



# Remote control of desorption by radiofrequency heating: Single pellet experiments

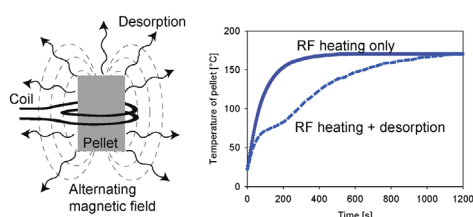
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## HIGHLIGHTS

- Composite adsorbent pellets with RF heating capability prepared.
- Rapid temperature rise in a 400 kHz alternating magnetic field achieved.
- RF induced desorption on three different systems demonstrated.
- Extent of desorption can be precisely controlled by the applied RF power.

## GRAPHICAL ABSTRACT



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## ABSTRACT

A method for controlled desorption by means of local heating due to high-frequency alternating magnetic field is described. The method has been demonstrated using three types of adsorbent–adsorbate systems: composite zeolite and silica-gel pellets for water adsorption, and activated carbon for the adsorption of toluene as a model volatile organic compound. The composite adsorbent pellets contained iron microparticles that played the role of susceptors that dissipated heat in the presence of a radiofrequency magnetic field. The ability to induce adsorbent regeneration and to desorb defined quantities of the adsorbate was confirmed by gravimetric measurements and by on-line monitoring of the desorption process by fibre optic temperature measurement. An energy balance has been used for calculating the instantaneous desorption rate.

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## 1. Introduction

Adsorption is a widely used separation process at both industrial and laboratory scale. Adsorption–desorption cycles can be used not only for the separation of gas mixtures (Liu et al., 2012) or purification of fluid streams, but also in conjunction with a reaction step for selective removal or dosing of reaction components, such as in sorption-enhanced steam methane reforming (Koumpouras et al., 2007) or in the so-called storage catalysts (Štěpánek et al., 1999). Recently, mesoporous silica based

adsorbents have also been proposed as carrier matrices for controlled drug delivery (Kovačik et al., 2012).

The regeneration (desorption) step during a periodic adsorption process is typically achieved by one of three methods: pressure swing adsorption (PSA) (Ray, 1986; Grande, 2012), temperature swing adsorption (TSA) (Clause et al., 2004) or concentration swing adsorption (CSA) (Rao and Sircar, 1993). In the case of adsorbent regeneration by temperature swing (Ko et al., 2002), common methods of energy supply into the adsorbent bed include direct heat transfer (e.g. using superheated steam) or contact heat transfer via a heating jacket or immersed heat transfer tubes. However, alternative methods such as microwave heating or heating using electric current (the so-called electrothermal swing adsorption or ESTA (Sullivan et al., 2004))

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have also been described. In the case of zeolite adsorbents, dielectric heating at lower frequencies (10 MHz as opposed to the typical GHz range used in microwave heating) has been recently demonstrated (Kraus et al., 2011).

In large-scale processes where the latent heat of desorption along with the heat capacity of the adsorbent bed exceed the heat capacity of the auxiliary equipment (vessel walls and piping), the above-mentioned methods can be considered energy-efficient. However, in situations such as small-scale adsorbent regeneration or controlled release of pre-adsorbed active substances from adsorbent carriers, it might be preferable to heat up selectively only the adsorbent particles for a short period of time, without raising the temperature of the environment in which the adsorbent particles are present. In that case, other means of energy transfer need to be considered. The direct heating of the adsorbent particles can also be beneficial due to the low thermal conductivity of the packed bed.

Radiofrequency (RF) heating offers the possibility to selectively heat electrically conducting materials (Duquenne et al., 1994) via Joule heating or magnetic nanoparticles via Neel and Brownian relaxation processes. Nowadays, RF heating is used in industrial applications such as metal processing or thermo-bonding (Li and Dickie, 1991), but more recently also in biomedicine for selective heating of tumours using magnetic nanoparticles (Zhao et al., 2012). In our recent work we have demonstrated that the principle of RF heating can also be used for local control of other temperature-dependent processes such as controlled release from stimuli-responsive composite hydrogels (Zdražil and Štěpánek, 2010), hollow silica microspheres (Kovačik et al., 2012) or liposomes (Hanus et al., 2013). A distinct advantage of RF heating is that it enables “remote control” of local temperature-dependent processes without a physical contact or direct line of sight. Additionally, alternating magnetic field in the radiofrequency range (100 kHz–10 MHz) does not interact with living tissues, which is not the case of microwave or infrared heating, for instance.

In the present work, the use of RF heating for the control of desorption is investigated. Microstructured composite adsorbents based on three different materials (silica, zeolite, and activated carbon) and containing a metal RF susceptor (iron) have been prepared, their RF heating properties have been characterised and the factors controlling the desorption rates of two different adsorbates (water and toluene) have been described.

