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# PROTOTYPE OF AN EXPERIMENTAL SETUP FOR THE STUDY OF ADSORPTION DRYING PROCESSES OF DIFFERENT GAS MIXTURES

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**Abstract:**

A setup has been developed for testing the prototype of an adsorption drying apparatus for gas mixtures R134a-SF6 and He-$CO_2$. The setup is intended for the preliminary evaluation of the selected adsorbent's performance, methods, and instruments for measuring the humidity of the working gas, as well as for measuring the parameters of the working cycle of the adsorber when working with a model wet gas-argon or nitrogen.

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## 1. INTRODUCTION

This work was carried out in preparation for the "Compressed Baryonic Matter" (CBM) experiment at the FAIR-SIS100 accelerator in Darmstadt, Germany [Deppner et al., 2023]. The experiment involves the use of two particle identification systems: Time-Of-Flight (TOF) and Transition Radiation Detector (TRD) [Seddiki, 2014]. To ensure the proper operation of these two systems, the working surfaces of the corresponding particle detectors and their elements must be in a gas mixture atmosphere. For the CBM-TOF detectors, this mixture is R134a and SF6 (97.5/2.5). For the CBM-TRD detectors, the mixture is Xe and $CO_2$ (85/15) or Ar and $CO_2$ (80/20). It is known that impurities may appear in the detector communication lines during their operation for various reasons. Of particular concern for the operability of the detectors is the appearance of water vapor ($H_2O$) and oxygen ($O_2$) impurities. To dry the gas mixture circulating through the detector communication lines, a short-cycle adsorption technology is proposed, based on the principle of

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periodic adsorption of water vapor from the gas mixture at a temperature close to ambient temperature. The setup can consist of two (or more) identical adsorbers, which operate alternately. Designing such a drying system requires knowledge of the duration of the main stages of the adsorber's operation cycle. Calculating these parameters is complicated due to the non-isothermal nature of the adsorption process and the lack of data on water vapor adsorption from the mentioned gas mixtures. Therefore, the necessary prerequisites for such a design are supposed to be determined not only by calculations, but also as a result of experiments with a model adsorber under conditions as close as possible to operating conditions. The organization of experiments with working gas mixtures R134a-SF6 and Xe-$CO_2$ requires special measures to prevent leaks of these mixtures into the environment. This significantly complicates the design and operation of the experimental setup. However, for a preliminary assessment of the selected adsorbent's suitability and the duration of the adsorber's cycle stages, experiments with humidified nitrogen would suffice. Therefore, an experimental setup was created to assess the performance of the selected adsorbent, methods, and means for measuring the working gas's humidity and to measure the parameters of the adsorber's working cycle.

## 2. APPROXIMATE CALCULATION OF THE PROTECTIVE ACTION TIME OF THE ADSORBER

The purpose of this calculation is: to determine the value of the concentration of water vapor $C_f$ in the gas flow at the outlet of the adsorber depending on time $t$ (output curve) - $C_f(t)$.

The basis of the numerical calculation is the mass balance of water passing from the gas to the adsorbent, contained in the elementary volume of the adsorber, and - the heat balance, providing for the distribution of the heat of adsorption between the adsorbent and the gas. Here, the elementary volume of the adsorber is the space enclosed between two cross-sections of the adsorber, located at a distance $\boldsymbol{\delta}$ from each other.

This calculation is an estimate, since errors are possible due to the assumptions made to simplify the calculations:

- the outer surface of the adsorber has ideal thermal insulation. That is, there is no heat exchange between the adsorber and the environment,
- the concentrations of water in the adsorbent and in the gas at the outlet of the elementary volume correspond to the equilibrium state.

### 2.1. Characteristics of the adsorbent.

Molecular sieve 3A (zeolite KaA) was chosen as the adsorbent for preliminary calculations and experiments, the parameters of which are presented in the following table.

| | | |
|---|---|---|
| Tipe* | Molecular sieve 3A (zeolite KA) | |
| Supplier | ThermoFisher (Kandel) GmbH | |
| Granule shape* | Spherical | |
| Granule diameter*, mm | 1 ÷ 2 | 2÷5 |
| Heat capacity, kJ/kg*K | 0,9 | 0,9 |
| Bulk density**, g/dm$^3$ | 777 | 743 |

*- from supplier documents; **- own measurements.

**Adsorption isotherms:**

Typical adsorption isotherms of water vapor on zeolite 3A (balls with a diameter of 2.36 mm), constructed according to the data of work [Lin et al., 2015].

