

## Nano-particle dynamics during capillary suction

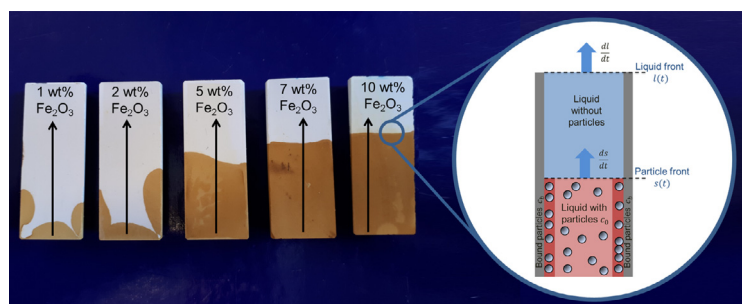
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### GRAPHICAL ABSTRACT



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### ABSTRACT

Due to the increased use of nanoparticles in everyday applications, there is a need for theoretical descriptions of particle transport and attachment in porous media. It should be possible to develop a one dimensional model to describe nanoparticle retention during capillary transport of liquid mixtures in porous media.

Water-glycerol-nanoparticle mixtures were prepared and the penetration process in porous  $\text{Al}_2\text{O}_3$  samples of varying pore size is measured using NMR imaging. The liquid and particle front can be measured by utilizing  $T_2$  relaxation effects from the paramagnetic nanoparticles.

A good agreement between experimental data and the predicted particle retention by the developed theory is found. Using the model, the binding constant for  $\text{Fe}_2\text{O}_3$  nanoparticles on sintered  $\text{Al}_2\text{O}_3$  samples and the maximum surface coverage are determined. Furthermore, we show that the penetrating liquid front follows a square root of time behavior as predicted by Darcy's law. However, scaling with the liquid parameters is no longer sufficient to map different liquid mixtures onto a single master curve. The Darcy model should be extended to address the two formed domains (with and without particles) and their interaction, to give an accurate prediction for the penetrating liquid front.

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## 1. Introduction

With the increasing number of engineered nanoparticles used in everyday applications ranging from inkjet printing (pigment particles), contrast agents in medical applications (Nuclear

Magnetic Resonance imaging) and removal of contaminants in the subsurface [1,2], there is an increasing demand to understand and predict particle transport in porous media. Whether the porous medium is the paper used in printing, different soil layers in the subsurface or the human body, the transport, and more specifically, the retention of particles in such media are of utmost importance. Particle transport in saturated porous media has been the topic of many studies in the last couple of decades, and theories

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to predict the particle retention based on colloid filtration theory (CFT) are developed for many liquid – media systems [3–6]. A review on the flow of dispersed particles through porous media is given by Zamani and Maini [7]. The retention of nanoparticles depends on both liquid and media properties, including the interaction between the liquid and the porous material. An analysis on the importance of the different liquid, media and interaction parameters defining nanoparticle transport in porous media is given by Babakhani et al. [8], who reanalysed data from published column experiments to determine the five key continuum model parameters to predict nanoparticle transport.

Validating the developed theoretical models and, in most cases, finding the attachment efficiency of a liquid – media system, requires experimental data [9]. Most of the experimental data available consist of breakthrough curves (BTC), where the outflow concentration of particles is measured over time using techniques like light extinction [10], X-ray fluorescence [11], turbidimeter [12] and atomic absorption spectrometer [13]. The advantage of these measurements is the high time resolution that can be obtained as well as the possibility to measure on any porous material. The main drawback of these methods, however, is the use of a sample average for the particle retention measurement. Furthermore, BTC's is often used for saturated sample measurements, which is not necessarily mimicking a real life application. For example in the case of inkjet printing, the attachment of particles in the paper porous system during capillary flow determines the penetration depth of the particles, which in turn affects the print quality and print robustness [14].

A secondary method that is often used to monitor particle retention in porous media is microscopy to measure particle transport in micromodels or columns filled with glass beads [15–17]. Similarly to the BTC methods, these yield high time resolutions, but the method can only be applied to transparent media. A technique that allows local measurements in non-transparent porous media is NMR imaging [18–21]. Paramagnetic particles for contrast enhancement offer new opportunities to study particle motion in porous media [22].

