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Effect of silicone oil contamination on imbibition characteristics of liquid through porous media

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Abstract:
Spontaneous imbibition is a common phenomenon in oil and gas reservoir extraction, environmental engineering and biomedicine. Studies generally assume that solid matrices possess smooth and clean surfaces. However, in most practical research scenarios, solid surfaces become contaminated, obscuring their inherent wettability. This research investigates the impact of silicone oil contamination on spontaneous imbibition characteristics. Spontaneous imbibition experiments were conducted using microporous filter membranes under silicone oil contamination. The results indicate that silicone oil contamination significantly prolongs the complete saturation time. Compared to uncontaminated membranes, contamination for 5, 10 and 15 days results in an increase in saturation time of 37.5%, 68.75% and 112.5% respectively. The spontaneous imbibition dynamics under silicone oil contamination still follow the form of the classical Lucas-Washburn equation. Calculations show that the contact angle increases with the duration of contamination, indicating a decrease in wettability. A novel theoretical model based on the Lucas-Washburn equation is established to describe the imbibition dynamics under silicone oil contamination. The conclusions drawn from this study are of significant importance for understanding and improving the performance of paper-based microfluidic devices in contaminated environments, enhancing the efficiency of oil and gas reservoir recovery, and assessing the accuracy of groundwater monitoring under contaminated conditions.

1. Introduction

Contamination is ubiquitous in daily life, exerting adverse effects on human health and industrial production (Kuster et al., 2008; Dougherty et al., 2010; Pal et al., 2010; Kumar et al., 2021). It can alter the surface wettability of the solid matrix (Iler, 1979; Roy and McGill, 2000; Li et al., 2013), affect the imbibition rate (Schütt and Spetzler, 2001) and greatly reduce the mobility of the contact lines between solids, water and gas (Paterson et al., 1995; Moerig et al., 1997; Waite et al., 1997; Schütt and Spetzler, 2001). Although many studies have taken steps to minimize contamination, the surface may still have been inadvertently contaminated, thereby obscuring its inherent properties, including wettability (Li et al., 2013).

Spontaneous imbibition plays a crucial role in the natural environment and daily life (Cai et al., 2022; Zhou et al., 2023; Wang et al., 2024), such as environmental monitoring (Rattanarat et al., 2013, 2014), paper-based microfluidic analytical devices (Martinez et al., 2010; Yetisen et al., 2013), water infiltration in soil and porous rocks (Tokunaga et al., 2017;

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Cai et al., 2020; Wang et al., 2023; Shao et al., 2024), CO_2 geo-sequestration (Bachu, 2013; Lyu et al., 2024) and enhanced oil recovery (Wei et al., 2024; Yang et al., 2024; Zhang et al., 2024). Numerous factors can influence the imbibition dynamics, such as molecular adsorption (Gruener and Huber, 2009), liquid viscosity (Quéré, 1997), dynamic contact angle (Siebold et al., 2000), gravity (Chan et al., 2004; Fries and Dreyer, 2008), surface freezing (Gruener and Huber, 2009), surfactants (Pan et al., 2021), evaporation (Dubé et al., 2000) and roughness (Bico et al., 2002; Shen et al., 2017). Most previous studies have assumed that the solid matrix has a clean surface. However, in most cases, solid surfaces are rough and chemically heterogeneous (Paterson et al., 1995). Contamination is an important factor causing surface roughness and heterogeneity.

Using infrared spectroscopy and X-ray photoelectron spectroscopy, it is demonstrated that airborne hydrocarbons adsorb onto the surface of graphene, leading to an increase in the water contact angle. Clean graphene surfaces become more hydrophobic after exposure to ambient air due to the adsorption of hydrocarbons, which can occur within minutes of exposure to air (Li et al., 2013). Using a Hele-Shaw cell, it is investigated that 2-propanol contamination reduces surface wettability, thereby reducing flow rates. It also creates local surface wettability heterogeneities leading to contact line roughness and air bubble entrapment (Schütt and Spetzler, 2001). However, the geometric shape of the solid matrix is too simple to represent the porous media. In addition, only the horizontal diffusion and imbibition of liquids are studied, which is different from the capillary rise in porous media.