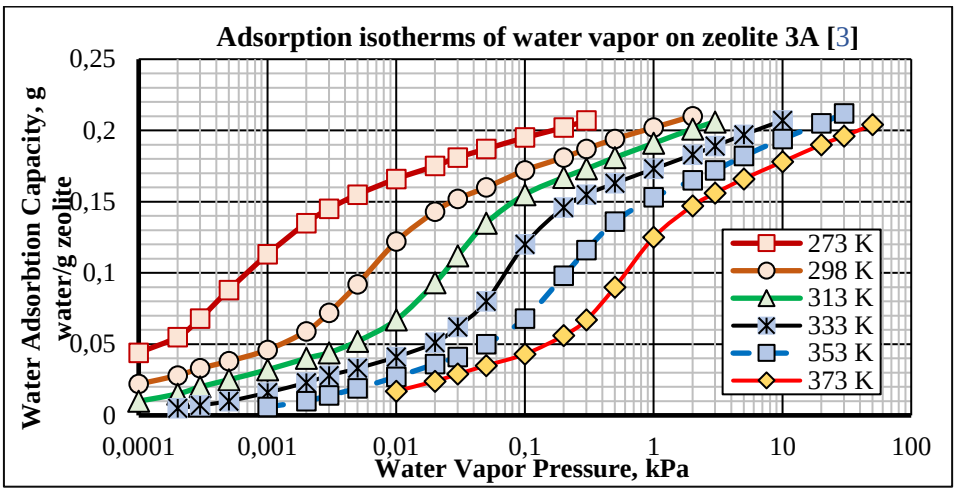


**Figure 1**. Isotherms of water vapor adsorption on zeolite 3A [Lin et al., 2015]

**Parameters of the experimental adsorber that were used in the calculations:**

Body material ……..…….…. stainless steel
Body ……..…….. pipe 76×2 mm
Length, mm ……..…… 510
Sectional area, $m^2$ ……..….. $4.07\times10^{-3}$
Volume occupied by adsorbent, $m^3$ ……..…….… $1.1\times10^{-3}$
Weight of adsorber with adsorbent, kg ……... 5.181
**Adsorbent mass, kg** …… **1.514**

The results of the calculations of the dynamics of water vapor adsorption from gaseous wet nitrogen are presented below in comparison with the experimental results.

## 3. EXPERIMENTAL SETUP

The schematic diagram of the experimental setup is shown in Figure 2.

The main components of the experimental setup include:

- The adsorber is made of a steel tube with an outer diameter of 76 mm and a wall thickness of 2 mm. The length of the adsorber is 510 mm. The design of the adsorber allows for the replacement of the adsorbent. The adsorber's casing has thermal insulation made of 20 mm thick mineral wool and two layers of aluminum foil.
- Flowmeter FM: Siargo MF5612-N-300-A-D-N 0…300 SLPM;
- Vacuum Pump VP: PFEIFFER ACP-28;
- Termometers T1, T4: Termocuplu Cupru-Constantan $T_{max}$= 400°C (located in the gas flow);
- Termometers T2, T3: ZOKURA model: Z1177, measurement range -20 - 300°C;
- Humidity Sensors DP3**:** MICHELL Instruments Easidew Transmitter -100/+20 B171-003 EAS (designed for measuring SF6 humidity, adapted for measuring humidity in various gas mixtures) [ELECTRONSYSTEM MD];
- Differential pressure gauge DM: APLISENS, Type: APR-2000ALW;
- Piping and fittings from Swagelok;

- Mixer is a cylindrical vessel made of stainless steel, with an inner diameter of 200 mm and a height of 1000 mm. It contains distilled water through which dry gas is bubbled. The vessel is closed with a flange, on which the piping, manometer, and safety valve are located;

- Heater is a copper coil made from a 6x1.0 mm tube, wound onto a stainless-steel shell with a diameter of 100 mm and a length of 300 mm. Three ring-type heaters L10060C97A5 D100/101 H60 970 W 230V are mounted on the outer side of the copper coil. The heater has thermal insulation made of 20 mm thick mineral wool, covered with aluminum foil. A universal PeakTech 2235 power supply unit is used for the heaters;

- Cooler is a coil made of a 6x1 mm copper tube (tube length 8300 mm, coil diameter 100 mm, 26 turns). The coil is cooled by natural convection of the surrounding air;

- Water Separator is a container for collecting liquid condensed from the working medium flow in the cooler. The liquid level is controlled visually through a sight window. The accumulated condensate is periodically drained through a drain valve located at the bottom of the separator.

