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VOLUME I

Edited by

Nadežda Ćalić, Ljubiša Andrić,
Igor Miljanović, Ivana Simović

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Foreword

Practically, all human societies depend on the availability and use of mined products. Mining and mineral processing has played a vital part in the history and economy of the Balkans.

In the world, mineral processing was an art till the 1920s, when it started to become a science. The achievements of fundamental science enabled the explanation of phenomena in the processes of mineral processing, or they started from fundamental science to come to an appropriate solution in mineral processing. In many respects mineral processing becomes fundamental science.

Balkan countries have more or less rapidly accepted innovations in the field of mining and mineral processing.

Generations of professionals from Balkan trained on the tradition of mining schools, afterward universities, (Schemnitz established 1702, Jachimov 1716, Banska Štiavnica 1725, Jekatarinburg 1730, L' Ecole Polytechnique 1794 in Paris, Politehnika in Prague, and certainly the most famous Bergakademie Freiberg founded in 1765, and much later, universities in the United States and Soviet Union) contributed to today's level of development of mineral processing, and contributed to the quality of studies of mineral processing, both in the world, and so in the Balkans.

After the Second World War in the Balkans a large number of universities, faculty, institutes and laboratories of mining industry with special departments for mineral processing were opened. In many Balkan countries remarkable impact on development of mineral processing had Russian and American schools.

A great number of researchers and specialists in Balkan area were occupied for more decades by the research in mineral processing. The goal of this research was establishment of concentration process in industry, capacity enlargement, optimization of processes, increase the energy efficiency of processes and devices, introduction or construction of new machines. Based on those activities, Balkan mining has been evolving and continuously operates up to nowadays. As a result, in the Balkan countries appeared a significant number of successful researchers in the field of mineral processing. They founded the first Balkan mineral processing Committee (1973), and then the Balkan Academy of Mineral Technology.

Balkan Congress on Mineral Processing is beening held for 40 years. Participation in the work of the Committee of the Balkan mineral processing is a strong link between the development of the science and profession with global trends, and it provides the possibility of establishing direct contacts between researchers, designers, equipment manufacturers and investors from the region and around the world. It has already become tradition to hold every second year an international event, "Balkan Mineral Processing Congress," in which participate, not only Balkan experts, than experts from the whole world.

Maintenance XVI Balkan Congress on Mineral Processing in Belgrade from 16 to 21 June 2015 is held under the auspices of the Ministry of Mines and Energy of Serbia, with the financial assistance of the Ministry of Republic of Serbia. Incomparably greater financial support Congress had from sponsors who strongly support the mineral processing industry all over the world.

Prof. dr Nadežda Ćalić

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SOLID WASTE AND WASTE WATER TREATMENT and SOIL REMEDIATION

FLOTATION EXTRACTION OF BLUE WATER

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Abstract: Flotation of ions and molecules represents a promising method of separating components from the solution, or in our case, gelled copper salt from the blue waters. The method of flotation of ions and molecules is basicly flotation extraction process which produces different concentration gradients of more component phase system. Different concentration gradients in the flotation extraction are due to the driving forces of the participating molecules driven by molecular interactions. More component phase system i.e. flotation pulp consists of water molecules of the solute, and a suitable reagent which has the role of connection of the liquid and gaseous phases dispersed in a biphasic system. Such a system leads to the interaction of the solute with a reagent to the formation of coagulated component in water as solvent. Separation in the polyphased system is in accordance with Van't Hoff-th equation, and spontaneously occurs in two-phase systems like gaseous-fluid, such as the system of flotation pulp. By use of the osmotic pressure in the columnn flotation machine comes to the separation of the flotation pulp to the heavy and light phase. This allows the molecules of water to form their natural distances which are only valid for molecules of pure water, apropos for heavy phase. Also, the solute forms it's light phase, copper – surfactant salt as insoluble product. For some poliphase systems such as colloidal systems like in flotation process are sufficient atmospheric pressure conditions and for others solutions should be applied overpressure while being aerated. The paper presents the results of the research of flotation extraction of blue water i.e. copper solution content of 0.2 g/l. From the results, it is concluded that there is a possibility of purifying of blue water from copper mine tailings using the flotation extraction technology.
Keywords: Concentration gradient, condensed phase, coagulation, osmotic pressure, colligend

