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## Review

# Laboratory core flooding experimental systems for CO<sub>2</sub> geosequestration: An updated review over the past decade

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## ABSTRACT

Carbon dioxide (CO<sub>2</sub>) geosequestration in deep saline aquifers has been currently deemed as a preferable and practicable mitigation means for reducing anthropogenic greenhouse gases (GHGs) emissions to the atmosphere, as deep saline aquifers can offer the greatest potential from a capacity point of view. Hence, research on core-scale CO<sub>2</sub>/brine multiphase migration processes is of great significance for precisely estimating storage efficiency, ensuring storage security, and predicting the long-term effects of the sequestered CO<sub>2</sub> in subsurface saline aquifers. This review article initially presents a brief description of the essential aspects of CO<sub>2</sub> subsurface transport and geological trapping mechanisms, and then outlines the state-of-the-art laboratory core flooding experimental apparatus that has been adopted for simulating CO<sub>2</sub> injection and migration processes in the literature over the past decade. Finally, a summary of the characteristics, components and applications of publicly reported core flooding equipment as well as major research gaps and areas in need of further study are given in relevance to laboratory-scale core flooding experiments in CO<sub>2</sub> geosequestration under reservoir conditions.

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## 1. Introduction

In accordance with Consensus for Action 2013 (IPCC, 2013), carbon dioxide (CO<sub>2</sub>) is a major contributor to a significantly accelerating rise in the average global temperature and the consequent effects of climate change (IEA-GHG, 2008; IEA, 2010). Moreover, CO<sub>2</sub> is also perhaps the most important factor in driving recent anthropogenic global warming (Bachu and Adams, 2003; IPCC, 2007; Burnside and Naylor, 2014; Bachu, 2015; Zhao et al., 2015), and further increases in atmospheric CO<sub>2</sub> are also projected to adversely affect future life on the Earth (Haszeldine, 2009; Bacci et al., 2011; IPCC, 2013). CO<sub>2</sub> emissions from fossil fuel combustion and industrial processes, such as coal-fired power generation and cement making, account for 78% of the increase in greenhouse gases (GHGs) emissions between 1970 and 2011 (Bachu and Adams, 2003; Olivier et al., 2014; Akbarabadi and Piri, 2015). To date, this trend is a continued rapid rise and therefore, controlling

the continuously increasing atmospheric CO<sub>2</sub> content has become an essential requirement (Olivier et al., 2014; de Silva et al., 2015).

Due to the fact that CO<sub>2</sub> release to the atmosphere is considered as the major drive behind climate change, CO<sub>2</sub> geosequestration in deep saline aquifers is proposed as a climate reduction way (Lackner, 2003; Schrag, 2007; Bachu, 2008, 2015; IPCC, 2013; Rao and Kumar, 2014; Soroush et al., 2014; Yang et al., 2014; Bhattacharjee et al., 2015). Carbon capture and storage (CCS) is recognized as one of the most effective technologies for reducing CO<sub>2</sub> emissions in the short to medium term (Li et al., 2003, 2011, 2015a; Jakupi et al., 2008; de Coninck and Benson, 2014; Rathnaweera et al., 2014; Taylor et al., 2015; Wei et al., 2015; Yamabe et al., 2015). The CCS consists of the main processes (Shukla et al., 2010; Li et al., 2014a; Song et al., 2014): (i) Capture and separation of CO<sub>2</sub> from point sources such as coal fired power plants and other high intensity CO<sub>2</sub> emission industries such as the steel and cement manufacturing industries; (ii) Transportation of the captured CO<sub>2</sub> to the injection sites after proper treatment (pressurization, liquefaction, or hydrate formation); and (iii) Injection of CO<sub>2</sub> into the geological formation (underground) for storage. CCS can take place in various geological formations, including sedimentary formations, depleted oil and gas reservoirs, deep unmineable coal seams and deep saline aquifers (Holloway, 1996; Gunter et al., 1998; Gale and Freund, 2001; Gale, 2004; Plug and Bruining, 2007; Lions et al., 2014). Among these, deep saline aquifers have unique advantages, one of which is the

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extended storage capacity (Gale and Freund, 2001; Nordbotten et al., 2005; Li et al., 2014a, b). Up to now, several projects have been successfully demonstrated at pilot and commercial scales (Bachu and Adams, 2003; Rodosta et al., 2011; US-DOE-NETL, 2012; Li et al., 2013, 2014c; Roettereng, 2014; Bachu, 2015).

CO<sub>2</sub>-brine displacement in sandstones, carbonates and shale has been studied extensively (e.g. Bachu and Bennion, 2009a,b; Akbarabadi and Piri, 2013; Bennion and Bachu, 2005, 2006a,b; 2008a,b, 2010; Deng et al., 2015), including the wettability of the rock (e.g. Krevor et al., 2012; Farokhpoor et al., 2013), flow directions (Ruprecht et al., 2014), and heterogeneities (e.g. Perrin and Benson, 2010; Pini et al., 2012, 2013; Pini and Benson, 2013a,b). In order to better understand the supercritical CO<sub>2</sub> (scCO<sub>2</sub>)-plume migration and front interface in geological reservoir formation at representative temperature, pressure, and salinity (Bennion and Bachu, 2005; Krevor et al., 2013), various core flooding experimental systems have been developed and implemented for simulating core-scale scCO<sub>2</sub> injection and migration in saline aquifers (e.g. Shi et al., 2009, 2011a,b; de Silva and Ranjith, 2013; Baldygin et al., 2014).

Precise knowledge of the CO<sub>2</sub>-induced interactions for supercritical CO<sub>2</sub>-brine-rock systems at elevated temperatures and pressures (Assayag et al., 2009), and of the resulting changes in the chemical and physical properties of the reservoir system is therefore a prerequisite for any secure operation of a storage site (Liu et al., 2014; Wang et al., 2015b). In order to better understand and estimate the CO<sub>2</sub>-injected space distribution and migration processes, the relative permeability of multiphase flow in porous media, and the phase status of the CO<sub>2</sub> plume, laboratory core flooding experiments such as those widely used in petroleum industry could better fulfill these requirements (e.g. Baldygin et al., 2014; Li and Fan, 2015). The aim of this paper is to systematically review the latest laboratory core flooding experiment and facilities available in the publicly reported literature since 2006 for investigating core-scale supercritical CO<sub>2</sub>-brine-rock interactions under geological reservoir conditions during the injection of CO<sub>2</sub>, and to conceptualize an optimal core flooding apparatus and experimental scheme for investigating CO<sub>2</sub> storage in deep saline aquifers. To date, few papers have systematically analyzed laboratory core flooding experimental systems, in particular for CO<sub>2</sub> geosequestration in deep saline aquifers, under simulated subsurface reservoir pressure and temperature conditions.

