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Short communication

In situ observation of wall effects in activated carbon filters by X-ray microtomography

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ABSTRACT

X-ray microtomography is a powerful non-invasive visualisation technique which can be advantageously used to get a better understanding of dynamic adsorption processes. In the present work, this technique is shown to be able to detect wall effects during the dynamic adsorption of methyl iodide on activated carbon filters. The analysis of transversal cross-sections along the filter height clearly shows the existence of radial concentration profiles. These radial adsorption profiles are directly linked to velocity profiles due to a higher permeability at the wall. Obtaining such *in situ* information constitutes a real progress in order to validate simulation models allowing predicting reliable breakthrough times.

1. Introduction

Activated carbons are largely used to adsorb organic compounds in industrial processes as well as for civil and military protection. Their main advantages are as follows: high specific areas, high affinity towards organic compounds and low cost. As organic vapours are potentially toxic, a correct estimation of filter breakthrough times is essential. Many authors have developed models to predict breakthrough times [1]. Most of these models are one-dimensional, use an average linear velocity for the gas and neglect radial velocity profiles within the carbon beds. Nevertheless, because of the higher porosity at the wall, the permeability is more important in that region, leading to gas flow through preferential channels [2]. Consequently, as the annular zone close to the wall becomes rapidly saturated, a part of the gas stream leaves the filter before elimination of organic compounds and the breakthrough time decreases.

Even though the knowledge and the quantification of wall effects are crucial for a correct filter operation, they remained quite difficult to determine until the development of non-intrusive visualisation techniques such as X-ray tomography. This technique has been extensively applied in chemical engineering for the last 10 years in order to determine the phase spatial distribution within large process vessels [3,4]. More recently, the development of microfocus X-ray sources and high resolution CCD detectors led to the commercialisation of microtomographs with improved resolution (around a few microns). These systems, dedicated to small objects (from the mm^3 to few cm^3) opened the way for many applications in domains such as material science [5–8], biomedical science [9,10], and food science [11,12]. In this work, X-ray microtomography is used to evidence the existence of concentration radial profiles during adsorption of methyl iodide. The use of this non-destructive imaging technique to follow a dynamic adsorption process within activated carbon filters has been recently validated [13,14].

The experiments were carried out with CH_3I. The selection of methyl iodide is justified by its low boiling point, 42 °C, and by its adsorption behaviour which is the same as its radioactive counterpart [15,16]. Organic iodine is one of the three main radioactive forms that are produced during nuclear incidents, with molecular and particulate iodine (aerosols) [17].

2. Materials and methods

2.1. Activated carbon filters

Polyethylene cylindrical canisters with an internal diameter of 15 mm whose inlet and outlet consist of a metallic grid allowing gas circulation were used (Fig. 1). Polyethylene was chosen

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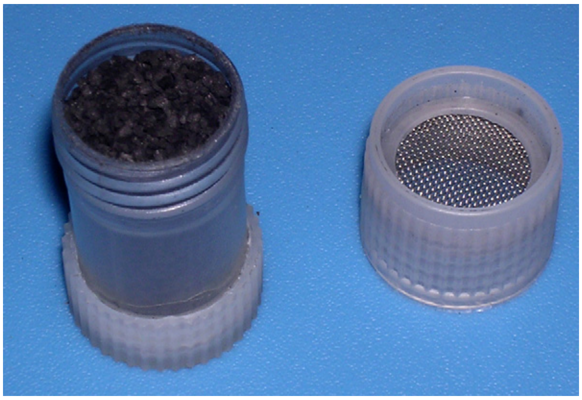

Fig. 1. Activated carbon filters.

because of its low X-ray attenuation, allowing obtaining good contrasted images of the carbon bed. The experiments were realised with BPL® 12 × 30 (Calgon Carbon Corporation) activated carbon the grain diameter of which ranging between 0.6 and 1.7 mm with an average of 1 mm. About 1.6 g of activated carbon were used for each trial, corresponding to an apparent density of 0.45 g/cm^3. The adsorption tests were conducted in a classical breakthrough measurement system [18] with CH_3I in dry air at a temperature of 293 K. The gas flowrate was fixed at 5 l/min, producing superficial velocities of 47 cm/s. The filters were analysed by X-ray µ-CT before and after exposure to CH_3I.

