

Ministry of Health of the Republic of Belarus
Educational institution
"Gomel State Medical University"

Department of General and Bioorganic Chemistry

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METHODOLOGICAL MANUAL
for conducting the laboratory class
in the academic discipline "Medical chemistry"
for students
the first-year of the Faculty of International Students (FIS),
studying in the specialty 7-07-0911-01 "Medical business"

Topic 14: Physico-chemistry of surface phenomena

Time: 2 hours

Approved at the meeting of the Department
of General and Bioorganic Chemistry
(Protocol No. 9 dated 31.08.2024)

THE TRAINING AND EDUCATIONAL GOAL, TASKS, MOTIVATION TO STUDY THE TOPIC

Training purpose:

- formation of students' basic professional competence for solving diagnostic, research and other tasks of professional activity based on knowledge about physico-chemistry of surface phenomena;
- familiarization with the surface processes occurring at the interface of the two phases, with special attention to the adsorption processes and their biological significance;
- to give an idea of adsorption therapy (hemo-, lymph-, plasmadsorption and enterosorption), as well as the use of surfactants in modern medicine.

Educational goal:

- to develop your personal, spiritual potential;
- to form the qualities of a patriot and a citizen who is ready to actively participate in the economic, industrial, socio-cultural and social life of the country;
- learn to observe academic and labor discipline, the norms of medical ethics and deontology;
- to realize the social significance of their future professional activities.

Tasks:

As a result of the training session, the student should

know:

- the concept of free surface energy and surface tension, as well as the factors affecting them;
- the concept of adsorption and its types;
- theoretical bases of surfactant's adsorption at the liquid-gas interface surfaces and mathematical description of this process by the Gibbs', Shishkovsky's and Gibbs-Shishkovsky's equations;
- natural and synthetic materials used as solid adsorbents;
- the concept of molecular adsorption on solid adsorbents and mathematical description of this process by the Langmuir's and Freundlich's equations;
- theoretical bases of the adsorption of electrolytes on solid adsorbents, described by the Panet-Phayans' rules;
- theoretical bases of ion exchange adsorption and the use of ionites in medicine and engineering;
- the use of sorbents in medicine. Hemo-, lymph-, plasma- and enterosorption as methods of treatment of some diseases;

be able to:

- explain the reason for the appearance of excess energy of molecules at the interface of the two phases compared with molecules in the interior;
- explain the structural features of surfactant's molecules and their orientation at the liquid-gas interface, as well as the effect of the length of the hydrocarbon radical on surface activity;
- perform calculations according to Gibbs', Shishkovsky's and Gibbs-Shishkovsky's equations describing the adsorption of surfactants at the liquid-gas interface;
- know the main statements of the theories of molecular adsorption and perform calculations according to the Langmuir's and Freundlich's equations;

– describe the selective adsorption of electrolytes from solutions in accordance with the Panet-Phayans' rules;

– describe ion exchange adsorption, give schemes of ion-exchange processes for cations and anions;

possess:

– skills in experimental determination the surface tension of liquids by the stalagmometric method.

Motivation to study the topic:

Surface phenomena and adsorption have a great importance in medicine and biology. The most important biological processes: enzymatic reactions, protein synthesis, etc., occur on the interface surfaces. Surfactants that reduce the surface tension of liquids play an important role in physiological processes. For example, bile acid salts, which have high surface activity, provide emulsification of fats and their absorption. Adsorption of toxic substances on hemo- and enterosorbent is the basis of adsorption therapy. Immunosorbents are widely used in the treatment of microbial intoxication, gas gangrene and tetanus.

NECESSARY EQUIPMENT

1. Methodological manual for students on the topic "Physico-chemistry of surface phenomena".

2. Training tables:

a) Periodic Table of chemical elements by D.I. Mendeleev;

b) table of solubility of acids, bases and salts.

3. Reference materials of basic physico-chemical constants.

4. Chemical reagents and equipment necessary for laboratory work.

CONTROL QUESTIONS ON THE TOPIC OF THE CLASS

1. Surface phenomena. The concept of free surface energy and surface tension.

2. Adsorption and its types (physical and chemisorption).

3. Surfactants' adsorption on gas/liquid interface surfaces.

4. Adsorption on solid adsorbents.

COURSE OF THE CLASS

The theoretical part

1. SURFACE PHENOMENA. FREE SURFACE ENERGY AND SURFACE TENSION

Surface phenomena are processes that run spontaneously at the border of two different phases (interface surfaces). Surface phenomena are adhesion, cohesion, adsorption and some others. Such phenomena play an essential role in metabolic processes such as respiration, digestion, and excretion.

