

DETERMINATION OF FOAMING ABILITY OF SURFACTANT SOLUTIONS AND SELECTED COSMETIC FORMULATIONS

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Task IX-S

DETERMINATION OF FOAMING ABILITY OF SURFACTANT SOLUTIONS AND SELECTED COSMETIC FORMULATIONS

I. Aim of the task

The aim of the task is the experimental determination of the influence of concentration and the surfactant structure as well as the water hardness on the foaming ability of the surfactant solution and selected cosmetic solutions (washing powder, bubble bath, liquid soap, sodium soap).

II. Introduction

1. Foams
 - 1.1. Mechanism of foam formation,
 - 1.2. Methods of foams preparation,
 - 1.3. Characteristics of foams,
 - 1.4. Stability and breaking of foams.
2. Foaming ability of surfactants.
3. Practical application of the foaming process.
4. Foaming agents used in cosmetics.
 - 4.1. Shampoos,
 - 4.2. Foaming bath agents.
5. Water hardness.

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III. Theory

III. 1. Foams

III. 1.1. Mechanism of foam formation

The **foams are dispersions** in which the continuous phase is a liquid and dispersed phase is gas. Both phases are in equilibrium, so that there is no cracking of bubbles on the liquid surface or, if it occurs, it is slow. The foam must have specified durability. Foam has a more or less stable honeycomb structure of gas cells whose walls consist of thin liquid films with approximately plane parallel sides. These two-sided films are called the lamellae of the foam.

In 1888. Quinke showed that pure liquids can not form stable foams. The gas bubble contained in the liquid or solution without the surfactant is not stabilized. As gas density is less than the density of the liquid, according to the Archimedes law existing under these conditions the buoyancy force causes the escape of gas bubbles from the liquid (degassing process).

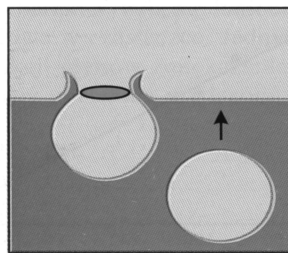


Fig. 1. Aqueous system – air without surfactant [4].

In order to obtain stable foams, in addition to the dissipation phase (gas) and the dispersion medium (liquid) the third compound must be dissolved in the liquid, i.e. foaming agent with stabilizing properties. Its action is due to adsorption of molecules on the interface, thereby reducing the surface tension. **Foaming agents** are mostly the amphiphilic surface-active compounds lowering the surface tension.

In the aqueous solution of the surfactant (liquid phase) the foam-forming agent adsorbs at the liquid-air interface forming thin layer of particles, wherein the hydrocarbon chains are directed perpendicularly to the air, and hydrophilic groups are located in the liquid (Fig. 2).

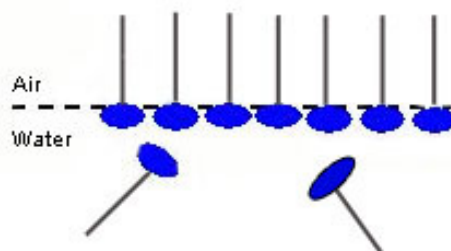


Fig. 2. The molecular orientation of the foam-forming substance at the liquid-air interface.

When the air is injected into the surfactant solution, it is dispersed in the form of bubbles and surfactant is adsorbed on the air-liquid interface forming an **initial monomolecular layer**. Because of their low density, the bubbles rise, lift the adsorption layer on the surface of the liquid and become covered by a second layer on their exterior. Water containing the foaming agent is trapped between these two layers (Fig. 3).

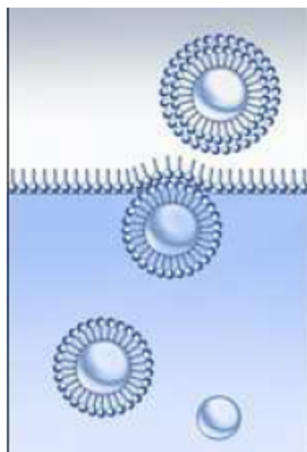


Fig. 3. Foam forming by blowing air into the surfactant solution [5].