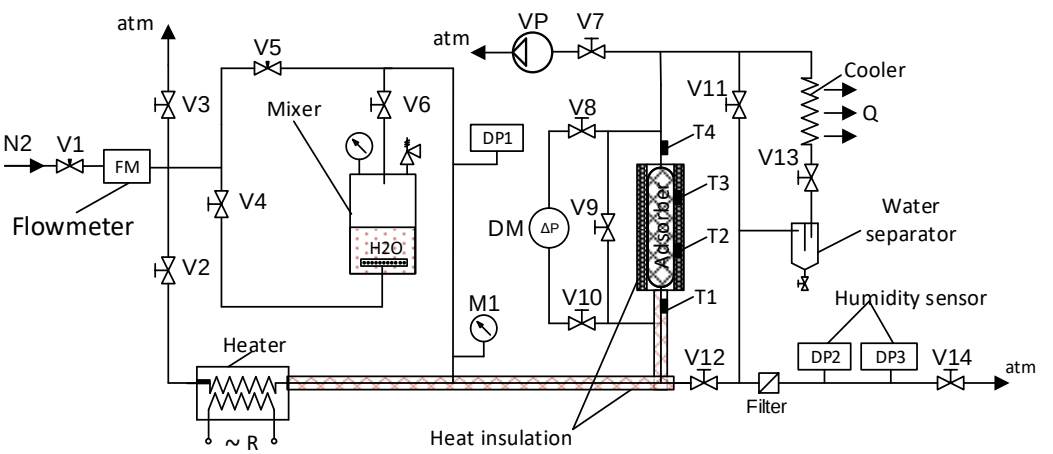


**Figure 2.** Schematic diagram of the prototype experimental unit

Photos of the main elements of the prototype experimental unit are shown in Figure 2.

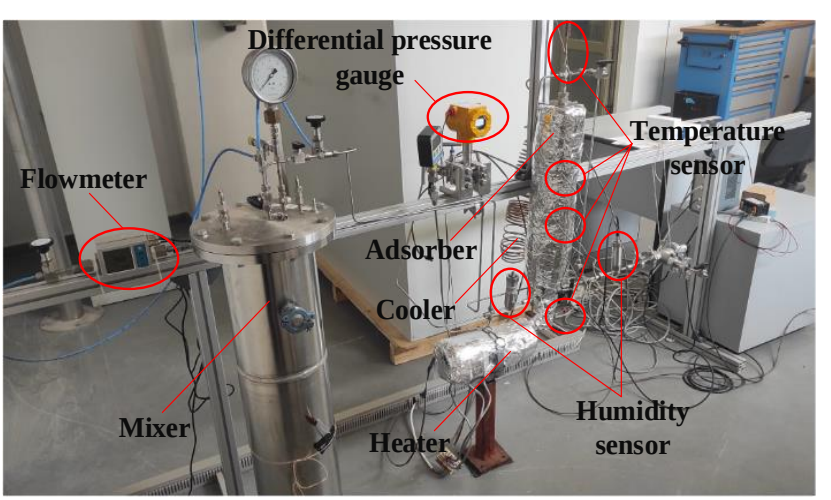


**Figure 3.** Photo of the main elements of the prototype experimental unit

## 4. METHODOLOGY FOR CONDUCTING EXPERIMENTS ON ADSORPTION DRYING OF GASES

The working cycle of the experimental adsorber consists of two processes (stages): regeneration (desorption) and adsorption. The purpose of the experiments is to determine the duration of each of these stages for different types of adsorbents and for different values of gas flow rate, its temperature, composition and pressure. The data obtained will be used to design a unit operating on the principle of short-cycle adsorption.

### 4.1 Regeneration (desorption) stage

The desorption stage is performed to prepare the adsorbent for operation and is the removal of moisture from the adsorbent accumulated during the adsorption stage. Desorption is performed by purging the sorbent layer with heated gas, during which the heated gas, passing through the sorbent layer, is saturated with moisture contained in the sorbent. Then, evacuation can be performed to remove residual moisture from the sorbent. The main criteria for the completion of this stage are: stabilization of the gas temperature at the outlet of the adsorber at a level close to the gas temperature at the inlet of the adsorber and a reduction in the moisture content of the gas leaving the adsorber to a stable minimum value.

Purge of the sorbent layer with heated gas is carried out as follows. Dry gas is fed to the inlet of the experimental setup (see Fig. 2). Then the gas flow is directed through the flow meter, heater into the adsorber. The gas leaving the adsorber is directed to the separator for separation of condensed moisture. Gas flow is regulated by valve V1. Flow measurement within the range from 0 to 300 normal liters per minute is carried out using a flow meter FM. The temperature at the inlet to the adsorber is regulated within the range from 20 to 260°C by changing the heater power. The moisture content in the gas leaving the adsorber is measured using sensors DP2 and DP3. After reaching a certain gas temperature at the outlet of the adsorber and humidity, the purging process is completed. The adsorber is evacuated to the minimum pressure. Then the adsorber is weighed.

