

Measurement Method for Determining Solvent Exchange in Aerogels During Realization and Functionalization

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Summary:

In this work a measurement method for determining solvent exchange in an aerogel-filled microfluidic channel is investigated. For this purpose, the dye Eosin Y is applied to examine the diffusion using a fluorescence microscope. The measurements confirm the suitability of the measurement method for determining the diffusion behavior in aerogel. This enables the optimization of solvent exchange after the aging step of the sol-gel process and will facilitate the use of aerogel-based sensors in microfluidic channels.

Keywords: aerogel, diffusion, solvent exchange, microfluidic channel, MEMS

Background, Motivation and Objective

Aerogels refer to a group of materials characterized by their high porosity, low density, and low thermal conductivity [1]. Aerogels typically have a porosity of 80% to 99.8%, a specific surface area of 500 m²/g to 1200 m²/g, and a density of 0.003 g/cm³ to 0.5 g/cm³. Silica-based aerogels are the most commonly used [2]. The use of aerogels as a material for sensors offers great potential. For example, gas sensors or bio sensors often rely on the use of sensing materials. Due to the high specific surface area and highly interconnected porous structure aerogels are attractive as a support material for these. The Agglomeration of the sensing material that is being taken up into the aerogel can be reduced by the porous network. Furthermore, the high porosity ensures that the active sites of the sensing material remain accessible for the stimuli [3]. In the sol-gel process for silica aerogels, one of the most important steps is the aging process. This process strengthens the network, resulting in a stable porous network [2]. After this process, different solvents are regularly replaced to achieve specific properties for the aerogel [4]. However, in order to enable the integration of aerogels in chips, there is a lack of fundamental research on the diffusion of solvents in aerogels. In this work the solvent exchange in aerogel within a microfluidic channel is investigated.

Description of the New Method or System

The silica aerogels used in this work were synthesized using the sol-gel process. This process

is done in two phases. First TEOS, ethanol, and water are mixed. To start the hydrolysis HCL is added to this mixture. After 1.5 h, ammonia is added to initiate condensation, causing the gelation. After gelation is achieved which takes around 50 min, the gel is filled into a microfluidic channel [4]. The used channels have a width of 1.6 mm and consist of a glass-silicon-glass structure with two openings in the top glass wafer. The aerogel in the channel was aged afterwards and kept in a beaker filled with ethanol. To be able to investigate the diffusion in a porous silica aerogel structure the dye Eosin Y from Merck is used. This dye has fluorescent properties which makes it possible to investigate the diffusion using a fluorescence microscope. For the measurement, the chip filled with aerogel was placed in a beaker with an Eosin Y ethanol solution. The aerogel was loaded with the dye for 30 min and then put into a Petri dish filled with ethanol without the dye to avoid evaporation of the liquid during the measurement. This Petri dish is then placed under a fluorescence microscope and images were automatically taken every 5 min. The camera settings were kept constant to avoid any influence from changing settings.

Results

In Fig. 1 a selection of images illustrating the diffusion into the aerogel is shown. Time intervals of 30 min, 45 min, and 5 h were selected. The reason for this is that the diffusion is significantly faster at the beginning and slows down as the dye concentration increases. After

approximately 5 h, the color is saturated, and no further change in intensity can be observed. It is visible, that the green color of the Eosin Y dye is not evenly distributed across the channel. The reason for this could be the inhomogeneity of the aerogel structure. The porous network is denser in some areas than in others, resulting in slightly different diffusion rates.

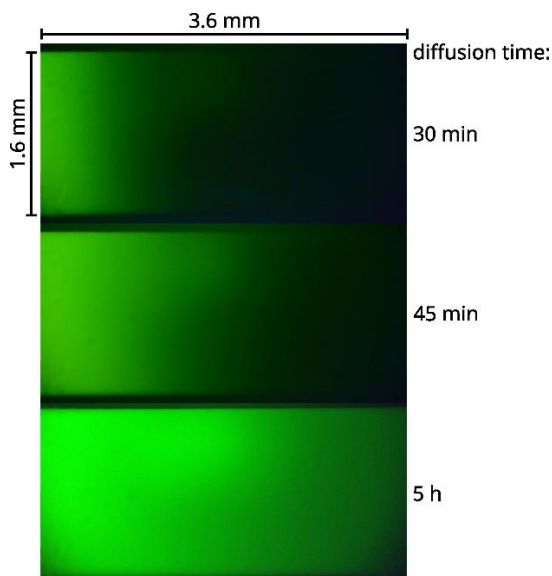


Fig. 1. Diffusion of Eosin Y in ethanol into aerogel in a 1.6 mm wide and 3.6 mm long microfluidic channel.

Using the images from the measurements the intensity of the green color can be extracted. The intensity is normalized to the maximum measured value using an 8-bit color profile. The resulting intensity profile can be seen in Fig. 2. The figure shows the average of the green color across the entire channel width. As the aerogels were loaded for 30 min the first measurement was taken at 30 min.

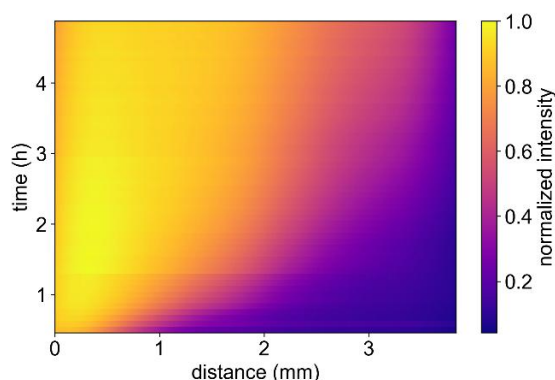


Fig. 2. Fluorescence intensity profiles of the diffusion measurement.

It can be seen that the diffusion is initially much faster and slows down as the dye concentration in the channel increases. To observe this behavior more closely, the diffusion distance at a normalized intensity of 0.75 is plotted over the time in Fig. 3. The measurement results were shifted to the origin to allow fitting the data.

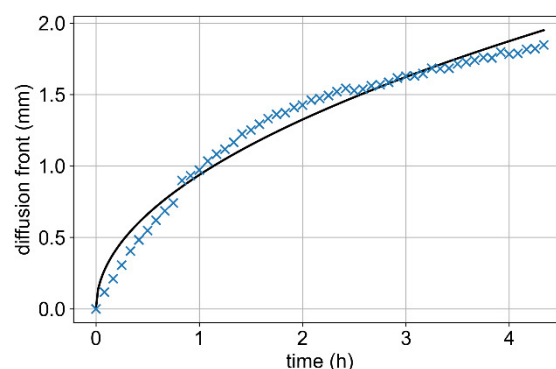


Fig. 3. Diffusion front at a normalized intensity of 0.75 over time.

The data shows that diffusion in a wet aerogel can be described by the function $x \approx \sqrt{2Dt}$. The curve fitting results in an apparent diffusion constant of $D = 1.2e^{-10} \text{ m}^2/\text{s}$. For comparison, the self-diffusion coefficient of ethanol at 25 °C is $D \approx 1.1e^{-9} \text{ m}^2/\text{s}$ [5]. This difference of about a factor of 10 is to be expected, since diffusion slows down noticeably due to the size of the dye molecules and the porous network of the aerogel. The measurement performed demonstrates the ability to determine the diffusion coefficient of Eosin Y in an aerogel-filled microfluidic channel. Using the presented method, it is possible to determine the diffusion of the solvents used for the solvent exchange. This would allow for adjusting the intervals between solvent exchanges or determining the maximum still practical length of the microfluidic channels.

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