A bubble, which rose to the surface of the liquid, called **elementary foam** is covered by a surface film forming two layers of adsorbent between which there is a liquid film. As floating on the surface of the liquid bubble deposit one on another to form a foam. Merging foam bubbles lose their spherical nature creating a foam cell, the walls of which are arranged according to the **Plateau's law**. Fig. 4 schematically shows the combination of three bubbles along the so-called **Plateau border**, which plays a significant role in the process of foam drainage.

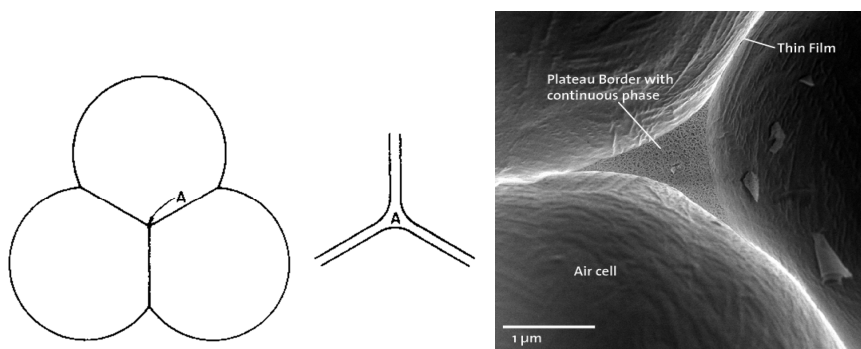


Fig. 4. Plateau border [2, 6].

To maintain the mechanical equilibrium between the three laminae a symmetrical set at an angle of 120° is required because the surface tension of each film is the same. For a two-dimensional system a set of three foam bubbles is permanent. The combination of four bubbles along one edge is shown in Fig. 5.

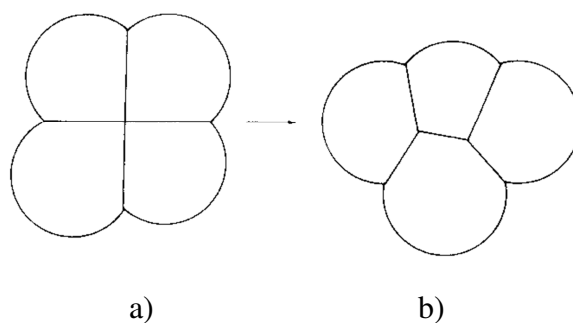


Fig. 5. Connection of 4 foam bubbles [2].

However, such arrangement is undurable and the smallest pressure difference in the adjacent bubbles is enough to change their position and establish a lasting configuration based on the model of three connected bubbles. For the **three-dimensional foam**, the situation is more complicated. In the ideal case where bubbles have the same dimensions, and the gas pressure is everywhere the same spatial structure composed of edge formed by the intersection of three surfaces at an angle of 120° can be expected. Intuitively, it is expected that such foam should consist of regular polyhedrons of pentagonal dodecahedron type. Different coloured foams are the result of interference of light at the surface membranes that behave like thin plates.

III. 1.2. Methods of foams preparation

As in case of other dispersions, foams can be prepared by condensation or dispersion methods.

The condensation methods for preparing foams include: the creation of gas bubbles in the solution by reducing the external pressure, the temperature increase (up to a supersaturated solution), or by chemical reaction.

In the **dispersion methods**, foam is formed by dispersing the gas in the form of bubbles through the capillary tube, porous plate, gauze or paper or by blowing gas through gauze soaked in the solution. The dispersion of gas in a liquid can also be obtained by shaking a vessel partially filled with the solution, a simultaneous flow of gas and liquid through the a glass tube, mechanical mixing of the gas and liquid using a stirrer, perforated disk or other device, pouring the liquid on the surface of the same liquid, suction (absorption) of gas by the flowing liquid.

III. 1.3. Characteristics of foams

The principal characteristics of the foam are: foaming ability, density and durability of foams.

Foaming ability is expressed by the amount or volume of foam formed under certain conditions (such as the method used to produce foam, temperature of solution, hardness of water used, and its electrolyte content).

Foam density is expressed by the ratio between the volume of the solution leading to foam formation and the volume of foam. This property may indicate if foam is wet or drier. **Foam stability** (rate of drainage and breaking) is expressed by its height or volume after a certain period of time after its creation.

