A noble method for rapid prototyping of porous micromodels applicable to enhanced oil recovery

N Haque¹, A Singh², U K Saha³

¹Department of Mechanical Engineering, Indian Institute of Technology Guwahati, India
²Department of Chemical Engineering, Indian Institute of Technology Guwahati, India
³Department of Mechanical Engineering, Indian Institute of Technology Guwahati, India

E-mail: saha@iitg.ac.in/anugrah@iitg.ac.in

Abstract. In this paper, a noble method for rapid prototyping of porous micromodel in Polydimethylsiloxane (PDMS) with good control over the pore geometry is described. An attempt has been made to demonstrate the making of PDMS micromodels using round hole perforated metal sheet as a master. The geometric shape of the micromodel in PDMS is the negative of the perforated sheet, which gives a section of cylindrical pillars separated by a uniform gap. After peeling off the PDMS from the porous plate, it is sealed with a glass plate to prepare the microfluidic porous channel. This protocol requires only materials that are commercially available, inexpensive and less time consuming. The size of the pores can be adjusted by selecting the perforated sheets having different hole size and thus micromodels with a variety of pore size distribution can be generated using this simple method. The optical visualization experiments were performed to test these fabricated micromodels for their applicability and reliability. Furthermore, Particle Image Velocimetry (PIV) technique is used to get the velocity distribution along the porous section of the micromodel. Identical flow patterns were seen at the different section of the micromodel which again indicated the reliability of these homogeneous micromodel fabricated by using this noble method. In addition, the velocity profiles are obtained near the throat region of the micromodel at three different flow rates.

1. Introduction
Micromodels are useful tool to study the fluid flow behavior in porous media at micron scale that is relevant to the petroleum recovery. During the last 30 years, micromodels have found to be the most precious tool, which allow the observation of fluid flow and transport at the micron scale in many processes related to chemical, biological, and physical fields of engineering. A micromodel is regarded as an artificial portrayal of a porous medium comprised of a network of connected pores made of a transparent material [1]. The early micromodels were very simple in geometry. One of the first micromodels fabricated and used to explore the behavior of fluid flow at microscale in porous media, was a single layer of glass spheres sandwiched between two flat glass plates [2]. Since 1980s, it has turned out to be conceivable to fabricate micromodels having complex geometries, and these are computer produced, based on statistical distribution and fractural designs [3]. So far, the most
convenient method to fabricate microchannel is photolithography technique [4]. In this method, a mask containing desired pore network of the microchannel is made digitally. Thereafter, the mask is projected to the silicon or glass substrate which has been already spin-coated with photoresist. It is then exposed to ultraviolet light whereby the wet or dry etching technique is used to generate the microchannel. Finally, the other opening side of the micro-channels is covered with a transparent material via bonding. In recent times, the use of thermosetting plastic as a substrate for the fabrication of micromodel has been observed due to its incredible chemical, optical, and mechanical properties. Hsu et al. [5] selected a rigid thermoplastic material cyclic olefin copolymer (COC) as the substrate and fabricated fractured micromodel via hot embossing and performed two-phase flow experiments. The surface micro-machining also has been evolved to produce microchannels with various perspective proportions and shapes in MEMS. Moreover, complicated techniques like stereo lithography [6], laser-chemical three-dimensional writing [7] and micro-joining [8] have additionally been developed to form complex microchannels on surfaces of different materials. Furthermore, three-dimensional microchannel in elastomer was fabricated using a module assembly strategy where 3-D layer by layer, microchannel configuration was formed with the help of photolithography technique [9].

Soft lithography is a method, which is used to replicate the micro patterns on the surface of soft materials like elastomeric stamps using moulds and photo-masks [10]. Among elastomeric stamps PDMS (Sylgard 184, Dow Corning) is regularly used to create microchannel through a procedure known as micro contact printing. Many authors have utilized PDMS micro-moulding methods to fabricate micro scale fluidic devices [11]. PDMS is a very low-cost material compared to the other substrates like silicon or glass that are used in traditional micro-fabrication methods. In addition, micro-moulding method is straightforward and quick compared to the conventional etching and bonding approaches. Due to the ease of bonding and excellent optical properties, PDMS is the most suitable material for micro fluidic devices. However, the fabrication of PDMS micromodels by soft lithography depends on a master with micrometric features that need to be made in a clean room using photolithography and microfabrication methods. In addition, the photolithography technique is comparatively expensive, time-consuming and beyond the reach of many researchers. In order to overcome the shortage of micromodel and to fabricate low cost micromodel, new advances or enhanced strategies have been explored gradually to diminish the high expenses that come from using conventional photolithography technique.