Accumulation of free energy at the interface surface is result of non-balanced intermolecular forces affecting on molecules at the interface surface.

Let's consider the energy state of molecules in a single component system composed of two phases: liquid water – water vapor.

A molecule in the interior of liquid phase is completely surrounded by other molecules, each of which effects on it with an intermolecular force f_1 . The resultant of these forces is equal to zero: $\Sigma f_1 = 0$ (Fig. 1):

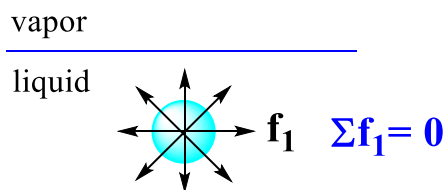


Figure 1 – A molecule in the interior of a liquid phase

On molecule at an interface surface affect molecules from liquid phase and molecules from gaseous phase with intermolecular forces f_1 and f_2 , $f_2 \ll f_1$. The resultant of forces f_1 and f_2 is equal to the force f_3 that pulls the molecule towards the interior of the liquid phase (Fig. 2):

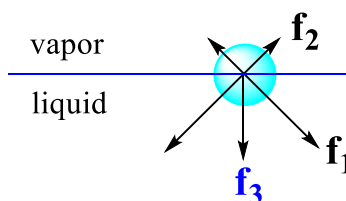


Figure 2 – A molecule at an interface surface

The difference between energies of surface molecules and molecules in the interior is defined as **free surface energy**:

$$G_s = \sigma \times S$$

S – the area of interface surface, m^2 ;

σ – proportionality coefficient, named surface tension.

Surface tension is the amount of free surface energy per unit area:

$$\sigma = \frac{G_s}{S}, \frac{J}{m^2} = \frac{N}{m}$$

An increase in temperature leads to a decrease in surface tension, as it leads to the break of some intermolecular bonds. The greater the polarity of the liquid, the greater its Surface Tension, since an increase in polarity leads to an increase in intermolecular forces.

Surface Tension is an important characteristic of liquids, solutions and biological fluids. For example, *surface tension of blood is $45.4 \times 10^{-3} N/m$* . Even negligible deviation from the normal value signals about serious diseases.

Stalagmometric method is wide applied in practice to measure surface tension of liquids [1-3].

2. ADSORPTION AND ITS TYPES (PHYSICAL AND CHEMISORPTION)

Adsorption is a process which involves the accumulation of one substance at the surface of another substance. It involves separation of substance from one phase and its accumulation at the surface of another.

Adsorption should not be confused with absorption. **Adsorption** is a surface phenomenon in which particles or molecules bind to the top layer of material. **Absorption**, on the other hand, goes deeper, involving the entire volume of the absorbent. Absorption is the filling of pores or holes in a substance.

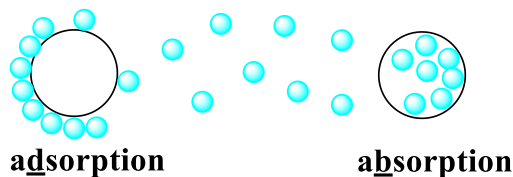


Figure 3 – Adsorption vs. Absorption

Two participants are required for the adsorption process:

- **adsorbate:** a substance that concentrates on the surface of another substance, for example, O₂, N₂ gases;
- **adsorbent:** an adsorbing substance (a substance on the surface of which the adsorbate is adsorbed), for example, charcoal, silica gel, aluminum oxide).

Adsorbed amount (Γ) is defined as:

$$\Gamma = \frac{n}{S}, \frac{mol}{m^2}$$

N – number moles of an adsorbate, mol;

S – area of an interface surface, m²

Depending on the nature of the forces existing between adsorbate molecules and adsorbent, there are two types of adsorption:

Physical adsorption (physisorption): the forces of attraction that exist between the adsorbate and the adsorbent are the Vander Waal's forces. For example, H₂ and N₂ gases are adsorbed on coconut coal.

