

USE OF MICROMODELS TO STUDY MULTIPHASE FLOW IN POROUS MEDIA

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ABSTRACT

Micromodels are transparent etched or molded porous media used to visualize multiphase flows and displacements. This paper presents the various techniques used to make micromodels and review the main results derived from visualization studies.

The history spans from pioneering work of Mattax and Kyte to the recent use in microfluidics. The techniques are mainly based on glass etching by hydrofluoric acid by using image patterns plotted by computer. The etched plate is then sintered with a glass cover or a mirror pattern inside an oven. Placed in a confinement cell these micromodels allow the visualization of fluid displacements at reservoir pressure and temperature. Etching on silicon wafers can reach much smaller pore sizes. Another method uses photopolymers for making the grooves; then a micromodel is casted in resin after molding. Pores are bigger, but shapes are more regular and visualization and interpretations are easier.

The main application of micromodels has been to identify the mechanisms of immiscible displacement of one fluid by another inside channels and at intersections of channels, for drainage and imbibition processes. The identified mechanisms have then been used to quantify the pore-scale physics and develop mechanistic pore network numerical simulators. Results from simulations at pore network scale have been compared to micromodel displacements, as a model validation step. One of the main advantages is to relate macroscopic transport properties to microscopic displacement mechanisms and fluid distribution. In earlier work, attention was paid on the description of flow patterns, invasion percolation, viscous fingering and stable flow regimes. Then, emphasis was placed on flow of wetting fluid film along the walls, trapped blob (ganglia) displacement, three-phase flow mechanisms as related to wettability, two-phase flow of shear-thinning fluids, particle flow and filtration, miscible displacement and hydrodynamic dispersion, flow of surfactants etc.

Further studies with micromodels can throw light in not-well understood flow mechanisms, such as oil displacement under conditions of neutral wettability, with contact angle close to 90°, and evident impact to digital rock simulations.

INTRODUCTION

Various techniques are used to visualize the pore scale properties of flow in porous media, starting from single tubes to recent micro-tomography. The purpose is to

understand the mechanisms in order to develop theoretical models or to elaborate the displacement rules to be used in network and pore-level simulators. Some visualization studies are also very useful for pedagogical purpose. This paper will concentrate on 2-dimensional planar transparent models of porous media called "micromodels". A description of other non-destructive imaging techniques for 3-D samples (gamma ray, X-Ray microtomography, NMR) are described in a detailed review by Werth [1].

This paper is not an exhaustive review. Our purpose is only to recall a few techniques for making micromodels and emphasize some representative results and give a list of several domains of application to oil industry.

OTHER VISUALIZATION TECHNIQUES

Single tubes have provided results on the snap-off mechanism (circular cross-section with Roof [2] or square cross section with Legait [3] showing the effect of inertial forces, even for very slow displacements. Transparent media using crushed glass either with matched refractive index (high concentration of $ZnCl_2$ solution), water/oil (Figure 1) or water/gas systems (Figure 2). This technique was mainly used to study the fingering patterns during unstable displacement but cannot reach pore scale resolution, at least in the historical version [4,5]. Crushed cryolithe gives a better result since its refractive index is close to that of water, but transparent crystals are difficult to find.

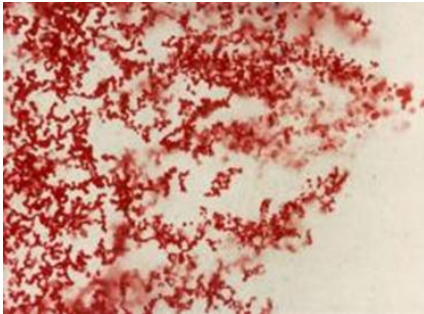


Figure 1 Oil (red) displaced by water at end of imbibition (length around 10 cm)

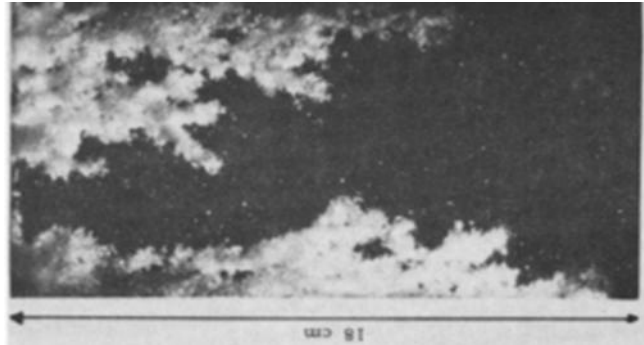


Figure 2 Gas displacing water in a crushed glass packing [6]