III. 1.4. Durability and breaking of foams

The foam is subjected to four major forces that determine its evolution over time:

- **the force of gravity** causes segregation of the bubbles according to their size and the drainage of liquid from the foam responsible for thinning the film separating the bubbles,
- **the capillary forces** which appear when the bubbles become polyhedral and which accentuate the drainage of liquid from the centre of the films toward the edges (called Plateau borders),
- **pressure difference** between the gas bubbles of different sizes; higher pressure inside the smaller gas bubbles causes gas diffusion to the bubbles of larger sizes, where the gas pressure is lower,
- **electric or steric repulsion**, which limits the thinning of the liquid film.

Liquid contained between the adsorption layers of foam trickling under the forces of gravity and surface layers, resulting in a membrane forming foam wall getting thinner. Two mechanisms of foams draining can be distinguished: drainage of gravity and subsequent tubular drain off. The draining rate depends on the viscosity of the film forming foam bubbles and liquid, and film flexibility. Foam drainage can take place either by the surface of the film or by liquid separation on the edge of the films. The drainage of foams forming thick film foams drain off occurs mainly due to gravity drain of the liquid from the foam with the average speed described by Equation (1):

$$v = \frac{\rho \cdot g \cdot \delta^2}{3\eta} \quad (1)$$

where : v – the average rate of foam drainage, δ – the film thickness, ρ – the liquid density, η – the viscosity of the liquid, g – the gravitation.

Foam stability depends mainly on the properties of adsorption layer of surfactant. The more the endothelial surface conformed to the solid construction, the more durable the forming foam is. Durability of the film depends on the speed with which the surface tension changes to allow adaptation to local film tensions and for the difference of tensions in its various areas. This property, called **film flexibility** determines its durability compared to mechanical perturbation and is proportional to the surface excess Γ of the surfactant. Stretching without film interruption must correspond to the increase of surface tension. Therefore, the most durable films are obtained for the concentrations at which fast surface tension changes can occur.

To reduce the foam stability, a film surface should be destroyed. Such effect can be obtained due to liquid **film drainage** under the gravity – foam film becomes thinner and

breaks (evaporation accelerates this phenomenon), or due to mechanical perturbations (under pressure, shake etc.).

Foam stability can also be reduced by replacing the substance forming resistant surface layer with easily adsorbing substance which forms a less stable film. In this way, the foam of the water-air-water type can be broken by the addition of boric acid or valeric acid, which replaces the surfactant molecules in the foam film causing the foam breaking.

The stability of the foam is also influenced by the gas pressure inside the foam bubbles. The domination of the gas phase (ca. 98% of the volume in the dry foam and 87% in the wet foam) means that the foam properties are similar to a gas, and its volume varies inversely to the external pressure. Foam is durable thanks to the gas vapour pressure, i.e. the smaller the bubbles are the higher gas vapor pressure and more stable foam are.

In many cases, also the addition of certain amounts of electrolyte causes foam breaking. This is due to the exchange of ions between the electrolyte and the foaming agent (the latter forms a salt with worse surface properties) or a change in pH to a value at which the foaming agent becomes less active. In the case of ionic surfactants, the addition of salts increases the value of **packing parameter of the surfactant**, increasing the surface activity and often results in an increased tendency for producing foams.

III. 2. Foaming ability of surfactants

The ability of surfactants of foam formation in aqueous solutions is determined by the following factors:

- chemical structure of surfactant and its concentration,
- pH of the solution,
- water hardness,
- the presence of other components in the solution.

The highest potential for foaming is found in the surfactants with an **alkyl chain length** in the range of **12 to 15 carbon atoms**. Surface-active agents with the alkyl chain below 10 and above 16 carbon atoms usually do not show good foaming properties. Similarly foam-ability optimum appears in the case of surfactants containing polyoxyethylene chains. The derivatives containing **10-12 oxyethylene groups** in one surfactant molecule are characterized by the best foaming properties. A kind of surfactant polar group also influences on foaming ability.

Table 1 shows a comparison of the foaming properties of the three main groups of anionic surfactants, for the molecules containing the same hydrophobic chain, the same position of the hydrophilic group and the same cation.

Table 1. Foaming properties of anionic groups in the surfactants [7].

Properties	Polar groups		
	-COO-	-SO ₄ -	-SO ₃ -
Foaming ability	satisfactory	good	very good
Foam stability	very good	good	satisfactory
Facility of foam formation	satisfactory	good	very good

Fig. 6 shows the dependence of the foaming capacity as a function of concentration for the three surface active compounds which are the base for the manufacture of shampoos. It can be seen that for all of them the foam volume increases with the increasing concentration of surfactant.