## 2. Mechanisms for CO<sub>2</sub> trapping in saline aquifers

When supercritical CO<sub>2</sub> is injected into structural reservoirs in deep permeable geological formations, various physical changes and geochemical reactions between the injected CO<sub>2</sub> and reservoir rocks occur under different subsurface geological conditions. Presently, CO<sub>2</sub> is stored in geological formations mainly by four trapping mechanisms (Bolster, 2014; Huppert and Neufeld, 2014; Cohen and Rothman, 2015; Manceau et al., 2015; Wang et al., 2015a). In this section, these mechanisms' basic principles will be described for subsequent analysis of the laboratory experiments.

### 2.1. Structural and stratigraphic trapping

Physical trapping consists of structural and stratigraphic trapping (Akbarabadi and Piri, 2015). This trapping occurs where migration of the CO<sub>2</sub> plume is impeded by regions of porous media with a concave-down structure and a low-permeability seal (i.e. caprock) (de Silva and Ranjith, 2013; Bolster, 2014). Structural and stratigraphic trapping is well established in geological literature, and is also the most dominant trapping mechanism at the early stages of CO<sub>2</sub> geosequestration.

### 2.2. Residual trapping

This phase of trapping happens very quickly as the fractured porous rock acts like a tight, rigid sponge (Cohen and Rothman, 2015). As the supercritical CO<sub>2</sub> is injected into the brine formations, it displaces fluid as it moves through the porous rock (Fig. 1a). Residual trapping is a relatively rapid process, occurring over time scales of days to months in core scale experiments (Pentland et al., 2011a; Shi et al., 2011a,b), and is predicted to contribute significantly to trapping within 10's of years following CO<sub>2</sub> injection (Sifuentes et al., 2009; Saadatpoor et al., 2010).

### 2.3. Solubility trapping

As CO<sub>2</sub> migrates through the target brine formations, some of it, up to 30% of the injected CO<sub>2</sub>, will dissolve into the formation water (Fig. 1b) (Doughty et al., 2001). The dominant short to medium term benefit of trapping of this type is that once injected CO<sub>2</sub> is soluble into host rock and saline aquifers, it no longer exists as a separate phase, and thereby eliminating the buoyant forces that drive it upwards (IPCC, 2005; Li, 2011). In the long term, water-rock interactions driven by acidification caused by CO<sub>2</sub> dissolution in groundwater will lead to mineral trapping of the CO<sub>2</sub> (Pang et al., 2012).

### 2.4. Mineral trapping

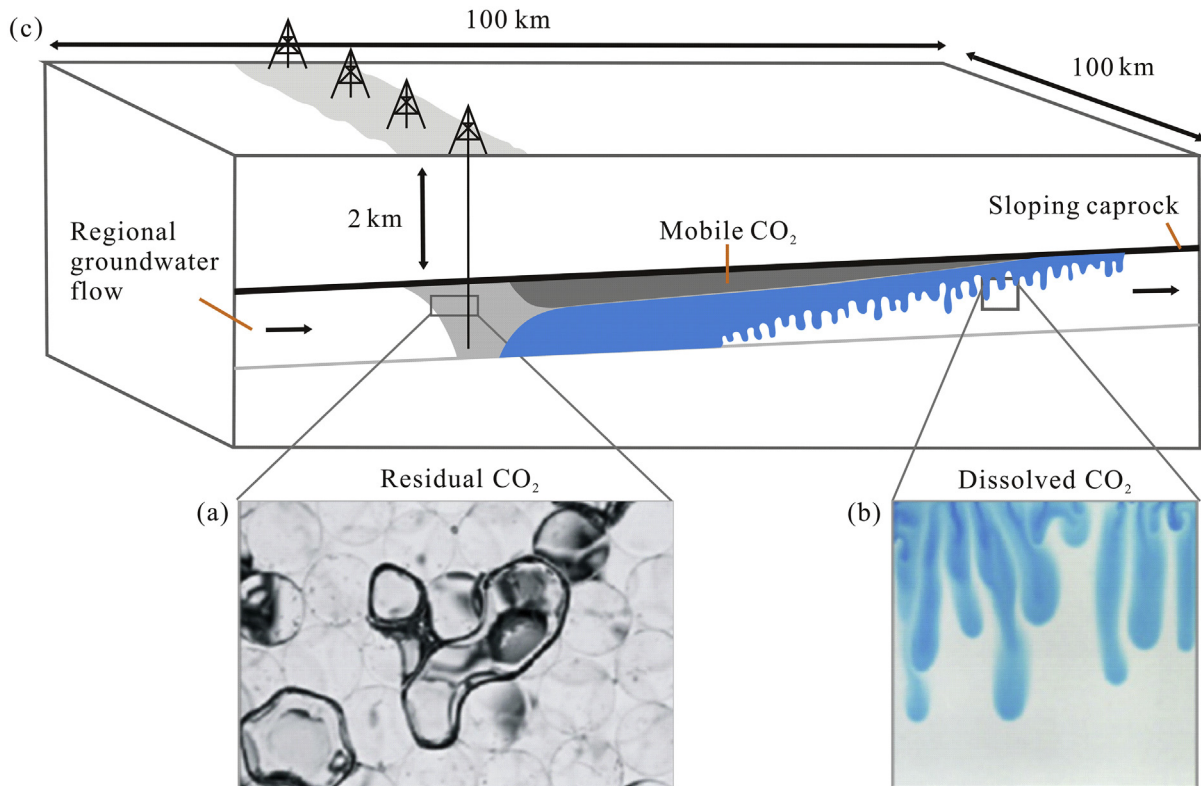
Mineralization reactions will ultimately trap the majority of injected CO<sub>2</sub> (Talman, 2015). However, this process generally operates on a very long time scale (Xu et al., 2004) and does not contribute in any significant way to CO<sub>2</sub> geosequestration during CO<sub>2</sub> injection and to the immobilization of the CO<sub>2</sub> plume within the initial decades (or century) of storage (Farokhpoor et al., 2013; Sell et al., 2013; Tsai et al., 2013; Bachu, 2015). Mineral trapping is believed to be comparatively slow, potentially taking a thousand years or longer (Peters, 2009).

### 2.5. Summary of trapping processes

CO<sub>2</sub> trapping processes take place over many years at different rates from days to years to thousands of years, but the general principle is that geologically sequestered CO<sub>2</sub> becomes more securely trapped with time (Zhang and Song, 2014). Various demonstrations of CO<sub>2</sub> geosequestration are already being carried out in a range of projects of varying scale (see Fig. 1c). Three industrial scale projects, i.e. Sleipner, Weyburn-Midale, and In Salah, which inject a minimum of around 3000 t a day of CO<sub>2</sub>, have been under the way for several years (Ringrose et al., 2013; Xie et al., 2013).

