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Flow measurements below 50 μm: NMR microscopy experiments in lithographic model pore spaces

Elke Kossel, Rainer Kimmich*

Universität Ulm, 89069 Ulm, Germany

Abstract

Quasi two-dimensional random site percolation model objects have been prepared using a synchrotron radiation lithography technique with a spatial resolution better than 50 μm and an aspect ratio of up to 17. Flow of water through the pore space was studied with the aid of an NMR velocity mapping method and compared with a computational fluid dynamics simulation. In order to be able to measure and map widely distributed flow velocities with microscopic resolution (typically 40×40 μm), an experimental protocol that permits one to cover an effectively very wide velocity field of view (0.6–10 mm/s) had to be developed.

Keywords: Magnetic resonance imaging; Flow imaging; Porous media; Percolation

1. Introduction

MRI techniques are routinely used for mapping flow in porous media [1,2]. However, the smaller the spatial resolution of the experiment is, the more difficult it gets to record complete and undistorted flow maps. Effects such as diffusion, voxel inflow and outflow and artifacts caused by high velocities limit the experimentally accessible velocity range or lead to distortions of the map. These effects can be particularly severe in systems with a broad distribution of velocities such as porous media. If the exact geometrical structure of a pore space is known, a numerical simulation of the flow reveals the velocity range that is expected to be present in the experiment. With the knowledge of this range, it is possible to adjust the pulse sequence and the experimental setup in order to obtain a more complete picture of the flow process.

2. Velocity mapping

Artificial porous media can be simulated by so-called site percolation models [3]. Sites on a specific grid are randomly defined as being part of the pore space. Fig. 1 shows a two-dimensional site percolation cluster on a 300×300 pixel square lattice. We used deep X-ray lithography [4] to imprint this pattern with a pixel size of 60×60 μm in 1-mm-thick PMMA sheets [5]. Since the structure of the sample is known, finite element simulation software (Fluent 6.1, Fluent, Lebanon, NH, USA) could be used to numerically calculate flow maps for this cluster. Fig. 2A shows the obtained map of the velocity magnitude while Fig. 2C shows the histogram of the velocity distribution. A broad range of velocities is present, covering several orders of magnitude. In Fig. 2B, the spatial distribution of a selected range of small velocities is presented. It reveals a large network of flow channels that do not show up in Fig. 2A because of the poor contrast of the gray scale at small velocities. It is obvious that such a finely resolved velocity map cannot be obtained experimentally. However, it is important to get an estimate of the range that actually can be detected and of the possibly disturbing impact of velocities outside the experimental window. A general limit of the detectable velocity range is given for high velocities by voxel inflow or outflow during one scan of the pulse sequence. At high spatial resolutions with small voxel sizes, this effect is already present at velocities in the range of millimeters per second. Small velocities up to a few 100 μm/s, on the other hand, are masked by diffusion. The experimentally accessible velocity range is defined by these boundaries. A flow experiment should be set up in a way that the largest detectable velocity present in the sample is covered by the corresponding velocity field of view

* Corresponding author. Tel.: +49 731 5023140; fax: +49 731 5023150.
E-mail address: rainer.kimmich@physik.uni-ulm.de (R. Kimmich).

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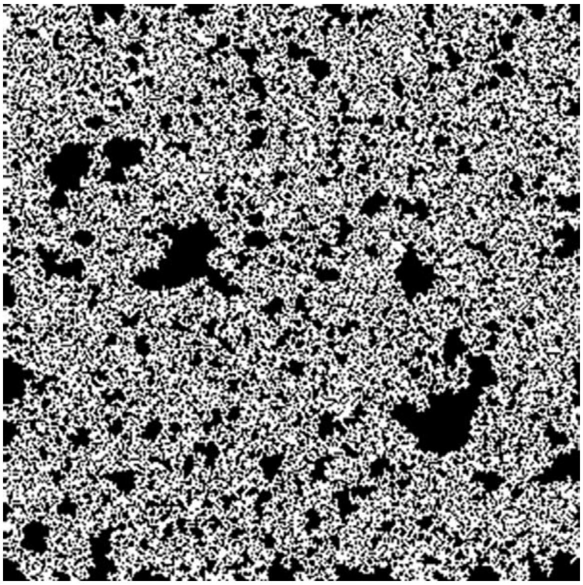

Fig. 1. Computer-generated percolation cluster that was used as a template for the sample. The pore space is shown in white.

