

VARIOUS PRECONCENTRATOR STRUCTURES FOR DETERMINATION OF ACETONE IN A WIDE RANGE OF CONCENTRATION

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Abstract. In this paper, the investigation results on preconcentration of acetone at various initial concentrations are presented. The structures were made of conventional materials, such as stainless steel, quartz tube as well as fabricated in MEMS technology – micro-preconcentrators. All structures have the same ‘active’ area to obtain more suitable comparison. The adsorbent materials were selected from commercial available Sigma-Aldrich Carbon Adsorbent Sampler Kit, consisting of 8 various adsorbents. The highest concentration factors were obtained by utilization of micropreconcentrator filled with Carboxen-1018, which is recommended for adsorption of C₂-C₃ compounds. The preconcentrators were placed into microsystem, and semiconductor gas sensor array was used as a detector unit. The microsystem was previously tested and designed for exhaled breath acetone analysis. The obtained results show that micropreconcentrator can be a useful tool for an increasing sensor sensitivity.

Keywords

Acetone, MEMS technology, micropreconcentrators, preconcentration.

1. Introduction

Acetone is widely used in many industrial processes, it can be found in many goods used in everyday life as well, such as e.g. lacquer, varnish, rubber, plastics, etc. The occupational exposure limit for ace-

tone in the EU, USA, Japan, as a time-weight average concentration over an 8-hour work shift, equals 2400 mg · m⁻³ (1000 ppm), respectively [1]. Based on guidelines provided by the National Institute for Occupational Safety and Health (NIOSH), concentrations of approximately 250-500 ppm were reported to cause slight irritations of noses, throats, lungs and eyes. After exposure to the concentration of 1000 ppm symptoms such as headache, headedness, dizziness, unsteadiness and confusion are common. Inhalation of concentrations higher than 2000 ppm can cause a feeling of drunkenness, drowsiness, nausea and vomiting. Higher concentrations can result into collapse, coma and death. Furthermore, acetone can be also determined in the exhaled breath with emphasis on type-1 diabetes mellitus subjects [2], [3], [4] and [5]. The exhaled acetone is usually in the range of 0.2 – 1.8 ppm for healthy people, and in the range of 1.25 – 2.4 ppm for people with diabetes [6]. Therefore, acetone is an attractive compound that have to be measured in a wide range of concentrations. The concentration range from 50 - 5000 ppm is covered by commercially available gas sensors, i.e. TGS 822, TGS 823 (Figaro Inc, USA), AF63 (Scimarec Ceramic Technology, UAZ). However, the gas sensors for acetone in the concentrations below 50 ppm are still under investigation [7], [8], [9] and [10]. Currently available instruments are based on laboratory systems such as: gas chromatography-mass spectrometry (GC/MS) [11], proton transfer reaction mass spectrometry (PTR-MS) [12], selected ion flow tube mass spectrometry (SIFT-MS) [13], etc. However, the size, cost and complexity associated with these instruments preclude their routine application. One of the cheap and very effective method to increase the

limit of detection (LOD) to measure low amounts of gasses is the application of gas preconcentrator structures [14], [15] and [16].

Conventional preconcentrators are usually made of glass or stainless steel tubes filled with an adsorbent material [17], [18]. These structures have lateral dimensions and power consumption, which is too high to be applied in portable applications. A solution that overcomes these limitations are micropreconcentrators made in various technologies [19], [20]. In this study, the preconcentrators made in various techniques for determination of acetone in a wide range of concentrations are presented and discussed. The main goal is to have a fast extraction while maintaining high sensitivity. Voiculescu et al. [21] have reported the comparison of the analyte concentration and time constant in micromachined and conventional preconcentrators. The authors reported that microfabricated preconcentrator devices are characterized by reduced dead volume and thermal mass and delivering a concentrated sample that has a narrow time-width pulse for analysis [21].

In this paper, the preconcentrators have the same volume of adsorbent material. The additive dimensions were as small as possible. In such solution, we compared the detector response for both conventional preconcentrators and micropreconcentrators (Fig. 1). As presented in the Fig. 1, the detector response is 5-times longer for conventional preconcentrators than for micropreconcentrators in both available technologies, i.e. MEMS (micro-electro-mechanical systems) [22], LTCC (low temperature cofired ceramics) [23].

