Methods of pollution control and waste management - laboratory

Adsorptive removal of volatile organic compounds from gases streams

Manual for experiment 17

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The phenomenon of adsorption of gases by solids is used in the methods of air cleaning for several years. The most commonly used adsorbents are activated carbon, silica gel, natural aluminosilicate minerals and zeolites (synthetic aluminosilicates). For example, silica gel, zeolites and aluminosilicates are used in separation and drying gases, as well as for the recuperation of solvents from the industrial tail gases. Activated carbon is used mainly for the recuperation of solvents and as a fill in exhaust hoods to protect the respiratory tract and for the removal of toxic substances from the air.

Adsorption is a phenomenon occurring on the surface of a solid material called "adsorbent". The true area taking part in the process of adsorption is the sum of the external surface of the adsorbent grains and the inner surface of all its pores. The adsorbent specific surface is expressed in m^2 per 1 g of a solid, and defines as total area participating in the adsorption process. The specific surfaces of so-called heavily porous materials are of several hundred square meters per gram. Because of so much developed surface such materials can absorb the amount of solvent vapors and gases many times higher than the volume of the adsorbent.

Exercise aim

The main goal of this experiment is to familiarize students with the adsorptive method of the removal of volatile organic compounds (alkanes) from gases streams. In exercise the dynamic adsorbing capacity for chosen adsorbents (zeolites 13X and activated carbon) is determined. After experiment adsorption efficiency of both beds will be compared and discussed.

Installation description

The experiment is performed on two identical installations differing with adsorbent beds. A schematic diagram of devices is shown in Figure 1 (containing zeolite) and in Figure 2 (with active carbon bed) as adsorbents. In Figure 3 adsorber construction is presented.
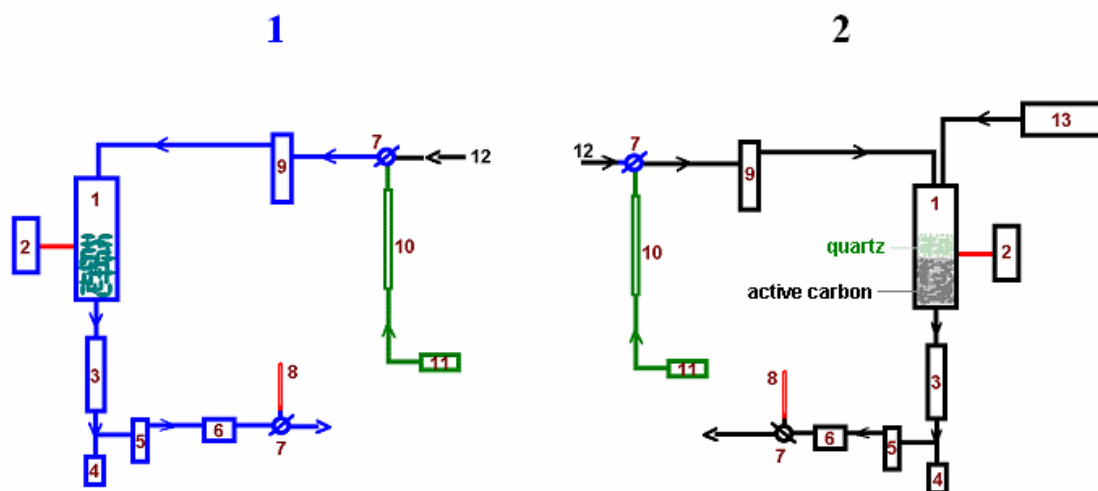
Figures: 1 (zeolite as adsorbent) **and 2** (active carbon as adsorbent).
1 - adsorber, 2 – temperature control unit, 3 – Liebig condenser, 4 - receiver, 5 – cold trap, 6 – washer, , 7 – three-way valve, 8 – indicator tube **K-2**, 9 – hydrocarbon container, 10 - rotameter, 11 – membrane pump, 12 – inert gas (Ar) supply, 13 – infusion pump