The Lucas-Washburn (L-W) equation is commonly used to describe the relationship between the liquid rise height and time (Lucas, 1918; Washburn, 1921):

$$H=\sqrt{\frac{\gamma R\cos\theta}{2\mu}t} \tag{1}$$

where H is the imbibition height, γ is the interfacial tension between the liquid and air, θ is the equilibrium contact angle, R is the capillary tube radius, μ is the liquid viscosity, t is the time. Subsequent studies have modified the L-W equation to take into account various factors such as liquid column momentum change (Bosanquet, 2009), gravity (Fries and Dreyer, 2008; Cai et al., 2012), saturation gradient (Lockington and Parlange, 2004), tortuosity (Gruener and Huber, 2009; Cai and Yu, 2011) and hydrostatic effects (Xue et al., 2006). Most capillary rise models are based on the L-W equation and adjust for dynamics with capillary and fluid parameters, and they often overlook the potential effect of contamination.

In this paper, the impact of silicone oil contamination on the spontaneous imbibition characteristics of liquids through microporous filter membranes is investigated. A new model based on the L-W equation is established to characterize the imbibition dynamics under silicone oil contamination.

2. Materials and methodologies

2.1 Microporous filter membrane

During the imbibition study, the microporous filter membrane, made from a network of mixed cellulose ester (MCE) fibers, forms a porous framework. Upon interaction with liquids, these pores engage in capillary absorption. The MCE membranes presented in this research maintain consistent pore dimensions and uniform thickness, with the specified pore diameter of the MCE sample being 45 μm and a thickness of 100 μm.

MCE consists mainly of three elements: Carbon, hydrogen and oxygen, and it is mainly composed of cellulose nitrate and cellulose acetate (Vaca-Garcia et al., 2001; Sun et al., 2023). These cellulose esters are pivotal in a wide range of applications, such as high-performance coatings, drug delivery systems, eco-friendly plastics, composite materials, layered structures, optical films, and membrane-based separation technologies (Edgar et al., 2001; Zugenmaier, 2006; Baker, 2023). The consistent and compact pore architecture of MCE supports the regulation and uniform distribution of fluid flow. In addition, the incorporation of polar, oxygen-rich functional groups like hydroxyl and carbonyl groups in MCE enhances its hydrophilicity, imbibition rate, and the significant influence of contamination on the imbibition process (Edgar et al., 2001; Sun et al., 2023).

2.2 Chemicals

Deionized water is used as the imbibition liquid in the experiments. Additionally, silicone oil with a viscosity of 0.65 mm^2/s and favorable volatility properties is obtained from Clearco Products Co, Inc.

Low-viscosity silicone oil has good volatility, which allows contamination levels to be controlled, thereby simulating varying degrees of contamination in practical engineering applications (Pan et al., 2021). Silicone oil shares certain elemental similarities and chemical structures with crude oil (such as the elements carbon, hydrogen and oxygen, as well as methyl groups). However, crude oil is extremely complex in composition, whereas silicone oil is more singular in composition, which facilitates the analysis of results (Kim et al., 2012; Gaweł et al., 2014). Silicone oil has a wide range of applications, such as lubricants (Aziz et al., 2018), clinical medicine (Barca et al., 2014), personal care products (Hu et al., 2012) and microfluidic experiments (Rostami and Morini, 2018; Wang et al., 2021), making this study highly informative for applications in related fields.

2.3 MCE membrane characterization

The pore size of the MCE is determined to be 0.45 μm, with a porosity of 79% and a thickness of 100 μm (Shirasaki et al., 2017; Song et al., 2021).