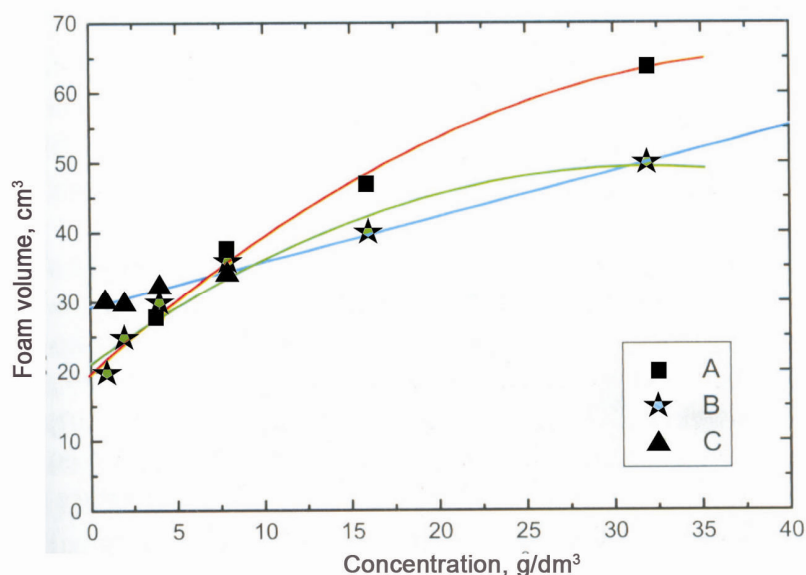


Fig. 6. Foaming ability as a function of the concentration for three bases of the shampoo
A - $C_{12}H_{25}OSO_3Na$, **B** - $C_{12}H_{25}(OCH_2CH_2)OH$, **C** - $C_{12}H_{25}(OCH_2CH_2)_{10}OSO_3Na$ [7].

The nonionic surfactant results in the formation of foams with low durability. The highest stability is exhibited by the foams produced using anionic surfactants, wherein the presence of a polyoxyethylene group in the molecule reduces the foaming capacity, however, it does not reduce the foam stability.

The effect of solution pH on the surfactant ability of foaming depends on the type of foaming surfactant. Within the pH range from 3 to 9 foaming ability for nonionic surfactants practically does not depend on the pH value. In the case of ionic surfactants the ability has a **maximum for a pH between 7 and 10**, depending on the HLB value and CMC of the surfactant.

Fig. 7 shows the stability of the foam for the shampoo bases shown in Fig. 6, at a concentration of 32 g/dm^3 at 20°C .

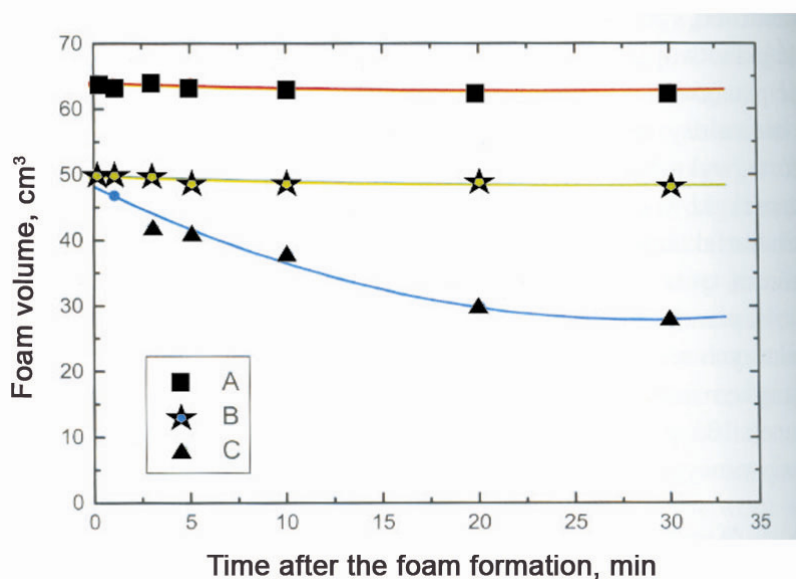


Fig. 7. Stability of the foam for the three shampoo bases at 20°C A - $C_{12}H_{25}OSO_3Na$, B - $C_{12}H_{25}(OCH_2CH_2)OH$, C - $C_{12}H_{25}(OCH_2CH_2)_{10}OSO_3Na$ (surfactant concentration of 32 g/dm^3) [7].