## 3. Laboratory-scale core flooding experiments

The multiphase flow properties of CO<sub>2</sub>/water systems in permeable rocks control the engineering design and management of industrial CO<sub>2</sub> geosequestration projects (Krevor et al., 2013; Song and Zhang, 2013; Manceau et al., 2015). Most laboratory studies on deep saline aquifers to date have been limited to the core flooding experiments (Suekane et al., 2009), which have been performed under simulated in-situ P-T (pressure-temperature) conditions ranging between 3 MPa and 150 MPa, and with working temperature of 10–70 °C (Table 1). Core flooding experimental research involving multiphase flow characteristics is the most effective method to figure out the scCO<sub>2</sub>-brine-rock interaction mechanisms during/after CO<sub>2</sub> injection and to determine the rate of CO<sub>2</sub> injection, the spread of injected CO<sub>2</sub> in subsurface storage formations, and the long-term immobilization of injected CO<sub>2</sub>. In



**Fig. 1.** Residual and soluble trappings are the key trapping mechanisms that contribute to CO<sub>2</sub> storage capacity (Szulczewski et al., 2012); (a) shows the blobs of gas immobilized by residual trapping in an experimental analog system, (b) displays the solubility trapping in a different analog system, and (c) models the trapping at the large scales relevant to a nationwide analysis and accounts for the injection and migration of CO<sub>2</sub>.

order to acquire meaningful results from core flooding tests, a series of experiments involving a variety of conditions should be undertaken (Bacci et al., 2011).

It is predictable that the multiphase flow characteristics of the fluid-rock system would change from one stage to another as it is subjected to any of the described flooding schemes. Pentland et al. (2011a,b) indicated that as to CO<sub>2</sub> sequestration in deep saline aquifers, there is an increasing awareness that detailed investigations are required to understand the role of the inherent heterogeneity of the rock samples used in the experiments on the measured multiphase properties. In fact, although the simulation studies clearly show the importance of small (sub-core) scale heterogeneity on fluid displacement (Chaouche et al., 1993; Plug and Bruining, 2007; Krause et al., 2011), experimental techniques are needed for quantitative observation of this phenomenon under a variety of conditions and of cores from different geological settings. A detailed investigation of the effect of such changes, using representative reservoir rock and fluid samples under in-situ reservoir P-T conditions, is vital before the commencement of any CO<sub>2</sub> geosequestration project during which a cyclic CO<sub>2</sub>-brine flooding is expected to occur. This would help to have a better understanding of the fate of the injected CO<sub>2</sub>, i.e. how the CO<sub>2</sub> plume evolves and migrates through the porous medium and potential change of the CO<sub>2</sub> injectivity in the target formations.

### 3.1. Overview of state-of-the-art core flooding experimental investigations

Understanding the multiphase flow properties of CO<sub>2</sub> and saline water in porous media is essential for successful large-scale geological storage of CO<sub>2</sub>. The pre-existing core flooding experimental systems, as reported in the previous literature (e.g. de Silva

and Ranjith, 2013; Baldygin et al., 2014; Stephen et al., 2014), have three main components: the upstream, the core block, and the downstream. Laboratory core flooding equipment is applicable to (1) analyze injection potential and storage capacity of field-scale subsurface geological formations (mainly sandstone and carbonate rock), (2) seize the migration front of laboratory-scale CO<sub>2</sub> plume, (3) estimate the petrophysical parameters and their profiles change under multiphase flow, (4) investigate the influence of scCO<sub>2</sub> dissolution on fluid displacement and imbibition, (5) monitor the behavior of CO<sub>2</sub> mass transfer and its transfer rate, and (6) examine the effect of varying CO<sub>2</sub> concentrations in injected water on both dissolution and displacement (Chang et al., 2014). Core flooding experiments are very important not only for acid gas injection (AGI) projects but also for CCS and CO<sub>2</sub> utilization (CCUS) projects such as CO<sub>2</sub> enhanced oil recovery (CO<sub>2</sub>-EOR) and CO<sub>2</sub> enhanced water recovery (CO<sub>2</sub>-EWR) (Huisingh et al., 2015; Li et al., 2015a,c). Nonetheless, advancements in field and experimental techniques for experimental setup indicate improvements for traditional core flooding systems (Baldygin et al., 2014).

Various investigations have examined the effects of different factors, such as temperature, pressure and salinity level, on CO<sub>2</sub> solubility reactions (e.g. Bando et al., 2003; Portier and Rochelle, 2005; Akiniev and Diamond, 2010; Darwish and Hilal, 2010; Taylor et al., 2015). The results are summarized in Table 1. These experimental and modeling results show that CO<sub>2</sub> solubility increases with elevated pressure and decreases with increasing temperature and salinity, which allows for a direct link between the observations in laboratory and the physics of the multiphase displacement process (de Silva et al., 2015). The main targets of these experimental studies are the injected scCO<sub>2</sub> and/or gCO<sub>2</sub> (supercritical CO<sub>2</sub> and/or gaseous CO<sub>2</sub>) in brines over a large range of temperatures, pressures and various core descriptions (Table 1).

**Table 1**A summary of published literature on core flooding experimental studies for CO<sub>2</sub> geosequestration in deep saline aquifers.

