, the result will be increased in water-wetting [15]. By the hydrophilicity of the surface, wettability is essentially controlled. Most hydrophilic surfaces have very less contact angle. The thermodynamic outcome of connected phase interaction is wettability. By controlling the values of surface tension, wettability can be managed with the help of surfactant and in addition to liquids selection and solid interface. Oils and other organic phases are repelled by hydrophilic surfaces while water is repelled by hydrophobic surfaces. As a result, even though oil phases may be present when water and oil are combined, the water in the combination will adsorb to hydrophilic surfaces like steel, corrosion and precipitated salts produced layers. But as a surfactant adsorb to a surface it might modify wettability. The character of surface can be changed by surfactants in some illustrations from hydrophobic to hydrophilic [25]. Because of its gradually growing practical importance in various applications, researcher find it tough to wet low energy solid surfaces is challenging, nevertheless, due to the energy mismatch between the solid surface and the polar liquid. Typically, various surface-active agents are utilized to enhance the wetting property [26]. The influence of surface energy and surface tension in wettability is shown in Fig. 1.6.

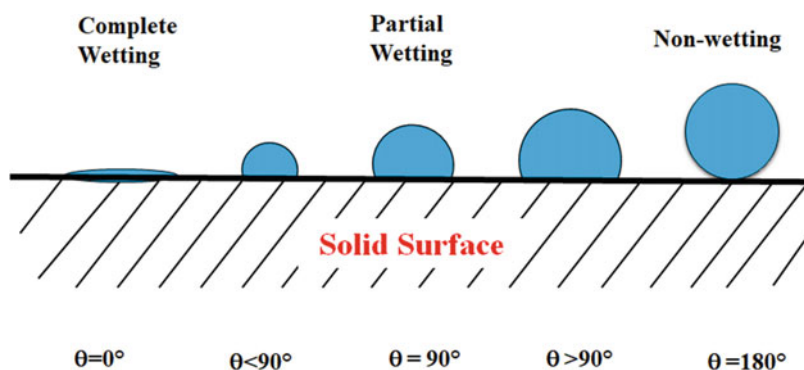


Fig. 1.5 Effect of contact angle on wetting of the surfaces

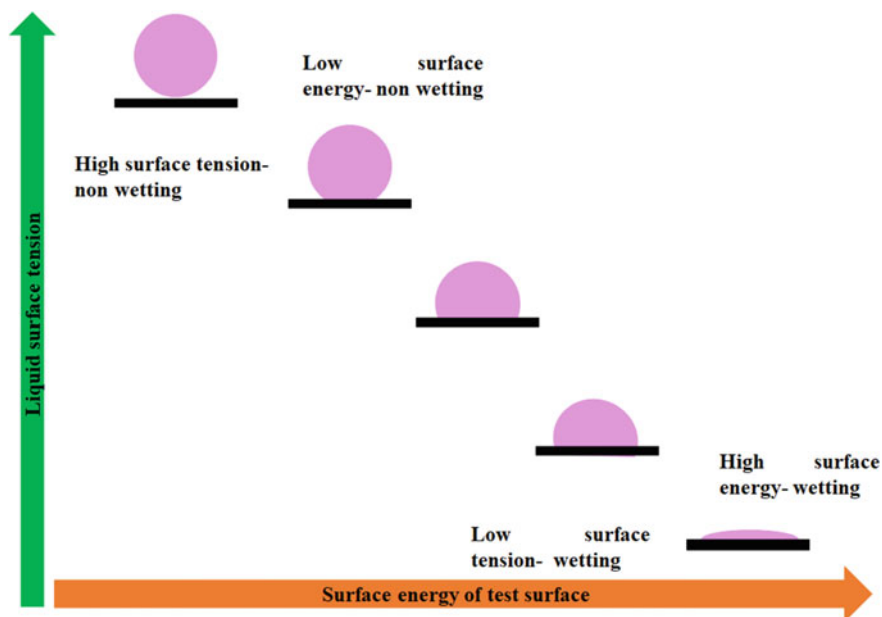
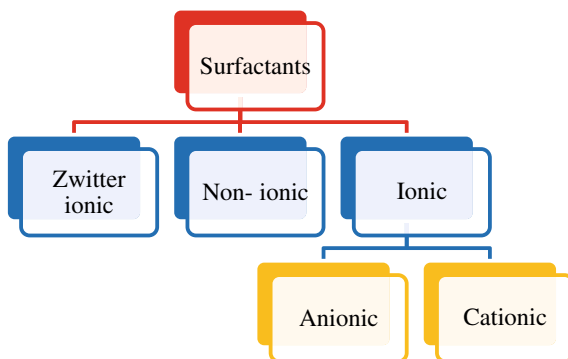