A simple method to fabricate microchannel with basic geometry would be to embed the microwire into the PDMS and to remove the microwire from the PDMS upon being cured. Of late, these techniques are extensively used to fabricate the microchannels because of their easier implementation and cost effectiveness. Ghatak et al. [12] utilized embedded nylon threads as a template to fabricate straight as well as a mesh of channels inside PDMS block. Channels having complex orientations like helices, knots, super-helices, and of different cross-sections can be produced using this low cost method and this method is also used to design microfluidic devices. A recent development brought by Shrirao et al. [13] was the use of scotch tape as a master to fabricate a PDMS microfluidic device. In this method, the scotch tape did not remain as a part of the microfluidic device after fabrication and the PDMS replica is peeled off and bonded to the transparent substrate.

In the present work, a noble method to fabricate homogeneous porous micromodel has been developed. Furthermore, an attempt has been made to reveal the technical practicability of this strategy, typically by using round hole perforated metal sheets as template to fabricate the micromodels. Thereafter, the fabricated micromodels are tested for fluid flow experiments. In addition, micro PIV analysis of fluid flow is performed to map the velocity field and to find the applicability of these micromodels thus fabricated.

2. Materials and Methods

The main materials used in this fabrication method includes polydimethylsiloxane (Sylgard 184, Dow Corning), commercial round hole perforated metal sheets of identical sized holes, silicone connecting
tubes, and silicone adhesive sealant. Microscope glass slides coated with thin layer of cured PDMS is used to cover the micromodel. Round hole perforated metal sheet is used in this method. These metal plates serve as master during the fabrication of porous micromodels. Figure 1 shows the image of the porous plate.

2.1. Fabrication of micromodel
Polydimethylsiloxane (PDMS) is widely used for the fabrication of microfluidic systems because it can be moulded into any desired shape. It is transparent, permits visualization and can be sealed easily with substrates like glass. The materials required to fabricate PDMS micromodels are silicon elastomer base (Sylgard 184 from Dow Corning) and the corresponding elastomer curing operator.

![Figure 1. Image of the porous plate having circular holes.](image1)

The silicon elastomer base, which is in fluid state, is blended with the curing operator at a mass proportion of 10:1. The two segments are blended altogether by mixing them in a container for couple of minutes. The amount of PDMS that is utilized decides the thickness of the slab made. In the present work, typical mass of the elastomer and curing agent were 40 g and 4 g, respectively. The subsequent amount of PDMS should ensure that the PDMS slab is sufficiently thick to make a rigid micromodel. In addition, it should be sufficiently thin enough so that it can be punched for making inlet and outlet holes of the micromodel. In present case, screws are placed vertically at the two ends of the perforated plate before pouring the liquid PDMS to make inlet and outlet holes of the micromodel. During the mixing processes some air bubbles are trapped. To remove these bubbles, the cup with the mixture is kept in a vacuum chamber. In the wake of degassing, the stuff of the cup is poured over the mesh pattern in a Petri dish or in a mould prepared by double sided tape. Karadimitriou et al. [14] described a similar method of pouring process in their work on the fabrication of the micromodel. The material must be poured as gradually as possible keeping in mind that there should not be any trapped air pockets. Additionally, the PDMS should not be poured directly on the top of the network pattern, so that the liquid flows slowly into and over the network pattern. Else, there would have been a risk of dust particles being caught in the structure of the network, bringing about a defective micromodel. Then the Petri dish is kept in an oven at 80°C for no less than 2 hours with a specific end goal to set the liquid PDMS.