### 4.2 Adsorption stage

This is the main stage of the adsorber operation. During this stage, water vapor is removed from the wet gas supplied to the adsorber. Gas drying occurs here due to the preferential adsorption of water vapor contained in the wet gas by the adsorbent located in the adsorber. The main criterion for the end of this stage is the concentration of water vapor in the gas leaving the adsorber.

Moisture adsorption on the sorbent layer is carried out as follows. Dry gas is fed to the inlet of the experimental setup. Then the gas flow is directed through the flow meter and mixer (humidifier) into the adsorber (see Fig. 2). The gas flow is regulated by valve V1. Flow measurement within the range from 0 to 300 normal liters per minute is carried out using the FM flow meter. The moisture content in the gas is regulated by valve V5 by mixing dry gas into the wet gas flow. The concentration of water vapor in the gas at the inlet and outlet of the adsorber is measured by sensors DP1 and DP2, respectively. During the adsorption stage, the concentration of water vapor in the gas at the inlet of the adsorber and its flow rate are maintained constant. As the sorbent layer is saturated with water vapor, the moisture concentration at the outlet of the adsorber increases. The adsorption process is considered complete when the concentration of water vapor in the gas at the outlet of the adsorber is equal to their concentration in the gas at the inlet of the adsorber. This allows determining the sorption capacity of a given adsorbent for water vapor under

these conditions. Continuous monitoring of the water vapor content in the gas at the outlet of the adsorber allows obtaining the output curve of the adsorption front movement and determining the time of the adsorber's protective action for water vapor under these conditions.

## 5. DISCUSSION OF THE CALCULATION RESULTS AND THE EXPERIMENTAL RESULTS

The following table presents the initial parameters of the wet gas at the inlet to the adsorber that took place in the experiment, and those that were adopted in the calculations.

| | Calculation | Experiment |
|---|---|---|
| Result date | 22.01.2025 | 26.02.2025 |
| Gas flow (N2), nl/min | 45 | 40 |
| Gas inlet temperature, K | 298 | 291 |
| ***Gas Humidity at the Inlet:*** | | |
| DP, °C | + 5,1 | + 10,6 |
| Concentration of water vapor in gas, g/g | 0,0058 | 0,010 |
| Partial pressure of water vapor, Pa | 880 | 1280 |

In the experiment described above, the adsorption process was continued until the concentration of water vapor in the gas flow at the outlet of the adsorber and at the inlet to it equalized. The amount of adsorbed water, determined by weighing, was 323g. This corresponds to an adsorbent capacity of 323g/1514g = 0.213g/g. The measured value of the capacity of zeolite 3A at a gas temperature of 291 K (+18°C) approximately corresponds to the maximum saturation of the adsorbent with water at a given temperature of 291 K and a pressure of 1280 Pa (see isotherms in Fig. 1).

The following is a comparison of the parameters of the adsorption process dynamics, determined as a result of the calculations and measured in the experiment.

It should be noted that the calculations were performed before the start of the experiment, since the purpose of the calculations was to plan the duration of the experiment and assess the possible limits of the values of the measured quantities.

It should also be noted that, unlike the calculation conditions, the thermal insulation of the adsorber in the experiment was not ideal. This means that during the experiment there was a radial heat flow in the adsorbent packing and, - the corresponding distribution of the flow and adsorbent temperature in the direction perpendicular to the direction of the gas flow in the adsorber.

### SO, COMPARISON:

The following Fig. 4 and Fig. 5 show the time dependences of the gas temperature $T_f$ and the concentration of water vapor in the gas $C_f$ at the outlet of the adsorber, obtained as a result of the experiment and as a result of the calculation.