Adsorptive removal of volatile organic compounds from gases streams is realize in the following way: the air stream polluted with the hydrocarbon vapors flows through an adsorbent bed (zeolite or active carbon) until tubular detector K-2 change the color. This characteristic moment indicates that adsorbent bed was breakdown, and from this moment not all amount of hydrocarbon deposits on the adsorbent surface.
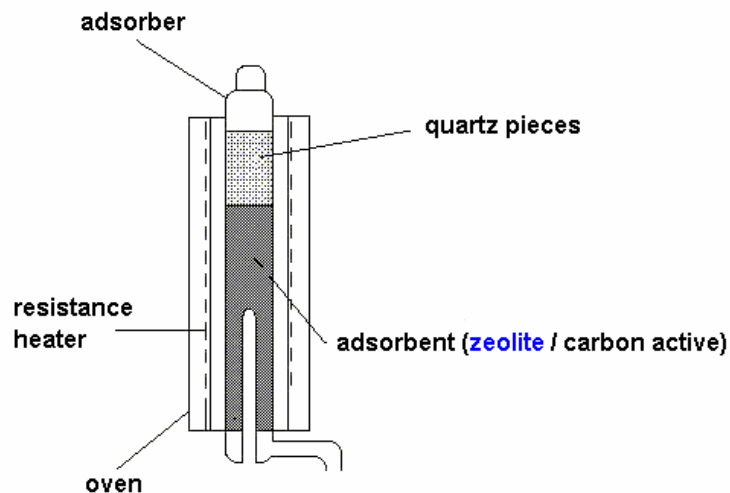
Figure 3. Adsorber scheme.

Saturation of the air hydrocarbon's is obtain by flowing a clean air stream of hydrocarbon through a hydrocarbon-filled container. Air stream saturated by hydrocarbon is obtained in the following way: a clean air stream passes through a container filled with this hydrocarbon (if necessary temperature should be increased). The hydrocarbon content in the air stream is adjusted by changing the temperature of the container and flow rate of the air. The speed of air flow is controlled using rotameter. In the first step of the process hydrocarbon is completely adsorbed. At that time to the indicator tube **K-2** goes on the air, completely devoid of impurities. After some time, when the capacity of the deposits is exceeded in the air stream leaving the deposit appear certain quantities of hydrocarbon. This is a moment of breakthrough adsorbent layer. Its detection is done visually by the changes color of the fill in tube **K-2**.

After adsorbent bed breakdown (or achieving acceptable by National Burro of Standards - Polskie Normy - the concentration of hydrocarbon) closes the stream flow of the polluted air, and performs the reverse process, ie. desorption of hydrocarbon.

Desorption of hydrocarbon from zeolite is carried out by raising the temperature of the deposit to around. $3200^{o}C$ and by flow a stream of clean air. In the case of deposits of activated carbon in the desorption process uses a stream of water

vapor. Water vapor stream receives by dosing using a syringe pump water on heated to a 130°C layer of quartz, On the end adsorbent is dried with clean air stream.

During the desorption process the stream of vapors is directed to the water cooler and cold trap (filled with a mixture of solid CO_2 and acetone) where vapors condensation occurs. In case of zeolite adsorbent, hydrocarbon removed from adsorbent is collected in the receiver. After the process is completed, the contents of the receiver shall be weigh and then analyze by gas chromatography.

In case of active carbon bed after desorption process receiver contents shall be transferred into a separatory funnel and after the separation of phases the organic phase shall be weight, and analyze it by gas chromatography[*].

The experiment ends regeneration fold. In both cases, it involves rinsing the deposits by streams clean air and argon.
So put up deposits of adsorbents can be used in the next cycle. Each cycle of work always precedes to irrigate the deposits of a stream of inert gas.

