

# Manufacturing of artificial core based on transparent soil technology: A preliminary experimental study on various grains and pore fluids

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Artificial cores are more representative than natural cores on heterogeneity in laboratory core analysis. This paper presents a new method to prepare transparent cores based on transparent soil. A preliminary experiment is carried out to determine the optimal combination of different grains and pore fluids. The basic parameters of mineral oil used in this paper are tested including the refractive index RI value, density, and dynamic viscosity. The relations between RI value, pH value, viscosity, density and concentration of calcium bromide solution are obtained as well. The results indicate that the RI and density of calcium bromide solution increase with the increase in concentration, whereas pH and viscosity decrease with the concentration. The effect of temperature on RI value increases with the increase in calcium bromide solution concentration. When the particle size of silica gel ranges from 49.7 to 74.5  $\mu\text{m}$  and the concentration of calcium bromide solution reaches 62%, the prepared mixed system will achieve the highest transparency.

Keywords: Artificial core; Transparent soil; Fluid flow; Geotechnical engineering; Petroleum

## INTRODUCTION

Core analysis plays an important role both in the petroleum industry and the geotechnical engineering field [1]. During laboratory testing on natural cores, the limitations lay on the weak representativity, strong heterogeneity, and low efficiency of drilling of cores, which greatly restrict the development of laboratory tests [2, 3].

What's more, the opacity of natural and artificial samples makes it impossible to directly capture the internal processes in the sample. Computerized tomography (CT) technology [4, 5] and microfluidics model [6, 7] have been used to capture the inner rock structure and flow process. However, the view size of these pore scale tests is too small when compared to mesoscale experiment field application, compared to mesoscale experiments operated on the core samples.

Laboratory experiment conducted on physical models manufactured from transparent soil has been regarded as a new tool for visual technique in geotechnical engineering. The earliest study on transparent soils can be traced back to 1982. Allersma studied the stress-strain relationship under the single-shear condition with a model made of broken glass [8]. Mannheimer studied the non-Newtonian fluid flow by using a transparent slurry [9]. After that, researchers have conducted fruitful work on preparation, engineering properties and applications of transparent soil [10, 11].

Previous studies on the physical and mechanical properties of the consolidated samples were performed, and the results indicated that the mechanical properties of these samples are similar to those of the weak cemented natural rock-soil mass [12, 13]. In the literature, researchers mainly focused on the mechanical and deformation characteristics of transparent soil [14-18].

In this paper, we test the RI value and basic physical parameters of mineral oil. Meanwhile, the basic parameters of calcium bromide solution with different concentration are obtained as well, including RI value, pH value, density, and dynamic viscosity. Different combinations of grains and pore fluids are evaluated to achieve optimal transparency. The work conducted in this study presents a theoretical basis for further manufacturing of transparent artificial cores.

## EXPERIMENTAL

### *Materials and methods*

The artificial core is manually manufactured under a given pressure in a mold of specific shape with a certain proportion of grain and binder. According to the manufacturing process and different type of binder, the artificial core can be divided into three categories including cementation of quartzite and colophony, cementation of aluminum phosphate and filling tube of quartz sand [19-21], as shown in Fig. 1.

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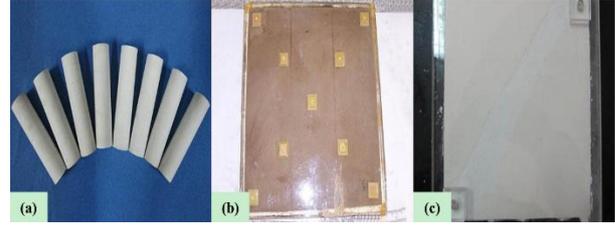
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Transparent soil is generally synthesized by two materials, i.e. grains and pore fluids which have a similar RI value to each other. In the early stage, the amorphous silica powder and silica gel were the most common grain material for transparent soil preparation [10]. More types of new materials were then used as grains including fused silica [14], hydrogel (which was renamed Aquabeads) [22], and Laponite [23]. The pore fluids used in transparent soil mainly include mineral oil (a mixed system of liquid paraffin and normal alkanes), calcium bromide solution and sucrose solution. [24, 25]. The physical properties and corresponding pore fluids to different grain materials in literature are summarized, as shown in Table 1.