Samples	Location	Core dimension		Experimental condition		Fluids <sup>b</sup>	References
		$\phi$ (mm)	L (mm)	T (°C)	P <sup>a</sup> (MPa)		
Brown coal	VIC, Australia	203.0	832	RT <sup>c</sup>	15–25	gCO <sub>2</sub>	de Silva and Ranjith (2013)
Clay	UK	38.0	49–60 <sup>d</sup>	40	20–60	Brine, scCO <sub>2</sub>	Edlmann et al. (2013)
Brown coal	VIC, Australia	54.0	832	RT	3–13	gCO <sub>2</sub>	Jasinge et al. (2011)
Limestone	Wyoming, USA	37.7	174	60	19.16	Brine, scCO <sub>2</sub> + SO <sub>2</sub>	Akbarabadi and Piri (2015)
Sandstone	Japan	36.8	145	40	10	Brine, scCO <sub>2</sub>	Shi et al. (2009, 2011a,b)
Limestone	UK	6.0	25–35	50	11	Brine, scCO <sub>2</sub>	Andrew et al. (2013a,b, 2014, 2015)
Sandstone	Japan	35.0	70	40	12	Brine, scCO <sub>2</sub>	Zhang et al. (2013)
Sandstone	China	50.0	40/60	40	>10	H <sub>2</sub> O, CO <sub>2</sub>	Chang et al. (2014)
Sandpack	Netherlands	25.0	84	21–40	P <sub>atm</sub> –8.5 <sup>e</sup>	Water, scCO <sub>2</sub> , N <sub>2</sub>	Plug and Bruining (2007)
Sandstone	OH, USA	38.0	100 <sup>f</sup>	10	20	Brine, scCO <sub>2</sub>	Levine et al. (2014)
Sandstone	VIC, Australia	50.8	63.5–203.2	50–63	12.4	Brine, scCO <sub>2</sub>	Perrin and Benson (2010); Perrin et al. (2011);
Sandstone	OH, USA	50.8	63.5–203.2	50–63	12.4	Brine, scCO <sub>2</sub>	
Sandstone	Israel	51.0	96	25, 50	12	Water, scCO <sub>2</sub>	
Sandstone	OH, USA	51.0	96	25, 50	12	Water, scCO <sub>2</sub>	Pini et al. (2012, 2013); Pini and Benson (2013a,b)
Sandstone	OH, USA	50.8	95	50	11.7	Brine, scCO <sub>2</sub>	Krevor et al. (2012)
Sandstone	Australia	50.8	96	50	11.7	Brine, scCO <sub>2</sub>	
Sandstone	IL, USA	50.8	100	50	11.7	Brine, scCO <sub>2</sub>	
Sandstone	AL, USA	50.8	108	50	11.7	Brine, scCO <sub>2</sub>	
Sandstone	France	75.0	150	50	140	Brine, scCO <sub>2</sub>	Ott et al. (2012, 2015)
Sandstone	Germany	10.0	50	45	10	Brine, scCO <sub>2</sub>	
Limestone	France	25.4	90	35	7.5 <sup>g</sup>	scH <sub>2</sub> S, scCO <sub>2</sub> , N <sub>2</sub>	Roels et al. (2014)
Sandstone	Germany	10.0	30	28	Variation	Brine, scCO <sub>2</sub>	Berg et al. (2013)
Sandstone	USA	75.7	149.7	45	15	Brine, scCO <sub>2</sub>	
Limestone	England	4.0	12	50	13	Brine, scCO <sub>2</sub>	Menke et al. (2015)
Sandstone	Japan	50.0	100	40	12	Brine, scCO <sub>2</sub>	Kitamura et al. (2013)
Sandstone	Canada	38.1	32–197.6	35–75	8.6–27	Brine, scCO <sub>2</sub> , H <sub>2</sub> S	Bennion and Bachu (2005, 2006a,b, 2008a,b, 2010); Bachu and Bennion (2009a,b); Bachu et al. (2009a,b)
Carbonate Shale							
Anhydrite							
Sandstone	OH, USA	38.0	69	40	15	Brine, scCO <sub>2</sub>	Gutierrez et al. (2012)
Sandstone	OH, USA	38.4	75.4	70	11.72	Brine, scCO <sub>2</sub>	Pentland et al. (2011a,b)
Sandstone	OH, USA	38.0	50	22	$\Delta P^h$	Oil, water, brine	Hadia et al. (2007, 2008, 2012, 2013)
Sandstone	OH, USA	38.0	200	28	11	liqCO <sub>2</sub> /scCO <sub>2</sub> , brine, N <sub>2</sub>	Niu et al. (2014, 2015); Al-Menhali and Krevor (2014); Al-Menhali et al. (2015)
Ferric iron-containing sediments		Wide range		<350	<25	scCO <sub>2</sub> + SO <sub>2</sub> , water	García et al., 2014
Sandstone	Germany	476	494.5	40	5–20	scCO <sub>2</sub> + SO <sub>2</sub> , brine	Kummerow and Spangenberg (2011)
Mixtures of CO <sub>2</sub> and H <sub>2</sub> O at various NaCl (aq.), different pressures and temperatures				12–100	<600	Water + scCO <sub>2</sub> , NaCl (aq.)	Spycher et al. (2003); Spycher and Pruess (2005)
Brown coal	VIC, Australia	203	1000	38	6–10 <sup>i</sup>	N <sub>2</sub> , gCO <sub>2</sub> , scCO <sub>2</sub>	Ranathunga et al. (2015)

<sup>a</sup> The confining pressure.<sup>b</sup> scH<sub>2</sub>S denotes supercritical H<sub>2</sub>S; gCO<sub>2</sub> denotes gaseous CO<sub>2</sub>; liqCO<sub>2</sub> denotes liquid CO<sub>2</sub>; aq. denotes aqueous.<sup>c</sup> RT: room temperature.<sup>d</sup> The lengths of samples used for the experiment are 49.6 mm, 54.1 mm, 60.5 mm, respectively.<sup>e</sup> P<sub>atm</sub> refers to the atmospheric pressure.<sup>f</sup> The actual lengths of samples are 101.7 mm and 102.8 mm.<sup>g</sup> The pore pressure.<sup>h</sup> The differential pressure is across the cores, more details see Hadia et al. (2013).<sup>i</sup> The injection pressure.

The laboratory experiments depicted in Table 1 have been used around the world both for academic research and for the oil and gas industry by reservoir engineers. In addition, Table 1 also lists detailed fluid categories and low-permeability rock cores with reservoir fluids in most of the core flooding systems.

Based on the statistical data in Table 1, histograms of core diameter, core length, pressure and temperature, which are the most important parameters considered in the core flooding experiments, are given in Fig. 2. In Fig. 2a and b, the optimal range of core dimension is 20–60 mm in diameter (a proportion of frequency in 79.3%) and 50–150 mm in length (around 83.3% central range of total frequency). As for the pressure and temperature, 10–30 MPa (95% in frequency) and 40–60 °C (about 69.6% in

frequency) are the best interval to choose, as shown in Fig. 2c and d. Thus, combined with geological settings of sampled cores, we should pay more attention to the parameters used (for example, 50 mm in diameter, 125 mm in length of sandstone core under 20 MPa injection pressure, confining pressure of 50 MPa), since the basic parameters determine the success or failure of the whole tests (Spycher et al., 2003; Spycher and Pruess, 2005).