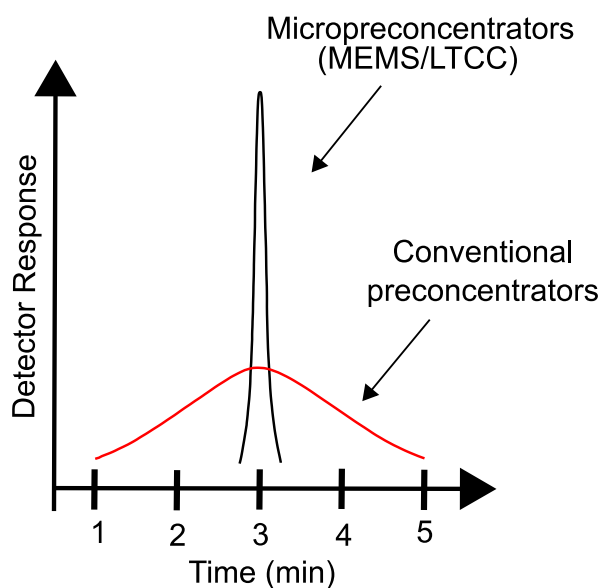


Fig. 1: Comparison of the detector response and time constant in micropreconcentrators and conventional preconcentrators.

2. Experimental Results

The preconcentration efficiency is measured using a concentration factor (CF) defined as the ratio of a gas concentration after and before the concentration process:

$$CF = \frac{V_{sample}}{V_{desorbed}} = \frac{V_{sample}}{W_h \cdot u}, \quad (1)$$

where V_{sample} is a sampled volume (liter), $V_{desorbed}$ is a desorbed volume (liter), W_h is a width of injection band (min), u is a desorption flow rate ($l \cdot \text{min}^{-1}$). Concentration factor theory is briefly described in the literature [24], [25] as well as various materials used in preconcentrators, i.e. polymer-coated microcolumns [26], Tenax polymer [14], [27]. The proposed preconcentrators were filled with different CMS (Carbon Molecular Sieve) adsorbent materials. The authors used a commercial available Carbon Adsorbent Sampler Kit (Sigma-Aldrich, St. Louis, MO 63178, USA) which contains 8 different adsorbent materials. This convenient kit allows a cost-effective way to evaluate several of special carbon adsorbents, i.e. Carboxen 569, Carboxen 1000, Carboxen 1012, Carboxen 1018, Carboxen 1021, Carbosieve G, Carbosieve S-III, Graphsphere 2016. As the authors focus on acetone analysis, after the pre-selecting, few promising adsorbent materials were selected: Carboxen-1018, Carboxen-1012, Carboxen-1003, Carboxen-1000 and Carboxen-569. They all are hydrophobic, have large surface area and grain diameters suitable to channel dimensions.

2.1. Conventional Preconcentrator – Quartz Tube

The preconcentrators were constructed from quartz-glass capillaries (0.2 mm i.d., 0.4 mm o.d.) cut to a length of 46.8 mm. The volume of the channel into preconcentrator is approximately 14.7 mm^3 . The mass of adsorbent packed into the tube ranged from 0.95 to 11.3 mg. A quartz wool was folded and inserted into each end of the preconcentrator to retain the adsorbent. Figure 2 shows the schematic view of the preconcentrator made of quartz-glass capillaries. The preconcentrator is based on a thermal desorption, therefore, a 650 mm Kanthal (Ni-Cr) wire was coiled around the tube.

2.2. Conventional Preconcentrator – Stainless-Steel Tube

The preconcentrator stainless steel internal and outer diameter are 0.7 mm and 1.2 mm, respectively. Therefore, the tube was cut to a length of 38.2 mm to provide approximately the same volume (14.7 mm^3) as in the

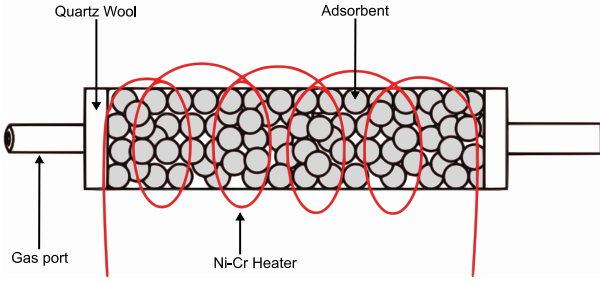


Fig. 2: Schematic view of the preconcentrator made of the quartz-glass capillary.

quartz-glass preconcentrators. As previously, the active carbon was held in place with quartz wool. The preconcentrator was heated using a coil of insulated nichrome wire.