Large water hardness reduces foaming ability of ionic surfactants. The use of soap in hard water causes formation of insoluble precipitates that increase consumption of soap. In the case of application in the textile industry or for laundering they cause stains on fabrics. Deposits of these salts may also block the skin gland duct, and in the case of injury can be settled even in the depths of the wound. Unlike soaps, synthetic anionic detergents with calcium and magnesium cations do not form insoluble precipitates.

Sometimes one can observe the phenomenon of foaming properties synergism for solutions prepared with the addition of surfactants, some insoluble in water nonpolar co-solvents and macromolecular non-electrolytes.

III. 3. Practical applications of the foaming process

The foaming ability of substances is of great importance in many industrial applications such as fire retardant foam, mineral flotation, foam extinguisher of good quality, in the production of cellular concrete (agent generating cells - generating the air or gas cells - improving durability of concrete by increasing resistance to progressive destruction of the following frost and defrost processes) in the food industry e.g. the production of whipped cream.

Froth flotation is a process for selectively separating hydrophobic materials from hydrophilic and is used in several processing industries such as the recovery and upgrading of ores. The ground ore is mixed with water to form a slurry. The desired mineral is rendered hydrophobic by the addition of a surfactant or **collector** and the slurry is aerated to produce bubbles. The hydrophobic particles (usually a useful, extracted component) attach to the air bubbles, which rise to the surface, forming foam which is removed from the flotation cell, producing a concentrate of the target mineral. The hydrophilic particles (gangue) remain in

suspension, which is discarded. Collectors (reagents causing the surface hydrophobisation of a useful mineral) cover almost all types of surfactants. Apart from the addition of surfactants acting as flotation collectors, also the addition of foaming agents is of great importance. These frothing agents, known as **frothers**, are introduced to the pulp to promote the formation of a stable froth on top of the flotation cell.

In the washing process foam is not critical, as previously thought. However, it plays a significant role in preventing the re-deposition of dirt. The aim of using a laundry detergent in the washing machines is to reduce foaming and more particularly changes the nature of the foam from rare of great volume to dense foam. However, in the case of cleaning upholstery fabrics, carpets, curtains etc. cleaning by the action of foam is used. Foam applied with a brush on the cleaning surface leads to the separation of a dispersion of dust particles and pigments and emulsification of fat dirt.

In cosmetics, besides the use of surfactants as emulsifiers, dispersants, wetting agents, of great importance are foaming properties (the ability to create abundant, dense and durable foam). Although there is no relation between foaming ability of detergent and its cleansing properties, different cosmetics can require foam of different properties and therefore also different foam-forming agents. Foaming properties of the surface active agents used in shampoos can be average because during washing friction between hair and fingers favours formation of foam. Foam formed by the bath foaming agents should be stable and "fixed", which is to delay cooling of hot bath, and gently massage the body. Foam produced by the shaving cosmetics must be "rigid" to keep the hairs raised.

However, in many processes, foam is a negative factor causing serious technological problems: foaming of drilling fluids, foam formation during electroplating processes, in the manufacture of paper, during the process of fermentation in the production of: antibiotics and other pharmaceutical products, beer, tomato juice, yeast, distillation and evaporation in the production of sugar, etc. Hence, conditions for creating durable foam and methods for their violations are of great interest.

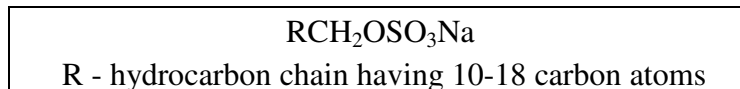
III. 4. Foaming agents used in cosmetics

The most common forms of cosmetic demanding the creation of rich foam are bath preparations and shampoos.