Introduction

Flotation was first applied in 1877 to enrich the graphite ore but immediately afterwards was abandoned. From the first patented process (elmore 1898). [1] it has been applied and constantly improving. The use of flotation in wastewater treatment is considered to be a revolutionary innovation because it runs 6-8 times faster than sedimentation and ends in 15-30 minutes. [2] In addition it provides a very high degree of removal of suspended material, significantly reducing the concentration of surface active materials in waste water and increases the oxygen content, all in a great extent the later stages of processing. Today, the flotation process is applied in many areas of industry: in the enrichment, in the separation of various metals in ores and fuels, in the separation of valuable components from the solutions, while in the wastewater treatment is used for removal of suspended and emulsified pollutants and biological sludge buildup. The solid particles, droplets, ions or molecules from the liquid phase, "glued" to the air bubbles pumped to the liquid phase in various ways, are carried to the surface by the driving force of the osmotic pressure, and concentrated to a foam which is removed from there.

Flotation process: The process of flotation is based on the fact that particles with hydrophobic surface in aqueous dispersions have the ability to stick with a gas bubble. During the process of mutual mixing of the dispersion and the bubbles, some particles are concentrated on the surface of the bubbles, while particles that do not have a hydrophobic surface remain in the volume of the dispersion. Separation based on these natural capabilities of the particles is used as an opportunity of getting separate particles based on their different abilities to concentrate at the surface of bubbles. Flotation is a process of molecular "attaching" of particulate materials on the interphase border area, usually gas (usually air) - water. Flotation is used as an alternative method to the other separation processes: sedimentation, centrifuge separation, filtration and the like, and it is often more efficient or economicaly justified [2]. Figure 1 illustrates the differences between these processes, and figure 2 shows the types of flotation process.

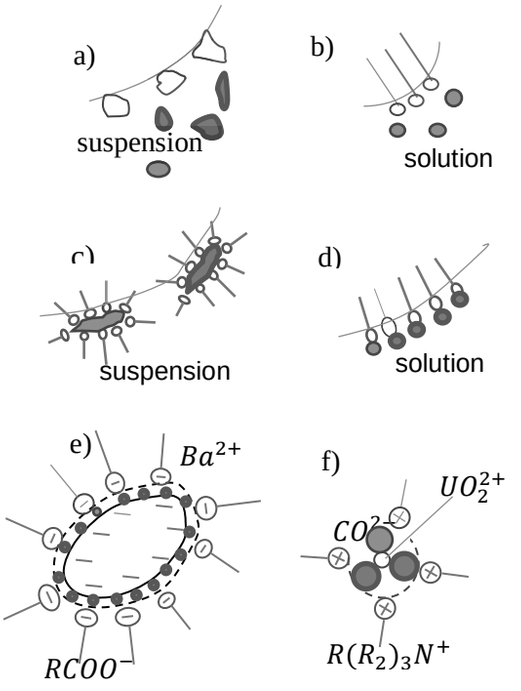


Figure 1. Illustration of similarity of flotation of ions and molecules with the flotation of minerals.

a) Flotation active mineral particles are adsorbed onto the surface of the bubbles while inactive mineral particles remain in the bulk;
b) Surface active components of the solution are adsorbed onto the surface of the bubbles, while other ones eg. suface inacitve remain in the bulk;
c) Onto the surface of the bubbles are adsorbed mineral particles covered with collector;
d) Onto the surface of the bubbles is adsorbed sublat (individual chemical compound), which consists of colligend (indicates the ion to be removed) and the collector;
e) Ba^{2+}ions are attracted by fatty acid anion collector ($RCOO^-$) adsorbed at the surface of quartz particles;
f) CO_3^{2-} ion are binder between UO_2^{2+}ion and four ions of alkylammonium collector ($R(R_2)_3N^+$).