2.3. Micropreconcentrator in Silicon Technology

The micropreconcentrators fabrication process was reported in detail in [28]. In this paper, we present investigation results obtained with the utilization of micropreconcentrator with microchannel dimensions: 0.35 mm x 0.35 mm x 120 mm. In this case, the microchannel volume is approximately 14.7 mm³. The approximate adsorbent weight in the microchannel equals 4 mg. The adsorbent material filled the microchannel from 80 % to 95 % and strongly depended on adsorbent material type. Figure 3 shows the SEM images of a Carboxen-1021 adsorbent material with marked grain dimensions (Fig. 3(a), Fig. 3(b)), sieve used for selection of molecules with specified grain size (Fig. 3(c)) and the channel filled with the Carboxen-1021 (Fig. 3(d)). To provide uniform temperature distribution inside the microchannel, the platinum microheater was deposited to cover the entire working area of the microchannel. The nominal resistance was 40 Ω ± 1 Ω. However, to achieve the selected desorption temperature the voltage supply of 13.4 V ± 0.3 V is required. Due to, portable electronic devices supply standards, the voltage supply should be decreased to 5 V or even 3.3 V. The micropreconcentrators with reduced capacity as well as with special designed thermal insulation, which can improve power consumption are under investigation.

2.4. Thermal and Electrical Measurements

As already mentioned, the preconcentrators are based on thermal desorption. While selected carbon adsorbents are stable to above 400 °C, the desorption temperature was set to 220 °C. A PID controller (Eu-

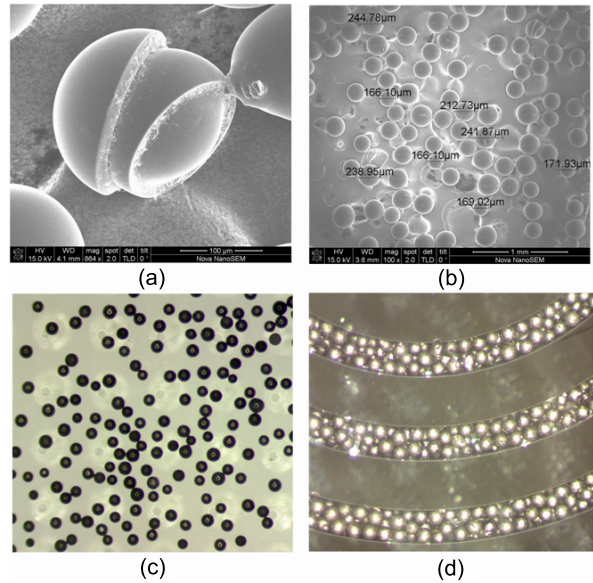


Fig. 3: The adsorbent Carboxen-1021: (a) SEM image of a single grain, (b) SEM image with molecule diameters, (c) sieve with adsorbent molecules, (d) the channel filled with the adsorbing material.

rotherm 2408, UK) and power supply (Motech PPS-1203, Taiwan) were used to control the heating profile for thermal desorption as well as during the adsorbent material activation. Figure 4 shows the relations between temperature and power consumption for three different designs: quartz-tube (Section 2.1.), stainless steel-tube (Section 2.2.) and MEMS (Section 2.3.). As presented in Fig. 4, the power consumption in MEMS micropreconcentrator is 4-times and 2-times lower than for stainless steel-tube and quartz-tube preconcentrators, respectively. It has to be underlined, that microchannel volume is the same for all compared designs.

