Problem IV.P ... sieves

10 points

Propose as many methods as possible for separating substances from a mixture and provide a detailed explanation of the physical principle and experimental procedure for at least three of them. Each one of the three methods described in detail should be based on a different physical or chemical property of the substances.

Káťa is preparing for the life of an experimental physicist :D

Introduction

Firstly we shall define types of mixtures and briefly describe their attributes. Afterwards nothing will stop us in discussing three chosen separation methods and to conclude we will shortly introduce a few more methods.

Mixtures

There are three basic types of mixtures being homogeneous, heterogeneous and kollodial. Although all of them are composed of at least two chemically pure substances, which do not react with each other; small differences can be found between them.

Homogeneous mixture Homogeneous mixtures are characterised by their attribute, that the size of dissolved particles of each substance is lower than $10^{-9}\,\mathrm{m}$ and there is no interface between them. Their key attribute is that in all their volume they have the same physical quantities (e.g. boiling point and solidification temperature, density, composition, etc.). They can exist in all states of matter, e.g. air as gaseous, seawater as liquid mixture (*mixture*) or bronze as a solid substance (*alloy*).

Heterogenneous mixture Quite differently from homogeneous mixtures, heterogeneous mixtures do not have clearly defined composition or physical quantities in all their volume. Mixtures are typically composed of multiple phases which are divided by interfaces. In these interfaces the physical quantities change rapidly between parts of the system. Particles of these systems are usually greater than 10^{-7} m. Two-component heterogeneous mixtures are classified based on the state of matter of their components into following categories:

- solid in liquid—*suspension*, e.g. sand in water;
- liquid in liquid—*emulsion*, e.g. mixture of oil and water;
- gas in liquid—*foam*, e.g. shaving foam;
- liquid in gas—*mist*, e.g. classic mist;
- solid in gas—*smoke*, e.g. smoke from fire.

It is essential to mention that sometimes the difference between colloidal and heterogeneous mixture is barely noticeable.

Colloidal mixture Colloidal mixture is a name for all the dispersion systems which according to the size of the particles until the interface between homogeneous and heterogeneous mixtures (from 10^{-9} m to 10^{-7} m). They have a number of interesting attributes for example their particles can execute weaker thermal movement and diffund. On the other not all of the colloidal mixtures are stable and able to sedimentate or agglomerate. Investigating coloidal mixtures is the point of interest of colloidal chemistry. Different gels, aerosols and colloidal solutions (colloidal silver and gold, nanodiamants etc.). In the future they could have significant usage in nanotechnologies.

Separation methods

This notion is the collective designation for physical-chemical processes by which we are able to separate individual components of the without changing their chemical essence. Therefore for example electrolysis is not classified as a separation method. Next let's describe the most notable separation methods.

Distillation Distillation is separation method which enables us to divide the of multiple liquids with different boiling temperatures. In the following paragraph we will try to clarify the principle which is responsible for proper functioning of the distillation process because it is often explained insufficiently in other problems.

To simplify we shall assume that we are working with homogeneous mixture of liquids which has its own boiling point. This boiling point being in between the boiling points of its components. While distillating we heat the to its boiling point where it starts to rapidly evaporate (both components are being evaporated at the same time, not only the one with lower boiling point). Gaseous however won't have the same composition as the liquid , because the component with lower boiling point will evaporate more quickly than the one with higher boiling point. This whole process is described by Raoult-Dalton's law. To simplify we won't explain the individual equations but we can show you how do the boiling point and weight ratios of components in steam change in relation to weight ratios of components in liquid for a concrete case. Graph of such relation is typically called distillation or component curve. For example the distillation curve of ethanol and water is shown in the given picture 1. [1]

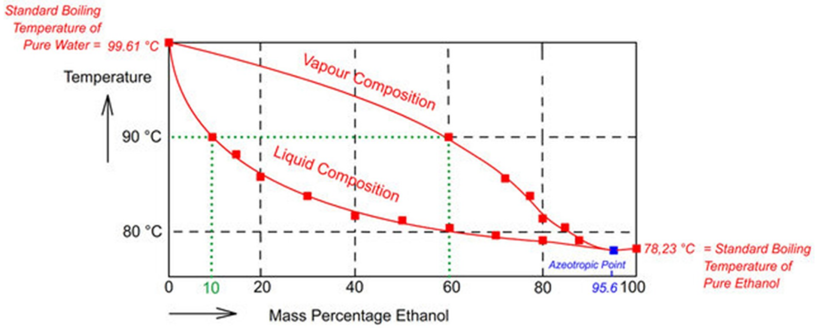

Figure 1: Distillation graph for the of ethanol and water.

