LAB EXPERIMENTS

Adsorption

Framework
This module explains the lab experiments on adsorption.

Contents
This module has the following contents:

1. Objective

Adsorption on activated carbon is used for the preparation of drinking water from surface water or riverbank filtrate. A good removal of taste and odour is achieved by dosing carbon powder or filtration over granular carbon. Another application of activated carbon is the removal of micro-pollutants (e.g. atrazine, a pesticide). Filtration over granular carbon beds is mostly used. In this experiment a test installation is used to learn about adsorption on activated carbon. The purpose of this test is the application of theoretical background. Design rules are developed using the measurement data that are evaluated with a computer model. This computer model is suited for the prediction of the behaviour of activated carbon filtration when loaded with specified matter. To apply this computer model specific constants that describe the adsorption process need to be known from literature or by experiments. With the help of obtained results a cost optimum is made for a full-scale installation.

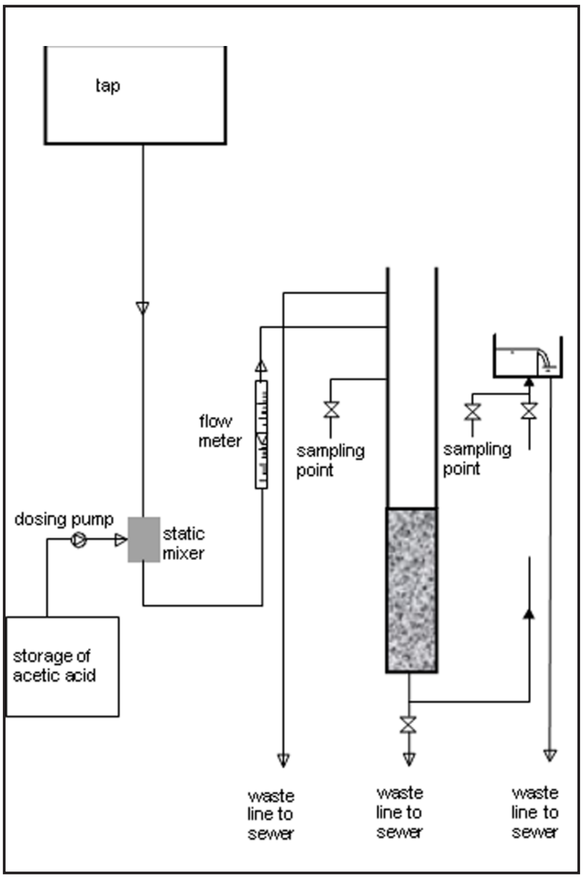

Figure 1 - Adsorption, experimental setup

2. Experiment set-up

The pilot plant (Figure 1) exists of a column, internal diameter 100 mm, filled with granular activated carbon and a storage tank with an acetic acid solution. Acetic acid (CH_3COOH) is the substance to be adsorbed. Tap water flows through the installation. The acetic acid is dosed into the influent with a dosing pump, whereupon it is thoroughly mixed with the tap water. The filtration rate is adjusted with a valve and controlled with a flow meter. For practical reasons an open column has been chosen for this experiment. More water is supplied than is filtrated through the activated carbon. The surplus flows over the top of the column into the sewer. A sampling point is installed on the column above the filter bed and on the effluent pipe. The acetic acid concentration of the samples is determined by titration with a sodium hydroxide solution.

3. Theory

The amount of a substance that can be adsorbed on activated carbon depends on the nature of the substance and its concentration, the surface structure of the activated carbon and the temperature and pH of the water. For a treatment system with a specific type of carbon and a known substance, there is a relationship between the amount of adsorbed matter per unit of weight of carbon and the equilibrium concentration in the water, when temperature and pH are constant. This relationship is called an isotherm. The shape of the isotherm can be described in various mathematical ways. The most well known is the Freundlich isotherm:

$$q_{max} = K \cdot c_e^n$$

in which:

q_{max} = adsorption capacity of carbon (g/kg)
c_e = equilibrium concentration of the substance in the water phase (mg/l)
K = Freundlich constant (g/kg / (mg/l)^n)
n = constant (-)

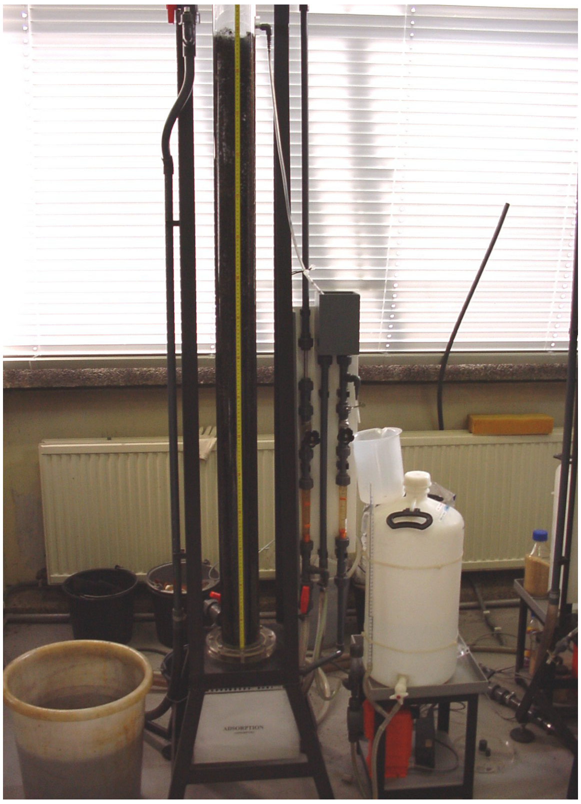

Figure 2 - Adsorption installation in the laboratory

Under specific circumstances the values of K and n can be found experimentally by determining the adsorption capacity of the activated carbon at different concentration of the substance.