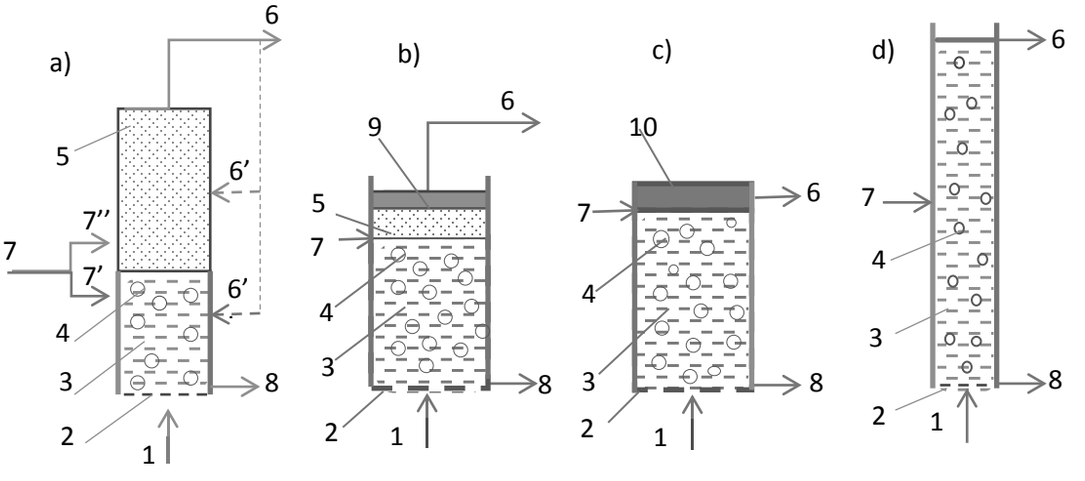


Figure 2. Scheme of foam separation (a), ion flotation (b), flotation extraction (c) and nucleate fractionating (d)

1. air, 2. bubble sprunger, 3. water solution, 4. surfacing bubbles, 5. The foam layer, 6. the above product, 6' i 6''. or the above product which is returned to the wet foam and upper product which is returned under the foam , 7. starting solution, 7' i 7'' added to the starting solution, ie the initial solution added above and below the level of foam , 8. bottom product , 9. foam, 10. the organic liquid phase

Adsorption on the surface of bubble

Adsorption on the surface of the bubble is the basis of the flotation of ions and molecules.[3]

Termodynamics of adsorption: The basic thermodynamic equation of interfacial boundaries liquid-gaseous gave Gibbs:

$$d\sigma = -\eta_s dT - \sum_{i=1}^{n} \Gamma_i^0 \, d\mu_i \qquad [1]$$

Where:

σ - surface tension
η_s - excess entropy in the system
T - absolute temperature
Γ_i^0 - the excess weight of *i-th* component per unit of surface of the interfacial area
μ_i - chemical potential
n - the number of the components

From the expression given in the form of equation 1 is derived adsorption isotherm equation:

$$d\sigma = -\sum_{i=1}^{n} \Gamma_i^0 \, d\mu_i \qquad [2]$$

Or

$$\Gamma_i^0 = \frac{\partial \sigma}{\partial \mu_i} \qquad [3]$$

When the

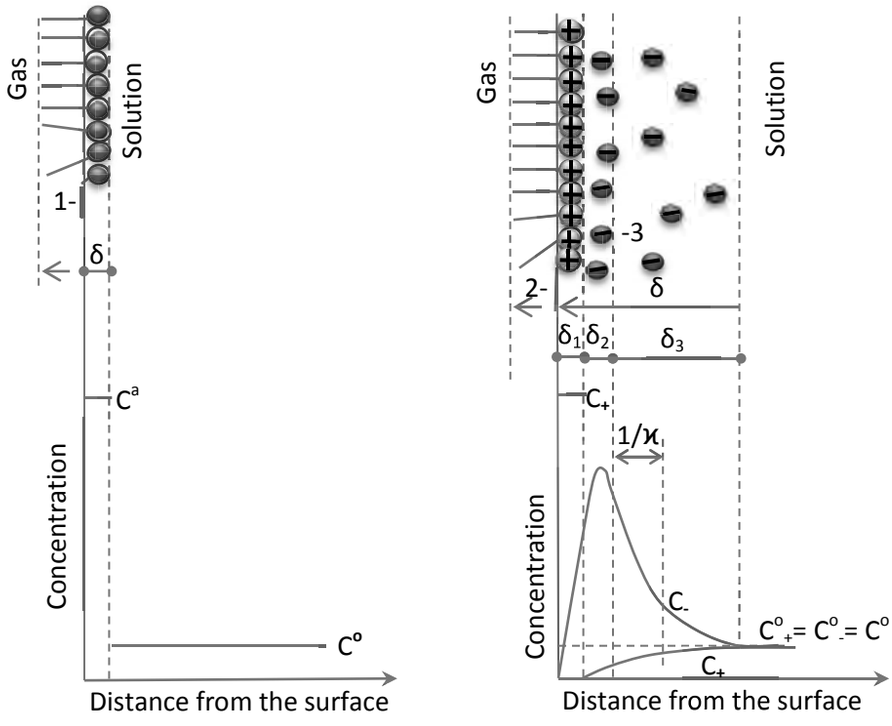
$$\mu_i = \mu_i^{\ominus} + RTlna_i^0 \qquad [4]$$