Chemical adsorption (chemisorption): the molecules (or atoms) stick to the surface, forming covalent bonds. For example, the formation of iron nitride on the surface when iron is heated in N₂ at 623 K [1-3].

3. SURFACTANTS' ADSORPTION ON GAS/LIQUID INTERFACE SURFACES

When substances dissolve in water, they can cause:

- a) lowering of its surface tension. Such substances are called surfactants (SAS);
- b) an increase in its surface tension. Such substances are called surface-inactive (SIS – inorganic acids, bases and salts);
- c) the surface tension of water does not change. Such substances are surface-nonactive (SNS – glucose, sucrose, etc.).

The dependence of the surface tension on the concentration of the dissolved substance at a constant temperature is called as isotherm of surface tension (Fig. 4):

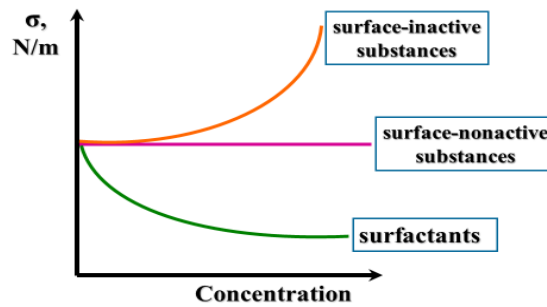


Figure 4 – Isotherms of surface tension

Surfactants or surface-active agents are substances that reduce surface tension of water. Surfactants are dual-nature molecules consisting of a polar hydrophilic head group and a non-polar hydrocarbon tail or radical (Fig. 5):

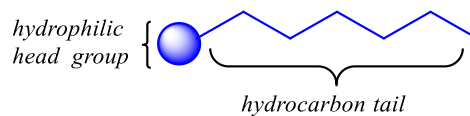


Figure 5 – Schematic representation of a surfactant molecule

Surfactants are divided into two categories:

- a) electrolytes;
- b) weak or nonelectrolytes.

Surfactants-electrolytes are divided into two groups:

- *cationic surfactants* are salts and hydroxides of alkyl ammonium. For example, cetyl trimethyl ammonium bromide $[\text{CH}_3-(\text{CH}_2)_{15}-\text{N}(\text{CH}_3)_3]\text{Br}$ is applied as antiseptic in surgery;
- *anionic surfactants* are salts of carboxylic acids ($\text{R}-\text{COOMe}$) and salts of sulphonic acids ($\text{R}-\text{SO}_3\text{Me}$).

Surfactants – weak or nonelectrolytes are abundant in nature. They are:

- carboxylic acids: $\text{R}-\text{COOH}$;
- thiols: $\text{R}-\text{SH}$;
- sulphonic acids: $\text{R}-\text{SO}_3\text{H}$;
- amines: $\text{R}-\text{NH}_2$.
- alcohols: $\text{R}-\text{OH}$;

Many bioactive substances exhibit surface activity. They are phospholipids, bile salts, amino acids and proteins. Their surface activity is responsible for their biological activity. *Phospholipids* are structural components of cell membranes (Fig. 6):

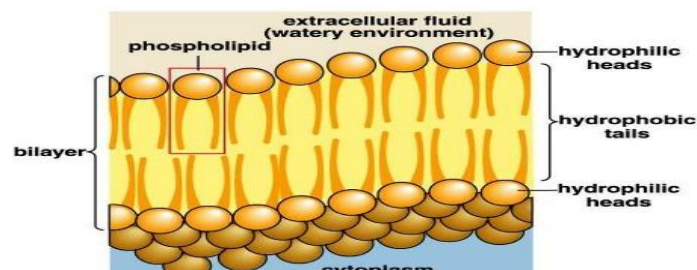


Figure 6 – The lipid bilayer of cell membranes is made up of two layers of phospholipids

Mechanism of surfactant's activity: adsorption of surfactants at gas/liquid interface surfaces obeys the rule: «Like dissolves like». Their polar hydrophilic head groups tend to be in water while non-polar hydrocarbon tails try to be in air. The adsorption of surfactants

reduces the surface tension of water, since this orientation reduces the difference in polarity between phases.