Nanoparticle penetration during capillary suction in porous media has been the topic of far fewer studies [23,24]. A review of particle retention and detachment in unsaturated porous media, especially at the air–water interface (for example at the liquid penetration front during capillary suction) is given by Flury and Aramrak [25]. The experimental techniques allowing to study liquid penetration in unsaturated porous media consist of microscopy for transparent porous media, ultrasonic measurements [26], and NMR.

In the field of inkjet printing, but also in the penetration of spills containing particles on porous media, the particle transport and retention in such unsaturated porous media is important. More precisely, the particle penetration and retention during capillary penetration of the liquid mixture determines the penetration depth of the particles. NMR can be used to study these kind of particle transport processes, where both the liquid penetration as well as the particle retention can be visualised using  $T_2$  relaxation effects similarly to the ones used in medical applications for  $T_2$  weighing [10]. This method allows a time and position resolved method to quantify the penetration depth of particles and liquid during the capillary suction process. Models describing these effects, where particles can move along with the liquid front or attach to a surface are developed in the field of chromatography [27–30].

The aim of this study is first of all to measure both the particle and the liquid components during imbibition in porous media. Secondly, we aim to develop a model that quantitatively describes the particle retention occurring during capillary suction of a complex liquid mixture containing particles.

This model is derived in chapter 2 and the theoretically predicted limiting cases are addressed. In Section 3 we discuss the used liquid mixtures, the porous medium and the experimental method. To measure the penetration of liquids containing paramagnetic nanoparticles, we use 1D NMR imaging, which is explained in detail in Section 3 as well. In Section 4, the main results of this study are presented. The liquid and particle fronts as a function of time are visualised and the binding constant and the maximum surface coverage for the used  $Fe_2O_3$  nanoparticles on the  $Al_2O_3$  surface are determined. Furthermore, the limiting cases predicted by the developed model are verified using sintered  $Al_2O_3$  samples with an average pore radius differing over two orders of magnitude in the micrometer and submicrometer regime.

## 2. Theory

When a liquid mixture containing nanoparticles is brought into contact with a porous medium, the mixture imbibes into the material. Depending on the interaction of the liquid with the porous material, the mixture can penetrate the material as a homogeneous liquid or the components can split into a liquid and a particle front. Several models exist for the penetration of liquids into porous media, most of them being based on Darcy's law [31]. When the particles have an attractive interaction with the porous medium, the particles can stick to the pore surface, delaying the actual motion of the particles and resulting in a separation of the liquid and the particle front. This effect is studied extensively in the field of chromatography [27–30].

### 2.1. Particle adsorption

We consider the particles as equally sized spheres with diameter  $d$ [m]. Particles either adsorb to the pore surface with an area  $A$ [ $m^2$ ] or stay in the solution with a volume  $V$ [ $m^3$ ].  $c$ [ $m^{-3}$ ] is the particle density in the solution and  $\Gamma$ [ $m^{-2}$ ] the surface coverage of the adsorbed particles. The latter can also be rewritten in terms of the bound particle concentration  $c_b$ [ $m^{-3}$ ]:

$$c_b = \Gamma \frac{A}{V} [m^{-3}]. \quad (1)$$

The total particle concentration is equal to  $\bar{c} = c + c_b$ [ $m^{-3}$ ]. Assuming that the sorption is only driven by particle–surface interactions, the maximum absorbed amount equals  $\Gamma_m = c_{b,m} \frac{V}{A} \sim d^{-2}$  with  $c_{b,m}$  the maximum bound particle concentration. In equilibrium, there is a unique relation between the absorbed amount and the concentration in solution. In case of monolayer adsorption, a widely used model is the Langmuir model [32],

$$c_b = c_{b,m} \frac{\kappa c}{1 + \kappa c}, \quad (2)$$

where  $\kappa$ [ $m^3$ ] is the binding constant. When  $\kappa c \ll 1$  a linear relation is left:  $c_b = c_{b,m} \kappa c$ . At very high particle concentrations,  $\kappa c \gg 1$ ,  $c_b = c_{b,m}$ .

### 2.2. Liquid motion

As a first approximation, it is assumed that the imbibition process itself is not influenced by the particle sticking process. In the case of particle retention during imbibition, two domains are present in the pore space. An upstream part without particles and a downstream part containing particles in the solution and attached on the pore walls. This situation is schematically drawn in Fig. 1.

For low particle concentrations, the liquid properties, such as the viscosity, surface tension and contact angle are similar for domains, where the liquid contains particles (before the particle front) and domains that do not contain particles (beyond the par-

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