Exercise no. 6. **Building: A2, room 110**

Environmental protection in chemical technology - laboratory

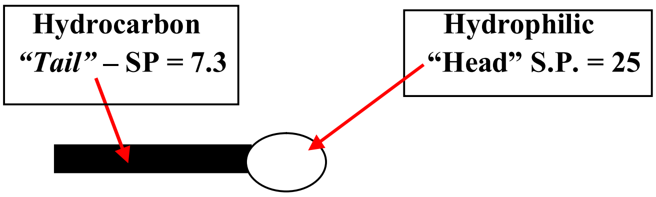
Cloud Point Extraction

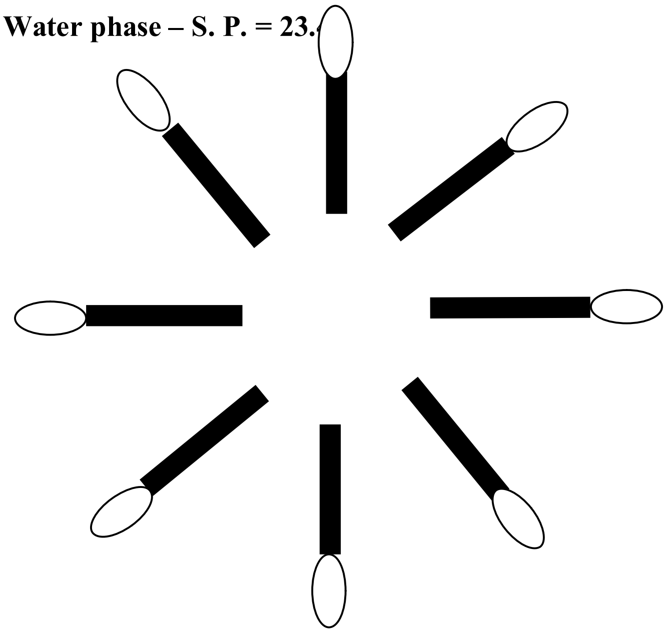
Prepared by Joanna Wolska

INTRODUCTION

In English the term **surfactant** (short for ***surface-active-agent***) designates a substance which exhibits some superficial o interfacial activity. It is worth remarking that all amhiphiles do not display such activity; in effect, only the amphiphiles with more or less equilibrated hydrophilic and lipophilic tendencies are likely to migrate to the surface or interface. It does not happen if the amphiphilic molecule is too hydrophilic or too hydrophobic, in which case it stays in one of the phases.

An amphiphilic substance exhibits a double affinity, which can be defined from the physico-chemical point of view as a polar-apolar duality. A typical amphiphilic molecule consists of two parts: on the one hand a polar group which contents heteroatoms such as O, S, P, or N, included in functional groups such as alcohol, thiol, ether, ester, acid, sulfate, sulfonate, phosphate, amine, amide etc… On the other hand, an essentially apolar group which is in general a hydrocarbon chain of the alkyl or alkylbenzene type, sometimes with halogen atoms and even a few non-ionized oxygen atoms [1].





The most accepted and scientifically sound classification of surfactants is based on their dissociation in water [1]:

- **Anionic Surfactants** are dissociated in water in an amphiphilic anion, and a cation, which is in general an alcaline metal (Na+, K+) or a quaternary ammonium.
- **Nonionic Surfactants** do not ionize in aqueous solution, because their hydrophilic group is of a nondissociable type, such as alcohol, phenol, ether, ester, or amide. A large proportion of thesenonionic surfactants are made hydrophilic by the presence of a polyethylene glycol chain, obtained by the polycondensation of ethylene oxide. They are called polyethoxylated non-ionics.
- **Cationic Surfactants** are dissociated in water into an amphiphiliccation and an anion, most often of the halogen type. A very large proportion of this class corresponds to nitrogencompounds such as fatty amine salts and quaternary ammoniums, with one or several long chain of the alkyl type, often coming.
- **Amphoteric** or **zwitterionic** when a single surfactant molecule exhibit both anionic and cationic dissociations. This is the case of synthetic products like betaines or sulfobetaines and natural substances such as aminoacids and phospholipids.