### 3.2. The representative core flooding experimental apparatus

Laboratory experiments such as CO<sub>2</sub>/brine core flooding and imbibition/drainage experiments are essential for observing and analyzing multiphase migration processes and trapping



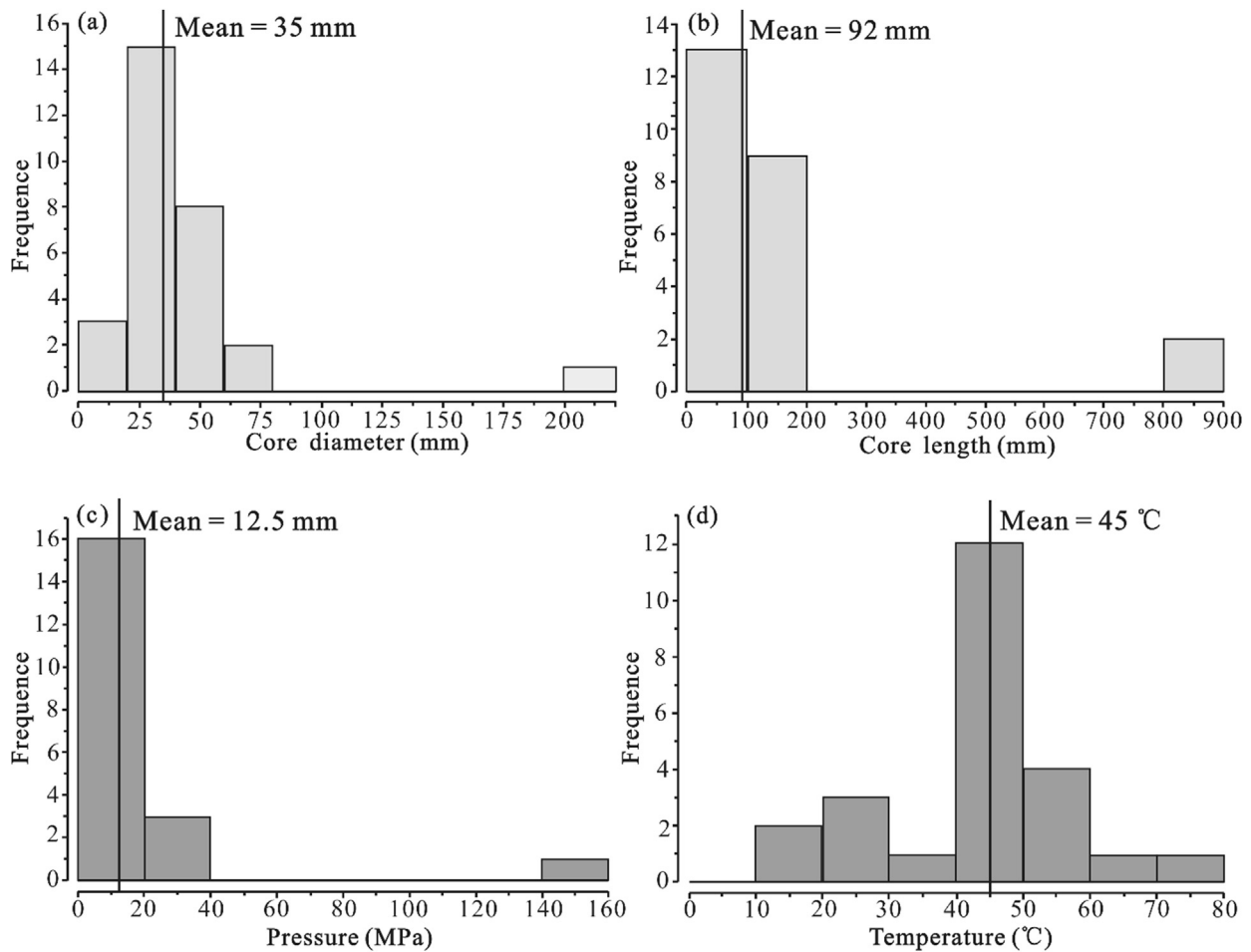


Fig. 2. Histograms of pre-existing experimental condition data, including (a) core diameter, (b) core length, (c) pressure, and (d) temperature for laboratory core flooding.

mechanisms of CO<sub>2</sub> in porous media (Al-Wahaibi et al., 2006; Bennion and Bachu, 2008a, 2010; Perrin and Benson, 2010; Shi et al., 2011a,b). In these experiments, the typical porous specimens can be real rock cores from target brine formations (e.g. Benson et al., 2006; Krevor et al., 2012; Elkhoury et al., 2013) or a packed bed of glass beads (e.g. Cinar et al., 2009; Luo et al., 2011; Xu et al., 2011; Jiang et al., 2015). CO<sub>2</sub> or brine (or their mixture if needed) is continuously injected into the porous media to displace the host fluids, which may be conducted at reservoir pressure and temperature. During the injection, CO<sub>2</sub>/water multiphase migration or CO<sub>2</sub> saturation inside the porous media can be observed or measured by different techniques.

### 3.2.1. Benson's testing apparatus

This setup emphasizes the strong influence of sub-core scale heterogeneities on the spatial distribution of CO<sub>2</sub> at steady state and provides useful relative permeability data on a sample originated from an actual storage site. Perrin and Benson (2010) designed a suite of two-phase core flooding experimental setups (Fig. 3), and experiments were performed through horizontally placed Berea sandstone cores while an X-ray CT scanner was used to image the interior of rock samples and visualize how the whole system behaves when other fluids or gases were flooded through the core. Table 2 lists the main configuration and petrophysical properties of the core samples.

The significance of these two-phase flow experiments with CO<sub>2</sub> and brine lies in using core flooding apparatus designed

independently, associated with an X-ray CT scanner on two heterogeneous rock samples to measure three-dimensional (3D) CO<sub>2</sub> saturation distributions in spatial and temporal aspects during a set of steady-state relative permeability measurements. The results demonstrate that small-scale heterogeneities, particularly low porosity inclusions, can yield a large influence on brine displacement efficiency.

### 3.2.2. Gutierrez's testing apparatus

This experimental study used a laboratory testing system to clarify the acoustic response of rocks to scCO<sub>2</sub> injection under deep saline aquifer conditions (Xue et al., 2003a,b, 2005; Shi et al., 2007; Jakupi et al., 2008; Lei and Xue, 2009; Nakatsuka et al., 2009; Kim et al., 2010; Ghosh and Sen, 2012; Shukla et al., 2012, 2013; Chen et al., 2013; Lebedev et al., 2013; Nakagawa et al., 2013; Cai et al., 2014; Kitamura et al., 2014; Mikhailsevitch et al., 2014; Li et al., 2015b,d). The main component of the system is a high-pressure and high-temperature (HPHT) triaxial cell that allows for injection of CO<sub>2</sub> in core samples of sandstone initially saturated with saline water, as shown in Figs. 4 and 5 (Gutierrez et al., 2012). Tables 3 and 4 list the main conditions and components involved in Gutierrez's core flooding tests.