According to a previous study [12], fused quartz and glass sand contain more impurities and

are relatively less transparent compared to silica gel. Silica gel is selected as the grain material in this study with a corresponding RI value ranging from 1.41-1.46, and the matching pore fluids are mineral oil and calcium bromide solution. The silica gel used in this study is prepared by the Qingdao Bangkai High-tech Inc.



**Fig. 1.** Various types of artificial core (a) cementation of colophony; (b) cementation of aluminum phosphate; (c) filling tube of quartz sand.

**Table 1.** Physical properties of different grain material and matching pore fluids

Grain categories	Amorphous silica powder	Silica gel	Fused silica	Hydrogel (Aquabeads)	Laponite
RI	1.442	1.442	1.458	1.333	1.336
Saturated unit weight (kN/m <sup>3</sup> )	9.4-16	11-14	13.4-16.4	10	10
Drained friction angle	19°-36°	29°-42°	44°-59°	—	—
Drained cohesion (kPa)	20-44	0	0	—	—
Compression index	1.6-3	—	0.34-0.54	0.1-0.15	16.6-20.6
Hydraulic conductivity (cm/s)	2.3×10 <sup>-7</sup> — 2.5×10 <sup>-5</sup>	1.5×10 <sup>-4</sup> — 7×10 <sup>-3</sup>	1.3×10 <sup>-5</sup> — 2.1×10 <sup>-5</sup>	7×10 <sup>-2</sup> — 6×10 <sup>-8</sup>	5×10 <sup>-9</sup> — 1.6×10 <sup>-6</sup>
Intrinsic permeability (Darcy)	0.015-0.16	1-45	24-40	—	—
Matching pore fluid	Mineral oil or calcium bromide solution		Mineral oil or sucrose solution (or STSI)	Water	Water

## RESULTS AND DISCUSSION

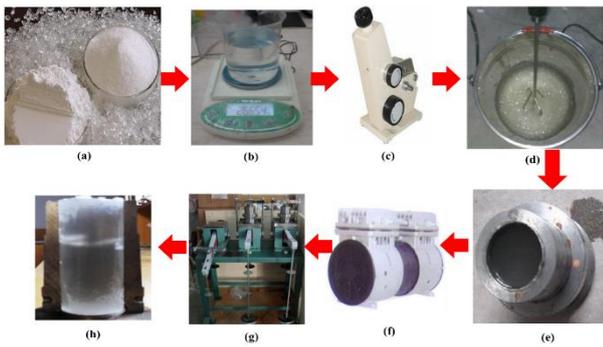
### General manufacturing process

The manufacturing process of the transparent core consists of three steps, i.e. the preparation of a mixed system of grain and pore fluid, molding and vacuuming of mixed system, and consolidation of mixed system. It should be noted that air bubbles must be removed from the mixed system to ensure the transparency of the core.

In addition, the pressure should be loaded step by step until the maximum load during the consolidation process.

### Laboratory test of mineral oil

The mineral oil is composed of two alkanes with different RI values, which generally depend on the RI value of the selected grain material. There are four types of alkanes selected and tested in this study, including liquid paraffin, 2#, 5#, and 10# white oil. Different types of white oil are



**Fig. 2.** General work-flow of the manufacturing process: (a) grain material; (b) pore fluid; (c) 2WJ type refractometer; (d) mixed system; (e) molding; (f) vacuuming; (g) consolidation; (h) removing of mold.

Generally, the RI value increases with viscosity at the same temperature. The test results of the four types of alkanes are listed in Table 2.

The RI value of a mixed system with two component liquids can be calculated by [26]:

$$\frac{(V_1 + V_2)}{n} = \frac{V_1}{n_1} + \frac{V_2}{n_2} \quad (1)$$

where,  $n$  is the RI value of the mixed system,  $n_1$  and  $n_2$  are the RI values of the component liquids, and  $V_2$  is the volume fraction of the component liquids.