Before the use of conventional micromodels, large-scale models were used in Institute of Fluid Mechanics, Toulouse, either with a single channel or 2-dimensional media (Figure 3). Grains of centimeter size were cut in plastic blocks with a shape following enlarged reproductions of rock thin sections and placed between two plastic sheets. The total size was around 1 m. Chauveteau [7] studied the inertial effects using colored fluid (Figure 4) and showed that the decrease of permeability at high velocities was due to the change of flow lines inside the pores. Ganoulis [8] described the mechanisms of pore invasion by a non-wetting fluid (Figure 5) and was the first who correlated drainage mechanisms with percolation theory in 1974.

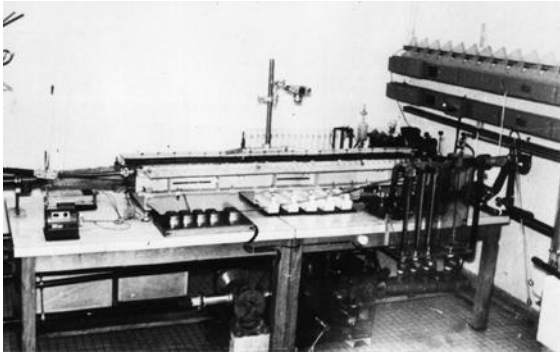


Figure 3 Experimental set-up for the analogical model used at IMF in Toulouse

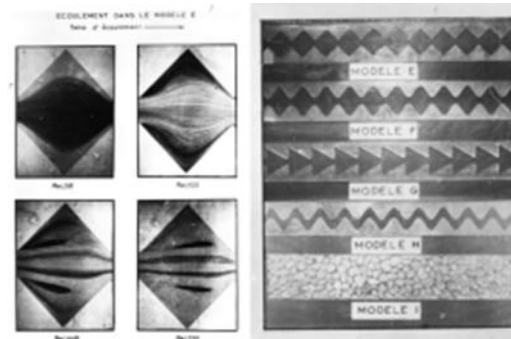
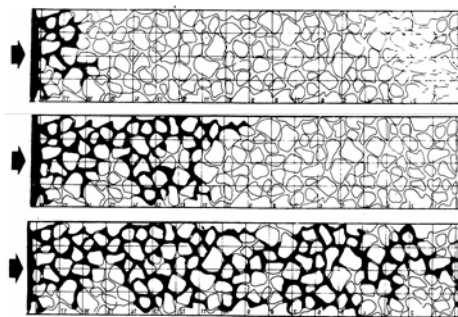
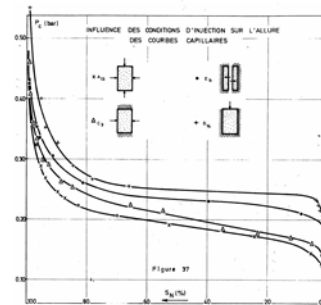


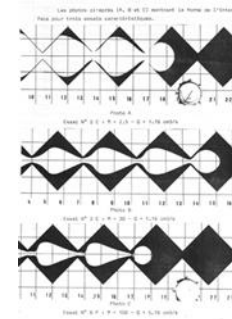
Figure 4 Study of inertial effects in analogical models by Chauveteau. The lengths of the channels are around 1 meter.



a)



b)



c)

Figure 5 a) invasion of mercury in Ganoulis analogical model (length around 1 m.); b) Pc curve derived from mercury injection with different boundary conditions; c) invasion of a non-wetting fluid at different flow rates showing the effect on the amount of trapped fluid.

MAKING MICROMODELS

Glass bead monolayers

Several authors have used experimental models containing a monolayer of glass beads between two glass plates (Chatenever [9], Moulu [10]). The main drawback was the fluid bypass between the plates and beads. To avoid this bypass, the research team in Oslo (Oxall [11]) used a thin plastic sheet pressurized by compressed air to make contact between the beads and keep them in place. This technique allows the use of very large samples (diameter~1m) without requiring any specific pattern of the porous medium. However, the difference of wettability between the glass beads and the plastic sheet can be a problem for oil/water displacements.