Where:

$\mu_i^{\ominus}$ - standard chemical potential
a_i^0 - the activity of the *i-th* component in the solution
R - universal gas constant

$$d\sigma = -RT\sum_{i=1}^{n} \Gamma_i^0 \, dlna_i^0 \qquad \text{i} \qquad [5]$$

$$\Gamma_i^0 = -\frac{1}{RT}\frac{\partial \sigma}{\partial lna_i^0} \qquad [6]$$



3. The structure of adsorption layers at the interphase boundary liquid - gas systems for water - dissolved surfactant

In very dilute solution where $a_i^0 \approx C_i^0$, C_i^0- concentracion of *i-th* component in the solution, instead of equation 6 we have:

$$\Gamma_i^0 = -\frac{1}{RT}\frac{\partial \sigma}{\partial lnC_i^0} \qquad [7]$$

Ili

$$\Gamma_i^0 = -\frac{C_i^0}{RT}\frac{\partial \sigma}{\partial C_i^0} \qquad [8]$$

From equation 8 it follows, if the components of the solution reduce the surface tension ($\frac{\partial \sigma}{\partial C_i^0} < 0$) then adsorption on the interphase surface ($\Gamma_i^0 > 0$) and vice versa. In the first case the substance is called surfactant, and contrary.

If we consider the adsorption layer as a single surface phase thickness δ, then for the *i-th* component of the solution can be written:

$$\Gamma_i^0 = (C_i^a - C_i^0)\delta \qquad [9]$$

If Γ_i^0 is adsorption extent, C_i^0 equilibrium concentracion in the solution, while C_i^a concentration in the adsorption layer (Γ_i^0) then from equations 8 i 9 it follows:

$$\frac{C_i^a}{C_i^0} = 1 - \frac{1}{\delta RT}\frac{\partial \sigma}{\partial C_i^0} \qquad [10]$$

Presented equaition shows that in the case of highly active surface active substance $\frac{C_i^a}{C_i^0}$ ratio can attain the value of 10^4 to 10^6 or more. Figure 3 illustratates the structure of the adsorption layer.

a) a nonionic surfactant;
b) ionic surfactant (symmetric binary electrolyte unassociated with surface-active cation);

C^o– concentration in bulk;
C^a – concentration at the adsorption layer;
C_+ i C. - concentration of anions and cations;
1 – surface active molecules
2 – surface active ions
3 – opposite charged ions
Based on the review of the structure of the adsorption layer is obvious that calculations based on equation 10 are illustrative only. More artificial is the notion of homogeneity thickness of adsorption layer in case of ionic surfactants

$$\frac{C_i^a}{C_i^0} = 1 - \frac{1}{\delta RT} \cdot \frac{\partial \sigma}{\partial C_i^0}$$

Driving forces and theoretical aspects of flotation extraction of ions and molecules