**Table 2.** Test results of mineral oil

Name	Refractive index	Density (kg/m <sup>3</sup> )	Dynamic viscosity (m <sup>2</sup> /s)
Liquid paraffin	1.4586	860-905	6-8
2# white oil	1.4205	816	2.8
5# white oil	1.4329	820	4.8
10# white oil	1.4485	828	9.8

#### Laboratory test of calcium bromide solution

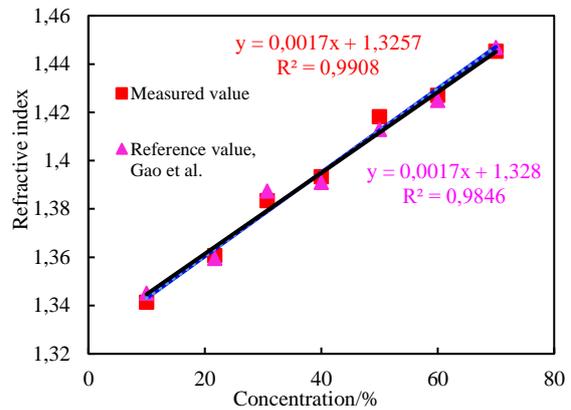
The anhydrous calcium bromide used in this paper is prepared by Tianjin Guangfu Fine Research Institute. The molecular formula is CaBr<sub>2</sub>, and the molecular weight is 199.89. It is white, granular or crystalline with strong hygroscopicity, easy deliquescence, and slight decomposition. The density, melting point and boiling point are 3.35 g/cm<sup>3</sup>, 730 °C, and 806-812 °C, respectively. It is soluble in water,

ethanol, and acetone, but is insoluble in ether and chloroform. It will turn yellow when stored in air for a long period and will decompose into bromine and calcium oxide at high temperature. The main technical parameters of CaBr<sub>2</sub> are listed in Table 3.

As is shown in Fig. 3, the RI values of calcium bromide solution are tested at various concentrations at room temperature conditions. It can be seen that the concentration of calcium bromide solution has a great effect on RI value which linearly increases with concentration. Besides, the test results are compared with the literature and are in good agreement [27].

**Table 3.** Technical parameters of CaBr<sub>2</sub>

CaBr <sub>2</sub> (%)	Chloride (%)	Sulfate (%)	Metal (ppm)	Insoluble (%)
≥	≤	≤	≤	≤
96.0	1.0	0.02	5.0	1.0



**Fig. 3.** Relationship between concentration and RI

Parameters such as density, pH value and dynamic viscosity of calcium bromide solution with different concentrations were tested as well, and the results are shown in Fig. 4.

Both density and viscosity increase with the concentration of calcium bromide solution, whereas pH decreases and shows acidic values.

The RI values of solutions of different concentration were determined at various temperatures, and the results are shown in Fig. 5. It is found that the RI value is insensitive to the temperature at low concentration. The sensitivity to temperature slightly increases with concentration, and the maximum increase is achieved at 40% concentration.