The effect of surfactant's concentration on their adsorbed amount from water solutions is represented in a graph form. This graphical representation is defined as isotherm of surfactant's adsorption (Fig. 7):

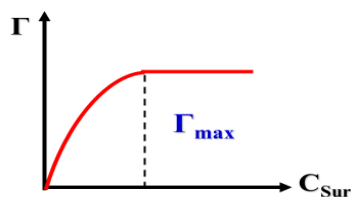


Figure 7 – Isotherm of surfactant's adsorption

In dilute solutions, an increase in surfactant's concentration leads to an increase in the adsorbed amount, but the adsorbed amount does not change with the formation of a saturated monolayer. The maximum adsorbed amount Γ_{max} corresponds to a saturated monolayer.

When the maximum adsorbed amount is determined, it is possible to calculate:

1) the length of the surfactant molecule (ℓ):

$$\ell = \frac{\Gamma_{max} \times M}{\rho};$$

ρ – density of a surfactant;

M – it's molar mass;

2) the area occupied by the molecule at the interface surface (S):

$$S = \frac{l}{\Gamma_{max} \times N_A};$$

N_A – Avogadro's number.

Surfactants are characterized by their **surface activity (g)**:

$$g = -\frac{\Delta\sigma}{\Delta C}$$

$\Delta\sigma$ is a decrease in surface tension corresponding to an increase in the surfactant's concentration by ΔC .

Surface activity of substances depends upon their nature. This dependence is expressed by the empiric **Ducklo-Traube's rule**: *the surface activity of organic compounds belonging to the same homologous series (carboxylic acids, alcohols, etc.) increases 3–3.5 times for each additional CH₂-group. At the same time, the surface tension of their solutions decreases.*

▪ The relationship between adsorbed amount (Γ) and surfactant's concentration in water solution at a constant temperature is expressed by the **Gibbs's equation**:

$$\Gamma = -\frac{d\sigma}{dC} \times \frac{C_{sur}}{RT}$$

C_{sur} – concentration of a surfactant, mol/l;

R – gas constant, 8.31 J/mol×K;

T – absolute temperature, K.

▪ The effect of surfactants on surface tension of solutions is expressed by the **Shishkovsky's equation**:

$$\sigma = \sigma_o - a \times \ln(1 + b \times C_{sur})$$

a and *b* are the parameters of the equation.

▪ The adsorbed amount may be calculated by the combined **Gibbs-Shishkovsky's equation**:

$$\Gamma = \frac{a}{RT} \times \frac{b \times C_{sur}}{1 + b \times C_{sur}}$$

Application of surfactants:

1. In everyday life anionic surfactants are used as soaps and detergents. They remove water-insoluble substances (butter, fat, oil) from solid surfaces. Surfactant molecules adsorb on the contaminated surfaces, turning hydrophobic to hydrophilic systems.

2. Cationic surfactants have a high affinity to bacteria and are used for medical purposes and cosmetics. Some surfactants are applied as antiseptics in surgery. Their antibacterial activity is much greater (~300 times) than that of phenol, traditionally applied in medicine for disinfection.

3. Surfactants are used for liposomes production. Liposomes may be used to transfer a wide range of medicines in blood (antitumor and antibacterial drugs, hormones and vaccines) [2, 3].

4. ADSORPTION ON SOLID ADSORBENTS

Solid sorbents are natural or artificial substances with a highly developed surface area. Porous and powdered substances have a higher adsorption capacity than nonporous ones. Sorbents are characterized by their *specific surface area* (*a_s*):

$$a_s = \frac{A_s}{m}, \quad m^2/g$$

A_s – surface area of the solid sorbent, m²;

m – mass of a sorbent, g.

Classification of solid sorbents:

1. Carbon-containing sorbents (activated carbon is most widely used).
2. Aluminosilicates: aluminum salts of polysilicon acids. For example, kaolin (white clay) Al₂O₃×SiO₂×2H₂O.
3. Zeolites are aluminosilicates with a high content of sodium and calcium.
4. Silica gel is a dehydrated gel of polysilicon acids (SiO₂)_n.
5. Oxides and hydroxides of some metals: Al₂O₃, Al(OH)₃, Fe₂O₃, Fe(OH)₃ and others.
6. Food fibers (cellulose, pectin and lignin) are essential components of food products. The main functions of food fibers are:
 - 1) activate peristalsis of intestinal tract;
 - 2) bind and remove toxins from the human body;
 - 3) contribute to the further growth of bacteria that synthesize B vitamins responsible for strengthening the immune system of people;

4) bind heavy metals and radio nuclides into stable chelate complexes easily removed from the intestinal tract.