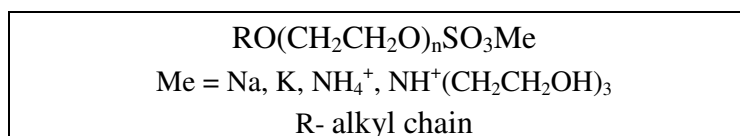
III. 4.1. Shampoos

Shampoo ingredients are divided into three main groups: ensuring cleaning action and the formation of foam, foam stabilizers and additional - thickeners, opacifiers, clarifying agents, colorants, preservatives, perfumes and medicines. The first group includes surfactants, which are the primary shampoo ingredients. The most commonly used surfactants in the manufacture of shampoo are:

- Alkyl Sulfates



- Polyethoxylated Fatty Alcohol Sulfates



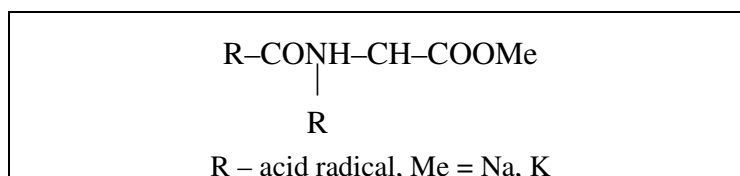
Their irritation properties decrease with the increasing alkyl chain length and the degree of ethoxylation. The most commonly used is a mixture of derivative of lauryl alcohol and myristic alcohol.

- Monoesters of sulfosuccinic acid and fatty alcohol



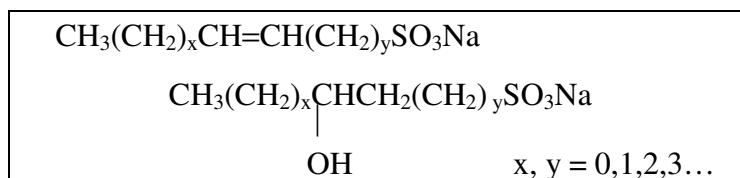
These compounds act gently on skin and mucous membranes, and ensure formation of abundant and stable foam.

- Condensates of fatty acids with amino acids or polypeptides



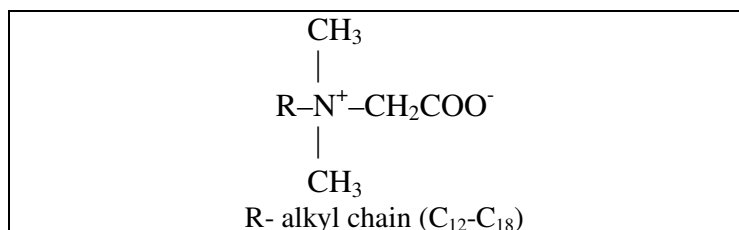
Acidic radicals derived typically from the acids: lauric acid, palmitic acid, undecyl acid or a mixture of fatty acids from coconut oil. These compounds cause formation of lower electrostatic charge on hair than other surface-active compounds.

- Alpha-Olefin Sulfonates

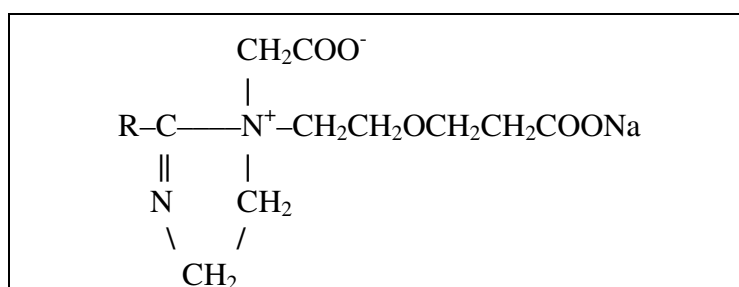


There is a mixture of approx. 60-65% alkyl sulfonates, 35-40% hydroxyalkyl sulfonates and about 10% of disulfonic derivatives. These compounds are characterized by advantageous properties detergency, foaming and good solubility and milder effects on the skin than alkyl sulfates.

- Betaines



- Imidazoline derivatives



Betaines and imidazoline derivatives are amphoteric compounds of a particular mild effect on the skin. These compounds have good wetting and cleaning properties in the pH range from 2 to 12, slightly antiseptic and fungicidal properties and form abundant, stable foam. Monoalkanolamides, dialkanolamides or amine oxides are usually used as foam stabilizers. They support washing ability of the composition, regulate its viscosity, and also have little conditioning effect. Amine oxides have also anti-static effect.

