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Interfacial Behavior of Saponin Based Surfactant for Potential Application in Cleaning

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Abstract

Amphiphilic molecules having a tendency to decrease the surface tension of the aqueous medium and those are widely used in the industrial and domestic sector. Nowadays they are in high demand to replace synthetic surfactants by naturally based molecules to reduce the environmental problem. Approx. more than 60% materials which are of surfactants-based enter into the marine water which dangerous for aquatic lives. We propose novel material which is a natural based surfactant which is biodegradable and eco-friendly alternatives. Here we are focused on tea saponin and investigated properties like surface tension, foaming, skin mildness, cleaning ability. This is maybe the first reporting a surfactant activity of tea based surfactant. Natural originated surfactants display well emulsion making capacity at the large amount as compared to synthetic. Tea is acidic in nature and it reduces surface tension to 31.4 mN/m, and greater foam, ultra-mildness, with excellent cleaning ability. The consequences suggest that tea have outstanding surface-activity which can be used as a green replacement for synthetic surfactants.

Keywords: green surfactant, saponin, foaming, emulsification, mildness

1. Introduction

Amphiphilic molecules contain hydrophilic and hydrophobic units that improve interfacial properties. This characteristic of surfactant makes it suitable for the fields of detergent, wetting, emulsification, oil recovery, froth flotation and other fields [1–3]. A large number of non-natural surfactants are used in industrial and domestic work which are spread over underwater, soil, sediment, etc. Studies have shown that more than 60% of surfactant derivatives are released into the aquatic environment. Global surfactant production was approximately 12.5 million tons in 2006 [4], while production in Western Europe that is more than 3 million tons in 2007 [5]. After that year of 2010, the use of non-ionic surfactants (polyethoxylated nonylphenol) in the United States was approximately 172,000 tons [6]. These synthetic surfactants can disturb the environment and cause health risks such as respiratory tract, dermatitis, eye irritation, etc. [7, 8].

Ostroumov expressed how non-biodegradable cleaning product can reduce the cleaning capacity of oysters and mussels. Reduce the Water purifying capacity of bacteria like *Crassostrea gigas* and *Mytilus galloprovincialis* which cause an important

impact on the ecosystem. Besides, aquatic plants, microorganisms, humans, also affected by the non-biodegradable surfactants [9].

Due to technological advancement methods for the mass production of surfactants are emerging, which have led to serious environmental problems [10]. Today's demand, surfactants should be biodegradable and have minimal toxicity to be surface active. Therefore, researchers are looking for environment-friendly surfactants, it may be a natural surfactant. Natural materials are gained from natural sources, such as plant, bacterial or fungal, animal fats. Various methods are needed, for extract and using them. The fatty acid esters or amides of these sugars can be used as substitutes for synthetic surfactants, as described by Salati et al. Humic acid extraction using biomass as a surfactant has been reported [11].

Saponins are well known plant-based surfactants. Natural surfactants have biodegradability, biocompatibility, and low toxicity, so they are less harmful to the environment [12]. These products can be manufactured with high production with cost effect which could be used in environmental control actions such as processing industrial emulsions, controlling oil pollution, detoxifying industrial wastewater, and bioremediation of contaminated soil [13].

Here we report that tea saponin is considered as a natural surfactant. A recent movement in the industry to rejecting used of synthetic products which inspiration for people to look for natural-based new materials. By these inspirations, we examined tea to estimate its physiochemical property such as critical micelle concentration (CMC), Foaming, viscosity behavior, emulsification, mildness pH, and conductivity. For comparison, we also examined the marketed product (ionic surfactants) just because they are commercially availability. We can reduce environmental issues just by replacing them with natural surfactant.

2. Experimental procedure

2.1 Materials

Tea saponin powder was purchased from King diamond (65% approx.). Surf excel (Hindustan Unilever Limited), as a marketed surfactant, Soya bean oil (refined, batch number (AF) SB06C04), Coconut oil (Lot No. KB003), Paraffin wax (candles from the market) were used. Hexane was obtained from Loba Chemie. Milli-Q water of surface tension 71.6 ± 0.1 mN/m was used. Bleached cotton cloth Sort No. 22425003 from Akash Textiles, Ahmedabad.

2.2 Surface tension measurements

Surface tension of the individual surfactant was determined by a force tensiometer (*Type: K20, KRÜSS, Germany*) using a platinum plate method. The experiment was directed by using 25 mL of surfactant solution in a sample container. In this method, the liquid to be inspected is placed in a container, and its position can be changed by using a screw to move up and down. There is a platinum plate on the top of the sample. During this process, to ensure the plate is accurately immersed in the liquid alignment of platinum plate adjusted at 3 mm distance from the liquid. In the tensiometer, a pre-programmed software archives the surface tension of liquid under examination. For accuracy before apiece measurement instrument was calibrated using Milli-Q water (71.5 ± 0.1 mN/m) at $27 \pm 0.5^\circ\text{C}$. After the completion of each reading, the platinum plate was carefully washed using deionized distilled water, followed by heating to ensure that the water evaporated from the surface of the plate. All measurements were recurrent three times to accord repeatability of the data.