Fig. 1.6 Influence of surface tension and surface energy in wettability

1.3 Classification of Surfactants

The classification is mainly based on the charge of the polar head groups. Broadly surfactants are classified into ionic, non-ionic, and zwitter ionic as shown in Fig. 1.7:

Fig. 1.7 Classification of surfactants



1.3.1 Cationic Surfactants

In these substances, head group is positively charged [27]. The surfactant's market as well as industrial formulations contain cationic surfactants as an important ingredient [28]. About 7% of the whole surfactant market share comprising of the cationic surfactants [29]. Self-assembling is one of the capabilities of these molecules and due to the cationic charge on these amphiphilic molecules; they are used in pharmacy and biomedicine as drug carriers [30]. The structure of cationic surfactant is comprised of cationic head group and hydrophobic tail, which is depicted in Fig. 1.8.

The characteristics of cationic surfactants are adaptable in many fields like corrosion inhibitor for metal, anti-electrostatic, in oil field as bentonite inhibitor and also act as tectonic and mesoporous template [5]. Beside their surface-active properties, these surfactants are used as lubricants, cationic softeners, antistatic and retarding agents and also show antibacterial activities [31]. Because of its bacteriostatic properties, this class of surfactants has become more significant. As a result, cationic surfactants are used as antiseptic and de-infectant agents in pharmaceuticals and personal care products [32]. Some of the examples of the cationic surfactants with their structures are depicted in Fig. 1.9 as follows.

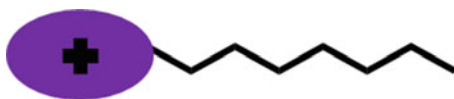


Fig. 1.8 Structure of cationic surfactant

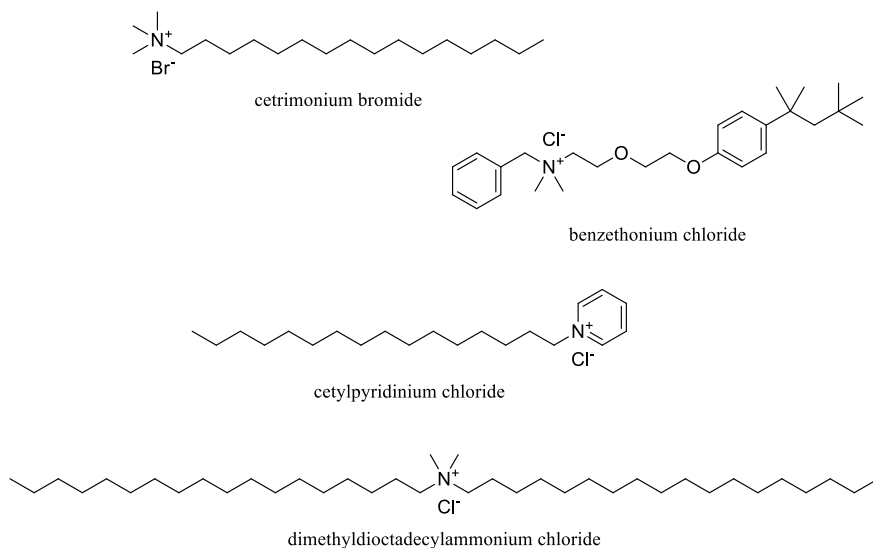
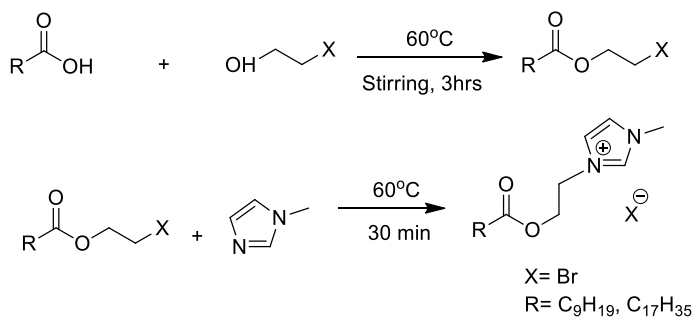