The osmotic pressure Π from Van't Hoff-e eq. is

$$\Pi = cRT \quad 11$$

In this equation, *c* is the molar concentration of the solute, *R* is the gas constant and *T* is the absolute temperature at which there are dissolved. The chemical potential of water must be in equilibrium and the same on both sides of the semipermeable **membrane**

$$\boldsymbol{\mu_P^{liquid} = \mu_{P+\Pi}^{solution}} \quad 12$$

In the case of the solution the following applies.:

$$\mu_{P+\Pi}{}^{solution} = \mu_{P+\Pi}^{liquid+RTLn(x_w)} \quad 13$$

Changes in the chemical potential related to changes in pressure are given by the equation:

$$\mu_{P+\Pi}^{liquid} = \mu_P^{liquid} + \int_P^{P+\Pi} V_m \, dP \quad 14$$

Combining equations 2, 3 and 4 assuming that the molar volume of water $(V_m, m^3mol^{-1}) =$ 1,80356E-05m^3mol^{-1} varies very little at a given pressure range, the following applies:

$$-RTLn(x_w) = \int_P^{P+\Pi} V_m \, dP = \Pi V_m \quad 15$$

Therefore

$$\Pi = -k_p \, T \, Ln(1 - x_s) \quad 16$$

Where k_p is piezoscopic constant ($= \frac{R}{V_m}$),

When the x_s is low, following approximation is:

$$Ln(x_w) = Ln(1 - x_s) = -x_s \quad 17$$

And is

$$\Pi V_m = x_s RT \quad 18$$

For small values of x_s

$$x_s = \frac{n_s}{n_w} \quad 19$$

Where n_s is the number of moles of solute n_w number of moles of water

$$\Pi = M_s RT \quad 20$$

Where the $M_s = \frac{n_s}{V}$, is molar concentration of the solution

At low osmotic pressure up to 0,5 MPa, ideal colligative equation has the form:

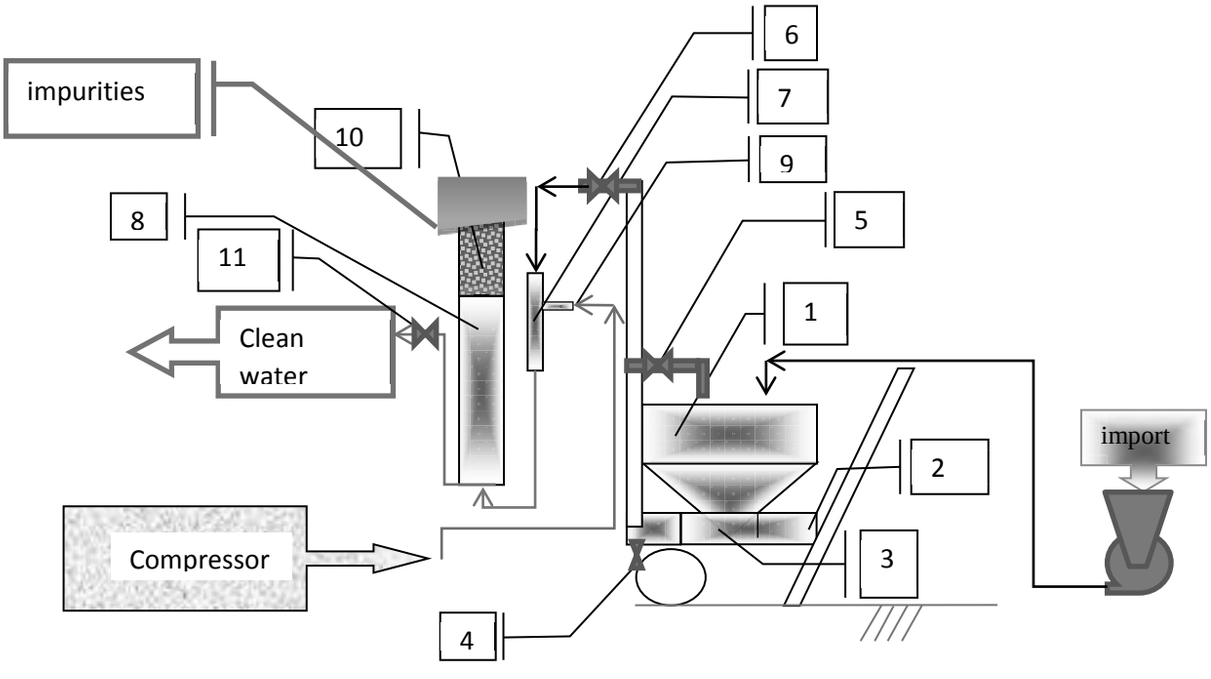
$$\Pi = M_s RT \left(\frac{x_w V^2}{1 + x_s V}\right) \quad 21$$

V is the volume (in liters) of solution containing one liter of water. [4]