Utilizing scanning electron microscopy (SEM), we examine the topographical features of the microporous filter membrane. The membrane is cut into appropriate small pieces and attached to a scanning platform with the aid of a conductive adhesive. To enhance electrical conductivity, the samples undergo a carbon coating process. The SEM captures images under a high-vacuum environment at an acceleration voltage

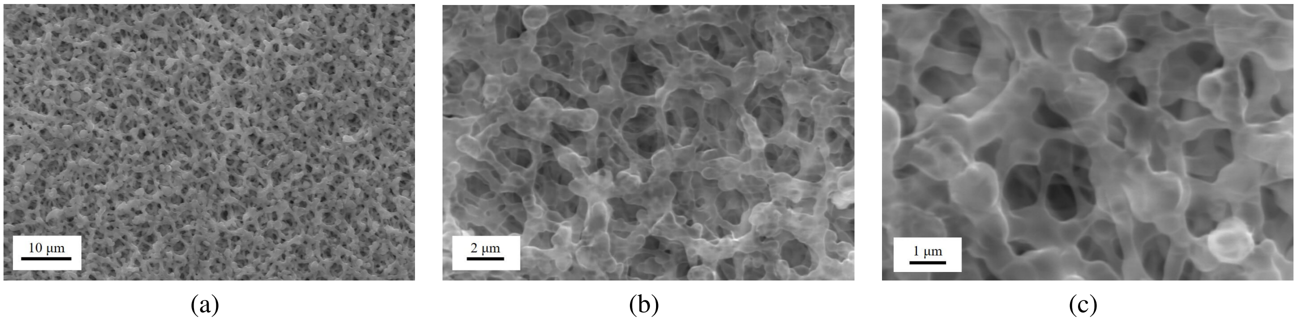

Fig. 1. SEM images of MCE membrane.

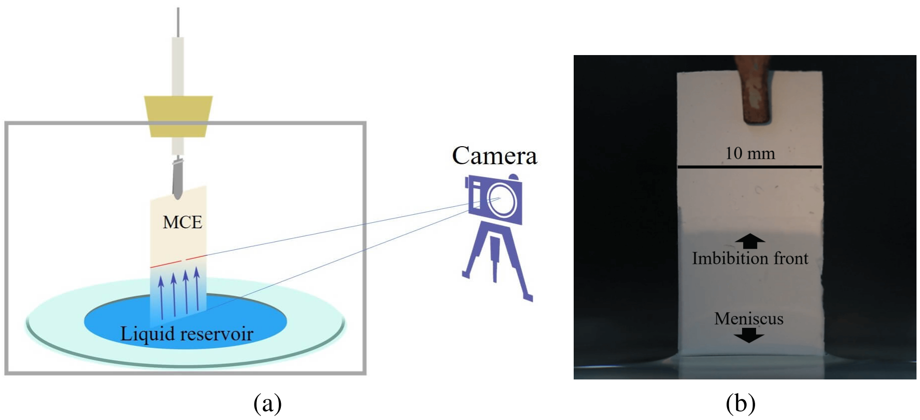

Fig. 2. (a) Schematic of experimental setup and (b) photograph of spontaneous imbibition of liquid in MCE membrane.

of 20 kV, providing detailed views of the fibrous structure within the microporous filter membrane material, as presented in Fig. 1.

2.4 Imbibition experiment

The imbibition process is executed with a microporous filter membrane, measuring 20 mm in length, 10 mm in width, and 0.1 mm in thickness, which is held above a liquid container by a clamping apparatus (Fig. 2(a)). Liquid is injected into the container and the injection is ceased once the liquid level contacts the filter membrane. The moment the underside of the membrane touches the liquid, an outer meniscus swiftly attains equilibrium (Fig. 2(b)). The imbibition of liquid within the microporous structure results in a distinct color contrast between the wet and dry regions, allowing direct observation of the imbibition front without the need for dye or contrast agent (Figs. 2(a) and 2(b)). The potential for a precursor film is ruled out. Moreover, the study disregards the influences of buoyancy, inertia, and gravitational forces.

For clean and slightly contaminated microporous filter membranes, the imbibition front remains relatively planar (Fig. 2(b)). The entire imbibition process is captured by a video camera (60 frames per second). Due to contamination causing discontinuity in the saturated areas, open-source software (ImageJ) is used to quantify the imbibition area. In this study, the meniscus height is factored out to concentrate on the imbibition dynamics within the porous medium.