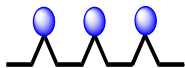
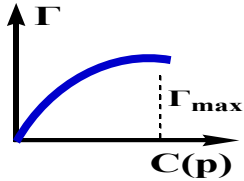
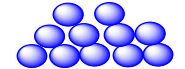
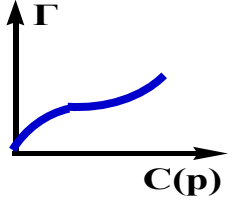
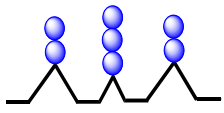
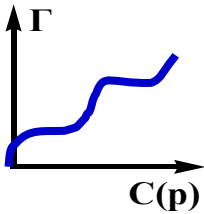
There are three main types of adsorption on solid adsorbents:

- molecular adsorption from gaseous and liquid phases;
- selective adsorption of electrolytes from aqueous solutions;
- ion exchange adsorption of electrolytes from aqueous solutions.

Molecular adsorption is the adsorption of nonelectrolytes or weak electrolytes from gaseous and liquid phases by solid adsorbents.

There is no unified theory describing all types of the adsorption on different adsorbents and different interface surfaces. The most common theories of molecular adsorption are given in Table 1.

Table 1 – The most common theories of molecular adsorption

The name of the theory	The physical model	Isotherm adsorption	Isotherm equation
1. Langmuir's theory of monomolecular adsorption	 <ul style="list-style-type: none"> ▪ solid surface is not uniform; ▪ its unevenness and roughness act as active adsorption centers; ▪ one center adsorbs one molecule of adsorbate; ▪ between the adsorbed molecules there is no interaction; ▪ adsorption is a reversible process 	 <p>C – concentration of an adsorbate; p – gas pressure of an adsorbate;</p>	$\Gamma = \Gamma_{\max} \frac{K \times C}{1 + K \times C}$ $\Gamma = \Gamma_{\max} \frac{K \times p}{1 + K \times p}$ <p>K – adsorption equilibrium constant</p>
2. Polyani's theory of polymolecular adsorption	 <ul style="list-style-type: none"> ▪ solid surface is uniform; ▪ this surface attracts adsorbate molecules forming polymolecular layers 		—
3. BET theory of polymolecular adsorption (Brunauer, Emmett, Teller)	 <ul style="list-style-type: none"> ▪ adsorption occurs only on the active adsorption centers; ▪ after the monomolecular layer formation, adsorption continues to rise giving S-shaped polymolecular adsorption isotherm 		—

In practice, the empirical Freundlich's equation is often used to calculate molecular adsorption:

$$\varepsilon = k \times C^{1/n}; \quad \varepsilon = k \times p^{1/n}$$

ε – mass of an adsorbate on one gram of an adsorbent;

k – Freundlich's constant;

n – empirical constant.

The definition of the Freundlich's equation parameters is represented in Figure 8:

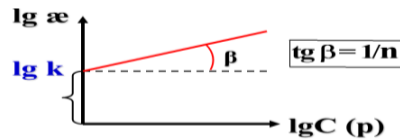


Figure 8 – Graphical definition of the parameters of the Freundlich's equation

The selective adsorption of electrolytes from aqueous solution obeys **the Panet-Phayans' rules**.

Rule 1. *Solid surfaces adsorb those ions that are included into their composition.* These ions are defined as potential determining ions, or PDI.

Example. Suppose that sodium chloride is treated with an excess of silver nitrate solution. The solid surface of silver chloride immediately starts to adsorb ions from the solution. It may adsorb either silver cations Ag^+ or chloride anions Cl^- , but only silver cations are available for adsorption since all chloride anions are precipitated as AgCl . Thus, Ag^+ cations behave as potential determining ions that give a positive charge to a solid surface (Fig. 9):