III. 4.2. Foaming bath agents

The primary function of the foaming bath formulation as liquids or creams is body cleaning combined with relaxation. They should therefore be characterized by good solubility in water, ability to create abundant and stable foam and neutrality in relation to the human body. Properly selected surface active compounds used in this type of formulations should have good solubility in water and effectiveness with a large percentage of dilution. Such requirements meet fatty alcohol sulfates or their ethoxylates. They are characterized by high foaming ability and stability in the presence of soap and high ability to disperse calcium soap. Sulfosuccinates with one ester group with a long hydrocarbon chain (C18) are also noteworthy. They are mild non irritating detergent with very good foaming properties. They increase the skin tolerance of other detergents, for example alkylarylsulfonates. In order to increase the mildness of action of other ingredients amphoteric detergents like betaine type or imidazoline derivatives are used in the recipe for bath preparations. Interesting compounds for this preparation are fatty acid condensates with proteins or their hydrolysates. However, due to the high cost they are used primarily in combination with other detergents. The role of foam stabilizers is fulfilled by mono- and diethanolamides or ethoxylated fatty alcohols, which also increase the viscosity of the composition, and due to the presence of free alcohol affect skin softening. When products of a lower viscosity are required and when larger amount of fragrance is need

to be stabilized, the ethoxylated alkanolamides are used, for example: ethoxylate monoethanolamide of fatty acid from coconut oil or fatty alcohol ethoxylates containing about 10 moles of ethylene oxide.

III. 5. Water hardness

Water hardness is caused by the presence of dissolved salts with a charge of +2, especially calcium and magnesium cations and in significant amounts Fe^{3+} , Mn^{2+} , Al^{3+} , Zn^{2+} . Mineral deposits are formed by chemical reactions resulting in formation of an insoluble precipitate or over the saturation level at a given temperature. The compounds which have the lowest solubility under the given conditions precipitate first. Water hardness is the most frequently expressed in milliequivalents of a given hardness component contained in 1 dm³ of water (mval/dm³).

With respect to hardness natural waters can be classified into five groups:

- very soft <1.5 mval/dm³,
- soft 1.5–3 mval/dm³,
- medium hard 3–6 mval/dm³,
- hard 6–9 mval/dm³,
- very hard > 9 mval/dm³.

Besides mval/dm³ water hardness can be also expressed as:

- **German degrees** (°dH) is defined as 10 mg of CaO per one liter of water
- **French degrees** (°fH or °f) is defined as 10 mg of CaCO₃ per one liter of water
- **English degrees** (°e, e, or °Clark) is defined as one grain (64.8 mg) of CaCO₃ per Imperial gallon (4.55 litres) of water

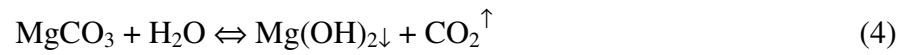
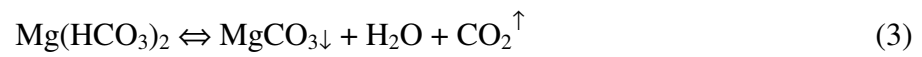
The hardness can be distinguished as follows:

- **Carbonate (temporary hardness)** - caused by the presence of acidic and neutral carbonates and hydroxides of calcium and magnesium,
- **Non-carbonate (permanent hardness)** - caused by the presence of sulphates, chlorides and silicates of calcium magnesium,
- **The total** - which is the sum of carbonate hardness and non-carbonate hardness.

Carbonate hardness is also called temporary hardness because it can be reduced by boiling water. The precipitate forming limescale deposits, whose main component is calcium carbonate formed by the decomposition of calcium bicarbonate:



As a result of carbon dioxide removal from the system sparingly soluble magnesium carbonate is precipitated, which after some time is converted to a harder soluble magnesium hydroxide.



Salts causing carbonate hardness precipitate in the form of sludge in the boiling water, and in the form of stone in preheated (not brought to boiling) water. In the first phase of precipitation solid ultraparticles are formed which, by coagulation transform into amorphous coagulates, or crystal nuclei. The rate of deposit formation depends on the number of nuclei produced per unit of time and linear speed of crystal growth.