Glass micromodels

In 1961, Mattax and Kyte [12] used chemical etching of glass plates. The plate was coated with a thin layer of wax and lines were scribed with a stylus. Then the glass was etched in hydrofluoric acid (HF). The drawing was a square grid with 350x350 lines. Intermediate wettability was obtained by saturating the model with crude oil for several hours. Davis and Jones [13] made 2-D networks using a photosensitive resist that enables

the reproduction of any network pattern made from a photograph or generated by computer. The technique, improved by Mckellar and Wardlaw [14], is still in use. It is based on the following succession of operations (see also [15]):

- A piece of mirror is placed in a hot NaOH solution to remove the backing protection.
- A photoresist coating (photosensitive polymer) is applied to the copper backing.
- A film of the pattern is applied to the photoresist and exposed to UV light to polymerize the resist. The channels are in black and are not exposed to UV.
- The image is developed, and unexposed photoresist is removed.
- Exposed copper is removed by a nitric acid solution (channels).
- The exposed portion of the glass is then etched by hydrofluoric acid.
- The remaining copper and photoresist are removed.
- A cover is fused on the top of the etched plate or two glass-etched plates of mirror images are sintered inside a programmable muffle furnace [16] .

The etching leads to a lenticular pore shape with a typical depth in the range 50-100 μm and the distance between channels of the order of 1 mm. The glass plates can be made oil-wet by chemical treatment, like silane.

Payatakes group developed two-layer glass micromodels [16]. A thin glass plate drilled with holes (~ 0.7 mm) was inserted between the glass-etched plates and the system was sintered. It seems that the non-planarity has small qualitative effect on the displacement mechanisms but significant quantitative effects on trapped saturations and relative permeabilities [16].

Silicon Wafer Micromodels

Micromodels with very fine structure, down to 1 μm have been successfully prepared in Stanford University (Hornbrook [17]) using a silicon wafer instead of glass. With this technique, the pore size in the micromodels is comparable to the pores in a real rock. The etching process oxidizes the silicon, producing a surface uniformly wetted, but not strongly water-wet. The cross-section is rectangular, almost perfect, that allows easier calculation of radius of curvature and flow properties. The cover is attached by anodic bonding.

Resin micromodels

This technique was developed in Institute of Fluid Mechanics, Toulouse and resulted from studies in microfluidics [18]. The principle of the technique is the following:

- A negative film is made from a computer generated pattern.
- A photosensitive plastic plate used for making printing plates is illuminated by UV light through the negative. The plastic under the transparent parts of the negative polymerizes and become hard.
- The soft part of the plate is washed out by a chemical solution.
- Since the plate opaque, a transparent replica made of polyester resin is cast in a rubber mold of the plate.
- The channels are filled with paraffin wax in vacuum and the surface is carefully cleaned.

- A thick layer of polyester resin is cast on the polyester plate to make the cover.
- The paraffin is removed by melting and rinsing with toluene.

One advantage of this technique is the well-controlled shape of the channels allowing the direct comparison of experiments with computer simulations: a rectangular cross section with a constant depth of 1mm and a minimum width of 0.2 mm. The main drawback of resin is the wettability hysteresis when oil and water are used together. Dawe and Wright [19] obtained a smaller channel depth of 50 μm by using another kind of photosensitive plate. Oxaal [20] used directly the etched polymer without replica.

PMMA micromodels

A new method of fabrication of pore network micromodels, using an excimer laser LIGA technique, was developed by Foundation for Research and Technology Hellas [21]. First, the microstructure was etched on a thin PMMA layer by using as input data the pore depth distribution (10–25 μm) and the pore width-to-depth aspect ratio distribution (~ 1 –4). Then, the void space was filled with a layer of nickel (total thickness $\sim 300\mu\text{m}$) which, in turn, was covered by a thick layer of copper ($\sim 1700 \mu\text{m}$) by using micro-electroforming. Finally, and after a series of mechanical treatments, a metal insert, which is a negative replica of the target microstructure, was produced and used for the printing of a large number of identical structures on PMMA plates with hot embossing. Each plastic model is glued with a thin PMMA cover foil by using a spin coating technique. In this manner, almost identical pore network models of well-controlled pore dimensions were produced and tested for two-phase flow processes [21].

DISPLACEMENT MECHANISMS

Invasion of a non-wetting fluid

For strong wettability, the displacement of a wetting by a non-wetting fluid is illustrated in Figure 6a: the neck of the pore is invaded when the pressure exceeds the capillary pressure implied by Laplace's law, and the pore body is invaded. Afterwards, other pores may be invaded if the pressure is high enough. At the scale of a large network of pores, this sequential pore-by-pore invasion is known as "invasion percolation" [22], [23] (Figure 7).