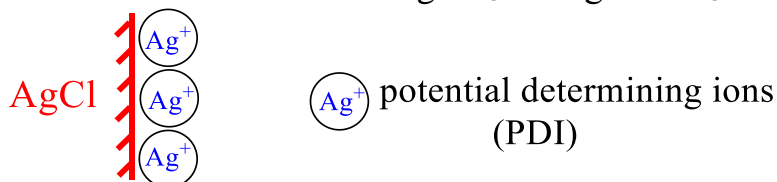
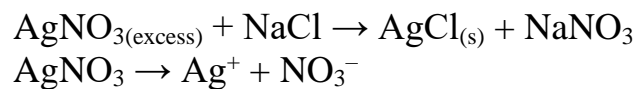


Figure 9 – Formation of a positively charged PDI layer on the AgCl surface

Rule 2. *The charged surface adsorbs only oppositely charged ions (counter ions).*

In the example, a positively charged surface adsorbs nitrate anions as counter ions. A double electric layer is formed on the solid surface (Fig. 10):

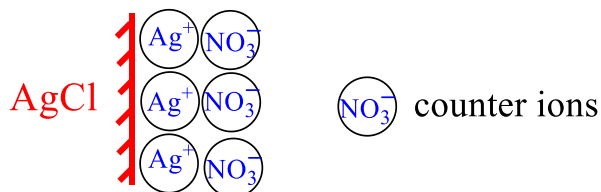


Figure 10 – Formation of a double electric layer on the surface of AgCl

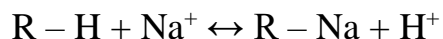
Ion exchange adsorption process is a process in which ions from a solid adsorbent are exchanged in equivalent quantities with similarly charged ions that are dissolved in aqueous solutions.

Solid adsorbents able to take part in ion exchange are called ionites or ionic exchangers. An ion exchanger is an insoluble substance containing weakly held ions that can be exchanged with other ions in solution that come in contact with it.

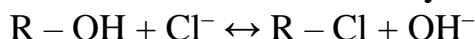
There are two types of ionic exchangers: a) acidic;
b) basic.

Acidic ionites contain loosely held hydrogen cations H^+ . Their structure can be represented as: **R – H**, where R is a polymer radical.

A large number of solid sorbents work as acidic ionites. These are aluminosilicates, zeolites, silica gels, cellulose and some other sorbents. Loosely held H^+ cations contained in their structure undergo reversible exchange with the cations from a solution:



Basic ionites ($R - OH$) contain loosely held hydroxyl anions OH^- . The examples of them are $Fe(OH)_3$, $Al(OH)_3$ and some other sorbents. They work according to the scheme:



In modern medicine solid sorbents are applied for:

- a) hemo-, lympho- and plasmasorption;
- b) enterosorption.

Hemo-, lympho- and plasmasorption are used for blood and other biological fluids purification from toxins by passing of the fluids through a column filled with activated carbon or some other sorbents (the method is applied since 60^s of the 20th century).

Enterosorption is a method to bind and remove toxins in intestinal tract of patients. Enterosorbents are solid sorbents (carbonaceous, aluminosilicates, zeolites and others) that bind and extract toxins, heavy metals, radio nuclides in gastrointestinal tract. Their use is an effective way to improve the life quality of people living in conditions of man-made pollution in areas with a high levels of chemical and radioactive contamination [1-3].

The practical part

Safety instructions before laboratory work.

LABORATORY WORK

Studying a relationship between surface tension of solutions and the length of hydrophobic radicals in surfactants

One of the main methods for determining the surface tension of liquids is the *stalagmometric method*. Stalagmometer is a capillary tube with an expansion (B) of medium height, ending in thick-walled capillary through which the liquid is not flowing, but dropping. There are A and C marks above and below the expansion.



Figure 11 – Stalagmometer is a capillary tube with an expansion in the middle

Stalagmometric method is based on measuring the number of droplets formed by a liquid liberating from the capillary. In this method, we compare the number of drops of the liquid under study and a number of drops of liquid with a known surface tension (for example, water) falling from the end of a stalagmometer. The greater the surface tension of the liquid, the greater the volume of forming droplets. Thus, the number of drops is inversely proportional to the surface tension of the liquid:

$$\sigma(X) = \sigma(H_2O) \times \frac{n(H_2O)}{n(X)}$$

1. Rinse the stalagmometer with water. Fill stalagmometer with water and count the number of water drops flowing from the volume of the stalagmometer between the upper and lower marks.