2.3 Foam ability and stability

The Ross Miles method was utilized to study the foaming behavior of surfactant. To study the foaming behavior 250 mL surfactant solution was prepared. Primary step to perform foam analysis was to wash the column with deionized water followed by rinsing of the column using surfactant solutions under examination. Afterwards, from 250 mL prepared surfactant solution gradually 50 mL of surfactant solution poured into the column and was stored at the bottom part of column. The remaining 200 mL surfactant solution was taken into the pipette and pipette containing surfactant solution positioned at top of the column. Then, faucet of pipette opened and at fixed flowrate surfactant solution run off into the column. As soon as the surfactant solution from pipette completely transfer to column, start the stop watch and note the initial foam height (termed as foamability) and after 5 min note foam height again (termed as foam stability).

2.4 pH and conductivity

pH and conductivity were measured by Laqua pH 1100 (Horiba) at room temperature. The pH and conductivity electrode was calibrated with the help of standard solutions previously measurements.

2.5 Viscosity

Vibration viscometer (SV-10 series, A & D Company, Limited) was employed for viscosity measurement of samples. The working principle of instrument to determine viscosity followed constant parameters like frequency 30 Hz and amplitude <1 mm, at these fixed parameters the required current to resonate sensor plates used to detect viscosity.

2.6 Emulsification

This measurement carried out by the Kothekar method [14]. Initially 10 mL of sample solution and 10 mL of refined oil was taken into a graduated cylinder followed by shaking of this mixed solution. Now for this emulsion time required to separate 5 mL of sample solution was determined and denoted as emulsion persistence.

2.7 Cleaning

To determine the cleaning efficiency the Sharma method [15] was considered, which involved four steps. Firstly 5 × 5 cm sized cotton cloth was soaked in water for 12 h, dried and weighed. In second step simulate dirt was prepared by maxing 0.5 g coconut oil and 0.5 g paraffin in 50 mL of hexane. Now, in third step the dry cotton cloth was two times dipped into the simulated oil, dried and weighed. Lastly the cloth was treated with surfactant by soaking it for 10 min followed by washing, drying and weighing. To determine cleaning efficiency different surfactant concentrations ranging from 0.01% to 0.1% were utilized at room temperature.

2.8 Protein solubilization

The protein (zein) solubilization potential of surfactant was quantified by the gravimetric analysis. The 1.0% by weight surfactant solutions were prepared in vials and approximately 2.0% by weight zein powder added to the solution. Now

these mixtures of surfactant-zein protein were continuously stirred for 24 h. After 24 h continuous stirring the solutions were filtered using Whatmann filter paper to collect insoluble zein from solution. Lastly the collected insoluble solid zein was desiccated at 80°C for 24 h and weighed. Using the insoluble zein powder weight the solubilized zein% has been calculated.

2.9 Lipid solubilization

The lipid (stearic acid) solubilization potential of surfactant was quantified by the gravimetric analysis. The 1.0% by weight surfactant solutions were prepared in vials and approximately 2.0% by weight stearic acid powder added to the solution. Now these mixtures of surfactant-stearic acid were continuously stirred for 24 h. The surfactant-stearic acid mixture was then filtered using Whatman filter paper. Finally, the collected solid was dried at 60° C for 24 h. The weight percentage of the solubility of the lipid in the surfactant was calculated from the weight of the insoluble stearic acid after drying for 24 h.

3. Microstructure

Microstructure of gas bubbles was measured through an Olympus STM7CB Digital microscope. The foam was generated by using the handshaking method.

4. Results and discussion

4.1 Surface tension

The surface tension creates an imbalance in the intermolecular force in an interface between liquid, vapor or liquid and solid. **Figure 1** shows that the reduction in surface tension due to surfactant concentration increment and after certain concentration surface tension value was constant as this concentration is called the critical concentration of micelles (CMC). The reduction in surface tension is due to the break of hydrogen bonds aqueous, the reason was increased adsorption of monomer

Figure 1. Plot of equilibrium surface tension versus total surfactant concentration at a temperature of 27 ± 0.5°C.

Name of surfactant	CMC (wt%)	γ_{CMC}	π_{CMC}
Surf excel (surf)	0.07	31.5	40.5
Tea saponin (tea)	0.04	31.4	40.6

Table 1.
Surface properties of surfactants.

at the air-water interface [1]. The surface tension reduction value between 32 and 37 mN/m consider as the material has good detergency and surface activity [16]. Therefore, tea has a decent washing ability.