2.2. X-ray microtomography

X-ray tomography is a non-destructive technique allowing to visualise the internal structure of the investigated objects. Transversal cross-sections are reconstructed from radiographs obtained at several angular positions, around 180° or 360°. The grey level of each pixel is related to the local X-ray attenuation. The attenuation mainly depends on the energy of the incident beam, on the material density and on its atomic number [19]. In this study, a "Skyscan-1074 X-ray scanner" (Skyscan, Belgium) was used. The cone-beam source operated at 40 kV and 1 mA. The detector was a 2D, 768 pixels × 576 pixels, 8-bit X-ray CCD camera giving images with a pixel size of 41 µm. The rotation step was fixed at the minimum, 0.9°, in order to improve image quality, giving total acquisition times close to 10 min. After the filters were scanned, cross-section images (Fig. 2a) separated by 205 µm were reconstructed along the plastic canister using a cone-beam reconstruction software based on Feldkamp's algorithm [20].

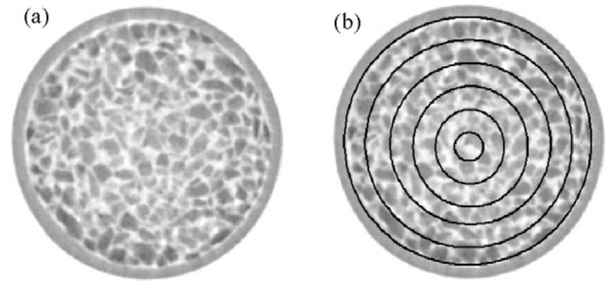

Fig. 2. (a) Transversal cross section in a virgin filter; (b) rings for the determination of concentration profiles.

2.3. Image analysis

The analysis of cross-sections is based on the fact that the grey level of a pixel full of carbon increases when adsorption takes place. This is related to the increase of the local X-ray attenuation coefficient with density (carbon + adsorbed CH_3I). Image processing was realised as follows: (a) a circular mask was applied to eliminate the filter walls; (b) the average grey level was determined on the

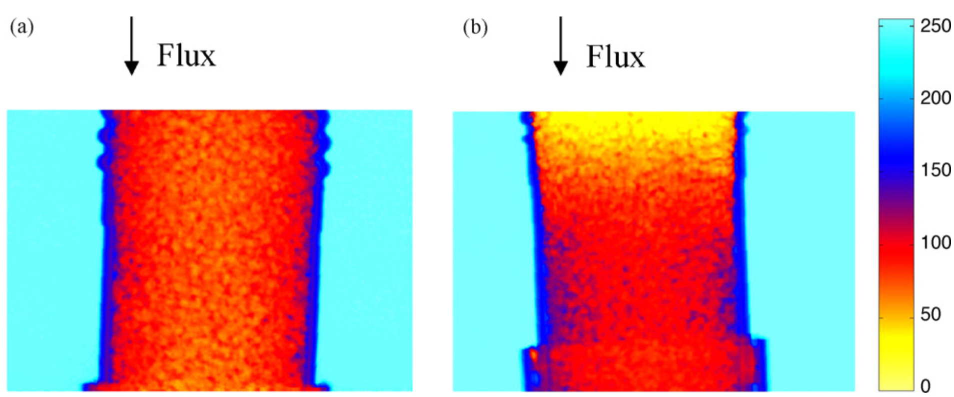

Fig. 3. Radiographs of a filter (a) before and (b) after 4 min exposure to CH3I.

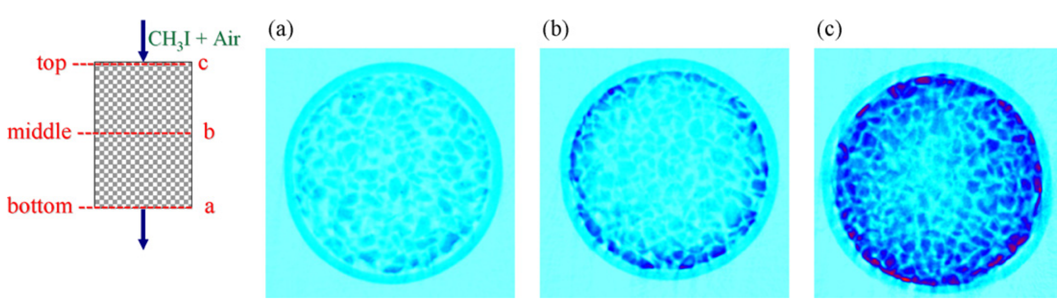

Fig. 4. Transversal cross sections located (a) at the bottom (b) in the middle and (c) at the top of the filter.