When a certain effluent quality is required the determination of a so-called breakthrough curve is important. The breakthrough curve is obtained by plotting the effluent concentration as a function of time or Bed Volumes (BV).

For a column with a cross section of A (m^2) and a bed height of H m the bed volume is V_b = A.H (m^3). At a flow of Q (m^3/h) the filtration rate is v = Q/A (m/h). The EBCT is the apparent residence time of a water particle in an empty bed with the same volume V_b. EBCT = V_b/Q = H/v (hours). After a run time of t hours the amount of treated bed volumes is BV = t.Q/V_b = t/EBCT. In this way BV is expressed as a time parameter

The change of concentration can be modelled as follows:

$$\frac{dc}{dt} = -\frac{u}{\varepsilon}\frac{dc}{dx} - k_2\left(c - \sqrt[n]{\frac{q}{K}}\right)$$

$$\frac{d\bar{q}}{dt} = -\frac{u}{\rho}\frac{dc}{dx}$$

in which:

c = concentration of the specific substance (g/m^3)
k_2 = mass transfer coefficient (s^{-1})
ρ = density of the activated carbon, bulk weight (kg/m^3)
q = loading of the activated carbon (g/kg)
u = filtration velocity (m/s)
ε = porosity (-)

4. Procedure

Execute the following procedure steps:

- check if the column is filled with activated carbon bed and write down the bed height.
- open the valve in the influent pipe a-d adjust the flow to 100 l/h. Check the flow regularly.
- open the valve in the effluent pipe and adjust the flow to 80 l/h. This gives a filtration velocity of 10 m/h (by overflow at the top, 20 l/h disappears). Check the flow regularly.
- switch on the dosing pump of HCl. The dosing rate is already set to the right value. Continue dosing until the pH of the effluent is below 3.5.
- Take a sample of the effluent and determine the buffering capacity of the water without acetic acid.Then switch of the HCl-dosing.
- measure the height of the water column above the bed and calculate the time lag (the time it takes for a water particle to flow from the inlet to the surface of the activated carbon bed). The

supernatant still doesn't contain acetic acid (CH_3COOH).
- switch on the dosage pump for the acetic acid and adjust the flow to 1 l/h. At the same time start the chronometer. Check the dosage flow regularly and adjust if necessary.
- after 10 minutes take an influent sample of 50 ml and determine the acetic acid concentration. Repeat this after 1.5 hours. Both concentrations should be the same.
- take after 15 minutes, followed by 15-min intervals effluent samples of 50 ml and determine the acetic acid concentration.
- stop the experiment when the effluent concentration exceeds 80% of the influent concentration.

Points of reference

Acetic acid concentration	= 80 g/l
	= 80.10^3 mg/l
Bed height	= about 1 m
Type of activated carbon	= ROW 0,8s Norit
Bulk density of carbon	= 400 kg/m³
Influent flow	= 100 l/h
Effluent flow	= 80 l/h
Filtration rate	= 10 m/h

5. Elaboration

Execute the following steps:
- draw a graph of the measured effluent concentration as function of time and BV.
- after how many bed volumes is the effluent concentration 50% of the influent concentration?
- calibrate the Stimela model, assuming the Freundlich constant n = 0.5.
- simulate the breakthrough curve of acetic acid at an influent concentration of 2 μg/l, using the Stimela model and the pre-calculated values for the calibration parameters.
- determine the most economical filterbed volume for a full-scale installation with an average flow of 5000 m³/h?
- the acetic acid concentrations of the influent concentration is 2 μg/l and the guidline value is 0.1 μg/l. Simulate EBCT of 5,10,20,30,40 and 50 minutes.
- regeneration costs for the activated carbon are 250 euro per m^3 of activated carbon.
- yearly costs for the filtration construction are € 180 per m^3 of activated carbon.
- what are the optimal dimensions regarding hight and diameter and number of the colums?

6. Analysis of acetic acid concentrations

Execute the following steps:
- use the titrator to obtain the ml NaOH needed to bring the pH above 8.4.
- make a correction for the buffering capacity of the water without acetic acid
- calculate the acetic acid concentration in all samples.

Data form

Group number: Date

Molarity sodium hydroxide : M
Influent flow rate : l/h
Effluent flow rate : l/h
Filtration velocity : m/h
Bed height : m
Correction in time : h

Acetic acid concentration of influent (titrate 20 ml samples):
After 15 min: pH: consumption NaOH: ml Concentration: mg/l
After 90 min: pH: consumption NaOH: ml Concentration: mg/l
Average : mg/l

Acetic acid concentration of effluent (titrate 50 ml samples)

Nr.	Time (min)	Time (h)	Time, corrected (h)	Sample (pH)	NaOH (ml)	Acetic acid (mg/l)
1						
2						
3						
4						
5						
6						
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