The properties of surfactants, such as detergents, solubilizers, etc., depending on their structure. The reduction in surface tension caused by natural surfactants is almost close [17] to the surface tension obtained by measuring surface tension (**Table 1**). The ionic surfactants tend to have relatively greater CMC value as of non-ionic surfactant (as observed for surf), such behavior of ionic surfactants may be attribute to the repulsion between neighboring analogous head group charge. The tea saponin reveal greater capability to reduce surface tension as compared to surf may be due to absence of elementary electrostatic interaction in tea saponin and this also resembles faster micelle formation in water.

The effectiveness of surfactant (π_{CMC}) is given by π_{CMC} where γ_0 is the surface tension of pure water and γ_{CMC} is the surface tension of solution at CMC. Natural surfactant shows maximum effectiveness followed by synthetic.

$$\pi_{\text{CMC}} = \gamma_0 - \gamma_{\text{CMC}} \tag{1}$$

The dynamic surface tension of surfactants solutions was studied by the pendant drop method. **Figure 2** displays the dependencies of dynamic surface tension for micellar solutions of natural and synthetic as functions of the effective lifetime [18]. The concentrations of surfactant are 0.005 wt% (below CMC). From the result shows that the natural surfactant leads to faster the surface tension changes because the Natural surfactant having no kind of change repulsive force at a head group like surf.

Figure 2.
Plot of surface tension as a function of time at a temperature of $27 \pm 0.5^\circ\text{C}$.

4.2 Foam ability and stability

Foaming behavior and cleaning action is not much interrelated, but foam behavior is a significant condition in cleansing agent assessment by the consumers. Foam creation and durability are imperative in numerous applications [19–21]. Foam ability is the amount of foam creation due to the constant formation of new interfaces. Higher power of foaming requires faster adsorption, high surface elasticity. The foam results obtain by Ross Miles test are shown in **Figure 3** at a concentration range from 0.02 to 0.10 wt% for both surfactant sample. Foam generation by pouring method and the amount of foam formation was considered as Foam ability and after 5 min foam height measures that are termed as foam stability. The tea solution produces dense, high-quality foam, which may be due to the presence of high amounts of saponin. The existence of saponin group contributes to significantly reduce the dynamic surface tension as well as supports to produce the large surface for foam formation [13]. Foam formation upsurges with increased surfactant concentration as greater number of monomers could custom in film to enhanced foam stability.

We have also observed under the microscope to get an insight into the bubble size and foam structures as depicted in **Figure 4**. The foam engendered by surf solution speckled to contain more liquid portion with larger bubble size, while the foam generated using tea solution contained less liquid portion with smaller bubble size. These experimental results lead to better understanding of foam formed using natural surfactant and it indicates that such natural surfactants could deliberately consider as foaming agent for various applications. The foaming behavior studied by utilizing the Ross Miles method [22] and similar results were observed for foam generated by hand shake method as indicated in a picture (**Figure 4**) at 0.1 wt% concentration for both tea saponin and surf.

4.3 Viscosity

Micellization affects the viscosity of a solution, depending on the size and number of particles in the solution. **Figure 5a** shows that the viscosity gradually increases with increasing concentration. The absence of charge repulsion in between the head groups of surfactant monomers may lead to induce viscosity with

Figure 3. Plot of foam height as a function of surfactant concentration in Milli-Q water at a temperature of 27 ± 0.5°C.

Figure 4.
Optical microscopy images of the foam generated from (a) surf (b) tea respectively at 27°C. Inset shows the photo of the foam for the corresponding surfactants.

increased concentration of tea. The hydrophilic part of surfactant monomers surrounded by water assists to a rise in viscous resistance. The viscosity progress may escalate rapidly above CMC due to micelle shape transition at higher concentration. The increase in viscosity far exceeds CMC, which is due to more interactions among micelles, the interactions among micelles are starting to get closer and reduce the critical packing parameters [23].

4.4 Conductivity

Detergent mostly made by ionic surfactants which are ionized in an aqueous condition so it will show conductance from low to high depends on concentration. The change in conductivity at lower concentrations almost constant further increases at higher concentrations for natural surfactant **Figure 5b**. At lower surfactant concentration, the headgroups of surfactant were encircled by water consequently results to lower conductivity. The conductivity upsurges with the increasing concentration of surfactant due to ionization of surfactant molecules [24]. The conductivity also associates with mildness, which demonstrates inversely

Figure 5.

Plot of (a) viscosity and (b) conductivity as a function of surfactant concentration at a temperature of $27 \pm 0.5^\circ\text{C}$.

proportional correlation [25]. The experimental outcome indicates that the conductivity increase at higher concentration for tea ensues to be more as compared to surf.