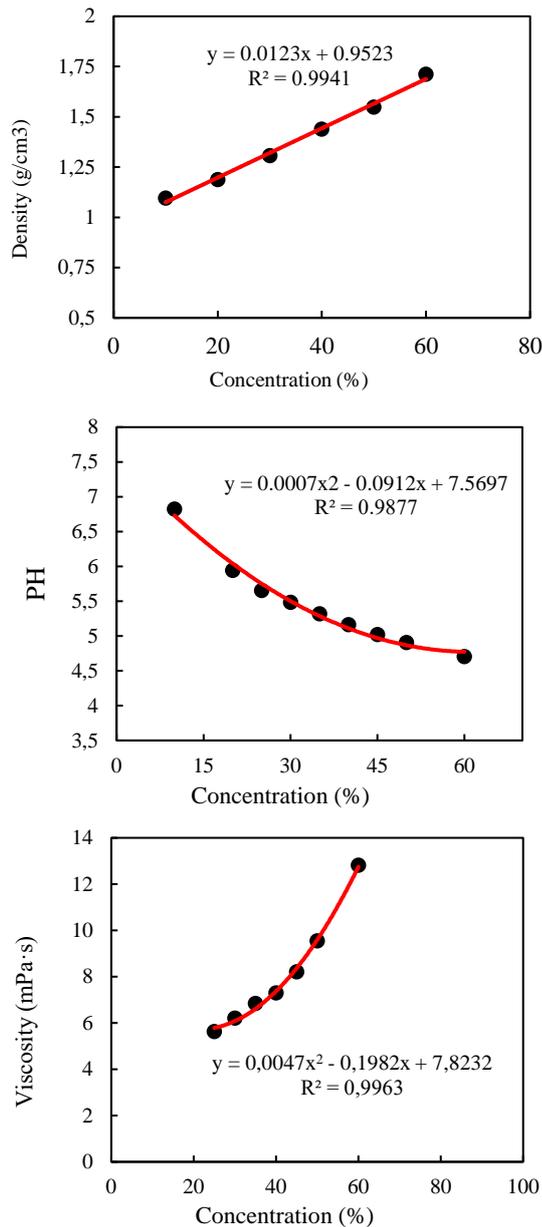


Fig. 4. Relationships between concentration & density, pH value, and viscosity.

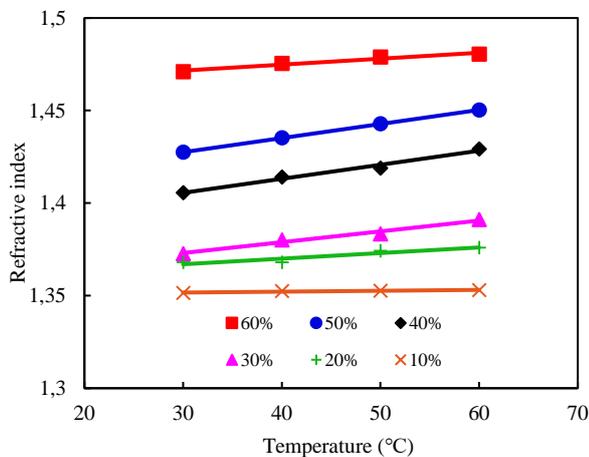


Fig. 5. Relationship between temperature and RI at various concentrations.

In summary, there is a good correlation between RI value and concentration of calcium bromide solution, and the RI value generally ranges from 1.34 to 1.47. The results indicate that calcium bromide solution is an ideal pore fluid material for transparent soil manufacturing, for it can satisfy a wide range of RI requirements. Considering the acidic alternation property of calcium bromide solution at high concentration, the mineral oil should be a better alternative selected in those cases when the experimental process is highly dependent on the chemical properties of the material.

*Effect of particle size and concentration on sample transparency*

The mixed systems of silica gel and mineral oil are prepared with four different types of particle size, as shown in Fig. 6. Four kinds of specifications of particle size including 1490 μm, 124.2 μm, 49.7 μm and 9.93 μm are used in this study.

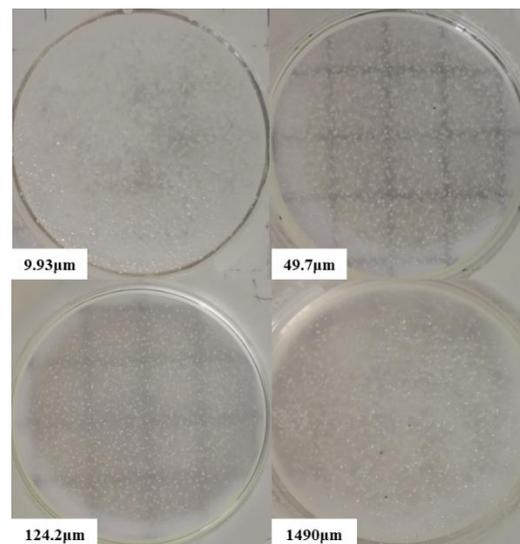


Fig. 6. Transparent mixed system of silica gel and mineral oil

In Fig. 6, it can be noted that the second group with particle size of 49.7 μm has relatively better transparency. The transparency decreases with the increase or decrease of particle size. When the particle size is too small, there will be more impurities which can significantly reduce the transparency of the mixed system. In addition, a large number of tiny air bubbles will be immersed in the mixed system during the molding and consolidation operation. On the other side, when the particle size is too large, segregation occurs easily between particles and pore fluids during the consolidation process, which can significantly reduce the transparency and strength of the final

cores. Currently, there are no products with a purity of 95% for a given particle size in the market. Therefore, the silica gel with particle size ranging from 49.7 to 74.5  $\mu\text{m}$  was selected as the grain material in this study, and the corresponding RI values range from 1.41 to 1.46.