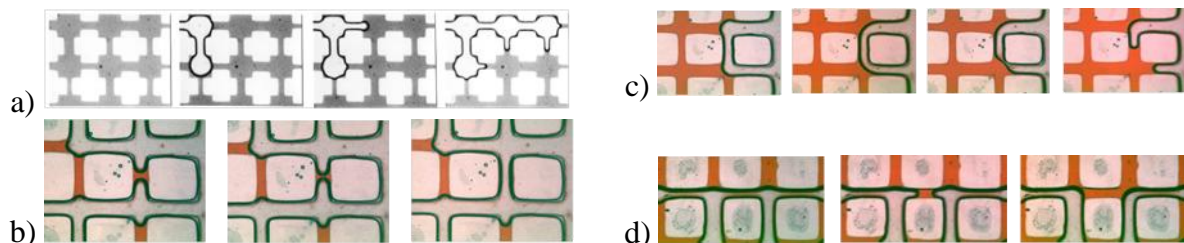


Figure 6 Basic pore-scale mechanisms in a resin micromodel: a) invasion of a non-wetting fluid (white) through the neck of the pore followed by the invasion of the pore body; b) invasion of a non-wetting fluid (white) showing the "escape" of the wetting fluid (red) by film in the corner of the pores; c) invasion of the wetting fluid (red) and collapse of the non-wetting fluid in a pore; d) similar as c) but collapse in a channel.

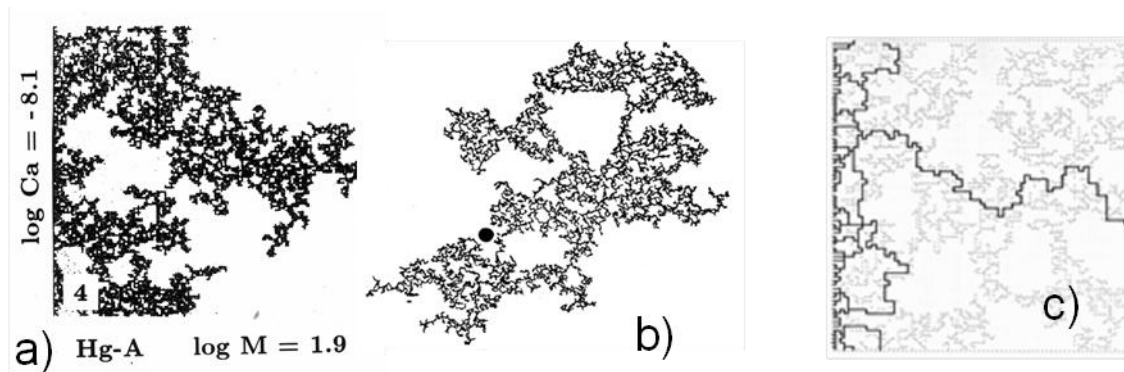


Figure 7 Invasion percolation: a) mercury displacing air in a linear resin micromodel; b) injection of mercury in a radial resin micromodel; c) numerical simulation of invasion percolation.

Invasion of a wetting fluid

The pore-scale mechanisms of imbibition are more complicated than those of drainage and micromodels have been proved very helpful in studying them. Several mechanisms have been identified depending of the topology of fluids; for instance collapse in pores (Figure 6c) and collapse in channels (Figure 6d). Micromodels have also shown the important role of flow by film in the corners and roughness of the pores (Figure 6b) Dong [24], Lenormand [25].