Fig. 1.9 Some of the important cationic surfactants with their structures



Scheme 1 Synthesis of capric and stearic acid-based cationic surfactants

The cationic surfactants are also attaining significance in the high technology field mainly in the preparation of liposomes for their application in non-viral gene liberation, cationic surfactants are used for the number of stabilization problem and have established many technical applications because of the most important negatively charged nature of normal colloids and surfaces. Some of the most essential application of cationic surfactants includes softeners, cosmetic product, electrode coating and the stabilization of epoxy resin polymer latexes as well as in removal and manufacturing [29].

Capric and stearic imidazolium surfactants have synthesized by using two steps. In the first step of the synthesis, alcohols reacted with fatty acids in the presence of catalytic amount of sulphuric acid to get ester and in the second step, the esters which are formed in the first step were reacted with 1-methyl imidazole to get monomeric imidazolium surfactants [33]. The electrochemical behaviour, antimicrobial activities, surface activities, and effect of temperature on micellization of these synthesized surfactants have been well reported [34], for electrical behaviour, cyclic voltammetry technique has been utilized [35, 36]. The synthesis scheme of capric and stearic acid-based cationic surfactants is depicted in Scheme 1 as follows.

1.3.2 Anionic Surfactants

The anionic surfactants contain a negatively charged head group with alkyl chain [37]. Anionic surfactants are powerful cleaners and even though these are superior in pull out the dirt and sebum [38]. The presence of sulphate, sulfonate, carboxylate, betaines groups, the anionic surfactants are the most usual for household purposes [39]. The structure of the anionic surfactant is depicted in Fig. 1.10.

The significant components of the presently accessible cleaning agents and other products are dodecyl benzene sulfonates (DDBS_o) and dodecyl sulphate (DDS), which are mostly used [40]. Some examples of anionic surfactants with their structures are shown in Fig. 1.11. These surfactants are found in toothpaste, laundry detergents, washing liquids as these are the everyday feature of most of the modern