To evaluate the effects of silicone oil contamination, clean microporous filter membranes are exposed to gaseous environments containing silicone oil for periods of 5, 10 and 15 days. After exposure, the membranes are removed and tested for imbibition in a silicone oil-free environment to observe any changes in the quantitative relationship between imbibition area and time. Clean microporous filter membranes are subjected to imbibition in a gaseous environment free of silicone oil, which serves as a baseline control group. The experiment monitors the evolution of imbibition area over time to establish a quantitative relationship between imbibition area and time.

The experimental procedure is as follows:

1) The clean MCE membrane samples are placed in a 50 mL beaker.
2) A 1 L beaker is used to simulate the gaseous environment surrounding the microporous filter membrane.
3) The 50 mL beaker containing the membrane samples is then placed inside the 1 L beaker.
4) Another 50 mL beaker containing 30 mL of silicone oil with a viscosity of 0.65 mm^2/s is also placed inside the 1 L beaker.
5) The opening of the 1 L beaker is sealed with cling film to maintain a controlled environment. The beaker assembly, including the microporous filter membrane samples and silicone oil, is left undisturbed to ensure equilibrium with the contaminated environment.

6) After predetermined exposure periods of 5, 10 and 15 days, the microporous filter membrane samples are removed from the silicone oil environment and subjected to imbibition tests in a clean gas environment. Each experimental condition is replicated 3 to 5 times.

3. Results

3.1 MCE membrane properties

The morphology of the MCE membrane is shown in Fig. 1. The microstructure of the MCE surface is observed by SEM. The SEM results show that the pore size is uniform and consistent with the given result of 0.45 μm.

3.2 The influence of silicone oil contamination

Compared to the uncontaminated microporous filter membrane, the complete saturation time increases by 37.5% after 5 days of contamination in a silicone oil environment, by 68.75% after 10 days of contamination and by 112.5% after 15 days of contamination (see Table 1). A higher level of contamination would result in a longer saturation time (Table 1), which is consistent with research by Schütt and Spetzler (Scht and Spetzler, 2001). However, in the previous studies, imbibition took place in the horizontal Hele-Shaw cell, the geometry of which is too simple to represent the imbibition process in porous media. In addition, the horizontal diffusion and imbibition of liquids in the Hele-Shaw cell is different from the capillary rise in this paper.

The linear relationship between the imbibition area and the square root of time applies to spontaneous imbibition under silicone oil contamination before the membrane is completely saturated (Fig. 3), which is consistent with the L-W equation. The slopes and correlation coefficients (from least squares linear regression) for the experimental data are given in Table 1. In addition, the longer the membrane remains in the silicone oil environment, the lower the imbibition rate (see slope in Table 1). The silicone oil in the air reduces the imbibition rate because the adhesion of the compound (here silicone oil) changes the wettability of the MCE surface (Li et al., 2013). The longer it is exposed to air, the more significant the change in wettability. Our research indicates that the MCE membrane becomes more hydrophobic than before, and the presence of silicone oil in the air is the reason for the change in wettability.

Table 1. Experimental and fitting data at different levels of contamination.

Contamination	Saturation time (s)	Slope ($mm^2/s^{0.5}$)	R^2	Contact angle (°)
None	240	12.9137	0.9997	84.68
Silicone oil 5 days	330	11.6366	0.9997	86.03
Silicone oil 10 days	405	10.3135	0.9996	86.61
Silicone oil 15 days	510	9.6422	0.9999	87.04

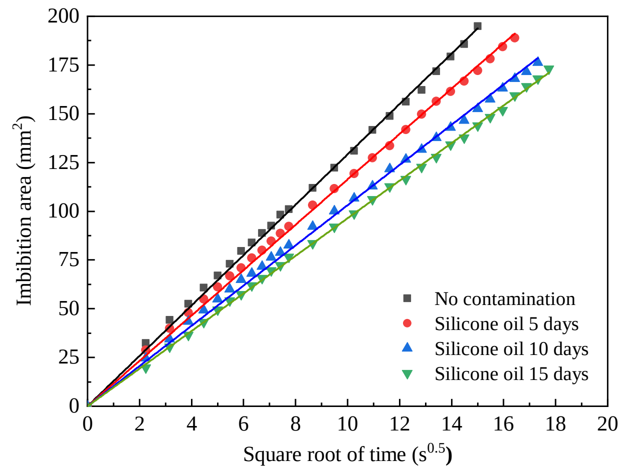

Fig. 3. The imbibition area of liquid as a function of square root of time in the MCE membrane at different levels of contamination.