Calcium bromide solution was used as a pore fluid in this study to manufacture transparent soil at four levels of concentration including 56%, 60%, 62%, and 65%. Silica gel with particle size of 47.9  $\mu\text{m}$  was added into the calcium bromide solution until saturation, and the mixed systems are shown in Fig. 7. Among the four mixed systems, the sample with 62% concentration has relatively good transparency.

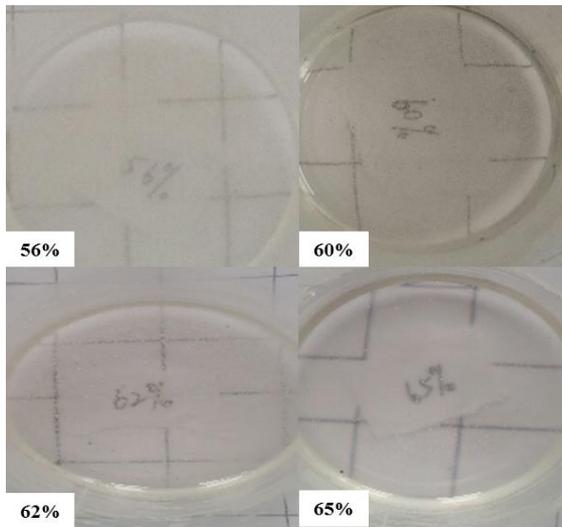


Fig. 7. Transparent mixture manufactured by calcium bromide solution with different concentrations.

Pressure sensors, flowmeter, and measuring ruler are utilized in the experiment to monitor the changes of pressure and flux inside the transparent sample. Meanwhile, the flow pattern could be imaged by multiple cameras which are arranged at different positions around the experimental setup, including front, side, and bottom of the sample box. These might be most helpful to realize the 3D analysis of the flow field during the test.

According to previous studies [28, 29], the transparent soil material not only has similar properties from the perspective of strength and deformation but also hydraulic property similar to some types of natural soil. By changing raw materials and operating conditions, transparent oil samples with different types of porosity, mechanical properties, and pore structure can be prepared. Then the samples can be adopted to conduct fluid flow experiments. A simple flow experimental setup was developed to model the 2D flow, as shown in Fig. 8.

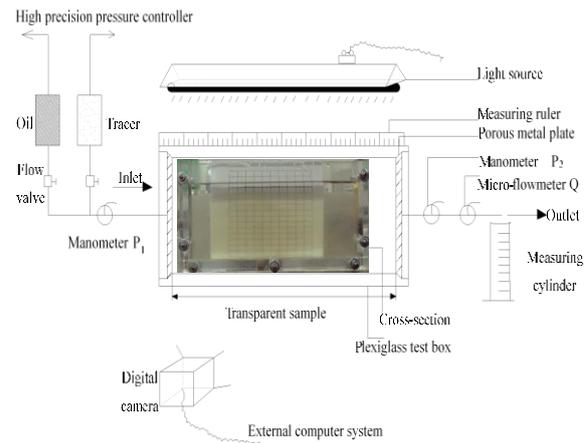


Fig. 8. 2D flow experimental setup used for transparent soil.

## CONCLUSION

In this paper, a new technology was proposed to prepare transparent samples in combination with artificial core and transparent soil technique. Several groups of grain materials and corresponding pore fluids were tested to determine the optimal combinations of different materials.

The basic physical parameters such as RI value, density, dynamic viscosity, and pH value were obtained both for mineral oil with a different combination of alkane compounds, and calcium bromide solution with different concentrations. The pH value of the mineral oil is more stable than that of the calcium bromide solution. Nevertheless, the calcium bromide solution has a wider range of RI values.

The effect of temperature on RI value is also studied. The results indicated that the general trend of the RI value of calcium bromide solution increases with temperature, especially at high concentrations. However, mineral oil is less sensitive to temperature.

The mixed systems of grains and pore fluids were prepared for further study. As a key indicator of the transparent sample, the effect of particle size and solution concentration on the transparency of the mixed system were quantitatively investigated. The results indicated that the silica gel of particle size ranging from 49.7-74.5  $\mu\text{m}$  and calcium bromide solution with a concentration of 62% will lead to relatively higher transparency of the mixed system.

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**Conflict of Interests:** The authors declare that there is no conflict of interest regarding the publication of this manuscript.

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