(FOV). This avoids distortions from the backfolding of velocities into the experimental velocity scale. As can be seen from the velocity histogram in Fig. 2C, small velocities give a major integral contribution to flow through porous structures. Therefore, small velocities should also be covered experimentally. If positive and negative velocities are present, the required number of gradient steps for the pulse sequence is given by $4v_{max}/v_{min}$ if no zero filling is applied. A sufficiently large Δk between two gradient steps can only be guaranteed by using very strong or very long gradients. But the presence of suchlike gradient pulses inevitably causes an almost complete signal loss due to diffusion. This problem can be reduced by adapting the pulse sequence to the velocity FOV: Fig. 3A shows the velocity map of a fictional sample that is to be determined experimentally. Instead of performing one experiment with a large number of gradient steps, it is possible to cover the entire range by two experiments with a significantly smaller number of gradient steps. A small number of gradient steps would allow for the use of relatively short gradient pulses while Δk is still sufficiently large. The first experiment has a velocity FOV that covers the entire velocity range in as few steps as desired (see Fig. 3B). The map is not distorted but small velocities are measured as zero velocity. The second experiment covers the velocity range between the minimum expected velocity and the minimum velocity detected by the first experiment (see Fig. 3C). It is distorted because of the backfolding of high velocities into the spectrum, but it properly shows the small velocities. The velocity map shown in Fig. 3D is a combination of the two maps. Its basis is the map of Fig. 3B. Where this map shows zero velocity, its value is substituted by the value of the second map (Fig. 3C). Therefore, backfolded velocities in the second map do not contribute. The whole velocity range is covered but the resolution is now velocity dependent. It is coarse at high velocities and fine at low velocities.

3. Experiment setup

Flow of $CuSO_4$-doped water through the model pore space was investigated with a Bruker DSX 400 spectrometer (Bruker Biospin, Karlsruhe, Germany), which is equipped with a microimaging gradient unit. The maximal achievable gradient strength of this unit is 1 T/m; the maximal gradient strength that was used in the experiments was 0.83 T/m. All measurements were performed in two sets of Fourier encoding velocity imaging (FEVI) experiments corresponding to the FOV-adapted pulse sequence that is described in the preceding paragraph. The velocity FOV of the two experiments was chosen to be $\pm$9.6 mm/s for the first experiment and $\pm$2.4 mm/s for the second. Each single measurement had as few as 6 velocity-encoding gradient steps. Zero filling to 16 steps was applied afterward. The digital spatial resolution was 26.3$\times$36.7 μm. For this specific

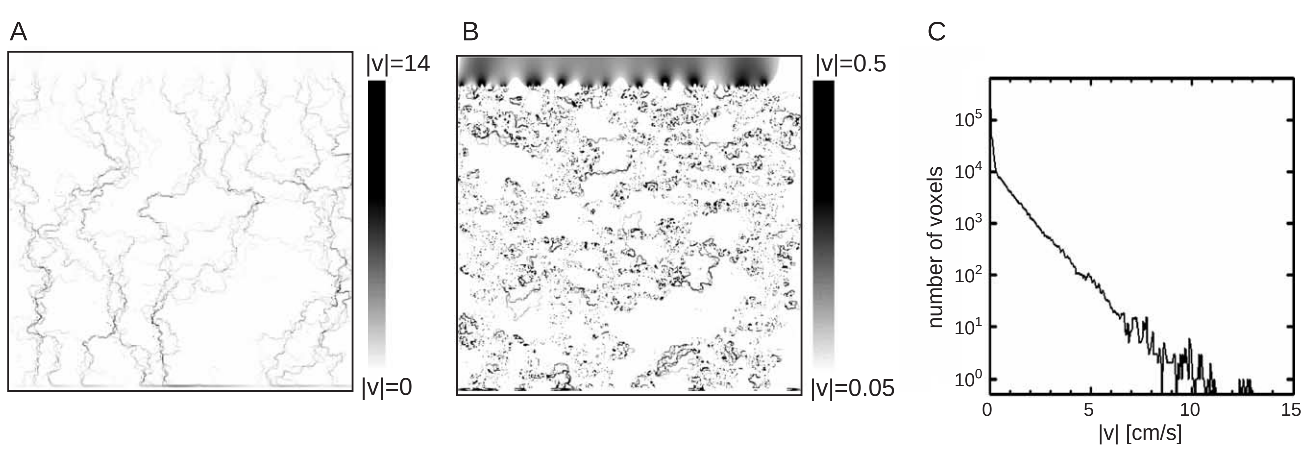

Fig. 2. (A) Numerically simulated velocity magnitude map for the sample geometry. The contrast of the color scale reveals only high velocities. (B) Velocity magnitude map showing a selected range of low velocities. (C) Histogram of the velocity magnitude obtained from the complete simulated map.