Recording a breakthrough curve of activated carbon

Aims of the experiment

- To carry out an adsorption experiment.
- To learn how to purify, i.e. clean water.
- To learn about activated carbon as an adsorbent.
- Experimentally determine the loading limit of activated carbon.
- To understand the interaction between adsorption and material transport.

Principles

To purify gases and separate liquid mixtures of substances, adsorption is often the method of choice. This process is used in environmental technology to clean exhaust gases or purify waste water. In the process, a solid is the adsorbent. The substance to be filtered from the mixture, in other words the substance to be adsorbed, is called the adsorbate. The adsorbent is the material already deposited.

Adsorption is differentiated into physisorption and chemisorption. In physisorption, Van der Waals forces are the reason for deposition onto the solid. Also, there is no chemical change in the structure of the adsorbate or the adsorbent. In chemisorption, on the other hand, the strength of the bond to the adsorbent is that of a chemical bond. The deposition is only one monolayer thick since there is no further opportunity for a bond. Also, the adsorbed particles can decompose due to the bond to the surface atoms.

Frequently used adsorbents include activated carbon and molecular sieves. Adsorbents always have a specific loading limit.

This depends on a number of factors, such as molecule size and the structure of the molecule that is to be adsorbed. Adsorbents also have properties which determine the loading limit, such as its internal surface area or the amount of the adsorbent.

Adsorbents cannot adsorb to an unlimited degree. A filter loses its filtering characteristics when the maximum of the substance to be adsorbed is reached. Then, what is known as “breakthrough” occurs. Breakthrough is when the filtrate has the same concentration as the solution to be filtered. Such breakthrough can be determined by a breakthrough curve. This is different for every material pair (adsorbent and adsorbate).

In this experiment, the breakthrough curve of indigo carmine on activated carbon will be determined. To this end, an indigo carmine solution is added to activated carbon. At defined intervals, the extinction of the filtrate is measured with a photometer until it returns to the starting value of the unfiltered solution.

Fig. 1: Set-up of the experiment.

Risk assessment

Ethanol is highly flammable. Therefore, do not work near sources of fire!

Indigo carmine	
 **Signal word: Caution**	**Hazard warnings** H302 Harmful if swallowed. **Safety information** P330 Rinse out mouth. P301+P312 IF SWALLOWED: Call a POISON CENTRE or doctor/physician if feeling unwell.
Ethanol	
 **Signal word: Hazard**	**Hazard warnings** H225 Highly flammable liquid and vapour. **Safety information** P210 Keep away from heat/sparks/open flames/hot surfaces. No smoking.

Equipment and chemicals

1	Pocket CASSY 2 Bluetooth	524 018
1	CASSY Lab 2	524 220
1	Saddle base	300 11
1	Stand rod, 25 cm, 12 mm diam.	300 41
1	Bosshead S	301 09
1	Universal clamp 0...80 mm	666 555
1	Immersion photometer S	524 069
1	Chromatography column, 235 x 20 mm	665 592
1	Volumetric flask, Boro 3.3, 1000 ml	665 796
1	Stainless steel spatula, spoon end, 180 mm	666 968
1	Electronic balance 440-3N, 200g: 0.01 g	667 7977
1	Beaker PMP, 25 ml, tall	604 009
1	Beaker, PP, 250 ml, squat	664 123
1	Glass wool, 10 g	672 1000
1	Activated charcoal, granulated, 100 g	670 2000
1	Indigo carmine, 10 g	672 3400
1	Ethanol, solvent, 1 l	671 9720
1	Stopcock grease	661 082

Set-up and preparation of the experiment

Set-up of the apparatus

1. The apparatus is set up as can be seen in Fig. 1.
2. Fix the stand rod in the saddle base to do so.
3. Fasten the chromatography column to the stand rod using a bosshead S clamp and a universal clamp.
4. Add a bit of glass wool to the chromatography column in addition to the glass frit.

Note: The glass wool is only used to keep the glass frit of the chromatography column from getting too dirty.

5. Connect the immersion photometer S to the Pocket CASSY.
6. Start CASSY Lab 2 and load the settings.

Settings in CASSYLab
Transmission: 445 nm active
Extinction: active
Recording: manual
Formula for converting the number of measurements to ml:
volume indigo carmine solution $V = 15*n-15$

Preparation of the experiment

1. Weigh out about 20 g of activated carbon and add to the chromatography column.
2. Close the valve of the chromatography column.

