DESIGN AND FABRICATION OF ROCK-BASED MICROFLUIDICS BY 3D
PRINTING: THE STRUCTURE CHARACTERIZATION AND PORE-SCALE
FLOW EXPERIMENT VALIDATION

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Although great progress has been achieved in numerical simulation on porous flow in rock in decades, experiments performed on Reservoir-on-a-Chip (ROC) have been emphasized as the most sufficient and direct way to investigate the subsurface fluid flow at pore scale. This paper applies the cutting-edge three-dimensional printing (3DP) technique into the fabrication of ROC and the visualized two-phase fluids experiments. The structure of 3D-printed ROC is quantitatively characterized using the surface

scanning analyzer and stereomicroscope, and validated by comparison with the origin digital structure. Then the immiscible (oil-water) two-phase flow experiments are conducted on the 3D-printed ROC and imaged using the high-resolution camera in real-time. The typical fingering phenomenon caused by the heterogeneity of pore-throat is observed, and the effects of surface wettability on the interfacial shape evolution are analyzed. Comparing to traditional fabrication methods (e.g., chemical etching and soft lithography.), 3D-printed ROC is approved to be a novel approach to manufacture the morphology, topology, and connectivity of pore network, and decline the cost and the time-consuming.

Keywords: ROC; 3D printing; waterflooding; wettability; pore-scale flow.

1 INTRODUCTION

Study on the fluid flow mechanism in porous media at the pore-scale is crucial to many engineering applications, such as reservoir exploitation, contaminant hydrology, CO₂ geo-sequestration, and so on (Al-Mukhtar et al., 2012; Blunt et al., 2013; Rathnaweera et al., 2019; Song et al., 2019a, 2019b; Li and Liu, 2020). The complex and disorder internal structure, and the non-transparency of the natural geo-materials (e.g., rock and soil), make it difficult to visualize the interfacial dynamics and the multi-physics coupling of the multi-phase flow under the in-situ conditions (Park et al., 2012; Li and Zhang, 2019). One of the current challenges is capturing the interface behavior between fluid phase and/or fluid (solid) phase accurately, to reveal the micro-scale interfacial dynamics which is controlled by the complex wettability of the solid surfaces and interfacial tensions between the fluids (Ju et al., 2019; Li and Zhang, 2019; Peng et

al., 2021).

Despite great progress in numerical simulation and experimental approaches for porous flow research in decades, the issues on sample heterogeneity and across-scales data are still challenging. Natural rock, for example, suffers a strong heterogeneity sample to sample in laboratory test. Besides, the data (e.g., thin-section, core sample, logging and seismic data) used for model reconstruction and simulation experiences across-scale problems (Lin et al., 2018; 2021). Reservoir-on-a-Chip (ROC) have been emphasized as the most sufficient and direct way to investigate the subsurface fluid flow at pore scale. Many scientific efforts have been devoted to fabricating, improving, and applying ROC to study the micro-scale flow behavior in porous media (Bonnet and Lenormand, 1997; Buchgraber et al., 2012; Zhou 2017; Jahanbakhsh et al., 2020). Chatenever and Calhoun (1952) firstly used glass beads packed micromodel to investigate the fluid behavior directly in porous media. Since then, the glass beads packed micromodel has been the most common micromodel in the early stage (Chuoque et al., 1959), followed by the micro-capillary channel model (Keller et al., 1997), the homogeneous pore network model (Cao et al., 2016), and rock-based pore network model (Liu et al., 2015; Lu et al., 2017).

The common procedure of conventional methods (i.e., the etching and soft lithography) for fabricating micromodel consists with (1) spin coating of photoresist, (2) photolithography with mask, (3) developing, (4) etching or casting Polydimethylsiloxane (PDMS), (5) stripping photoresist or peeling PDMS off the Si wafer, (6) binding to glass slide (Zhang and Austin, 2012). The schematic of the

photolithography process using positive photoresist and negative photoresist are shown in Fig. 1. Once the micromodel is prepared, the micromodel can be processed and modified to obtain a water/oil wet surface with a specific contact angle. The development of micromodels has made tremendous progress, and it has attracted much attention for application in chemistry (Matthew et al., 2004), food processing (Atalay et al., 2011), biotechnology (Sabuncu et al., 2012), medicine (Lee et al., 2020), and energy industry (Gunda et al., 2011; Lei et al., 2019a).

Figure 1. Schematic of developing designed structure onto a substrate using negative and positive photoresist (modified from Anbari et al., 2018).