## 2. Materials and methods

### 2.1. Composite adsorbent preparation

Three types of adsorbents were fabricated in the form of pellets for the adsorption of two adsorbates: water (silica and zeolite based adsorbents) and toluene as a model VOC (activated carbon based adsorbent). The composite pellets contained the adsorbent material, a binder (colloidal silica Bindzil), and iron particles that had the role of RF susceptors (i.e., heat generation under RF field). The ratio between the adsorbent material and the binder was kept constant in each case, while the mass fraction of Fe particles in the composite matrix was varied at three levels in each case in order to investigate the effect of RF susceptor concentration on the heating rate. The full compositions of each type of adsorbent are given in Table 1.

Silica gel was purchased from Sigma-Aldrich (particle size 63–200  $\mu\text{m}$ , mean pore size 60  $\text{\AA}$ , specific surface area 500  $\text{m}^2/\text{g}$ ). For fabrication of composite pellets the silica gel was milled in a vibration mill (RETSCH MN400) for 100 min at 100 Hz. This reduced the mean size of the silica gel particles to 10  $\mu\text{m}$ .

**Table 1**

Composition of adsorbent pellets on a dry basis.

	Absorbent [g]	SiO <sub>2</sub> from Bindzil [g]	Fe [g]
Zeolite	1.7	0.6	0.300/0.062/0.032
Silica	1.0	0.375	0.150/0.075/0.038
Carbon	1.5	1.07	0.428/0.234/0.112

The zeolite was a potassium KA type (composition 0.6 K<sub>2</sub>O: 4.0 Na<sub>2</sub>O: 1 Al<sub>2</sub>O<sub>3</sub>: 2.0–0.1 SiO<sub>2</sub>: xH<sub>2</sub>O) purchased from Sigma-Aldrich (particle size < 10  $\mu\text{m}$ , absorbing molecules smaller than 3  $\text{\AA}$ ). The activated carbon (powder) was purchased from MespoX CZ. Its mean particle size was 0.25 mm and specific surface area 750  $\text{m}^2/\text{g}$ .

The silica, zeolite and carbon based adsorbents were fabricated by the following method. A given amount of adsorbent was mixed with iron particles (mean size 25  $\mu\text{m}$ ), colloidal silica binder (Bindzil) and water (if required) to form a slurry. After homogenisation, the slurry was poured into cylindrical polydimethylsiloxane (PDMS) moulds with a 9 mm diameter and a height of 13 mm. A thin central cavity was formed in the axis of the cylindrical pellet to provide space for a fibre-optic thermometer that was later used for on-line temperature monitoring. The moulds were placed into an oven for 12 h at 50 °C to remove water from dry porous pellets. After 12 h the pellets were removed from the moulds and further dried at 200 °C for 12 h to remove the remaining moisture and prepare the pellets for adsorption experiments. The final pellets had a cylindrical shape and weight of approximately 1 g for silica and zeolite, and 0.5 g for carbon based adsorbents. The SEM images of the internal pellet structure are shown in Fig. 1.

### 2.2. Radiofrequency heating set-up

The experimental set-up used for radiofrequency heating of the adsorbents is shown in Fig. 2. It consisted of a two-turn, 24 mm ID water-cooled copper coil connected to a 400 kHz RF generator (PowerCube 32/400, CEIA, Italy) with a field amplitude of 20 mT, in which the adsorbent pellet was placed. Temperature of the pellets during the induction heating was measured and recorded at 1 s intervals by means of a fibre-optic thermometer (Neoptix ReFlex, Canada). The fibre optic temperature probe was inserted into the central cavity of the pellet, therefore the measured temperature corresponds to the middle of the pellet. The RF coil with the pellet were exposed to ambient air. The desorbed vapours were removed and did not accumulate in the vicinity of the pellet.

### 2.3. Desorption kinetics measurement

The desorption kinetics for each adsorbent–adsorbate pair was measured as follows: the adsorbent was equilibrated with saturated vapours of the adsorbate in a desiccator for at least 12 h at 24 °C (in all cases, the weight of the pellet became constant after 5 h of adsorption). The partial pressure of water and toluene vapours at 24 °C is 2959 and 3600 Pa, respectively (calculated from the Antoine equation). By comparing the weight of the composite pellet before and after adsorption, the initial quantity adsorbed was evaluated.

The progress of desorption was investigated by gravimetric and temperature measurements. During the gravimetric measurement, the pellet was placed into the alternating magnetic field for a specific time period (depending on the desorption progress in the previous step), removed from the RF coil and weighted. The pellet was then returned into the RF coil and the magnetic field was switched on again. The time interval for which the pellet was not presented in the magnetic field was 30 s in all cases.

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