Note: Make sure that the valve is easy to turn. If necessary, add some stopcock grease.

3. Then, rinse the activated carbon with enough ethanol until the collected liquid is clear.
4. For the indigo carmine solution, weigh out 0.03 g of indigo carmine and dissolve completely in 1 l of water.

Performing the experiment

1. First, calibrate the immersion photometer S. To do so, add some water to a beaker (25 ml), dip the immersion photometer in and enter the value →0← in the Extinction menu. Afterwards, throw out the water.
2. Initially, record one point for the indigo carmine solution at $\lambda = 445$ nM. To do so, add 15 ml of the solution to a beaker (25 ml) dip the immersion photometer S in and record a measurement by pressing  . Return the solution and rinse the beaker out with some water.
3. Fill the chromatography column with the indigo carmine solution. Wait briefly until all cavities in the activated carbon are filled.
4. Open the valve and collect 15 ml in a beaker (25 ml). Then close the valve.
5. Dip the immersion photometer S into the collected liquid and record a measurement by pressing the button.
6. Throw out the liquid and collect another 15 ml by opening the valve.
7. Add indigo carmine solution to the chromatography column so that it is continuously covered with liquid.
8. Repeat the measurement step and the fluid collection step long enough for the measurement to approximately match the initial value of the unpurified indigo carmine solution again or until it no longer changes.

Observation

Before the indigo carmine solution was passed through the activated carbon column, it had a dark blue colour. At the beginning of the measurement, the initial collected fractions are nearly colourless. However, the more solution to be purified, the more the colour returns to that of the initial solution.

Evaluation

In Fig. 2, the breakthrough curve of a 0.03 g/l indigo carmine solution can be seen, with activated carbon as the adsorbent.

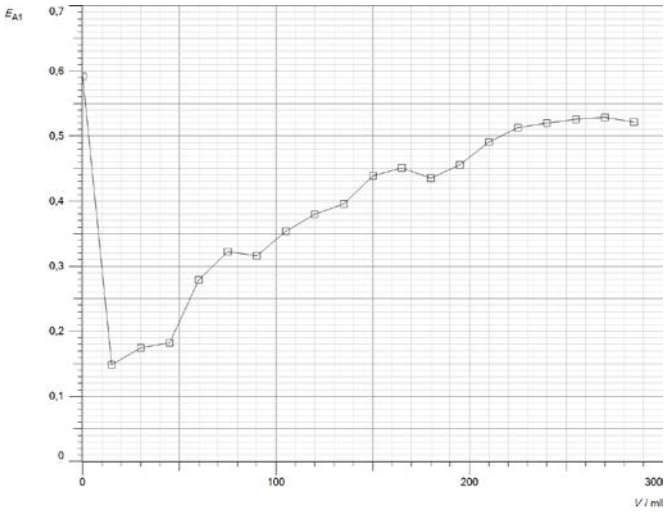

Fig. 2 Breakthrough curve for indigo carmine solution.

The first value at 0 ml indicates how high the extinction is, and thus the concentration of the original 0.03 g/l indigo carmine solution.

The second value is the first measurement of the indigo carmine solution that has passed through the activated carbon. It can be seen that a rapid decrease in extinction (i.e. concentration) is occurring. This clearly indicates that Indigo carmine is being adsorbed well.

However, the more the solution passes through the activated carbon column, the more the extinction increases, until it has finally almost reached the initial value again. Activated carbon can only adsorb a specific amount of a trace material, such as indigo carmine. The loading curve of the activated carbon is reached relatively quickly in this experiment, which is why breakthrough occurs at the end of the experiment. This indicates that the initial value has nearly been reached again and the solution is flowing through practically unfiltered.

The breakthrough in this set-up has been reached after approx. 250 ml of indigo carmine solution (c=0.03 g/l). The 20 g of activated carbon used were therefore able to adsorb m = 75 mg of indigo carmine.

Results

In this experiment, the breakthrough curve of indigo carmine on activated carbon was determined. When the value at 0 ml is compared with the first measurement points on the curve, a clear decrease in indigo carmine concentration can be seen. This increases again over time gradually, since the loading limit of the activated carbon is being continuously exhausted until it is finally reached.

The loading curve is at approx. 250 ml of indigo carmine solution, or 75 mg of indigo carmine.

Cleaning and disposal

The solutions can be disposed of in the laboratory sink. The activated carbon can be disposed of in the waste.

These experimental instructions were issued at the request of Prof. M. Niethammer and U. Krauße, TU Dresden.