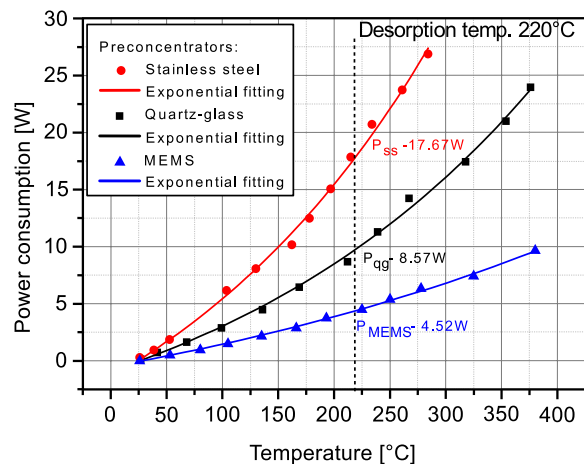


Fig. 4: Power consumption vs. temperature for preconcentrators made in different technologies.

2.5. Measurement Setup

Figure 5 shows a schematic view of the measurement system. As the sample gas, acetone with a concentration of 80 ppm, 8 ppm and 0.8 ppm was used. As the carrier gas, nitrogen (99.999 %) was used. The mass flow controller MFC (MKS Instruments, USA) provides a flow rate of 50 sccm. To obtain the best separation behaviour, temperature and flow rate are important parameters. Based on previously reported results [23], the desorption temperature was set to 220 °C and flow rate to 50 sccm. The first step is a purging phase of the system. In this step, the system is switched to desorption and the nitrogen flows through the system to clean (purge) all gas connection for at least 15 minutes at the room temperature. Before using the preconcentrators the adsorbent material has to be activated. The authors used typical time–temperature profiles for adsorbent activation: 0.5 h – 100 °C, 1 h – 200 °C, 1 h – 300 °C, 0.5 h – 350 °C and nitrogen as a carrier gas. After activation, the adsorption can be performed. The adsorption is performed at the room temperature and the gas flows past the preconcentration in adsorption direction (see Fig. 5). The breakthrough of acetone and thereafter the maximum possible acetone concentration can be calculated from Wheeler-Jonas formula:

$$t_b = \frac{W_e W_b}{C_0 Q} - \frac{\rho_B W_e}{k_v C_0} \ln \left(\frac{C_0 - C_X}{C_X} \right), \quad (2)$$

where t_b is breakthrough time (min), W_e is the equilibrium adsorption capacity ($\text{g} \cdot \text{g}^{-1}$), W_B is the bed mass (g), C_0 is the challenged concentration ($\text{g} \cdot \text{l}^{-1}$), C_X is the fraction of C_0 where breakthrough is measured ($\text{g} \cdot \text{l}^{-1}$), k_v is the kinetic rate coefficient (min^{-1}), ρ_B is density ($\text{g} \cdot \text{cm}^{-3}$), the Q is gas flow rate ($\text{l} \cdot \text{min}^{-1}$). In the case of study, t_b was reached after approximately 60 min.

In the case study, after the adsorption process, the valves are activated (by applying voltage) and the system is switched to desorption process (see Fig. 5). The preconcentrator temperature was ramped at controllable rates under homemade software control by monitoring the temperature and adjusting the voltage to the resistive heater. The microconcentrators have deposited a platinum heater, which was also used as a rapid temperature detector. To control the temperature in conventional preconcentration designs the thermocouple type-K was used. The nitrogen flows through the preconcentrator and acetone is desorbed from the adsorbent material. Thereby, it causes output signal on the gas sensors. The gas sensors responses are measured by electrometer and transferred to the PC.

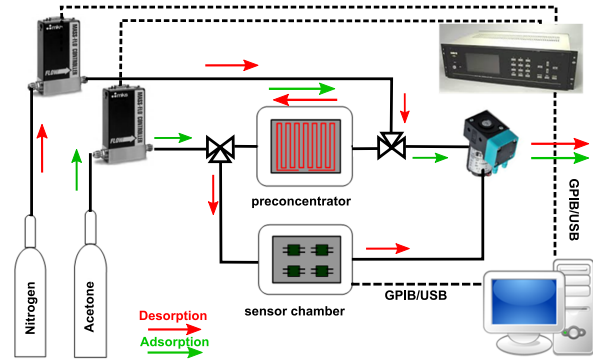


Fig. 5: Schematic view of the measurement setup. Red and green arrows show gas flow through the microsystem at desorption and adsorption, respectively.