In the experiment, ultrasonic wave velocity changes due to the change in CO<sub>2</sub> saturation were measured using Berea sandstone core which was initially saturated with saline water and was then subjected to constant CO<sub>2</sub> injection rate. The results demonstrated that the distribution of multiphase pore fluids has some effect on

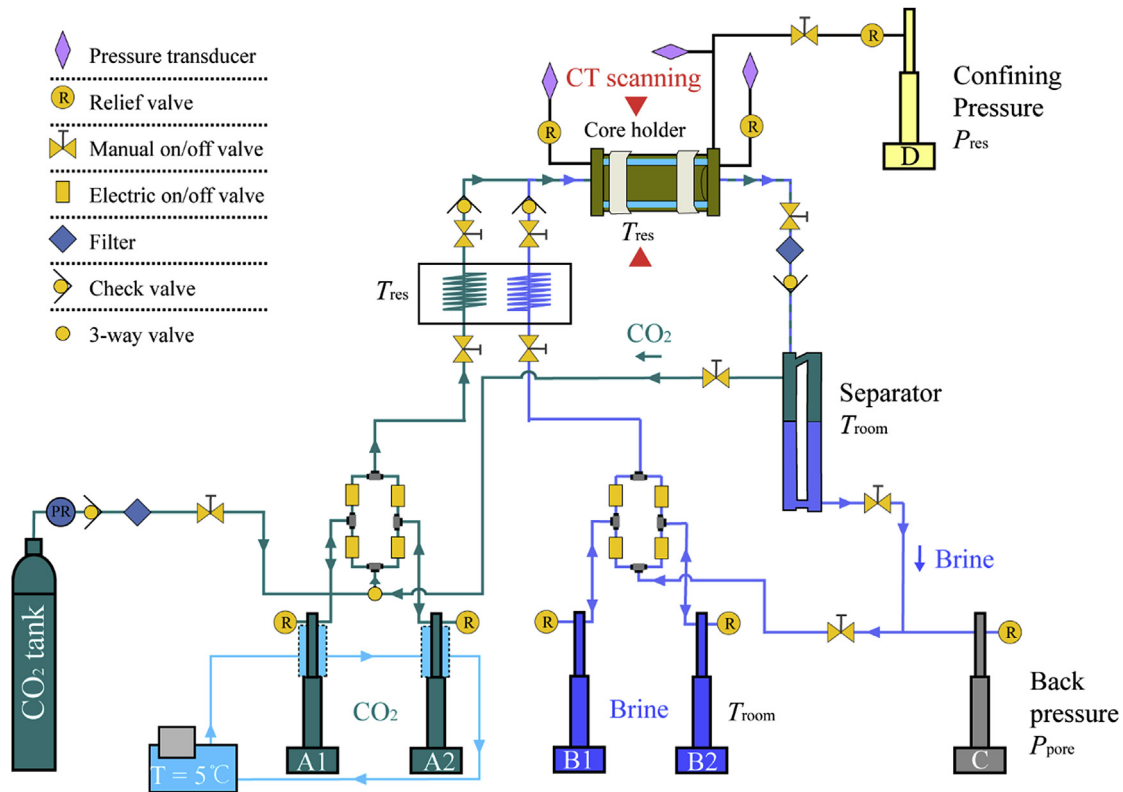


Fig. 3. A schematic of a typical core flooding experimental setup with X-ray CT scanner ( $T_{\text{room}}$ : room temperature, Perrin and Benson (2010)).

Table 2

Properties of the testing apparatus and cores used in Perrin and Benson (2010).

Experimental configurations (also see Fig. 3)				Samples	Core properties			Experimental condition	
Upstream	Core holder	Downstream	Acquisition		Dimension (mm)	$\bar{K}$ (mD)	$\bar{\varphi}$	$T_{\text{res}}$ (°C)	$P_{\text{res}}$ (MPa)
Two dual-pump systems (Pump A1 and A2 for CO <sub>2</sub> and Pump B1 and B2 for brine)	Heat-shrinkable Teflon sleeve, aluminum holder, Pump D for $P_{\text{res}}$ , two electric heaters for $T_{\text{res}}$	High pressure separator and Pump C for $P_{\text{pore}}$	Intelligent transmitter and X-ray CT scanner	Core #1 Core #2	$\phi 50.8, L83.0$ $\phi 50.8, L152.4$	$45 \pm 2$ $430 \pm 7$	0.182 0.203	63 50	12.4 12.4

$P_{\text{res}}$ : Confining pressure.

$T_{\text{res}}$ : Designated reservoir temperature through a heat exchanger.

$P_{\text{pore}}$ : Pore pressure, also back pressure in this configuration, see Fig. 3.

$\bar{\varphi}$ : Averaged porosity.

$\bar{K}$ : Averaged permeability.

the seismic velocity of porous rocks. Increasing CO<sub>2</sub> saturation affected the P-wave velocity which was observed to decrease; whereas the S-wave velocity was almost constant during the CO<sub>2</sub> injection. In addition, these preliminary results confirm that the Biot-Gassmann theory can be used to model the changes in the acoustic P-wave velocity of sandstone containing different mixtures of scCO<sub>2</sub> and saline water, provided that the distribution of the two fluids in the sandstone pore space is accounted for in the calculation of the pore fluid bulk modulus (Lei and Xue, 2009; Gutierrez et al., 2012; Vanorio, 2015).

### 3.2.3. Ranjith's testing apparatus

The 3GDeep-Research Laboratory in Monash University, Australia, includes state-of-the-art facilities for microscale to macroscale testing applied to deep CO<sub>2</sub> geosequestration. To obtain more realistic information on fluid behavior in multi-porous media, 3D X-ray CT can achieve high resolution measurements and is

mainly used for 3D reconstructions of porous structure and fluid distributions in natural and artificial porous media (e.g. Gunde et al., 2010; Perrin et al., 2011; Li et al., 2015d). Major core flooding test specifications designed for investigating CO<sub>2</sub> geosequestration in Dr. Ranjith's group are presented in Table 5.

