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Adapted MR velocimetry of slow liquid flow in porous media

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ABSTRACT

MR velocimetry of liquid flow in opaque porous filters may play an important role in better understanding the mechanisms of deep bed filtration. With this knowledge, the efficiency of separating the suspended solid particles from the vertically flowing liquid can be improved, and thus a wide range of industrial applications such as wastewater treatment and desalination can be optimized. However, MR velocimetry is challenging for such studies due to the low velocities, the severe B_0 inhomogeneity in porous structures, and the demand for high spatial resolution and an appropriate total measurement time during which the particle deposition will change velocities only marginally. In this work, a modified RARE-based MR velocimetry method is proposed to address these issues for velocity mapping on a deep bed filtration cell. A dedicated RF coil with a high filling factor is constructed considering the limited space available for the vertical cell in a horizontal MR magnet. Several means are applied to optimize the phase contrast RARE MRI pulse sequence for accurately measuring the phase contrast in a long echo train, even in the case of a low B_1 homogeneity. Two means are of particular importance. One uses data acquired with zero flow to correct the phase contrast offsets from gradient imperfections, and the other combines the phase contrast from signals of both odd and even echoes. Results obtained on a 7T preclinical MR scanner indicate that the low velocities in the heterogeneous system can be correctly quantified with high spatial resolution and an adequate total measurement time, enabling future studies on flow during the filtration process.

1. Introduction

Deep bed filtration [1–5] is a separation process with the aim of clarifying a liquid. To this end, the liquid with suspended solid particles is slowly streaming downwards through the porous filter with local velocities of submillimeters to millimeters per second. Two groups of mechanisms underlie the filtration process, one leads to routes of particles towards interfaces between the liquid and the porous filter (*e.g.* gravity settling, diffusion, interception), and the other causes immobilization of particles at the filter surfaces (Van-der-Waals and electrostatic forces, straining). Due to the large amount of particles which can be retained and the slow increase of pressure drop, deep bed filtration is widely used in industrial applications, such as wastewater treatment and desalination [4,5].

Recent studies [6–14] have shown the potential of X-ray *Micro-Computed Tomography* (μCT) for characterizing the particle deposition processes. With 3D spatial resolution in the micrometer to submillimeter range, serial X-ray μCT images of porous structures and deposited particles over time can provide microscopic knowledge of the particle deposition processes.

While the correlation between the particle deposition processes and the velocity fields of the flowing liquid has been studied for other filtration processes [15–17], this correlation has not been studied yet for deep bed filtration. Insights into this correlation will certainly lead to a more profound understanding of deep bed filtration mechanisms and thus an improved design of deep bed filters with regard to filtration efficiency. Quantifying the velocity fields of the liquid can be achieved by *MR Velocimetry* (MRV) [18,19], which is particularly preferred for opaque structures, where optical velocimetry techniques cannot be applied. MRV is a highly versatile tool for velocity mapping of fluid flow, since it contains a variety of adaptable methods for laminar and turbulent, single-phase and multi-phase, fast and slow flow [18,19]. For the specific case of deep bed filtration, dedicated MRV is required for measuring slow liquid flow of submillimeters to millimeters per second in macroporous media with 3D sub-pore-scale spatial resolution.

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Additionally, this MRV method should allow an adequate total measurement time, because the velocity fields of the liquid can alter significantly due to substantial changes of pore filling after a few hours of filtration.

MRV can be achieved by adding a displacement encoding module to a standard MRI pulse sequence. There are two main MRV methods, *q-space* MRI [20] and *phase contrast* MRI [21–23]. In q-space MRI, the probability distribution of fluid displacements is spatially resolved as voxel-wise propagator spectra. In phase contrast MRI, the voxel-wise average propagator is directly measured to provide the average fluid displacement within each voxel. Though inferior in velocimetry accuracy to q-space MRI, phase contrast MRI is widely used due to the much smaller number of required displacement encoding steps, leading to a shorter minimum total measurement time.

In phase contrast MRI, the *Signal-to-Noise Ratio* (SNR) of the complex-valued MR images is not only a measure of image quality, but also determines the accuracy of measured velocity maps, because the *STandard Deviation* (STD) of the velocities is reciprocally proportional to the SNR [24–26]. Therefore, phase contrast MRI methods using a *Spin Echo* (SE) based magnetization preparation module for velocity encoding are superior to those using a STimulated Echo (STE) based magnetization preparation module, because the latter implies a general 50% signal loss.

MRV can be accelerated by different methods, *e.g.*, compressed sensing, Bayesian MR, and parallel imaging [19]. Another strategy with respect to MRI pulse sequence design is the use of multiple echoes for multiple phase encoding steps, *i.e.* the *Rapid Acquisition with Relaxation Enhancement* (RARE) method [27]. RARE MRV is a reasonably fast MRV approach [28–37] and has been applied for displacement mapping of liquid in porous systems like plants [28,29] and rocks [37].