4.5 pH measurements

In surfactant science research investigation of pH is an indispensable study. The pH of a surfactant solution relies on the overall charge of the headgroup, which consequently changes the repulsion between headgroups [7]. The pH values for both tea and surf solutions for range of concentration 0.02–0.10 wt% in Milli Q water were observed to be 6.7 ± 0.2 . The pH values of the surfactant solution at various concentrations are shown in **Table 2**. Tea with an acidic pH, possibly due to the hydrolysis of nonionic glucuronic groups. This pH of the solution near the skin (~ 5.5) causes less damage to the hair and skin. The surf shows alkaline in nature. The pH of the tea solution decreases with concentration, while that of surf increases.

4.6 Emulsification

The emulsion is a fine dispersion of one liquid into another stabilized by emulsifier such as protein, surfactant, polymers etc. The surfactants can be solubilized non-polar substances into polar due to its amphiphilic nature, e.g. Surfactant

Concentration (wt%)	Surf	Tea
0.02	10.2	5.3
0.04	10.3	5.2
0.06	10.5	5.5
0.08	10.8	5.9
0.10	11.2	6.1

Table 2.
The values of pH at different concentrations of surfactants solutions.

monomers adsorb between the water-oil interface and reduce its interfacial tension. Due to this reduction in interfacial tension less energy is required to form new interfaces that are essential to the preparation of a stable emulsion. In these studies, we make simple oil-in-water (o/w) emulsion, which showed the emulsifying power of the surfactant's solution **Figure 6**. Emulsion stability increases with concentration increases, natural surfactant formed more stable (almost 30 min) emulsions at high concentration. The results of surface tension show that the stability of the emulsion decreases in the area of micelle formation, possibly because less surfactant is adsorbed at the oil-water interface. Tea has the best emulsion stability, followed by the surf. Stable emulsions occur when the adsorbed surfactant makes repulsive interactions among the drops and generates an energy barrier against breakage.

4.7 Cleaning

Cleansing activity means, the removal of unwanted substances such as soil, grease and dirt, is the main target of any detergent. The cleaning activity was calculated by the below equation

$$C = \left[\frac{(W_2 - W_3)}{(W_2 - W_1)} \right] \times 100\% \tag{2}$$

where W_1 = initial weight of cloth, W_2 = weight of the cloth with simulated dirt and W_3 = weight after cleaning with surfactant solution and water. As shown

Figure 6.
Plot of emulsification as a function of surfactant concentration at a temperature of $27 \pm 0.5^\circ\text{C}$.

in **Figure 7** The cleaning ability of the tea showed better at lower concentrations. The cleaning ability increases with concentration increases for natural surfactant. Although all surfactants have similar trends in cleaning performance, there is a significant difference in the amount of soil removed. The tea demonstrations decent cleaning efficacy at high concentration as compared to the surf, probably due to tea revealed greater efficiency to reduce surface tension.

4.8 Skin mildness of surfactants

In the skin structure first part is known as epidermis and the outer most layer of epidermis is stratum corneum (SC), this layer provides an important barrier function for skin. Surfactant can amend the function of SC by interacting with proteins and lipids of SC. These interactions lead to swelling and denaturation, however the comprehensive mechanism involved for such interaction has not been reported yet. But, predisposition of surfactant to interact with proteins could relate with its impact on human skin, generically termed as mildness to human skin [26].

Figure 7.
Plot of cleaning ability as a function of surfactant concentration at a temperature of $27 \pm 0.5^\circ\text{C}$.

Figure 8.
Plot of protein and lipid dissolution by surfactants (1.0 wt%) at a temperature of $27 \pm 0.5^\circ\text{C}$.

To comprehend impact of tea saponin and surf on skin, the solubilization potential of protein and lipid were determined using 1.0 wt% surfactant solution by dissolving model protein zein and model lipid stearic acid represented in **Figure 8**. The dissolution tendency of natural surfactants, zein and stearic acid is small compared to surf. This indicates that natural surfactants are milder than synthetic.

5. Conclusions

Herbal saponins, tea, were studied to find alternatives to synthetic surfactants that are commonly used and were make comparison with marketed available surfactant (Surf Excel). The outcomes demonstrated that saponins are naturally acidic and decomposable. Natural materials are considered to be biodegradable as plant extracts. The tea, which was probably examined by the first time, shows good effectiveness, in addition to high foaming capacity, decent cleaning capacity and ultra-soft. Although lots of works account on isolation, characterization, etc. Tea has good emulsifiers and can find some industrial uses. We also quantified the cleaning capacity of surfactant solutions. Therefore, our studies can offer a simple and inexpensive method to measure the general cleaning method for the evaluation of detergents. We conclude that tea comes with good surface-active properties. These studies can offer useful information for the food industry as well as the cosmetics industry reason was that these plant-based materials were biodegradable organic surfactant. We propose new biodegradable and renewable alternative from plant-based material which act as a surfactant.

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