IV. Experimental

A. Equipment and chemical reagents

1. **Apparatus:** mechanical stirrer BML-04.
2. **Equipment:**
 - 25 ml volumetric flask – 6 pcs,
 - volumetric pipette of volumes 5 ml – 3 pcs,
 - volumetric pipette of volumes 5 and 10 ml – 3 pcs,
 - glass cylindrical vessel for foam preparation – 1 piece,
 - wash bottle,
 - stopwatch,
 - scale.
3. **Chemicals. Aqueous solutions of:**
 - sodium dodecyl sulfate – SDS ($C_{12}H_{25}SO_4Na$) – 10^{-1} M,
 - sodium dodecylbenzene sulfonate – SDBS ($C_{12}H_{25}C_6H_4SO_3Na$) – 10^{-1} M,
 - washing powder – $0.5\text{ g}/100\text{ cm}^3$,
 - bubble bath – $0.5\text{ g}/100\text{ cm}^3$,
 - sodium soap – $0.5\text{ g}/100\text{ cm}^3$,
 - liquid soap – $0.5\text{ g}/100\text{ cm}^3$,
 - calcium chloride – 10^{-2} M.

B. Scheme of experiment

1. Preparation of aqueous surfactant solutions of the concentrations: 10^{-3} , $5 \cdot 10^{-3}$, 10^{-2} , $2 \cdot 10^{-2}$, $4 \cdot 10^{-2}$ M.
2. Measurement of foam height.
3. Plotting the curve of foam height as a function of the surfactant concentration $h = f(c)$.
4. Comparison of the effect of concentration and the surfactant structure on the foam formation process.
5. Plotting the curve of foam height as a function of water hardness $h = f(Tw)$ for four aqueous solutions of commercial detergents.
6. Comparison of the effect of water hardness on the foaming process of commercial detergents and cleaning preparations.

C. Experimental setup

Determination of the amount of foam depending on the structure and concentration of surfactant.

1. Prepare 25 cm^3 of 10^{-3} M SDS aqueous solution by dilution of a stock solution having a concentration of 10^{-1} M (before sampling a stock solution with a concentration of 10^{-1} M should be heated).

2. Pour into a glass cylindrical vessel an aqueous solution of SDS at a concentration of 10^{-3} M, insert the tip of the mixer so that it touches the surface of the liquid (**without dipping**).
3. Set the mixer speed to level 3.
4. Turn on the mixing. After 1 min switch a stirrer off, wait 15 sec and read the foam height.
5. During mixing the former solution prepare a 25 cm^3 of surfactant solution of a further concentration.
6. Pour out the solution from the measuring vessel and thoroughly rinse it with distilled water.
7. Take measurements for the SDS solutions of concentrations: $5 \cdot 10^{-3}$, 10^{-2} , $2 \cdot 10^{-2}$, $4 \cdot 10^{-2}$ M.
8. Make the analogous measurements as for aqueous solutions of SDBS.

Determination of foam height as a function of water hardness for washing and cleaning preparations.

1. In a 25 cm^3 volumetric flask prepare aqueous solutions containing:

Table 2.

Flask number	Powder/ bubble bath/ liquid soap/sodium soap	$\text{CaCl}_2\ 10^{-2}\ \text{M}$
1	5 cm^3	0
2	5 cm^3	2.5 cm^3
3	5 cm^3	5.0 cm^3
4	5 cm^3	10.0 cm^3

2. Measure the foam height like in part 1.

D. Results and conclusions

D 1.

1. Present the obtained results in Table 3.

Table 3.

Surfactant	Foam height – h [mm]				
	$10^{-3}\ \text{M}$	$5 \cdot 10^{-3}\ \text{M}$	$10^{-2}\ \text{M}$	$2 \cdot 10^{-2}\ \text{M}$	$4 \cdot 10^{-2}\ \text{M}$
SDS					
SDBS					

2. Plot the changes of the foam height as a function of surfactant concentration of $h = f(c)$.

- Based on the obtained results conclude about the impact of the concentration and structure of the surfactant on the foaming process.

D 2.

- Present the obtained results in Table 4.

Table 4.

Aqueous solution of detergent	Foam height h [mm]			
	without Ca^{2+}	+ 2.5 ml CaCl_2	+ 5 ml CaCl_2	+ 10 ml CaCl_2
Bubble bath				
Liquid soap				
Sodium soap				
Washing powder				

- Plot the changes of the foam height as a function of water hardness $h = f(^{\circ}T_w)$ expressed in German degrees.
- Based on the obtained graph conclude about the water hardness influence on the foaming process for the investigated cosmetic formulations.