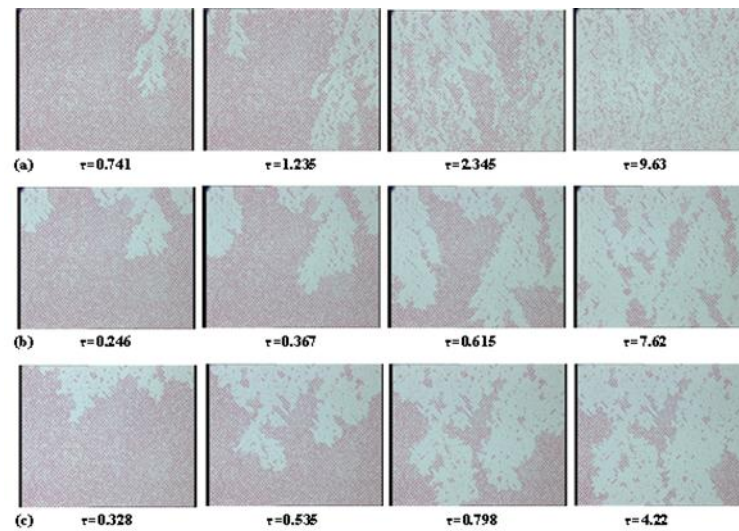


Figure 8 Imbibition growth pattern where paraffin oil colored with oil red (Newtonian non-wetting) is displaced by distilled water at (a) $Ca = 10^{-5}$, (b) $Ca = 10^{-6}$, (c) $Ca = 10^{-7}$.

Large scale patterns can also be described by statistical models depending on the aspect ratio (pore-to-throat-size ratio) [26]:

- Without film flow: compact growth (faceted) for small pore aspect ratio and invasion percolation in the dual network of the pores.

- With film flow: isolated clusters for small aspect ratio and bond percolation in dual network for large aspect ratio.

The effects of capillary number (defined by $Ca = \mu_{inv} u_{inv} / \gamma_{ow}$, where μ_{inv} , u_{inv} are the viscosity and superficial velocity of invading fluid, and γ_{ow} is the interfacial tension) on the displacement pattern (Figure 8) and dynamic capillary pressure and relative permeability curves of micromodels have extensively been studied (Tsakiroglou [27], [28]).

More recent papers from University of Regina have revisited the pore-scale displacement mechanisms for high and low interfacial tension, with an exhaustive literature review on the subject [29], [30].

Viscous fingering

Figure 9 shows three examples of miscible displacement with different viscosity ratios M , defined as the ratio of viscosity of the displaced fluid (yellow) divided by the viscosity of the injected (red) fluid. The fluid is injected at one corner and produced at the opposite corner of a resin micromodel (quarter five spot injection pattern).

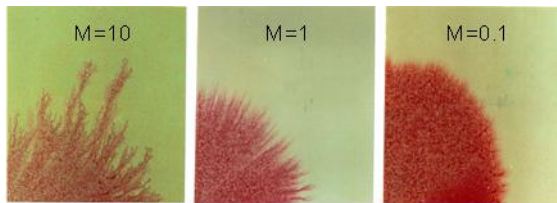


Figure 9 Examples of miscible displacements as function of the viscosity ratio. The case $M=1$ corresponds to dispersion; $M=10$ to viscous fingering; $M=0.1$ to flow stabilized by viscosity

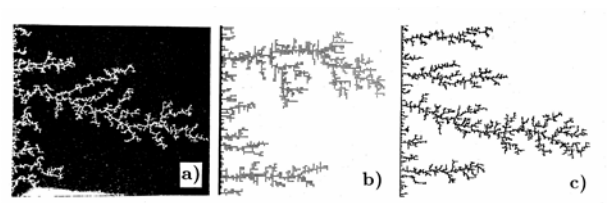


Figure 10 Viscous fingering with immiscible fluid: a) experiment in a resin micromodel, b) network simulation; c) pattern obtained by DLA model.

Both miscible and immiscible displacements lead to viscous fingering when $M \gg 1$. This mechanism has been studied by the Oslo group using micromodels made of a monolayer of glass beads [31]. It has been shown that viscous fingering was similar to a statistical model called Diffusion Limited Aggregation (DLA). Figure 10a shows an example of viscous fingering in a resin micromodel, b) simulation with a network of pores and c) pattern obtained by DLA.

Dispersion fronts

Observation of dyed fluid in a micromodel is mainly used for pedagogical purpose to explain the contributions of convective flow and molecular diffusion (Figure 11). At a larger scale, Feder [32] studied the displacement of two fluids with same viscosity in their monolayer glass bead model. From the position of the interface at different times, they were able to derive the fractal dimension of the dispersion interface (Figure 12).

OTHER STUDIES

Wettability

Mattax and Kyte [12] used their glass micromodel with restored wettability to study the flow by film and large scale fingering. They also measured relative permeabilities and found that the curves were similar to those of real rocks (in 1961!).

In Institut Francais du Petrole studies of wettability by selective silane grafting on the glass surface were performed on glass micromodels [33]. The model permits to impose a heterogeneous wettability by assigning different water/oil contact angles according to the desired wettability pattern. The results were used to improve modeling of wettability mechanisms in network simulator (Figure 13).