Surfactants find applications in the various fields of industry, agriculture, and household. The versatility of the applications is enormous. Some applications are connected with adsorption abilities of surfactants having an amphiphilic character. Other applications exploit the abilities of surfactants to form micelles.However, surfactants are often used in such concentrations that both adsorption and micellization phenomena are exploited [2].

Extraction is a well-known technique used to recover and separate various substances including undesirable organic pollutants. The use of hydrocarbons as diluents is the main drawback of extraction [2].

Nonionic surfactants or polyoxyethylene glycols dissolved in aqueous solutions can be separated as a surfactant-rich phase or a glycol phase by the breaking of the hydrogen bonds between oxygen atoms of polyoxyethylene chains and water molecules. That breaking can be achieved by an increase in temperature or by the addition of appropriate electrolytes. The former is used to separate the surfactant phase above its cloud point. The separated surfactant-rich phase is less hydrophilic than the micellar aqueous phase. As a result, various organic substances can be transferred to the surfactant phase and significantly enriched as the volume of the surfactantphase is one order lower than the volume of the aqueous phase. The technique can be also used to recover heavy metals. However, a hydrophobic complexing agent must be used to decrease the hydrophilicity of the hydrated metal species [2].

Polyglycol systems are also suitable for separations of both organic substances and metal species after their complexation. The use of the cloud point technique seems more advantageous as the clouding is reversible and transparent solutions are obtained after cooling [2].

EXPERIMENTAL:

1. Weight into the two beakers (1 and2) 2.5 g of TritonX-100 (2.5% w/v solution)
2. Weight a p-nitrophenol, in order to obtain 0.05g/Land 0.2g/L solution of p-nitrophenol
2. Add the weighed amount of p-nitrophenol to individual beakers 1and2, then add more portions of water to obtain 100 g of the solution in each beaker. The content of each beakers stirred at room temperature for 30 min.

3. Put 1 mL of obtained solutions (1 and 2) to the flasks 0-1 and 0-2 with a capacity of10 mL, and full to the line with water.
4. Put to the small beakers 1a and 2a a 1 g of NaCl.
5. Solutions of the big beakers 1 and 2, divided into two equal parts, and transfer measured amounts of solutions to the 4 small beakers (1, 1a, 2 and 2a).
6. The solutions in the beakers 1, 1A, 2and 2A heated by observing changes in the appearance of each sample depending on the temperature and time. Observations note in the report.
7. After the clouding of each solution cool them down and then taken from the top layer of each sample 1mL of the mixture to flasks (1, 1a, 2 and 2a) with the capacity of 10mL and full them with water to the line.
8. Then measure the UV spectrophotometer extinction value for the solutions before and after clouding at a wavelength of λ= 318nm.

CALCULATIONS

Make the calibration curve for the sample 0-1 and 0-2.
Determined the concentration of p-nitrophenol in the aqueous phase after the clouding in each of the solutions.
Determine the degree of separation for each of the following formula:

$P = (1 - \frac{C_K}{C_O}) \bullet 100\%$, in which:

P- the degree of separation
C_K- the concentration of p-nitrophenol in the aqueous phase after clouding in each beaker 1, 1A,2, 2A,

C_0 – the concentration of p-nitrophenol at the beginning

References:

[1] http://www.nanoparticles.org/pdf/Salager-E300A.pdf
[2] K. Materna, J. Szymanowski, *J. Coll. Interface Sci.*, **255** (2002) 195-201.

Attention

The report should contain:

1. **the object of exercises,**
2. **short introduction,**
3. **the measurements results in the tables,**
4. **all calculations,**
5. **conclusions**