Description of the equipment and processes

Flotation extraction device consists of three functionally incorporated continents [5].:

1. Worm pump
2. Injector or sprinklers
3. Column container



Slika 4. Scematic representation of the flotation extraction device

Functional connection of the device is shown in Figure 1. The mobile worm pump (Mozly) has a basket (1) for the incoming pulp, the engine (2), the worm wheel (3) and three outlet orifices from the valve (4) (5) and (6) . In the pump basket the pulp is conditionated with reagent (usually a flocculant) so that the output from the pump is returned into the basket through the return line (5). At the moment when the prepared pulp is put into the column through the injector (7) discharge valve of clean water is closed (11) and valve of the return line (5). Batch filling of the column (8) is carried out through injector element that sucks in the air (9) or to the air being blown through the compressor. In the higher part of the column above the condensed phase (10) is formed a two-phase system characteristic for isobaric and isothermal macroscopic flow of the pulp. The two-phase system increases its volume by continuously full column injector, a meniscus condensed phase, which is actually a semipermeable membrane is moved upward. Dynamic equilibrium of the water molecule as a solvent is established on the semipermeable membrane or meniscus of condensed phases. Mass transfer of the substance or flocculated gel in the case of lareger quantaties of the dispersion is founded in the nature of osmotic presure thermodynamic.

Materials and methods

Tailings resulting in exploatation of copper ore contain dissolved copper salt i.e. the so-called blue water. In order to inhibit precipitation of the copper salts it is necessary to form a compound with sodium-stearate from the soap. Sodium stearate is a salt of sodium and stearic acid. This is one is the basic raw material in the manufacture of soap. The process of obtaining is known since ancient times. [5] obtained by reacting vegetable oils with sodium hydroxide. In addition to the manufacture of cleaning stuffs it is also used as an emulsifier in various creams, shampoos and foodstuffs. It is obtained by saponifying the ester with sodium hydroxide, thus obtaining sodium stearate, and glycerol. In the reaction of sodium stearate and cooper from blue water in the process of flotation extraction, is formed gel-like structure i.e. copper stearate, which is collected on the surface and removed mechanically.

$$2C_{17}H_{35}COO^- + 2Na^+ + Cu^{2+} + SO_4^{2-} = Cu(C_{17}H_{35}COO^-)_2 + 2Na^+SO_4^-$$

Table 1. The values for the calculation of the osmotic pressure of blue water, and soap as a collector which was added to the flotation extraction process

1	2	3	4	5	6	7	8
n, mol	R, $Jmol^{-1}K^{-1}$	T, K	P, Pa	V, m^3	M, $gmol^{-1}$	m, g	h, m
Blue water, a solution of copper sulphate							
The amount of salt in solution	The molar gas constant	Temperature	Osmotic pressure	Volume in which disolved salt is	Molar mass of $CuSO_4$	The weight of dissolved salts	Foam height
0,00414	8,314	293,0	10073,0	0,00100	159,610	0,660	1,027
Sapun							
The amount of soap in solution	Molar gas constant	Temperature	Osmotic pressure	Volume in which soap is dissolved	Molar weight of soap	The weight of dissolved soap	Foam height
0,000424	8,314	293	10333,4	1E-04	306,46	0,13	1,053
9,79E-05	8,314	293	2384,65	1E-04	306,46	0,03	0,219
4,89E-05	8,314	293	1192,32	1E-04	306,46	0,015	0,118
1,63E-05	8,314	293	397,442	1E-04	306,46	0,005	0,040

Osmosis is a colligative property and depends on the amount of solute particles [6]. This feature is used to calculate the required weight of soap as a reagent, for the successful flotation extraction of the blue water. The osmotic potential of pure water is defined as zero. Osmotic potential of the solution has a negative value in units of pressure. At low concentrations of the solution osmotic potential is expressed by Van't Hoff's equation:

$$\psi_\Pi = -M_S RT$$

Blue water with 0.662 $CuSO_4$ g/l has an osmotic potential of about 10000 Pa, table 1, which is enough to hold the water column of about one meter. The experimental procedure is clear in a sense that it is obvious that when froter was added surface tension of liquid is decreased and the foam is formed. Above the surface of the water, upgraded gaseous liquid two-phase system (foam) is formed which can potentially carry the the copper compound. Of course it is to create a copper stearate scum in the form of gel was necessary to add a soap solution. Calculating the amount of soap is done by equalizing the osmotic pressures of blue water and soap as a collector as shown in table 1, column 4. Four different doses of soap in four separate experiment are used, but the best results gave the experiment in which osmotic pressure of soap coincided with the osmotic pressure which produces a solution of blue water. Ordinary laboratory flotation cell with a height of 30 cm would not meet the conditions set height of foam, so this account evidence that there is at least one reason why the column is used in flotation extraction process. A device that is used for flotation extraction of blue water has an important characteristic that highly aerate (in our case) solution, which is only possible to achieve by increasing inlet pressure in the ejector. With the help of worm pump is realized the flow of materials and required excess pressure of 5 bar in order to effectively and efficiently carry out the necessary flotation extraction.

Analysis of the results achieved

After treatment of the blue water with major soap compound (sodium-stearate), a chemical analysis of the product was carried out and is shown in table 1. The FTIR analysis was performed on the device - Thermo Fisher Scientific Nicolet IS-50 by ATR (attenuated total reflectance - plastered total reflection) technique in the range of 4000 to 400 cm^{-1} at a resolution 4. Upon completion of the measurement baseline correction were performed - atmospheric (for elimination of signal gases CO_2 and H_2O) [7].

Table 2. Distribution and chemical analysis of flotation extraction of blue water

	M %	Cu %	Fe %	S %	M*Cu	M*Fe	M*S	R Cu	R Fe	R S
Concentrate	0,74	2,65	0,13	8,23	1,961	0,096	6,090	94,49	33,77	9,87
Output	99,26	0,00115	0,0019	0,56	0,114	0,188	55,58	5,500	66,22	90,1
Feed	100,00	0,0208	0,0028	0,6168	2,075	0,284	61,67	100	100	100

IR (infra-red) spectroscopy is used primarily as a contribution to the structural analysis of organic molecules (as well as inorganic anions) in a manner that is based on the position of absorption bands, by wich functional groups that are present in the molecule can be identified. IR spectrum are vibrational spectrum, i.e. absorption of IR radiation comes from the vibrational transitions in the molecule. When the absorption of radiation occurs there is a change in oscillation frequency of chemical bond between the two atoms. On the basis of the radiation energy that is required for a given vibrational transition, (IR spectrum is ordered sequence of such energies) i.e. from the vibrational frequency change of the chemical bond, it can be concluded that between two atoms linked by chemical bond vibrational transition occurred.

In the case of the gel which during ionic flotation of blue water comes to the surface and for which it is assumed that is the insoluble salt of copper and organic acid soap (it's mostly stearaic, although they are always present to some extent, palmitic, oleic etc.- since the commercial soap was used), in addition to standard AAS analysis to determine the percentage of copper in the sample ("solid" phase which floated up), IR analysis is done to unequivocally shows that the gel is actually insoluble salt containing Cu(II) cations, and organic nature anions (originating from the water-soluble sodium and potassium salts of higher fatty acids in the soap). Therefore, if one looks at the IR spectrum of the dried gel, where one should have in mind that the in drying air all the trapped water can not evaporate completly, we can conclude the following:

The intensive band at (~ 1100) probably originates from the S = O bond (valent vibration of S = O bond) of sulphate anions, that are co-precipitated with the formation of "solid phase".

Band at (~ 1450) comes from the C-H bonds (CH_2 - deformation vibrations, i.e. change in the angle of chemical bonds; the band is in nature of medium intensity, but since the number of CH_2

groups in the molecule is high in this case it is clearly visible).
Poor visible band at (~ 1650) originates from COO^- group – it is a valent symmetric vibration of resonance structures of carboxyl anion).
The band, which should be the highest intensity at (~ 1720), i.e. valent C=O vibration, lacks because separated gel does not contain the fatty acids but fatty acids salts.
Band at (3000 - 3500) originates from the O-H bond (valent vibration, extended due to hydrogen bonds between water molecules - at 3600 there is a small peak of O-H bond which belongs to water molecules that are not conected by hydrogen bond). Therefore the IR spectrum clearly shows that the sample contains molecules with hydrocarbon chain (C-H bond), carboxyl group (COO^-), and water molecules (figure 5).