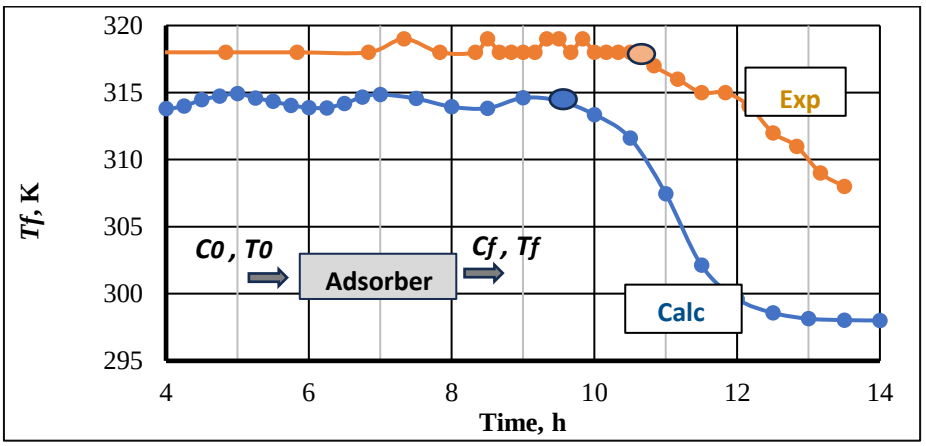


**Figure 4.** Change in temperature *Tf* of the gas flow at the adsorber outlet based on the calculation results (Calc) and the experimental results (Exp).

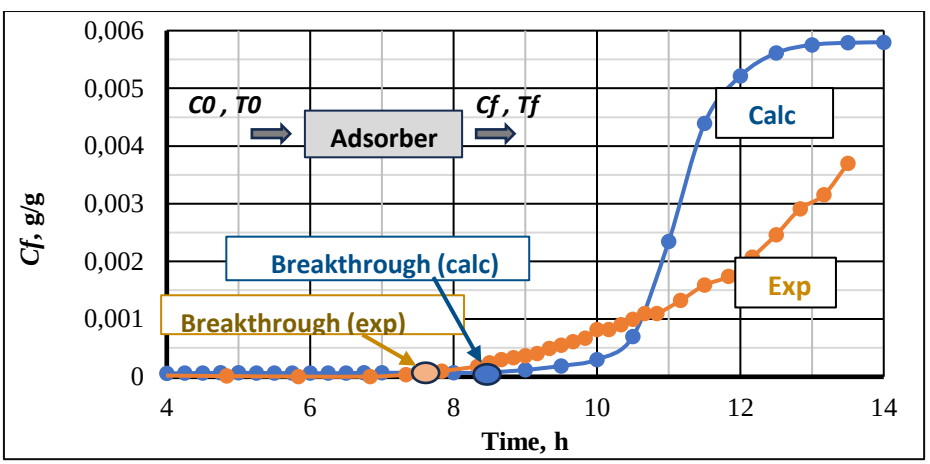


**Figure 5.** Change in the concentration of water vapor in the gas flow *Cf* at the outlet of the adsorber based on the calculation results (Calc) and – based on the experimental results (Exp).

As can be seen, in both cases the temperature in the working layer of the adsorber is noticeably higher than the temperature of the gas at the inlet to the adsorber, which is obviously a consequence of the high value of the heat of adsorption of water vapor on this adsorbent. It seems that a significant difference in the humidity of the incoming gas is the reason that in the experiment the gas at the outlet of the adsorber in the experiment is "warmer" than in the calculations.

The analysis of the dynamics of the adsorption process usually involves comparing the propagation velocity (movement) of the front of water vapor concentration in the gas $C_f$ in the adsorbent layer and the velocity of the temperature front. The ratio of these velocities can obviously be judged by the time dependences of the concentration of water vapor in the gas and the gas temperature $T_f$ at the outlet of the adsorber.

As can be seen from these figures, the time of the adsorber's protective action (the moment of the onset of "breakthrough" - an intensive increase in the concentration of water vapor in the gas at the outlet of the adsorber) according to the calculation results (8,5 hours) is very close to the experimental result (7,5 hours). This degree of coincidence of the calculation and experimental results can be considered satisfactory taking into account the

difference in the initial conditions and the attributes of the status of the preliminary experiment.

From a comparison of the $C_f$ and $T_f$ graphs in these figures, it follows that the concentration front (of water vapor in gas) moves in the adsorbent layer somewhat more slowly than the temperature front.

In addition, it is important that both in the calculations and in the experiment, the moment of decreasing the gas temperature at the outlet of the adsorber exceeds the protective action time of 2 ... 3 hours. This circumstance, in particular, indicates that simple monitoring of the gas temperature at the outlet of the adsorber cannot be used to identify the breakthrough of water vapor from the adsorber.

## 6. CONCLUSIONS AND FINDINGS

1. The results of the preliminary tests of the experimental setup showed the operability of its elements and methods for studying the parameters of the working processes of drying wet gas in a non-isothermal adsorber.

2. The results of the experiment confirm the adequacy of the model and the developed numerical method for calculating the dynamics of processes in the adsorber.

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