Horizontal axis shows weight ratio of the ethanol in water and the vertical axis represents boiling point of the mixture. The graph itself is composed of two curves. The lower curve describes the equilibrium of the liquid (we are able to determine how the boiling point of the mixture changes according to its composure) and the higher curve describes the equilibrium in saturated steams (for any given temperature and composure of the liquid we can predict the composure of the formed steams).

In the picture 1 there are nicely visible limits of this separation method. Using distillation we can never fully divide the components of homogeneous mixtures of liquids, the resulting distillate will always be impure (on the other hand many separation methods have similar disadvantage resulting from thermodynamics). The closer we get to acquiring a pure distillate the closer will the two curves on our graph get to touching each other and the effectivity of distillation will decrease accordingly. Furthermore in some cases the curves may connect and thus form an azeotropic point which will decrease the efficiency even further. To solve the problem of azeotropic point there are a few tricks for example adding a different component into the mixture. This leads to changes in distillation graph.

For common distillation we need a source of heat (usually a burner), distillation flask, thermometer with a stopper, refrigerator, refrigerant (in most cases cold water) and a beaker for collecting the distillate. Simple apparatus is displayed in the picture 2. [2]

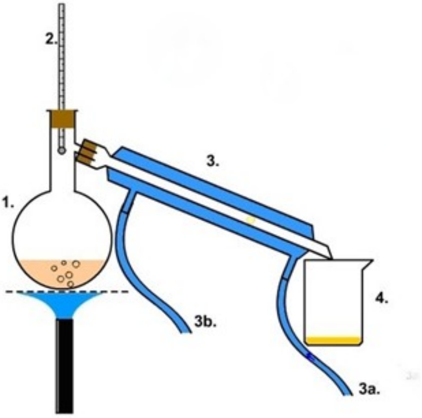

Figure 2: Schematic representation of the distillating aparatus: 1. distillation flask with the mixture, 2. thermometer with a stopper, 3. refrigerator, 3a./3b. inlet and outlet of the refrigerant, 4. beaker with the distillate.

The procedure is not hard, firstly we construct the apparatus according to the picture 2, next we pour the mixture into the distillation flask and we close it with a stopper with a thermometer. We open the inlet of water and we start to heat the mixture. At the same time we keep an eye on the temperature of the steams and when it considerably increases above the formerly established temperature we end the distillation and collect the distillate from the beaker. This process is only effective if the components of the mixture have boiling points sufficiently distanced from each other and the concentration of distilled substance is not high from the get go. Otherwise it is needed to repeat the procedure.

To increase the effectivity in the industry they use a rectification column. That is composed of multiple tiers where the steams condense and evaporate which results in higher concentrations of distillated substance at the end of the column. Essentially it is repeated simple distillation but it happens all at once in a single column. If we use it to distillate a mixture with more components (e.g. oil) and from certain tiers we will take away individual components then we are speaking of fractional distillation column. Schematic sketch of a rectification column with assigned points from distillation graph is shown in the picture 3. [3]