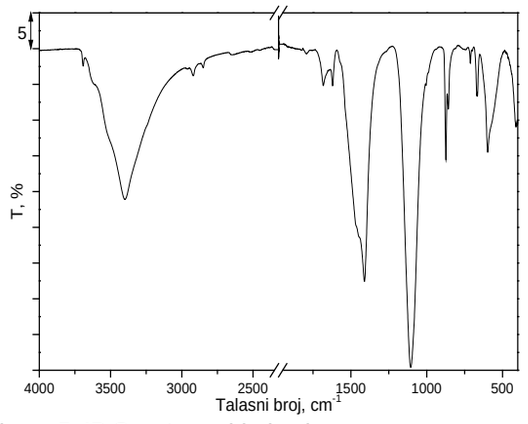


Figure 5. IR Spectrum dried gel

The second IR spectrum is the spectrum of a residue which was crystallized upon evaporation of the solution. Based on the appearance of deposits is assumed that the sediment is mostly blue stone ($CuSO_4*5H_2O$). IR spectrum confirms it (figure 6). It is certain that in the solution of "blue water" there are other salts that were deposited during evaporation together with clearly present (copper(II)sulphate), but those in the IR spectrum are not visible i.e. the IR spectrum indicates the sulfate (sulfite), water, and optionally nitrate (nitrite).
Based on the IR spectrum of the sample obtained by evaporating the solution one can conclude the following:
The intense band at (~ 1100) comes from the S =O bond (valent vibrations) of sulphate anions SO_4^2. Broad band at (~ 3600-2700) comes from the O-H bond. Everything that has been said regarding the presence of water in the sample applies to the residue.
The medium intensity band at (~ 1680) probably comes from nitrate (N=O) valent vibrations. Therefore the IR spectrum clearly shows that the sample contains molecules with hydrocarbon chain (C-H), carboxyl group (C-O band and C=O band), and water molecules.

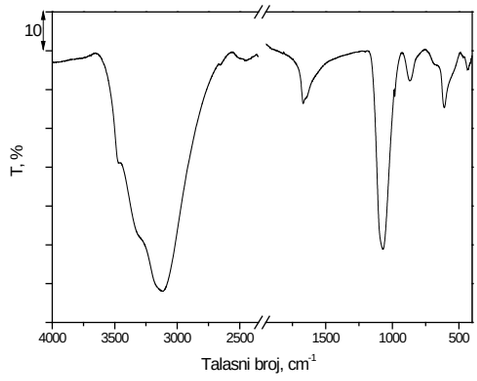


Figure 6. IR spectrum of evapotes solution

CONCLUSION

Any physical system conains the energy of a certain amount. The amount of energy of the system is not an absolute value, but relative to the reference condition or reference level. At the beginning of the twentieth century, *W. Nernst* has performed various fields of chemistry classification based on the type of energy introduced into the system: thermochemistry, electrochemistry, photochemistry, etc. Name mechanochemistry referred to the reactions initiated by mechanical energy eg the reactions that were initiated by the process of friction, breaking, during the mechanical treatment of solid components. The activation energy of mechanochemical treatment system is required to enable the external force to increase the surface of the material, to affect the reagents in the flotation of various pneumatic suspension. The processes of enrichment of mineral resources in flotation systems are based on electrochemistry, adsorption and catalytic functioning of the individual reagents to separate the components of such systems. In order to achive solute transport to form the for the gel to the surface in the flotation extraction process, the activation energy must have such an impact on the system and the energy barrier that resists that transport should be overcome by the external force. Adsorption extent refers to the gaseous fluid, and in a solid there is a change of intermolecular and intramolecular forces. Intramolecular forces are the forces that hold together the atoms and

thus form a molecule or compound. Intramolecular forces include all types of chemical bonds. They are stronger than the intermolecular forces that occur between the atoms or molecules which are not bound. The activation energy of the reacting system in one device (reactor) plays a very important role in the physical characteristics of the system in terms of the selective separation of components of the system. It is shown that one can calculate the intensity of the intramolecular force as the force of osmotic pressure, and it can be used to calculate the necessary amount of reagents for successful flotation extraction.

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