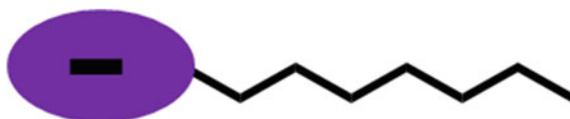


Fig. 1.10 Structure of anionic surfactants

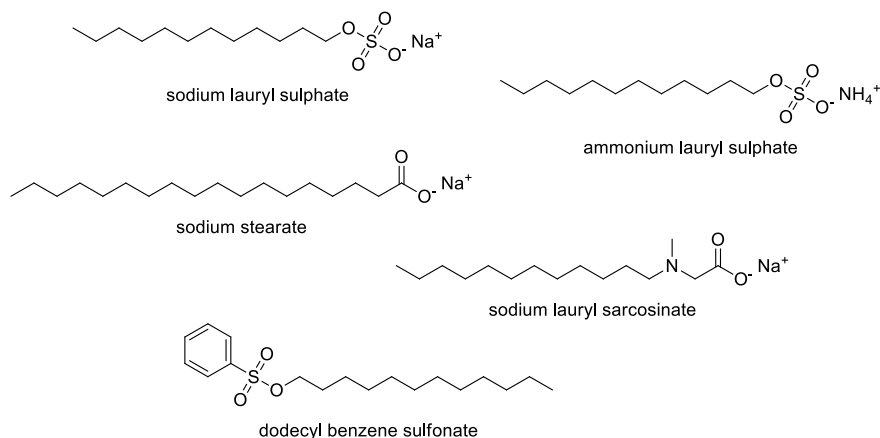


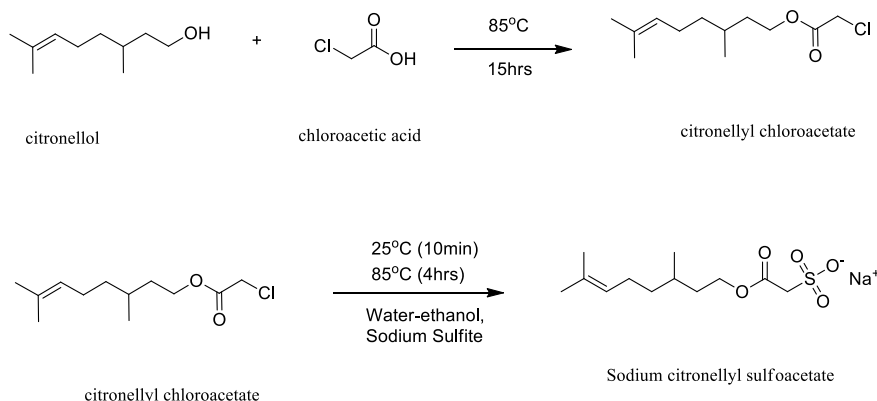
Fig. 1.11 Major anionic surfactants with their structures

households [41]. At low concentration, anionic surfactants are found in monomeric form in both polar and apolar solvents [42].

The citronellol-based biodegradable anionic surfactants have been synthesized [43]. Citronellol is reacted with chloroacetic acid under solvent-free condition for 15 h at 85 °C. Then, work-up was done to get citronellyl chloroacetate. Then, this citronellyl chloroacetate is stirred with ethanol at room temperature and aqueous solution of sodium sulphite is slowly added to round bottom flask and the reaction is stirred for 4 h at 95 °C and work-up was done to get the desired product. The synthesis scheme of anionic surfactants is depicted in Scheme 2.

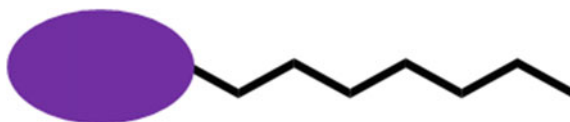
1.3.3 Non-ionic Surfactants

Surfactants that are non-ionized in aqueous solutions because their head group are non-dissociable [44]. Numerous factors, including hydrogen bonds and van der Waals interactions, have an impact on the solubility of non-ionic surfactants. Polyoxyethylene stearyl ether, nonylphenol, propylene glycol monostearate, alcohol ethoxystearate, fatty acid ethoxylates and alkylphenol ethoxylates etc. are a few examples of non-ionic surfactants [45]. Non-ionic surfactants are typically used as emulsifiers, predominately in cosmetic products meant for sensitive skin, baby skin, as well as for



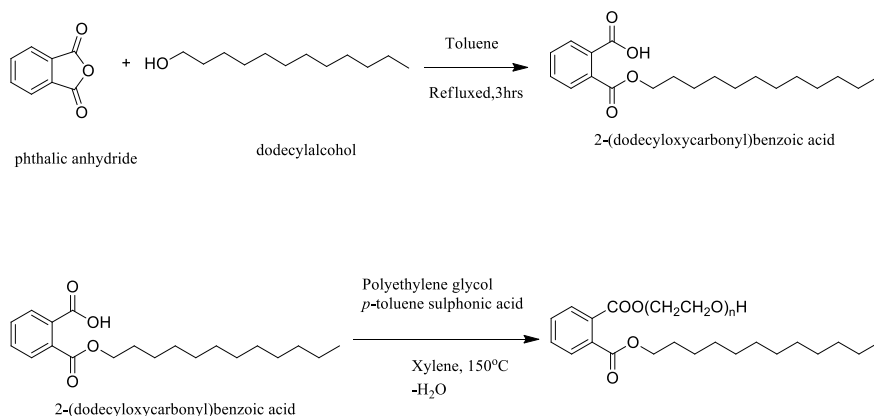
Scheme 2 Synthesis of citronellyl-based anionic surfactants