In petroleum engineering, the ROC contributes much for enhancing oil recovery (EOR), by revealing the micro-scale mechanism and interfacial dynamics of the surfactant-polymer flooding (Yun and Kovscek, 2015), foam flooding (Bonsu et al., 2017, 2018), micro-gel particle flooding (Lei et al., 2019b), and waterflooding (Aadland et al., 2020). With the achievements in visualization of cut-off and viscosity fingering phenomena, the tests on ROC are beneficial to better understanding of the formation, distribution, trapping, and migration of the residual fluids (Wang et al., 2019; Suo et al., 2020). Besides, compared to micro-computer tomography (CT) scanning, micromodel also provides a more direct, higher resolution, and cost-effective way to

visualize structural evolution and flow process, especially when multi-phase/multi-components and multi-physics are coupled, e.g., hydraulic fracturing and methane hydrate decomposition and transport (Huang et al., 2019; Song et al., 2021a, 2021b).

One of the biggest challenges for the conventional ROC techniques lies in that it is difficult to simultaneously fabricate a batch of micromodel with identical structure and stable surface properties (Cao et al., 2019). In addition, the conventional techniques are difficult to accurately manufacture the topology of the complex pore network structure in rock, and the cost is expensive (Berg et al., 2013). Besides, traditional glass, silicon, or PDMS-based ROC are limited in mechanical strength, thermal endurance, sealing, dimensions, and most importantly, operational complexity (Li and Zhang, 2019). Compared to chemical etching and soft lithography, 3D printing provides a new opportunity to fabricate ROC with complex geometry directly from digital data. 3D printing, also known as additive manufacturing, is a cutting-edge technique for rapid prototyping with higher fabrication efficiency and lower cost (Song et al., 2020). Recently, 3D printing has been employed to fabricate ROC with complex structure. Bonsu et al. (2017, 2018) investigated the effects of pore geometry on flow dynamics during the foam flooding using 3D printed models. Ju et al. (2019) investigated the immiscible oil-water displacement both in homogeneous and heterogeneous 3D printed ROC with transparent resin. These 3D printed micromodels reported in literature are limited in model dimensions and low printing resolution. To display the pore-throat connectivity, Li and Zhang (2019) successfully fabricated ROC at a much higher printing resolution (up to 2 μ m), and experimentally investigated the effect of wettability

on the interfacial dynamics via direct visualization of flow process. However, there are only 6 pores in the flow domain of Li's ROC, which is too small to investigate the effect of pore network heterogeneity on fluid flow.

In this study, a novel methodology is presented to fabricate micromodel via 3D printing simultaneously considering the dimensions of the pore network domain and the printing resolution (up to 10 μ m). The designed pore network embedded into the ROC is extracted from a scanning electron microscopic (SEM) image of Berea sandstone. The structure of the 3D-printed ROC is quantitatively characterized using the surface scanning analyzer and stereomicroscope and compared with the digital structure. The immiscible (oil-water) two-phase flow experiments are conducted on original and surface modified printed micromodel, respectively. The flow pattern is directly imaged to study the effect of wettability on fluid flow, particularly flow path, interfacial shape, residual trapping.

2. MATERIAL AND METHODS

2.1 Pattern extraction and digitalization

The porous structure of the micromodel is extracted from a natural Berea sandstone which is collected from the literature (Sirivithayapakron and Keller, 2003; Auset and Keller, 2004). The structure is a real pore structure of rock only operated by morphological processing to obtain a smooth outline, as shown in Fig. 2. The dimensions of the selected region are 640 μ m and 320 μ m in length and width, respectively.

Figure 2. Binary image of the original pore structure

2.2 ROC design

The binary image is firstly transformed into Stereolithography (STL) format file through contour extraction, vector file conversion, and mesh generation. STL file is a universal data format that can be recognized by most 3D printers. The detailed information of mesh generation can refer to our previous study (Song et al., 2017; Liu et al., 2017). Considering the shape of the cylindrical ROC holder used in this study, the extracted pore network is first upscaled 20-fold magnification and embedded onto a cylindrical plate with 12.5mm in radius and 10mm in height. The depth of the porous region, i.e., the flow domain, is designed to be 0.2mm. The schematic of the designed micromodel is shown in Fig. 3. To reach the compromise between the maximum resolution of the 3D printer and the imaging camera, as well as to meet the requirements of the sample holder, the upscaled 20-fold magnification is adopted to manufacture the model using the Arch S140 printer with 10μm optical resolution. Meanwhile, according to our previous study (Song et al., 2021c), the insufficient removal of the support material and the invasion of original pore space caused by matrix material expansion can significantly affect the reproduce of pore-throat channel which can reduce the

connectivity of pore network. The up-scaling printing scheme (20-fold magnification) is expected to decrease the effects of the post processing by 3D printing and reserve the connectivity of the pore-throat channel. In addition, the dimensionless capillary number (Ca) is adopted to determine the flow rate of the injected water, to reduce the effects of the model modification, which can be expressed as (Melrose, 1974).