Like saline aquifers, coal seams have been suggested as potential CO<sub>2</sub> storage reservoirs (e.g. Shimada et al., 2005; Jessen et al., 2007; Mazumder and Wolf, 2008; Connell et al., 2011; Wang et al., 2013; Zhou et al., 2013; Ranathunga et al., 2015). Furthermore, core flooding experiments on intact coal samples provide an opportunity to observe the processes involved in enhanced gas drainage (e.g. Sander et al., 2014; Connell et al., 2015; Masoudian, 2016). For the reasons mentioned above, a macroscale core flooding experimental equipment designed by Dr. Ranjith's group is aimed at understanding the storage potential of coal seams by using reconstituted coal samples of 203 mm in diameter and up to 1 m in length, which are compatible with pressures up to 25 MPa. This

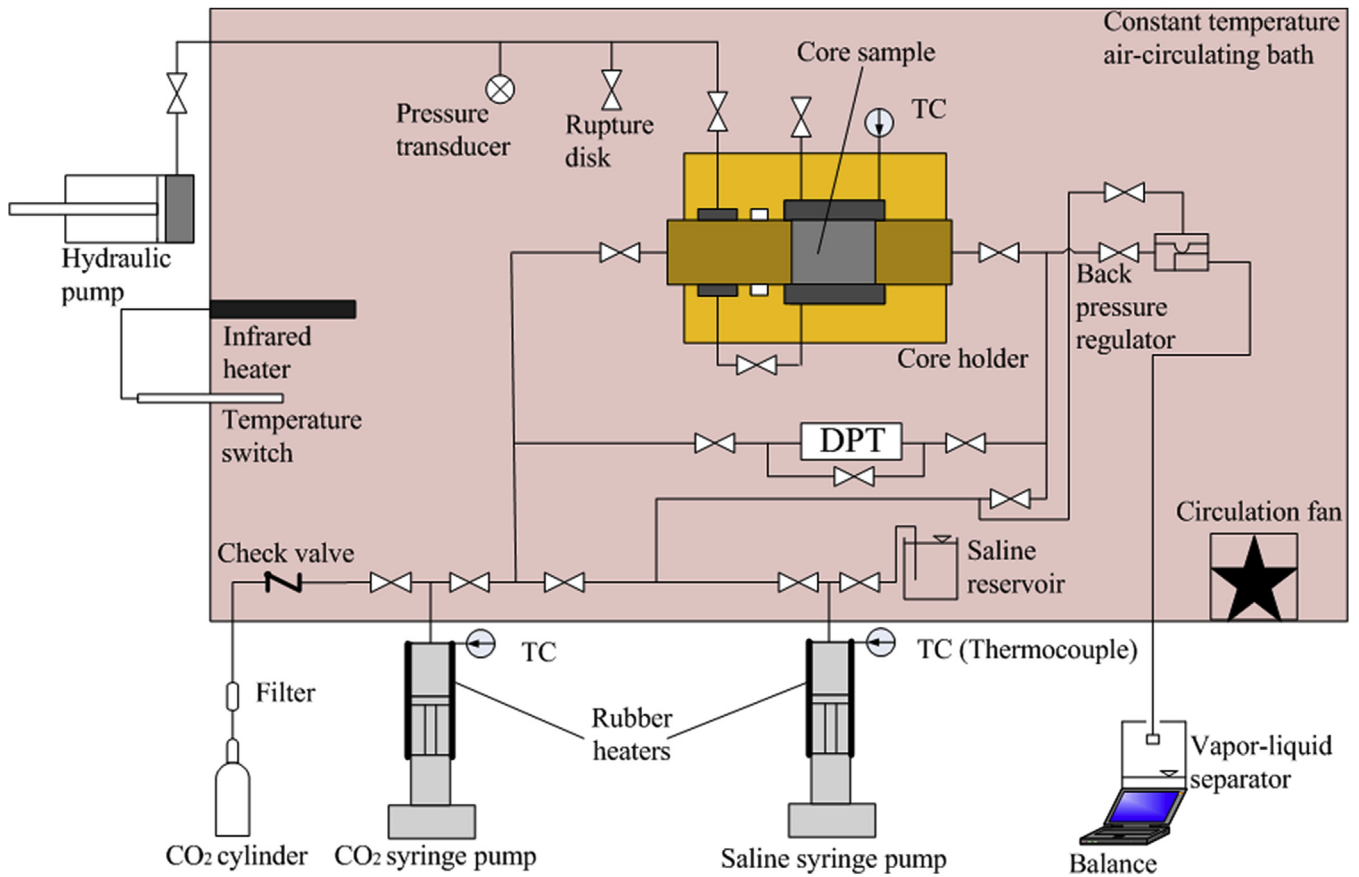


Fig. 4. A testing system for high-pressure and high-temperature testing of scCO<sub>2</sub> injection in fractured porous rocks (DPT: differential pressure transmitter, Gutierrez et al., 2012).

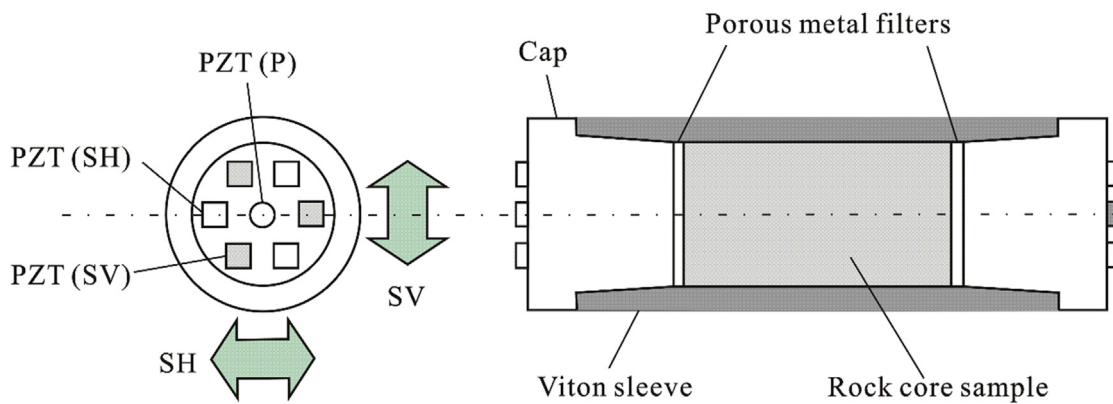


Fig. 5. Arrangement and polarization of piezoelectric transducers (PZT) for seismic wave velocity measurement. Arrows indicate the direction of oscillation of two different shear waves (horizontal “SH” and vertical “SV”), and “P” denotes compression wave (Gutierrez et al., 2012).

equipment can also offer the ability to observe the pore pressure distribution along the core length of the sample. A schematic diagram of the testing apparatus is illustrated in Fig. 6. In this figure, the apparatus consists of four main elements: (1) hydraulic loading system; (2) pressure cell; (3) high-pressure plumbing network, and (4) data acquisition system.