Fig. 1.12 General structure of non-ionic surfactant



everyday skin care. Non-ionic surfactants may work as cleansing agents (mostly in combination with anionic surfactants). Their main quality is compatibility with skin and eyes, and depending on the kind, they have weak to moderate foaming properties [46]. The hard water contaminants do not react with non-ionic surfactants (Ca and Mg ions). Compared to anionic, these are more useful for replacing organic dirt and oil. Non-ionic surfactants are utilized in a variety of industrial processes, including the formulation of agrochemicals, emulsion polymerization, and fabric-washing detergents [45]. Non-ionic surfactants are regarded as the most gentle surfactants as these are less toxic in nature [47] (Fig. 1.12).

The simplest non-ionic surfactants are synthesized by two-step processes [48]. In the first step, the mixture of phthalic anhydride and dodecyl alcohol was heated and refluxed for 3 h in dry toluene to get 2-(dodecyloxycarbonyl)benzoic acid. In the second step, the obtained product in the first step was esterified with polyethylene glycol of different molecular weights in the presence of xylene as a solvent at 150 °C and *p*-toluene Sulphonic acid was used as catalyst. The mixture was refluxed and stirred until the theoretical amount of water was collected. Then, workup was done to get the final product. The synthesis scheme of non-ionic surfactants is depicted as follows in Scheme 3.



Where:

n=4,6, compound I

dodecyl 14-hydroxy-3,6,9,12-tetraoxatetradecyl phthalate

n=9,0, compound II

dodecyl 26-hydroxy-3,6,9,12,15,18,21,24-octaoxahexacosyl phthalate

n=13,6, compound III

dodecyl 41-hydroxy-3,6,9,12,15,18,21,24,27,30,33,36,39-tridecaoxahentetracontyl phthalate

Scheme 3 Synthesis of non-ionic surfactants

1.3.4 Zwitter Ionic Surfactants

These surfactants possess both negatively and positively charged species within same molecule having long hydrocarbon chain [49]. These surfactants are more stable against acids, bases, electrolytes, and water hardness causing substances. Zwitter ionic surfactants are compatible with cationic, anionic, and non-ionic surfactants [50]. They have long hydrophobic tail and hydrophilic groups with both positive and negative-charged species [51]. The pH has a significant impact on their surfactant characteristics. For instance, the cationic form predominates in acidic circumstances, enabling surfactant substantivity. Normally utilized as foam stabilisers, thickeners and tend to lose these qualities when present with acidic substances [46]. These amphoteric surfactants have pH balance qualities and are relatively gentle, making them ideal for usage in shampoos and cosmetics [45]. The structure of the Zwitter-ionic surfactants with positive and negative head group and hydrophobic tail is depicted in Fig. 1.13.

Some common examples of zwitter-ionic surfactants with their structures depicted in Fig. 1.14.