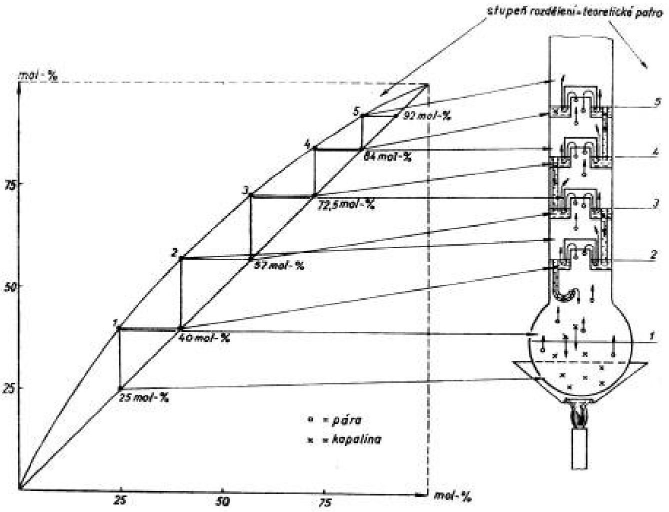

Figure 3: Scheme of a rectification column with assigned points from distillation graph.

Distillation is an important industrial method and it is the basis for most of the chemical industry. On the contrary it is less frequently used in the laboratories for its higher difficulty and lower effectivity and if possible is substituted by easier and most importantly more effective method.

Chromatography Chromatography is an interesting separation method which serves to separate substances based on their different affinities for stationary and mobile phase. Stationary phase is typically formed of more porous solid substance (e.g. silica gel, calcium carbonate, etc.), the mobile phase is either liquid or a gas, according to this chromatography is classified as liquid chromatography or gas chromatography. Stationary and mobile phases have different physicochemical attributes, so the examined will have different affinity towards individual phases (it is about attributes of the elementary molecules and atoms—chromatography is able to divide even multi-component homogeneous mixtures).

To perform a simple chromatography on a thin layer a TLC plate (aluminium plate on which a silica gel is applied—stationary phase), eluent (of multiple components—mobile phase), beaker, laboratory watch glass and a pencil. Schematically shown in the picture 4. [4].

First step is the preparation of the TLC plate, this lies in drawing two thin parallel lines with a pencil. The first of them will represent the starting line on which we will apply the researched mixture. The second line will be the finish line—when the eluent solution is applied, we can finish the chromatography. On starting line we will apply (ideally with a capillary) drop by drop the analyzed mixture and we are keeping an eye for the mixture not to soak. After it dries off we will insert it into the pre-prepared beaker with eluet solution while its

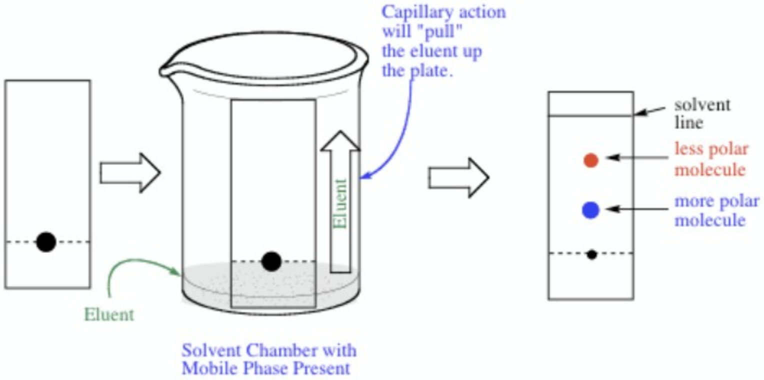

Figure 4: Sketch of an experimental arrangement for a chromatography on a thin layer.

surface must be under the starting line on the plate. The beaker should be covered during the chromatographic process to prevent evaporation. We shall cover it with a laboratory watch glass or Petri's dish and we observe the development.

When the eluent solution's surface matches with the finish line, we will take the plate out of the beaker and let it dry up. If it does not match the finish line we mark the spot to which the surface of the eluent solution really rose. After it dries off we observe how high from the starting line did the individual components of the mixture get. The ratio of these heights to the final height of the eluent solution is called retardation (retention) factor. From its value using tables we can estimate which substances we have separated from the mixture (the estimate is clearly dependent on the used mobile and static phase and that is why it is necessary to use a proper chart). The component with higher affinity towards stationary phase will rise higher than the component which has higher affinity towards mobile phase.

Another way of conducting a laboratory chromatography is creating a column which we will fill up with stationary phase that will push on the mobile phase. The advantage is that on the end of the column the separated components of the mixture will emerge one after another.

