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Experimental improvements for ferrofluid impregnation of rocks using directional forced impregnation methods: results on natural and synthetic samples

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The magnetic pore fabrics method is a useful technique to investigate the pore fabric of rocks. The method is based on impregnating porous samples with ferrofluid, a colloidal suspension of magnetic nanoparticles (particle size of 10 nm) in water or oil carrier fluid, and measuring the anisotropy of magnetic susceptibility. It successfully provides the average pore shape and orientation. This information is important to determine the preferred direction of fluid flow. A crucial step in magnetic pore fabric studies is ferrofluid impregnation; several studies pointed out the importance of forced impregnation methods to enhance impregnation efficiency. This study compares directional impregnation techniques (applying external forces along the ferrofluid flow along the axis of the core sample) with standard vacuum- and immersion-based methods. The newly developed or adapted techniques include: (1) pressure experiment: a cylindrical sample is placed in a metal tube under confining pressure of 12 bar, and an external pump-syringe system injects ferrofluid at a constant rate of 100 ml/min that generates a differential pressure of 5 bar; (2) resin flowthrough: vacuum is applied at the bottom of the sample and a mixture of resin and oil-based ferrofluid supplied at the top, so that the resin drags the fluid into the pores, where it hardens; (3) magnetically assisted flowthrough: fluid flow is enforced by the combined action of a hydraulic pressure gradient in the ferrofluid reservoir (~10 kPa) and the magnetic force exerted by the field gradient of about 2 A/m² in the vicinity of an electric coil. These impregnation methods were tested on natural and synthetic samples, for which previous experiments employing standard impregnation methods exist. The natural samples include calcarenite from Apulia, Italy (50% porosity) and sandstone from Schupfheim, Swiss molasse (20% porosity). Synthetic samples consist of calcite and quartz sand in different proportions, consolidated with liquid glass (sodium silicate) in a cubic consolidation cell (specifically designed for the experiment), applying uniaxial pressure along the z axis, to create uniaxial anisotropy. The cube was dried in the oven for three days and three cylindrical cores were drilled along the x, y and z axes. For each impregnation method, the magnetic anisotropy of the samples was measured before and after impregnation. Impregnation efficiency was tested using bulk susceptibility measurements, visual microscopic investigations and susceptibility profiles along the flow direction. Initial results show that (1) directional forced impregnation is more efficient than traditional methods in impregnating smaller pores, avoids particle aggregation, and allows viscous fluid such as resin to access the sample's pores; (2) directional impregnation methods require less fluid; (3) the distribution of the ferrofluid

after impregnation is more uniform, overcoming the difficulty of impregnating the centre of the sample; and (4) the fluid flow rate must be faster than the particle aggregation rate. For future studies, directional forced impregnation systems are recommended over standard vacuum- and immersion-based impregnation methods.