$$Ca_{micro} = \frac{\mu_w u_w}{\phi \sigma_{ow}} = \frac{\text{viscous force}}{\text{capillary force}} \quad (1)$$

where μ_w and u_w are the viscosity and velocity of the induced water, respectively, σ_{ow} is the interface tension, and ϕ is the porosity of rock.

To realize a stable and uniform flow of the injected water, a buffer zone is designed to bridge Inlet/Outlet and porous section on the right and left side (shown in right Fig.3) of the porous domain.

Figure 3. Schematic of micromodel

2.3 3D printer and material

A high resolution micro-SLS printing system (nano Arch S140 printer with optical resolution up to 10 μ m, BMF Inc., Shenzhen) is adopted to fabricate micromodel in this study. This micro printing system uses advanced Projection Micro Stereolithography (P μ SL) technique. It owes the advantages of ultra-high resolution, accuracy, efficiency, and cross-scale fabrication, as well as a wide range of the photosensitive resins to

choose. The general printing procedure of the P μ SL system consists of model slicing, projection and exposure of single layer pattern, integration, and post-processing. The schematic of the printing system is shown in Fig. 4.

Various types of photosensitive resin can be used for porous model fabrication including hard resin, toughness resin, high temperature resistant resin, biocompatible resin, flexible resin, transparent resin, and hydrogel. A light-yellow transparent resin (HTL) with high-temperature resistant property is used in this study (manufacturer: BMF Inc., Shenzhen). Detailed properties of the resin are listed in Table 1.

Figure 4. Schematic of the P μ SL printing system

Table 1. Properties of the resin

Indicator	Test value	Test standard
Viscosity	80CPs@25°C	GB/T 5561-1994
Tensile strength	79.3MPa	ASTM D638
Modulus	4.2GPa	ASTM D638
Elongation	2.23%	ASTM D638
Bending strength	120.6MPa	ASTM D790
Bending modulus	3.96GPa	ASTM D790
Glass transition temperature	168°C	DSC
Impact strength	30J/m	ASTM D256-10

Distortion temperature@ (0.45MPa)	142°C	GB 1634-79
Hardness	90 Shore D	ASTM D2240

2.4 Experimental setup

Two WHB-II micro-flow pumps (minimum injection rate 0.001ml/min) are used to control displacing and defending fluids respectively. A high-resolution camera probe is used to continuously capture the displacement process. In addition, the experiment setup consists of some other auxiliary devices including the intermediate vessel, external light source, and confining pressure system (nitrogen), as shown in Fig. 5(a). The schematic of pipeline connection in the flow cell is illustrated in Fig. 5(b). The printed ROC is sealed in a rubber set prior to embed in model holder and is highlighted in Fig. 5(c). The entire flow chamber is embedded in a constant temperature oven, in which the model can be heated up to 150°C to simulate the formation temperature. The model holder is sealed in a stainless-steel chamber, in which the model can be pressured to simulate the formation pressure (up to 70MPa).

Figure 5. Device and schematic diagram of pore-scale flow experiment

The fluids used in this study are simulated formation water (displacing phase) and kerosene (defending phase). Since the ROC is light-yellow, and the light source is white, to enhance the contrast, the formation water and kerosene are dyed as blue and red, respectively. The experiment fluids and the fluids properties are shown in Fig. 6 and Table 2, respectively.

Figure 6. Experiment fluids (left: kerosene; right: formation water)

Table 2. Properties of fluids

Fluids	Viscosity (mPa·s@25°C)	Density (g/cm ³)	Interfacial tension (mN/m)
Formation water	1.16	1.2	24
Kerosene	2.5	0.8	

The seal test of the flow chamber is conducted before the water injection. Then, the experimental setup is flushed by the ready-for injection fluids to vent residual air in the pipeline. The main experimental procedures consist of the following four steps:

(1) Applying confining pressure and vacuum the model, and injecting the water with a rate of 0.005ml/min to saturate the micromodel.