3. Results and Discussion

The acetone concentration factor (CF) was evaluated for the preconcentrators, using different initial acetone concentrations (0.8, 8, 80 ppm) and different adsorption times (from 5 to 60 min).

Figure 6 shows CFs obtained for microconcentrator filled with various adsorbent materials at constant adsorption time (60 min), and various acetone concentration, i.e. 0.8 ppm, 8 ppm, 80 ppm. The CFs obtained for 0.8 ppm were multiplied 25 times for better comparison. The results have proved that Carboxen-1018 is the best adsorbent material for acetone preconcentration from Carbon Adsorbent Sampler Kit. However, Carboxen-1003, Carboxen-1000 and Carboxen-1021 can be also worth considering, mainly due to the fact that the Carboxen-1018 is hardly available.

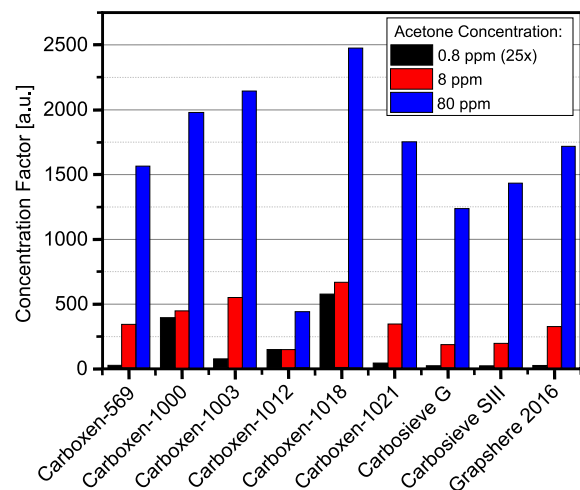


Fig. 6: Concentration Factors for different adsorbent materials from Carbon Adsorbent Sampler Kit at various initial acetone concentrations obtained by utilization of microconcentrator (adsorption time: 60 min, flow rate: 50 ml/min). CF obtained for acetone at 0.8ppm were multiplied 25 times.

Figure 7 shows the concentration factors for Carboxen-1018 for various preconcentration realization in the function of adsorption time. As it can be noticed, the CFs measured by utilization of micropreconcentrators are at least 3- and 4-times higher than for quartz-tube and stainless-steel tube preconcentrators, respectively.

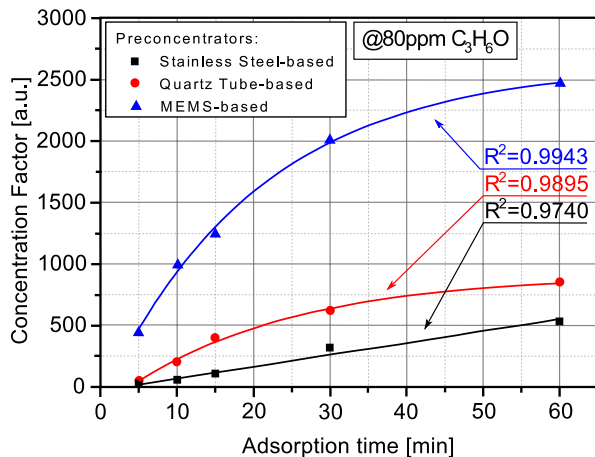


Fig. 7: Concentration Factor (with regression lines) for various preconcentrator designs in function of adsorption time for 80 ppm of acetone.

4. Conclusion

In order to overcome the limits in the sensitivity, the various preconcentrator structures were used, i.e. quartz-tube-based, stainless steel-based, micropreconcentrator. The adsorbent material has been chosen from a commercial available Carbon Adsorbent Sampler Kit. The adsorption-desorption cycles have been programmed applying power supply pulses to the heater and stabilized by PID controller. The results demonstrated that the Carboxen-1018 is suitable adsorbent material for acetone concentrations. The results prove that CFs obtained in micropreconcentrators are higher than in conventional ones. Moreover, micropreconcentrator require less power to achieve dedicated desorption temperature. Therefore, the micropreconcentrator can certainly be applied to the microsystems for low gas detection applications.

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