Fig. 1.13 Structure of Zwitter-ionic surfactants



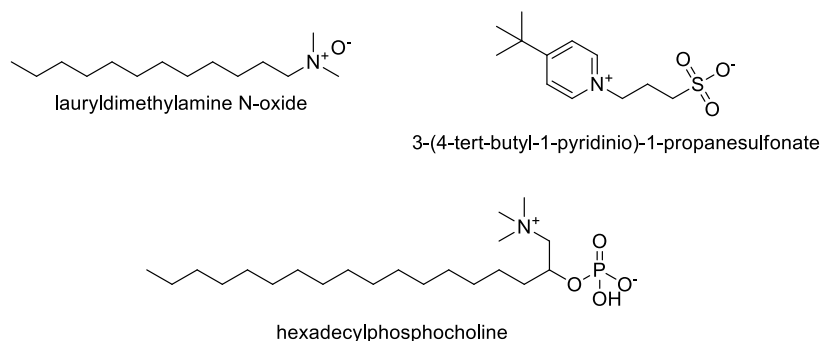
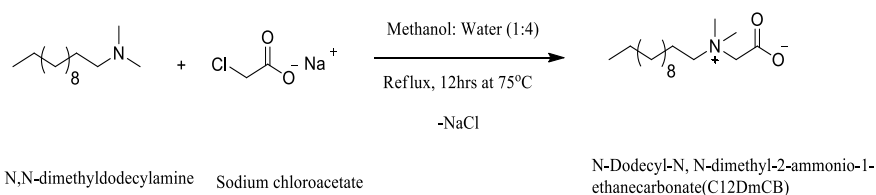


Fig. 1.14 Common zwitter-ionic surfactants along with their structures



Scheme 4 Synthesis of zwitterionic surfactants

Kumar and Mandal [52] synthesized zwitterionic surfactants in step using *N,N*-dimethyldodecylamine and sodium chloroacetate using the mixture of methanol and water as solvent. The reaction mixture was refluxed at 75 °C for 12 h. Then, the workup was done to get the pure products. The reaction scheme for the synthesis of C12DmCB (*N*-Dodecyl-*N,N*-dimethyl-2-ammonio-1-ethanecarbonate) is depicted in Scheme 4 as follows.

A new class of surfactants has come to the market, i.e., gemini surfactants. Gemini surfactants are getting attention from various fields like academic, research and industrial. They have gained the global community attention due to better physico-chemical properties and better applications in multiple fields in contrast with conventional monomeric surfactants [53, 54]. The general representation of structure of gemini surfactants is shown in Fig. 1.15.

These gemini/dimeric surfactants have shown exceptional characteristics in CMC, interface property and better wetting properties [55]. Gemini surfactants have shown good applications in the fields like skin care, life sciences, medicinal and petrochemical industry, construction of porous material etc. [56]. Gemini surfactants have applications in nanoparticle synthesis because the size and shape of nanoparticles can be handled more easily by selection of headgroup, spacer and counterion of the gemini surfactants. Gemini surfactants are used as phase transfer catalysts in reactions due to their amphiphilic nature. They are also used as microemulsion stabilizers. Gemini surfactants have good applications in environmental protection due to their unique features such as low CMC, low kraft temperature and better

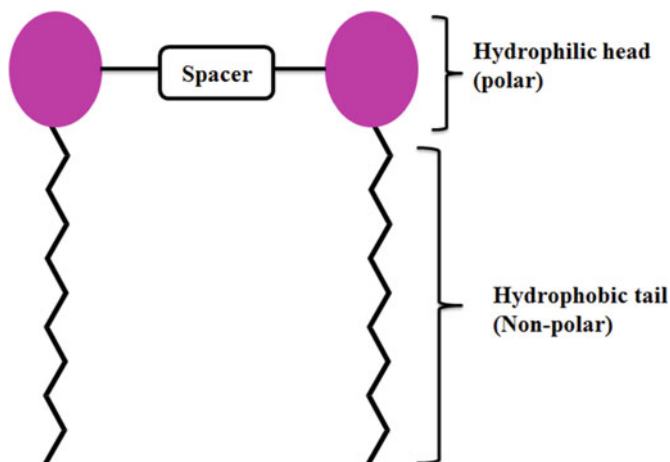
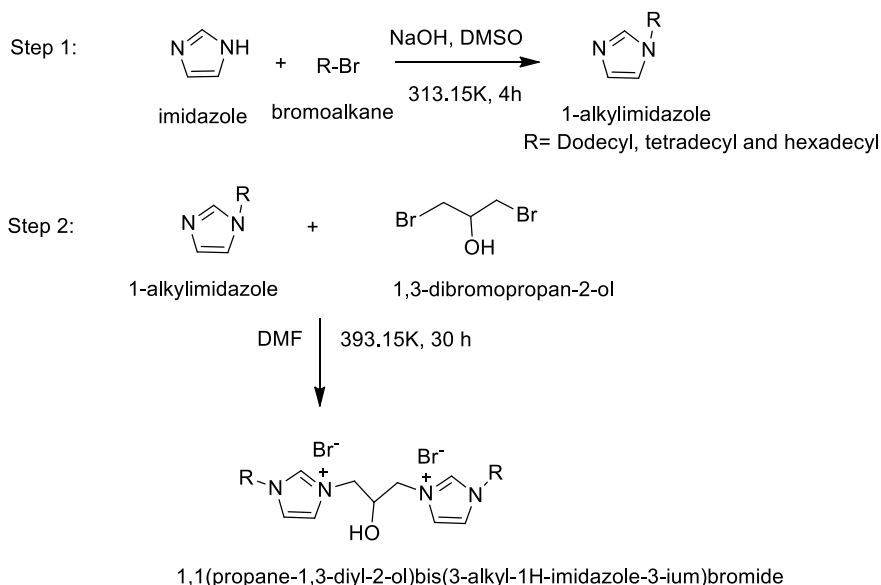