(2) Changing the pump and inject the oil with a rate of 0.002ml/min to displace water until there is no water at the outlet.

(3) Changing the pump, and start the waterflooding process with an injection rate of 0.001ml/min; capture the whole experiment process via video probe; when the

oil/water distribution state is stable, and then increase the injection rate to 0.05ml/min (lasts 10min), and then 0.5ml/min lasts 5min.

(4) Extracting the oil/water distribution image frame by frame, calculating parameters including saturation, recovery efficiency.

3. RESULTS AND DISCUSSION

3.1 Structure characterization

To evaluate the accuracy of the 3D printed ROC, a stereomicroscope is used to capture the detail of the printed model and compared with the digital image. The comparison between the digital structure and printed model is shown in Fig. 7. Four typical areas located at image corner are highlighted. The results indicate that the printed model perfectly reproduces the contour features of micro-channel and grain particles in the digital image.

Figure 7. A detailed comparison between printed model and digital structure

The surface scanning analyzer is used to measure the printed model quantitatively with the original digital image. The scanning results and the labeled image are shown in Fig. 8. Seven test points are selected and measured (6 pore channels and 1 grain particle), and the locations are labeled in Fig. 8(a). Fig. 8(b) and 8(c) are the

corresponding printed model and the scanning results. The measured and digital calculation results are listed in Table 3. The test results indicate that the error of all test points is less than 10%. Considering the size of the model, the error is within the acceptable range. The flatness measurement of the model is shown in Fig. 8(d). And the results indicate that the maximum difference of the depth on the tested area is less than $5\mu\text{m}$, which means the model surface is smooth enough for fluid flow.

Figure 8. Scanning results of the printed model

Table 3. Comparison of feature size between printed model and digital image

Test point	Digital structure (μm)	Printed model (μm)	Error (%)
1	181.4	196	8%
2	387.4	426	9%
3	118.5	126	6%
4	201.2	206	2%
5	189.2	186	1.6%
6	144.8	156	7.8%
7	575.4	526	8.6%

3.2 Effect of ROC wettability on oil/water distribution

Since the photosensitive resin adopted in this study has strong oil-wet property, the surface wettability of the printed model is modified by silane coating to evaluate the effect of wettability on fluid flow. According to the manufacturer, the silane coupling agents used for surface treatment consist of 2-[methoxy(polyethyleneoxy)6-

9propyl]trimethoxysilane (MPEOS) and n-octyltriethoxysilane (OTS). The silane solutions are prepared following a standard procedure [Hodder and Nychka, 2018]. The 3D-printed micromodel is placed in a cup full with silane solution, and the cup is placed into the vacuum container. And then vacuum-pumps the container manually until the bubbles occur in the cup. After about 2min, the container is vented, and then puts the silanized micromodel into an oven at 110 °C to post-processing for 10min. The wettability is characterized by contact angle (θ), and is measured by sessile drop method at the sample surface of printed ROC, which indicates that the micromodel transformed from strong oil wetted to weak oil wetted after silane treatment (contact angle of kerosene increased from 15.84° to 72.19°). The test results are shown in Fig. 9. The test is conducted at room temperature, and the test fluid is kerosene.

Figure 9. Contact angle measurement of (a) original and (b) modified ROC

The typical images of oil-water distribution at different stages are shown in Fig. 10. Fig. 10(a) is the original printed ROC with strong oleophilic ($\theta=15.84^\circ$), and Fig. 10(b) is the modified with weak oleophilic ($\theta=72.19^\circ$).

Based on the oil/water distribution images, the parameters of irreducible water saturation, residual oil saturation, and recovery can be obtained by calculating the oil/water area through multiphase segmentation operation, as labeled in Fig. 10.

Figure 10. Oil/water distribution at different displacement state.

Using the open-source ImageJ, the specific targets (oil phase) can be manually delineated. It can help to quantify different types of residual fluids, especially in throats and dead-end pores. An operational example is shown in Fig.11.

Figure 11. Saturation calculation based on target delineation

To quantify the effect of wettability on flooding process, the relationship between oil saturation and normalized displacement time (T^*) is shown in Fig. 12. T^* is defined as the ratio of absolute displacement time and the breakthrough time (T_b). Note that the zero point ($T^*=0$) is selected to correspond to the time when the injected fluid's front first arrived at the porous area.