4. Discussion

From the dynamic results of spontaneous water imbibition in the microporous filter membrane, it can be seen that there is an ideal linear relationship between the spontaneous imbibition area and the square root of the imbibition time throughout the imbibition process. Therefore, the spontaneous imbibition of water in the contaminated microporous filter membrane still follows the form of the L-W equation.

The L-W equation is a well-regarded model for illustrating the relationship between capillary rise height and elapsed time. However, this model conventionally employs the equilibrium (static) contact angle, which assumes a constant contact angle throughout the imbibition process (Siebold et al., 2000). Previous research has shown that contamination can affect the wettability of substrate surfaces, thereby affecting their contact angles (Iler, 1979; Roy and McGill, 2000; Li et al., 2013). Consequently, the presence of contamination can significantly reduce the predictive accuracy of the traditional L-W equation. We have refined the traditional L-W equation by incorporating a contact angle model under contamination conditions, thereby improving the predictive accuracy.

The imbibition area is divided by the width of the microporous filter membrane as the average imbibition height during the imbibition process. The contact angles at different levels of contamination are obtained using the L-W equation. A compilation of the contact angle data can be found in Table 1. The relationship between contact angle and contamination time is shown in Fig. 4. Nonlinear fitting is performed to derive the relationship between contact angle and time:

$$\theta(t) = 87.38 - 2.69\mathrm{e}^{\left(-\frac{t_c}{6.5}\times 10^{-5}\right)} \tag{2}$$

where t_c is the time of contamination in a silicone oil environment. The true contact angle of the contaminated membrane can be calculated using

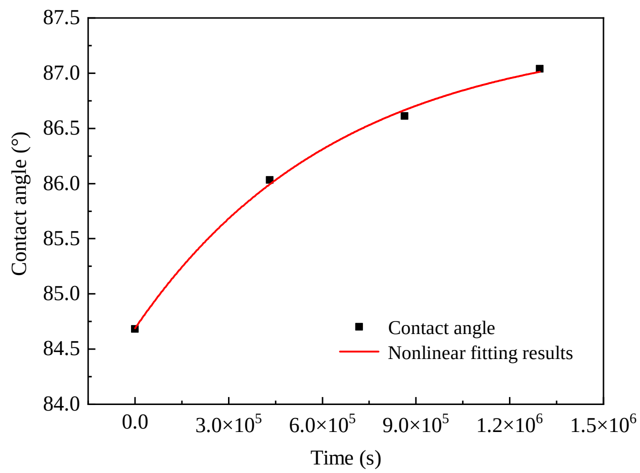

Fig. 4. The contact angle as a function of the contamination time for the calculated data shown in Table 1.

Eq. (2). The calculated true contact angle is then substituted into the L-W equation to characterize the spontaneous imbibition dynamics of water in the microporous filter membrane under silicone oil contamination. The results of our current research are mainly focused on the systems involving silicone oil and microporous membranes. Imbibition patterns under different reservoir conditions and different contamination sources will be investigated in our future studies to provide more insightful guidance for practical applications.

5. Conclusions

Spontaneous imbibition in porous media plays an important role in biomedicine, environmental monitoring, enhancing oil and gas recovery and improving electrolyte transport efficiency. This research investigates the effect of silicone oil contamination on the spontaneous imbibition characteristics. The following conclusions are drawn:

1) Compared to clean microporous filter membranes, the complete saturation time in a silicone oil environment increases by 37.5% after 5 days, by 68.75% after 10 days and by 112.5% after 15 days. The higher the contamination level, the longer it takes to reach full saturation.
2) Before complete saturation of the microporous filter membranes, there is an ideal linear relationship between the spontaneous imbibition area and the square root of the imbibition time. Consequently, spontaneous imbibition under silicone oil contamination still follows the form of the L-W equation. A new model is developed to modify the L-W equation to account for the impact of contamination on spontaneous imbibition.

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Conflict of interest

The authors declare no competing interest.

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