![Figure 2. (a) Schematic description of steps for fabricating the micromodel, (b) enlarged view of the cylindrical pillars of the micromodel.](image2)
In general, various combinations of temperature and curing time can be employed. The two extreme combinations are 10 min at 200°C and 48 hours in ambient temperature. However, longer curing times and less curing temperature are suitable to reduce stresses within the material. In addition, curing it at ambient temperature is not a suitable choice as it requires more time to solidify and hence the material might be in contact with dust particles before it sets. PDMS slab is permitted to reach ambient temperature before bonding to avoid permanent distortion that can occur due to excessive stress. Afterwards PDMS is cut around the edges and tenderly removed from the Petri dish with the assistance of a sharp pointed blade. The PDMS slab should be separated from the pattern very carefully and slowly so that it retains the complete pore network features. To seal the micromodel, a glass slide is used and it gives a firm base. Before sealing the glass slide with the PDMS slab, a thin layer of PDMS is coated above the glass surface to keep uniform wettability in the micromodel. Then the PDMS slab bonded with the glass slide are kept again in an oven for another hour at 80°C. Whereby silicone adhesive glue is used to seal the channel. Figure 2 shows the steps for fabrication of PDMS micromodels.

3. Experimental Setup
The flow visualization experiments are conducted using the prepared 2D porous micromodel. Fig. 3a shows the photograph of the experimental facility. The PDMS micromodel comprises of an inlet, an outlet and a 2D porous section (length, \( L = 40 \) mm; width, \( W = 20 \) mm; and depth, \( H = 450 \) μm). The porous section of the micromodel comprises of uniform-sized cylindrical shaped pillars (diameter, \( D = 788 \) μm, and height, \( H = 450 \) μm) organized in a zigzag order is shown in Fig. 3b. The maximum distance (pore size, \( s \)) and the minimum distance between the pillars (throat, \( t \)) are 1209 μm and 343 μm respectively yielding a porosity of 0.62. A glass slide is bonded to cover the open side of the micromodel and it is then sealed using silicone adhesive sealant. The micromodel is placed on the microscope stage and a syringe pump (NE 1000 single syringe pump, New Era Pumps, USA) is connected to a 50 mL syringe containing glycerol-water mixture (75%+25% by weight). The micromodel is then placed under a Leica DM IL LED inverted microscope to investigate the flow field. A 10x magnification lens (numerical aperture = 0.25) is used for imaging and an Nd:YAG double-pulsed (532 nm) laser provides the illumination. A double-shutter PIV CCD camera (FlowSense EO 4M camera, 2048×2048 pixels at 16 fps) is used to acquire the sequence of images. Entire experiments are performed at normal room temperature and pressure. Single-phase flow experiments through the micromodel are studied at three different flow rates viz. 10mL/hr, 20mL/hr and 30 mL/hr at two different sections of the micromodel (Reynolds number, Re < 1). The fluid is seeded with PMMA (Polymethyl methacrylate) particles (1-20 μm diameter) coated with Fluorescent dye Rhodamine B. These particles have density around 1.19 g/cm³ that is close to glycerol-water mixture, which minimizes sedimentation. The seeding concentration is set at roughly 0.05% by volume.

Figure 3. Details of (a) Experimental setup, (b) image of the micromodel and (c) a part of the homogeneous porous pattern with white solid pillars and dark pore space.
4. Results and Discussion

4.1. Micro-PIV measurements

During the last decade, the technique of PIV has found increasing applications as a non-intrusive diagnostic tool for the study of complex flow fields [15]. In hydrodynamics, micro-PIV technique is broadly used to map the velocity field of the flow as it can provide accurate velocity vector of flowing fluid quantitatively over multiple points simultaneously [16]. The working principal of a micro-PIV system is very simple. The working fluid is seeded with neutrally buoyant tracer particles. These tracer particles are illuminated by a light source, and the images of these tracer particles at a certain interval of time are recorded. Images are subdivided into many small areas, which are called interrogation windows, and the displacement of the particle images is then estimated statistically by correlating the particle image pairs. Thus, the fluid velocity is calculated by dividing the particle displacements by the time interval between consecutive image pairs.