Experiment steps

I. Adsorption

1. Irrigate the deposits of a stream of inert gas
2. Weight container with hydrocarbon (9) and connect it to the installation.
3. Properly install the indicator tube **K-2** for three-way valve (7).
4. Start passing air through the bottle with the hydrogen container (9). Turn on the diaphragmatic pump (11). The speed of the air flow shall be determined with the rotameter (10).
5. Hydrocarbon-contaminated air stream direct on the adsorbent bed.
6. Observe the indicator tube **K-2**, checking that there is no breakthrough the bed.
7. At the moment of the color change of substance that fills the tube inform please a teacher and then breakdown process (cutting off the supply of contaminated air into adsorber.
8. Remove the container (9) with the residual hydrocarbon and weigh.

9. Carry out the desorption process. Process conditions select depending on the type of adsorbent bed.

II. Desorption

Zeolite bed	Active carbon bed
1. Increase bed temperature until 320°C using regulator (2). 2. Cold trap (5) fill with dry ice (solid CO_2) and acetone mixture. 3. Turn on membrane pump (11) and start clean air dosing into adsorber (1). 4. Desorption products collect in receiver (4). 5. After around 30 minutes wash the bed with inert gas (Ar). 6. Turn off adsorber heating using regulator (2). 7. Disconnect receiver (4) and weigh. 8. Desorption product analyze by gas chromatography.	1. Increase bed temperature until 130°C using regulator (2). 2. Turn on infusion pump (13) and start water dosing into adsorber (1). (the speed dosing, time and water volume gives instructor). 3. Desorption products collect in receiver (4). 4. Turn off infusion pump (13). 5. Turn off adsorber heating using regulator (2). 5. Wash the adsorbent bed with inert gas (Ar). 6. Disconnect receiver (4) and weigh. 7. Receiver contents transfer into separatory funnel for phase organic and water separation. 8. Compute organic phase mass and then analyze it by gas chromatography.

All data as well as remarks should be noted in results sheets.

After the experiment it should be:

- calculate hydrocarbon flow in [mg/min],

- prepare a material balance,

- calculate dynamic absorptivity for both bed and compare them,

- discuss and compare chromatograms,

- determine the amount of hydrocarbon recovered in [%].

Experiment report should be prepared according to pattern attached to manual and contained:

- the purpose of the experiment,

- summary of the experiment

- glc results description and interpretation (including glc analysis parameters),

- results discussion, and comments (including: chemical equations, yield of reaction, sources of errors and errors discussion),

- conclusions (among, if the purpose of the experiment has been achieved or not),

- student's remarks and suggestions if any (for example how to improve some steps of experiment).

  The resulting sheet signed by instructor should be attached to the report.

* glc basic principles are described below

Gas-liquid chromatography is a form of column chromatography in which the absorbing medium is a liquid of low volatility, called the liquid phase, and this is dispersed over the surface of an inert solid support. The latter is usually a granular material which does not itself adsorb the components but which merely acts to hold the liquid phase in a stable dispersed form. A gas stream, known as a carrier gas, flows continuously through the column and the temperature of the system is controlled. When a small quantity of a volatile mixture is applied to the column the components are distributed between two phases and therefore moved more slowly than carrier gas stream, due to the retarding effect of the liquid phase. The column should be sufficiently long due to separating components as a result of differences in this retarding effect. When the separated components are eluted from the column a sensitive device, known as a detector, converts the concentration or mass substance contained in he exit gas stream into an equivalent electrical or other measurable signal (depending on detector type). This signal is usually measured by some continuous recording device to produce a chromatogram. Typical chromatogram consists of a number of peaks ach of which corresponds to a component of the mixture. The position of each of these peaks is characteristic of the component to which it is due. These characteristic positions are measured in terms of the volume of carrier gas which has passed through the column between the time application of the sample to the column and the time of emergence of the component. This gas volume is called the retention volume, V_R , and is a fundamental gas chromatography property in qualitative analysis. The size of each peak (its area or height), is normally proportional to the amount of component causing it. This property is an important prerequisite for accurate quantitative analysis.