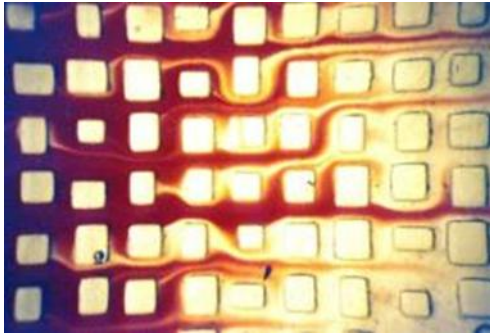


Figure 11 Example of dispersion in a resin micromodel showing the colored liquid following the flow lines (on the right) and the molecular diffusion taking place in the channels on the left.

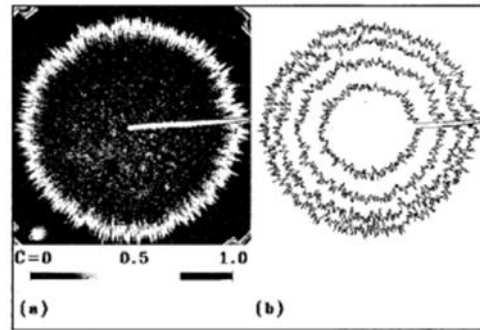


Figure 12. Injection of a colored fluid in a radial micromodel made of a monolayer of glass beads. The position of the front at different times was used to derive the fractal dimension of the diffusion front.

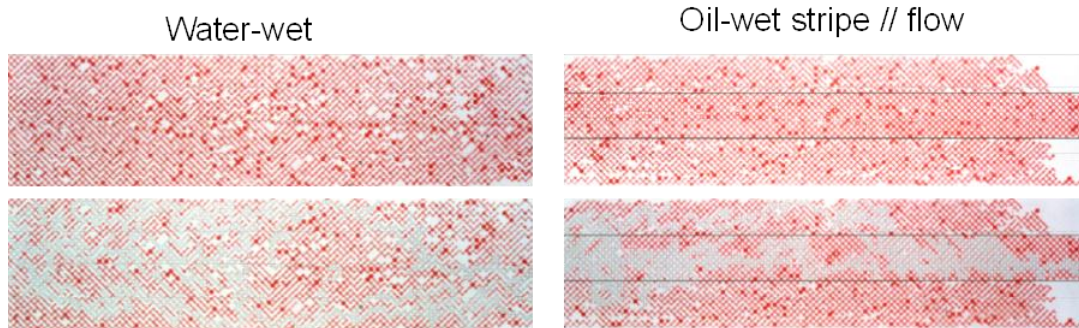


Figure 13 – Comparison of water displacing oil (in red) from Swi to Sor in a water wet micromodel and a micromodel with an oil wet stripe.

In his book, Dullien [34] has outlined the successive fluid menisci shapes within pores under conditions of neutral wettability (Figure 14). However, so far there is no study of the pore level rules for displacements under neutral wettability. This would be a real improvement for pore level simulators since the wettability is mainly neutral (90° contact angle) in the pore invaded by oil in reservoirs. Magghzi [35], have studied the use of nanoparticle to alter wettability in order to improve recovery.

Trapped phase -Blob displacements

Figure 16 presents some examples of residual fluid at the end of a displacement in a micromodel. Payatakes and his co-workers [36],[37] have used glass micromodels to

study the effect of capillary number, viscosity ratio, and wettability on the steady-state two-phase flow regimes and relative permeability curves (Figure 17) and the results were confirmed by pore network simulations [38].

Yortsos [39], Sahloul, [40] studied experimentally the dissolution of a trapped NAPL (non-aqueous phase liquid) in a micromodel.

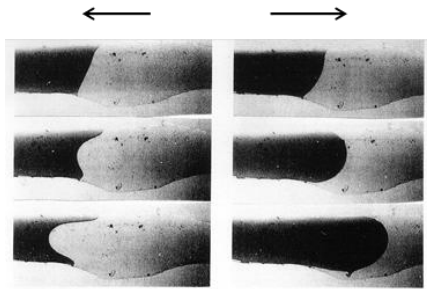


Figure 14 Displacement of a meniscus in a glass micromodel with neutral wettability (from Dullien).