A recent study [37] showed that RARE phase contrast MRI with 3D super-pore-scale spatial resolution can still be accurate for very slow liquid flow in porous media. However, the implementation of the method in [37] was not considered optimal for the current work. First, a specific STE based magnetization preparation module [38] was used in [37] due to very small velocities of submillimeters per second and strong internal magnetic field gradients within each super-pore-scale voxel, while an SE based magnetization preparation module can be used in the current work to improve the SNR because of relatively larger velocities and weaker internal magnetic field gradients within each sub-pore-scale voxel. Second, a rather small RARE factor (2) and thus a short echo train length were used in that study due to short T_2 in rocks, while longer T_2 in deep bed filters enables a larger RARE factor. However, several additional strategies need to be considered for velocimetry accuracy with a relatively longer echo train length, such as RF filtering [34] or phase cycling [33,36] for recovering the correct complex-valued signals, as well as phase encoding winder and rewinder gradients for suppressing motion artifacts.

In this work, an efficient and accurate MRV approach for slow liquid flow in porous media was achieved by adapting existing RARE phase contrast MRI methods. For SNR reason and due to a moderate velocity range to be measured, an SE based velocity encoding module was used. A two-step phase cycling containing CPMG and CP pulse trains [33,36] was applied to preserve the propagator information within the magnetization in the echo train. Velocity offsets caused by gradient imperfections [39–41] were measured with zero flow for correcting the velocity maps of the flowing liquid. Velocity maps of odd and even echoes in the echo train were combined to increase velocimetry accuracy. The proposed MRV approach can be a helpful tool for improving the understanding of deep bed filtration and may also be of interest for further MRV applications.

2. Methods

2.1. Porous filter sample

A templated foam derived from polysiloxane was produced as a cylindrical filter sample (diameter 9 mm, height 18 mm) with spherical pores with a mean diameter of about 2 mm according to [42]. Expanded polystyrene beads (~2 mm, Klassen Vitali, Germany) were used as templates and packed in a poly-propylene mold. This mold was infiltrated with a mixture of 86 wt% of methyl phenyl polysiloxane (H44, Wacker Chemie AG, Germany) and 14 wt % of 3-aminopropyltriethoxysilane (APTES, abcr GmbH, Germany) in a few milliliters of ethanol. The beads packing in the originally completely filled mold was compressed by 6.25 vol%, while excessive polymer solution was drained. After cross-linking at room temperature and 100 °C, the pyrolytic conversion of the cross-linked material was carried out at 1000 °C under nitrogen. This SiOC ceramic was shown to have a hydrophilic surface characteristic by performing water and n-heptane adsorption experiments.

Distilled water was used as the liquid flowing through the sample. The sample was mounted in a dedicated filtration cell made of polytetrafluoroethene (PTFE). The inlet and outlet in the filtration cell have an inner diameter of 8 mm. The filtration cell was connected to a peristaltic tubing pump (ISMATEC REGLO Analog MS-2/6, Cole-Parmer GmbH, Germany) by tubes. The pump permits steady liquid flow through the filtration cell with the linearly controllable *Volumetric Flow Rate* (VFR) between 1.26 and 125 mm^3/s. The linearity and the VFR range were measured by monitoring the volume of the distilled water flowing out of the filtration cell. Due to the long tube length between the pump and the filtration cell (>3 m), the pulsation effect of the peristaltic pump is regarded as negligible. Before MR experiments, the filtration cell was soaked within the distilled water in the bottom part of a desiccator to achieve pore pre-saturation, and then the desiccator was sealed and evacuated from the top by a vacuum pump for pore saturation. Due to the low pressure, remaining gas within the pores degassed and the pores were eventually saturated.

2.2. MR hardware

All MR experiments were performed on a horizontal 7T scanner (BioSpec 70/20 USR, Bruker BioSpin MRI GmbH, Germany), which is equipped with a 114 mm bore gradient system (B-GA 12S2, Bruker BioSpin MRI GmbH, Germany) and controlled by ParaVision 5.1 (Bruker BioSpin MRI GmbH, Germany) on a Linux workstation. The gradient system contains room temperature shim coils of up to second-order, a maximum gradient amplitude of 440 mT/m and a maximum slew rate of 3440 mT/m/ms.

Due to the vertically orientated filtration in deep bed filters, a vertical MR scanner would certainly be a good choice. However, the drawback of limited vertical space available in a horizontal MR scanner with a horizontal RF coil was reduced by using a home-made, vertically orientated RF coil. This dedicated coil consists of two identical thin rectangular copper plates fixed on the outer surface of a hollow PTFE cylinder resulting in a loop-gap resonator. A fixed-value capacitor was soldered on one gap and a trimmer capacitor on the other gap to link the two copper plates. A pick-up loop covering the resonator bore was mounted on a PTFE slider, which supports the movement of the pick-up loop along the longitudinal direction of the loop-gap resonator. Tuning and matching of this linear transceiver RF coil is performed by adjusting the trimmer capacitor and sliding the pick-up loop longitudinally, respectively.

In order to achieve high RF efficiency and SNR, the filling factor of the coil was maximized by adapting the resonator diameter to