2. After that, rinse the stalagmometer with a solution of ethyl alcohol and count the number of drops of ethyl alcohol solution flowing from the same volume of the stalagmometer. Repeat the experiment by filling the stalagmometer with solutions of propyl, butyl and amyl alcohols, respectively.

3. Write the obtained data into the Table 2.

Table 2 – Surface tension of alcohols solutions

No	Water / Alcohols	Number of drops	$\sigma, \text{N/m} \times 10^3$
1	H ₂ O		72.75
2	C ₂ H ₅ OH		
3	C ₃ H ₇ OH		
4	C ₄ H ₉ OH		
5	C ₅ H ₁₁ OH		

4. Calculate the surface tension of each solution using the equation:

$$\sigma(X) = \sigma(H_2O) \times \frac{n(H_2O)}{n(X)}$$

$n(H_2O)$ – number of water drops; $n(X)$ – number of drops of a test solution.

Surface tension of water at 20 °C: $\sigma(H_2O) = 72.75 \times 10^{-3} \text{ N/m}$

5. Represent the obtained data in a graph form:

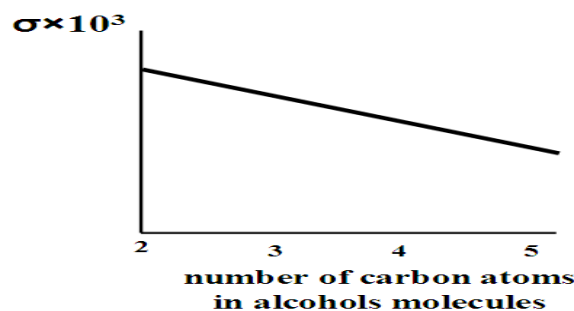


Figure 12 – The effect of hydrophobic radicals' length at surface activity of alcohols

6. Make a conclusion of how the length of hydrophobic radicals affects surface activity of alcohols.

Control over the assimilation of the topic

It is conducted in the form of an oral conversation with students.

METHODOLOGICAL RECOMMENDATIONS FOR THE ORGANIZATION AND PERFORMING OF INDEPENDENT WORK OF STUDENT (IWS)

The time allotted for independent work can be used by students for:

– preparation for laboratory classes;

- taking notes of educational literature;
- performing tasks for self-control of knowledge;
- preparation of thematic reports, abstracts, presentations.

The main methods of organizing independent work:

- studying topics and problems that are not covered in the classroom;
- writing an abstract and making a presentation;
- performing tasks for self-control of knowledge.

List of tasks of IWS:

1. Specify the formulas of surfactants: $C_{12}H_{22}O_{11}$; C_2H_5OH ; $NaHCO_3$; C_3H_7COONa ; $C_6H_{12}O_6$; $C_4H_9NH_2$.

2. Of the following compounds, which will be the best and which will be the worst surfactants: a) propionic acid C_2H_5COOH , b) lauryl alcohol $CH_3(CH_2)_{11}OH$, c) sodium lauryl sulfate $CH_3(CH_2)_{11}OSO_3Na$?

3. What will be the surface tension of an aqueous solution of pentanol-1, if the number of the drops of this solution flowing from the stalagmometer is 72, and the number of water drops is 60? The surface tension of water at the experimental temperature of 293 K is $72.8 \times 10^{-3} J/m^2$.

Answer: $60.7 \times 10^{-3} J/m^2$

4. At 20 °C, the surface tension of 0.2 M of an aqueous SAS solution is $55 \times 10^{-3} J/m^2$. Calculate the value of SAS adsorption (the surface tension of water at 20 °C is $72.75 \times 10^{-3} J/m^2$).

Answer: $7.3 \times 10^{-6} mol/m^2$

5. The surface tension (σ) of an aqueous solution of butyric acid at 20 °C obeys the empirical Shishkovsky's equation: $\sigma = \sigma_0 - 29.8 \times 10^{-3} \ln(1 + 19.64 \times C)$, where σ_0 is the surface tension of pure water equal to $72.75 \times 10^{-3} N/m$.

Calculate the adsorption of butyric acid from its 0.01 M solution.

Answer: $2 \times 10^{-6} N/m$

The control of the IWS is carried out in the form of:

- evaluation of an oral answer to a question, message, report or presentation;
- individual conversation.

LIST OF SOURCES USED

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