Dye adsorption on charcoal

THEORY

➢ ABSORBANCE

When light flux of monochromatic beam of intensity I_0 passes an environment, a certain part of the radiation intensity will be absorbed and the radiation is reduced to a value of I. The percentage ratio of the initial light intensity and the intensity of that radiation after passing through a substance is called radiation transmittance T:

$$T = \frac{I}{I_0}$$

In measurements and calculations logarithm of the inverse value of the transmittance is frequently used, it is called absorbance A (older term extinction; E):

$$A = \log\frac{I_0}{I}$$

Absorbance (and hence the transmittance) is dependent on the wavelength of the radiation, absorbing material and its concentration, on the layer thickness which radiation passes, and the direction of rays. The relationship between the absorbance, the layer thickness and the concentration of certain substances, defined for monochromatic light, expresses **Lambert-Beer law**:

$$A = \varepsilon \cdot c \cdot l\,,$$

where c is the concentration of the substance (mol · dm^{-3}, g · l^{-1}), l is the thickness of the absorbing layer, ε is the molar decadic absorption coefficient (when the concentration is in mol · dm^{-3}), or specific absorption coefficient (if the concentration is expressed otherwise, eg. g · l^{-1}).

It is therefore evident that if we have a device that can measure the absorbance, we are also able to detect the concentration of absorbing substance. This can be used to determine the adsorption capacity of activated carbon.

➢ ADSORPTION

Adsorption of substances takes place at the interface of phases. The most common cases are adsorption at the interface solid – gas and solid – liquid. To ensure maximum adsorption of a substance (adsorbate) a solid phase (adsorbent) should have the largest surface possible. As adsorbents are therefore used porous powdery materials. Some of these substances are used as intestinal adsorbents in diarrheal diseases (eg. activated charcoal or diosmectin).

Adsorption from solution can be observed e.g. in organic dyes. When activated charcoal is added to the solution, after shaking portion of the dye is adsorbed on its surface and after filtering the color of the solution is therefore less intense. After a sufficiently long contact time with the surface dye

adsorbent establish adsorption equilibrium. Then at a constant temperature it is possible to express dependency of adsorbed amount of dye a on its equilibrium concentration c in a solution by the **Langmuir adsorption isotherm**:

$$a = a_{max} \frac{\omega c}{1+\omega c},$$

a is the amount of dye adsorbed; a_{max} is the maximum adsorbed amount, ω is so-called adsorption coefficient characterizing the ability of the substance to adsorb onto the sorbent. It corresponds to the inverse value of the concentration at which half the coverage is achieved ($a = a_{max}/2$). Adsorbed amount of colorant a is quoted often in mg of dye per g of adsorbent:

$$a = \frac{m_1 - m_2}{m},$$

where m_1 is the weight of the dye in solution before adsorption (mg), m_2 is the weight of dye in solution after adsorption (mg), and m is the sample weight of adsorbent, here activated charcoal (g).

Parameters of adsorption isotherm (a_{max}, ω) are determined graphically and for the given adsorption system they are obtained from the linearized form of the Langmuir isotherm:

$$\frac{1}{a} = \frac{1}{a_{max}\,\omega} \cdot \frac{1}{c} + \frac{1}{a_{max}}$$

If $1/c$ is chosen as the independent variable and $1/a$ as the dependent variable, then the graphical representation of this dependence is a line with slope $1/(a_{max} \cdot \omega)$ and the intercept on the axis $1/a$ equal to $1/a_{max}$. From the slope and the intercept of linear regression of dependence $1/a$ on $1/c$ can therefore be these adsorption parameters calculated.

TASK

- Construct a calibration curve of dependence methyl orange absorbance on its concentration in solution.
- Determine the dependence of adsorbed amount of methyl orange on its equilibrium concentration in solution at constant temperature (i.e. adsorption isotherm). Determine the Langmuir isotherm constants for methyl orange.

EQUIPMENT AND CHEMICALS

- Spectrophotometer; shaker; 12× 100ml volumetric flask; 12 × titration flask (or Erlenmeyer flask); graduated pipette (5, 25 ml); pipette extender; 6× funnel; filter paper; weighing boat; spoon; 2× beaker; 4× spectrophotometer cuvette; stock solution of methyl orange (1 g · l^{-1}); activated carbon.

PROCEDURE

- To construct the calibration curve prepare six solutions containing progressively 0.1 to 1.6 mg of methyl orange (0.3 mg step) per 100 ml solution. Calibration solutions are

prepared by dilution of the stock solution of methyl orange of concentration of 1 g · l^{-1}. At the highest concentration solution we find an absorption maximum in the wavelength interval 435 – 495 nm; measurements are performed by 10 nm. At the value of the maximum absorption wavelength we measure each calibration solution 3 times, so that to the cuvette a new dose of solution is to be poured in. A calibration graph contains the dependence of the mean absorbance values on concentration.

- Into six 100 ml flasks we prepare solutions containing 15 to 30 mg of methyl orange (3 mg step), again by dilution of the stock solution. To six titration (or conical) flask, add 80 mg of active carbon which we weighed on the weighing boat on an analytical scales with an accuracy of one milligram. All prepared methyl orange solutions are then poured into individual flasks with charcoal at the same time, the flask are stoppered and finally placed on a shaker for 35 minutes for adsorption equilibrium. Then the solutions are filtered and the filtrates measured at spectrophotometer. In the case that the measured absorbance values of one of the solutions exceeds the absorbance of the most concentrated calibration solution, the solution is diluted appropriately and during the data evaluation these concentrations values are recalculated. **To save time, it is recommended to start with this part of the task!**

PROTOCOL

- Calculation of dilution for preparation of all solutions.
- Table of measured absorbance for each wavelength of the most concentrated calibration solution.
- Calibration table and graph: methyl orange absorbance dependence on calibration solution concentration (mg · ml^{-1}).
- Table with columns: sample number, active charcoal weigh m (g), solution absorbance A, the equilibrium concentration of dye in solution c (mg · ml^{-1}) calculated from the equation linear regression calibration curve, dye weigh in solution before adsorption m_1 (mg) and after adsorption m_2 (mg), adsorbed dye weigh a (mg · g^{-1} of active charcoal), $1/a$, $1/c$ values.
- Graph of the dependence of a on c.
- Graph of the dependence of $1/a$ on $1/c$. From the linear regression calculation of parameters of the adsorption isotherm a_{max} and ω, including units.

Operating instructions for Spectrometer SPEKOL 11

- Connect the device to the mains supply. Press the button (1). Light diodes above the buttons T, E, C, CAL, FL, KIN start blinking.
- Press the button (E) for selection the required measurement method (E is a symbol for extinction, which means A or absorbance). The device is let by for **20 minutes to warm up**.
- Set the wavelength by rotary knob (3).
- Insert the cuvette (5) with the reference solution (distilled water) into the right side of the cuvette space (4).
- R (6) is blinking. Press the button R to reset the device. The display (7) shows the value of 0.000. Green diode above R goes out.
- Insert the cuvette with the measured solution into the left side of the cuvette space (8). Write down the measured value.
- When changing the wavelength it is always necessary to reset the absorbance using reference solution (press the button R).
- After the measurement remove the cuvettes from the instrument, wash them, press button (1) and disconnect the device from the mains supply.