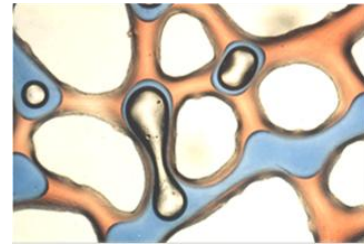


Figure 15 Three phase flow in a pore: water is the wetting fluid in the contact with the solid (yellow), oil in blue and gas in white (R. Dawe, personal communication)

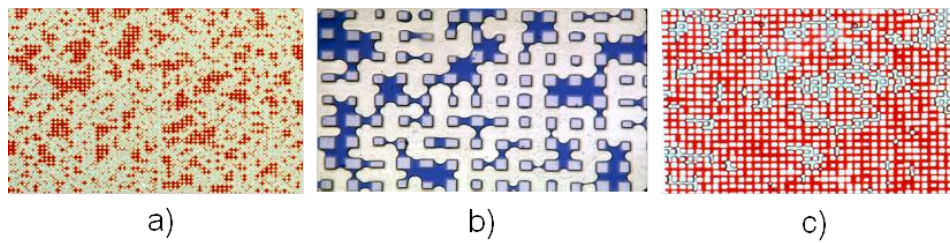


Figure 16 Examples of trapped phase: a) wetting phase trapped in a resin micromodel; b) wetting phase trapped in a glass micromodel; c) non-wetting phase trapped in a resin micromodel.

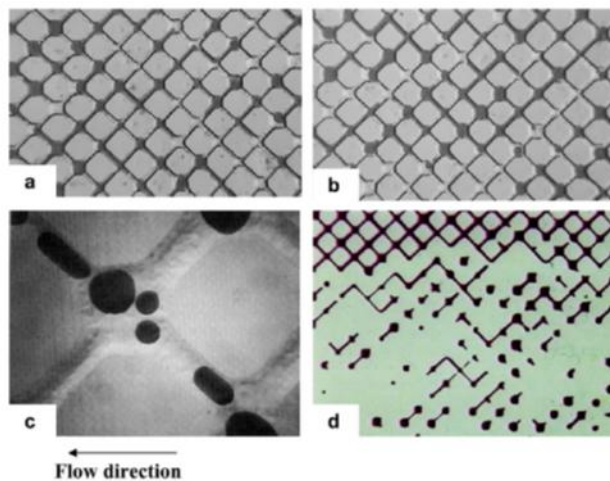


Figure 17 (a) Large ganglion dynamics (LGD) (fluid system 1); (b) small ganglion dynamics (SGD) (fluid system 1); (c) drop traffic flow (DTF) (fluid system 1); (d) coexistence of connected pathway flow (CPF) and small ganglion dynamics (SGD) (fluid system 2)

EOR

University of Waterloo used micromodels to explain the high recovery in gas tertiary recovery. They found that the injected gas was able to reconnect the oil phase and create an oil bank ahead of the gas front [41]. This mechanism is illustrated in Figure 15 where oil spreads between gas and water (R. Dawe, personal communication). Jamaloei, [42] studied polymer flooding for heavy oil, linked to an experimental study of viscous fingering. Glass micromodels were also very useful for surfactant [43], [44], alkaline flooding, [45], [46] and microbial recovery [47].

Critical Flow – Bubble Nucleation

The study of CO₂ nucleation in glass micromodels by El Yousfi [48], [49], have changed the understanding of the nucleation process in porous media. He has shown that the nucleation was a reproducible process and was driven by pre-existing microbubbles, always present in water, rather than thermodynamically fluctuations as usually assumed. This interpretation was confirmed by Yortsos [50].

Non-Newtonian flow

In Foundation for Research and Technology, the two-phase flow studies in glass-etched pore networks were extended to which cases the non-wetting fluid (oil) is a shear-thinning fluid, both the flow pattern and capillary pressure / relative permeability curves were analyzed for drainage and imbibition processes, and the results were interpreted in terms of scaling laws of the gradient percolation theory (Tsakiroglou [51],[52],[53]).

CONCLUSION

The main information gained from pore level micromodel observations is that we cannot forget that a porous medium has a granular and a network structure. Even if the transport properties of porous media are described by continuum laws such as Darcy's law, the physical mechanisms are governed by the pore-scale properties. In addition, large scale observations of micromodel displacements are very useful for the verification of all the models used for network or pore-scale numerical simulations.

Further studies with micromodels can bring a unique insight in complex flow mechanisms, especially in the domain of neutral wettability when contact angle is close to 90°, with an evident impact to digital rock simulations.

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