Figure 12. Change of saturation during displacement

The oil/water distribution in Fig. 10 indicates that due to the heterogeneity of the pore-throat structure, the original model with strong oleophilic shows obvious fingering phenomenon in the early stage of displacement, i.e., the injected water advances rapidly along the central connected macropore. The upper and lower part of the model, as well as the corners cannot be swept by the injected water. There are many residual oil blocks remaining in the model after breakthrough. Compared to the original model, the displacement performance in modified model is much better. Although the fingering still occurs in the early stage, the interface shows a relatively stable advanced front, and there are only few oil blocks trapped in throats and dead-end pores after breakthrough.

According to Fig. 12, the modified model not only has a higher recovery, but also a higher displacement efficiency than original model.

3.3 Analysis on the dynamic displacement

According to previous study (Li and Zhang, 2019), in the immiscible two-phase flow, there are two main flow patterns of the interface, i.e., convex shape and concave shape in the pores with different wettability. The convex shape interface occurs in non-wetting fluids invading the wetting fluid. In the contrary case, the interface is concave shape. During wetting fluid invasion, the fluid wets the wall surface firstly, and the capillary force is served as the driving force. The relationship of interfacial shape and wettability in a single capillary tube can be illustrated in Fig. 13, in which the total displacing force refers to F .

Figure 13. Relationship of wettability and fluid front in a capillary tube

The strong oil wetted model is selected to analyze the evolution of interfacial morphology and identify the preferential flow path. According to Young-Laplace equation, the capillary pressure is greater where the pore-throat size is smaller. Hence,

during the non-wetting fluid invasion, the invasion fluid will occupy the larger pores firstly since the capillary force acts as the resistance. Due to the fractal characteristics of pore structure, the initial stage of flooding is selected for analysis. The domain close to the inlet of the ROC is selected to analyze the preferential flow path, as shown in Fig. 14. To describe the interfacial shape and flow path during the water flooding, the pores and throats are numbered with letter and Arabic numerals respectively, and the red arrow represents the flooding direction.

Figure 14. Selected domain for analysis

Fig. 15 is a collection of screenshots on the fluid's distribution of the selected domain for analysis at different time. The evolution of the interfacial shape is depicted by dashed line. The fluid invasion process can be described as: (a) the displacing front firstly arrives at pore A through channel 3, and the interface shows typical convex shape (non-wetting fluid invasion), and the front stops at channel 4/5/6; (b) the displacing

front continue advances into the upper corner of the buffer zone and stops at channel 1/2/4/5/6; (c) the fluid enters pore B through channel 6 (the size of channel 6 is larger than channel 1/2/4/5) since the resistance is the smallest at channel 6; (d) the fluid enters pore C through channel 7 and stops at pore C; (e)-(f) the fluid starts to occupy pore D through channel 4 and stops at pore D; (g) the fluid enters pore E through channel 1, and then occupy pore F through channel 17 (h). As described above, the preferential flow path can be identified during non-wetting fluid invasion, i.e., the first flow path is: 3-A-6-B-7-C; the second flow path is: 3-A-4-D, followed by: 1-E-17-F.

In Fig. 15(g) and (h), it can be noted that oil is trapped in channel 10 and channel 11, highlighted in pink circle. This is caused by the unstable advance of interface dominated by capillary force, which is one of the causes of residual oil.

Figure 15. Screenshots of oil/water distribution at different flooding stage

4. CONCLUSIONS

This paper presents a novel method to fabricate the transparent ROC with designed, surface property controllable, and heterogeneous internal structure by 3D printing technology. The SEM image of natural Berea sandstone is adopted as the input for the designing and 3D printing. The ROC is printed with an ultra-high resolution up to 10 μ m. The printing accuracy of the ROC is quantitatively characterized and compared with the origin digital data. The wettability of the ROC is modified as strong and weak oil-wetted. The visualized immiscible oil/water flow experiment is conducted on the origin model and the model after the modification of the wettability.

The typical fingering phenomenon caused by the heterogeneity of pore-throat is observed, and the effects of surface wettability on the interfacial shape evolution are analyzed. Both the heterogeneity of pore-throat and surface wettability have significant effect on interfacial dynamics and residual fluids distribution, for which can cause unstable advance of fluids interface during displacement.

Comparing to traditional fabrication methods (e.g., chemical etching and soft lithography.), 3D-printed ROC is approved to be a novel approach to manufacture the morphology, topology, and connectivity of pore network, and decline the cost and the time-consuming.

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CONFLICTS OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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