During the experiments, a series of images containing tracer particles have been recorded. The sample raw images at different section of the micromodel are shown in Fig. 4. These images can not be used directly for PIV measurements due to the back-ground noise and low illumination. Therefore, to identify the grains and to remove the background noise image pre-treatment is required. After pre-processing, the images contain only bright particles that holds information regarding particle displacement. The cross-correlation of the acquired images have been performed using the PIVlab software [17]. Image pre-processing is performed using this tool to enhance the quality of the images that improves the measurement accuracy. For the cross-correlation of paired images, a fast Fourier transform correlation with several passes is utilized. The first pass uses relatively bigger interrogation window to calculate the particle displacement. Better signal to noise ratio can be obtained from large interrogation windows. However, it leads to poor vector resolution. Hence, to increase the resolution smaller interrogation windows are used in the subsequent iterations. This kind of iterative procedure provides significantly larger vector resolutions, a large dynamic velocity range and a high signal-to-noise ratio [18]. In present case, two passes are used starting with a bigger interrogation window and reducing this size by 50% for the following pass. To perform PIV analysis, smallest (last) interrogation window should contain at least 5-10 particles within the area. Thus, the area of the interrogation window is made small enough so that the displacement of the particle within the area becomes uniform. On the other hand, the interrogation window should be made large enough so as to contain sufficient information for the calculation [19]. Finally, an area of interrogation windows of 75% are overlapped and the post processing is performed to get the velocity vector field [20].

![Figure 4. Image of the fluorescent particles at different section of the micromodel.](image-url)
4.2 Velocity field
After the preprocessing of raw images, the enhanced images are analyzed to get the velocity fields at various section of the micromodel. At each section of the micromodel, 50 images are taken and their averaged values are used to obtain both velocity vector field and velocity profile. Identical flow patterns are seen at the different sections of the micromodel. Figure 5 shows the velocity fields at two sections of the micromodel. The velocity contour is almost similar at all the three flow rates. As it can be seen, the velocity vector magnitude is nearly zero near the solid boundaries and becomes maximum at the centerline. Figure 6 shows the flow velocity profile at the two sections of the micromodel at different flow rates. It is found that with the increase in flow rates velocity of the fluid increases.

It is also to be noted that the velocity distribution at the throat of the pores obtained by micro PIV analysis shows deviation from the parabolic nature. In spite of the fact that it looks straightforward yet this task must be performed precisely. In addition, the time gap between two consecutive images has to be taken judiciously to allow sufficient displacement of the tracer particles between consecutive image frames with considerable resolution. In the present case, the time interval between two consecutive images is varied from 8-10 ms with respect to the given flow rates. The tracer particles and the optical system must be selected very carefully in micro-PIV experiments to get actual velocity of the flow. The objective and camera properties should be chosen in such a way that the image of the particles are resolved over at least 2–3 pixels [21]. In our case, the diameter of the particle image size is 4–9 pixels, which satisfies the required condition. The time interval between images is chosen in such a way that the particles should not move more than 3 particles diameter from one frame to the other. The process is repeated at several locations at the field of view to get the instantaneous velocity vector field at different porous sections of the micromodel.

![Figure 5. Velocity vector map of the micromodel at locations corresponding to the images shown in Figure 4.](image)

![Figure 6. Velocity profile along the red line in as shown in Figure 4.](image)
5. Conclusion
In the present work, a straightforward and highly economical method has been developed to construct porous PDMS micromodels. Through a series of steps, it has been demonstrated that the round hole perforated metal sheets can be used as a master for the rapid construction of porous micromodels. This method usually takes only 5-6 hours. In addition, this method is cost-effective, flexible and convenient as compared to traditional techniques like photolithography or soft lithography.

With the help of this technique, it is also possible to construct porous micromodel having different pore geometry and pore sizes very easily. This method will increase the adaptability of fabrication and utilization of microfluidic devices. The visualization setup that provides images of the distribution of fluid seeded with tracer particles at different porous sections of the micromodel has also been shown. Furthermore, the micro-PIV analysis of the particle flow in these micromodels have been performed using PIV lab (MATLAB tool) to get the velocity distribution. The analyzed velocity profiles indicate the reliability and applicability of the fabricated micromodel. Therefore, this type of micromodel can be used in an efficient way to investigate the fluid flow at pore scale, which is crucial to the enhanced oil recovery (EOR) applications.

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