Chromatography found its application primarily as an analytical method due to its ability to effectively seperate components from even a small sample of a mixture which allows for the rest of the mixture to be examined by some other method. It is widely used in criminalistics, healthcare, organic synthesis (review of purity of a sample) etc. It is not commonly used in industry because its incapable of separating large volumes of mixtures.

Extraction Extraction is a separation method which uses variable solubility of a substance (or substances) in various solvents immiscible among themselves. Typically polar and non-polar liquids with different densities are used. As a polar liquid we can use water or some water solutions of salts, diluted acids and hydroxides. As non-polar liquids most commonly used are organic solvents which cannot be dissolved in water or otherwise dissolve water. Similarly to distillation also in extraction we can never really get a pure substance or extract whole component from a mixture.

The goal of extraction is transmission of dissolved substance from non-polar solvent to polar or vice versa. In laboratory we can carry it out using separation funnel which we can place on a laboratory stand with a holder. Firstly we will pour the liquid with a higher density (usually polar water phase) into the funnel, secondly we pour the lower density liquid (usually non-polar organic phase). Afterwards we will close the funnel and carefully shake it using this motion we transfer a certain part of the dissolved liquid into the second solvent.

We can perform this process up to 30 seconds. Next we shall place the funnel back into laboratory stand, we will remove the lid and wait until the interface between liquids is formed again. Then we will place a beaker underneath the funnel and by opening the ventil on the bottom part we will pour the extract—the denser component in this case enriched by the dissolved substance. The valve must be closed such that the interface of the liquids is kept visibly inside of the valve (by sticking to this rule we can evade the contamination of the solvents). Lastly we will drain the refined product—solvent with lower density (impoverished by the dissolved substance).

The extraction also has an industrial usage instead of using a separation funnel it is being done in an extraction column. The example of such a device is displayed in the picture 5. [5]

Figure 5: An example of industrially used extraction column.

To increase the effectivity the extraction can be repeated. Sometimes while conducting an extraction we can help ourselves with a chemical reaction. We can for example let some organic substances react with an acid or a hydroxide by doing this we can increase their polarity and solubility in water. If we want to get the original composition we must conduct the reaction in an opposite direction during the next extraction.

Other than liquid-liquid extraction, which we have described earlier we know solid-liquid extraction (more commonly known as leaching). That however requires a specific experimental equipment.

Some other examples of separation methods To conclude we provide a few more of the separation methods with a short description and a practical application.

Filtration: separates the components based on the size of the particles (mostly solids from liquids or from gasses). We encounter it everyday in water purification or air filters, vacuuming or fuel filters of transport vehicles.

Sedimentation and centrifugation: both methods are separating the components based on their different densities. Sedimentation is working only with a gravitational force and compared to centrifugation based on centrifugal force it is significantly slower. Furthermore if the densities of components are too similar it makes it impossible to separate them using sedimentation due to diffusion and thermal motion. On the other hand centrifugation requires usage of technology. We can encounter sedimentation in wastewater treatment plants. Centrifugation is usually used in medicine and in biochemical laboratories.

Magnetic separation: uses different ability of substances to interact with magnetic field. Typically it is used to separate ferromagnetic components from ores or to extract the metals out of the waste.

Crystallization: uses different solubility of two different substances in liquid. It is used in mining salt from sea water and in various purification processes.

Electrophoresis: is based on a principle of different mobilities of substances in electric field. It has an application in biochemistry in studying and dividing of proteins.

Sublimation: it is similar to distillation, it works on the principle of one substance to sublimate or to sublimate faster than the other substance. It is usually used to clean a sublimated substance.

Osmosis and reverse osmosis: is used to separate a soft membrane through which only a solvent can cross. If we add some dissolved substance then the newly created osmotic pressure will begin to suck in water from the other side of membrane (the one without a dissolved substance). If this pressure is mechanically overcome, we are dealing with a reverse osmosis which has found its purpose in producing a drinking water from sea water.

Dialysis: similarly to how osmosis uses a soft membrane which can also be crossed by some soluble substances up to a certain size. Typically these include salts and simple compounds such as urea; more complex organic components are unable to cross the membrane. It is being applied in medicine.

References

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