Fig. 1.15 Structure of gemini surfactant

wetting properties. Removal of heavy pollutants, hydrophobic organic pollutants and radionuclides from soil also come under the applications of gemini surfactants [57]. A series of imidazolium-based gemini surfactants with different chain lengths have been synthesized by two step reaction pathway. In the first step, deprotonation of imidazole with sodium hydroxide in the presence of DMSO takes place followed by the addition of 1-Bromododecane to mixture and stirred continuously for 5 h formed 1-dodecylimidazole. In second step, 1-dodecylimidazole and 1,3-dibromopropan-2-ol as a spacer added with 5 ml of DMF and refluxed with stirring at 393.15 K for 30 h to give the 1,1(propene-1,3-diyl-2-ol)bis(3-alkyl-1-*H*-imidazole-3-ium)bromide gemini surfactants [58]. The synthesis scheme of gemini surfactants is depicted below in Scheme 5.

1.4 Applications

One of the most utilized families of chemical molecules and a significant group of industrial compounds called surfactants is employed in practically every industry today. Within the US chemical sector, the demand for surfactants surged by nearly 300% during the previous 10 years. Global production already exceeds three million tonnes annually (at an estimated cost of \$4 billion USD), and by the end of the century, it is anticipated to reach over four million tonnes. Only 32% of the overall surfactant output is intended for industrial usage, with approximately 54% going into laundry and home detergents [59, 60]. Surfactants are widely used in industry (textile, biotechnology, and pharmaceutical) and households due to their unique qualities (detergents, health, and beauty aids) [61]. It has many applications in the field of research, industries and in household products like soaps, detergents, cosmetics,



Scheme 5 Synthesis of imidazolium gemini surfactants

personal care, cleaners, pesticides etc. [62]. The making rate of surfactants expands by 5.4% in the middle of 2016 and 2021 registered by CAGR (Compound annual growth rate) [61]. These surfactants are excellent candidates for a variety of bio-medical fields, including dermatology, drug delivery, anticancer treatment, surfactant therapy, vaccine formulation, personal hygiene care products, and many more due to their special qualities and capacities [63] (Fig. 1.16).

1.4.1 Cosmetics and Personal Care

The science of cosmetics can be summed up as the study of substances that are applied to the human body or any of its parts with the intention of enhancing, encouraging, or otherwise changing the appearance [64]. Hundreds of chemicals are used in the production of products of consumers in personal care industry (Lukic et al. 2016). Surfactants are widely used in the ingredients of the cosmetics and personal care products. Their affordable properties which are used as a function of cosmetics make stable and comparable possible products [62]. Surfactants are mostly used in cosmetics as moisturising thickeners, cleansing agents, conditioners, foaming agents, and solubilizers. In addition to these functions, they are also used to create a wide range of dispersed systems. Almost all the surfactant uses in the process sector, such as the formulation of cosmetics and personal care products, emulsion polymerization,