The investigation results of the coal core flooding experimental apparatus are mainly as follows: (1) Due to CO<sub>2</sub> injection, coal samples were swelled and shown to compact longer during the compaction stage. (2) Due to adsorptive weakening, the

compaction process had to be increased by 3–4 days to obtain a stable compaction level prior to CO<sub>2</sub> injection. (3) The coal swelling occurs due to adsorption of CO<sub>2</sub> as reconstituted coal does not contain any cleats or fractures. (4) The CO<sub>2</sub> storage capacity of large coal core samples in laboratory studies is estimated and the permeability variations along the sample length of a coal/sandstone core up to 1 m long are obtained.

Hence, the large-scale coal core specimens ( $\phi 203 \text{ mm} \times L1000 \text{ mm}$ ) under in-situ pressure and temperature conditions might be better to characterize CO<sub>2</sub> geosequestration in depleted or

**Table 3**  
Properties of Berea sandstone and fluid.

Berea sandstone core				CO <sub>2</sub>	Injected saline water saturated with scCO <sub>2</sub>		
Dimension (mm × mm)	Porosity	Permeability (mD)	Dry density (kg/m <sup>3</sup> )	Purity (%)	Salinity (%)	Pore pressure (MPa)	Temperature (°C)
φ38 × L68	0.17	20–30	2200	99.9999	3.4	10	39.85

**Table 4**  
Specifications of the main experimental components.

HPHT triaxial core holder <sup>a</sup>	Seismic wave velocity measurement <sup>b</sup>	
$P_{\text{Max conf}} = 70$ MPa, two syringe pumps, one back-pressure regulator, one hydraulic and differential pressure transducer	PZT	(1) Natural frequency: 250–1000 kHz (2) P-wave, SH-wave, and SV-wave (3) Mounted titanium end caps
Viton sleeve enclosing cores, porous plate installed both core ends to homogenize the fluid flow	Pulser/Receiver	(1) 100–400 V square pulses (2) Typical rise time: $< 1 \times 10^{-8}$ s (3) Maximum bandwidth: 35 kHz
Silicon rubber blanket heaters for $T_{\text{fluid}}$ , on-off infrared heater and air-circulating fans for $T_{\text{system}}$	Oscilloscope	(1) Bandwidth: 100 MHz (2) Vertical resolution: 8 bits (3) Real time sampling rate: $1 \times 10^9$ s <sup>-1</sup>

<sup>a</sup>  $P_{\text{Max conf}}$  denotes the maximum working confining pressure;  $T_{\text{fluid}}$  and  $T_{\text{system}}$  denote the temperatures of fluid and system, respectively.

<sup>b</sup> PZT: piezoelectric transducers.

abandoned coalfield. This research could also guide the field-scale CO<sub>2</sub> storage in deep saline aquifers and enlighten successful development of the devices on core flooding laboratory tests using CO<sub>2</sub>, CH<sub>4</sub>, N<sub>2</sub> and brine.

Similar studies to those of Drs. Sally Benson and Marte Gutierrez have been implemented by Dr. Ranjith's group, who presented the design, development and application of a new multiphase high-pressure and elevated temperature rock hydromechanical testing apparatus for investigation of reservoir and caprock behavior in CO<sub>2</sub> geosequestration projects (Shukla et al., 2012; Rathnaweera et al., 2015c).

The testing apparatus was primarily composed of a true triaxial cell (Perera et al., 2011a,b) and an AE device (Vishal et al., 2015). The former would be applied to support high confining stress, injection pressure and higher temperature to mimic the natural thermo-hydro-geomechanical (THM) conditions of deep underground geological formations (Li and Ito, 2010), and the latter was employed to identify the stress threshold values of crack closure, crack initiation and crack damage for each testing condition during the whole deformation process of the specimens. A detailed description of the apparatus is addressed in the studies (Jasinge et al., 2011; Ranjith and Perera, 2011; Shukla et al., 2012;

Rathnaweera et al., 2014, 2015a,b), with some initial results of the hydromechanical testing under triaxial stresses of rock subjected to water and scCO<sub>2</sub> injection.

### 3.2.4. IRSM's testing apparatus

As mentioned earlier, three categories of representative core flooding experimental apparatuses have respective advantages and applicable conditions, but they are facing some challenging issues such as component complexity, low temperature and low pressure, and electromagnetic interference.

The integrated high resolution laboratory core flooding apparatus (Fig. 7) has been designed for energy storage and acid gas subsurface disposal and is under testing in the Institute of Rock and Soil Mechanics (IRSM), Chinese Academy of Sciences, Wuhan, China (Li et al., 2015b; Sun et al., 2015). This integrated core flooding equipment can couple three monitoring techniques of AE probes, strain gage and fiber optical sensors into typical reservoir rocks in core-scale CO<sub>2</sub>/brine flooding experiments under simulated in-situ P-T conditions. The main objective of this tailored system is to continuously seize the front of CO<sub>2</sub> plume migration during the coupled process of scCO<sub>2</sub> displacing brine in brine-saturated sedimentary core samples.

**Table 5**  
Summary of related core flooding testing facilities used for investigating scCO<sub>2</sub> subsurface geologic storage (Source: [www.3gdeep.com](http://www.3gdeep.com)).

Properties	HPHT test rig	Shale gas/CBM rig	Core flooding system	3D X-ray CT
Capabilities <sup>a</sup>	Diameter: φ200–500 mm Length: L400–1000 mm $T = 100$ °C $P_{\text{pore}} = 100$ MPa $\sigma_r = 70$ MPa $\sigma_a = 2000$ kN	Diameter: φ50–200 mm Length: L100–1000 mm $T = 100$ °C $P_{\text{pore}} = 50$ MPa $\sigma_r = 32$ MPa $\sigma_a = 1000$ kN	Diameter: φ25–38 mm Length: L50–1300 mm $T = 150$ °C $P_{\text{pore}} = 40$ MPa $\sigma_r = 40$ MPa AE and P & S waves	Diameter: φ5–50 mm Length: L50–1300 mm $T = 200$ °C $\sigma_r = 15$ MPa $\sigma_a = 15$ kN Resolution: 700–0.70 μm
Major applications <sup>b</sup>	AE and P & S wave THMC measurement Wellbore stability Fault simulation Hydraulic fracturing EOR ECBM CO <sub>2</sub> geosequestration	$P_{\text{pore}}$ monitoring Hydraulic fracturing Tight gas stimulation Rock breakage Wellbore stability Well casing Fault simulations EOR	Shale gas CBM CO <sub>2</sub> geosequestration Relative permeability measurement Multiphase flow High acidic environment testing	THMC load stage 3D reconstruction Pore structure Microscale observation of flow path and pore connectivity Pore collapse during the loading
Research scale	Macroscale	Macroscale	Mesoscale	Microscale

<sup>a</sup>  $P_{\text{pore}}$  denotes the pore pressure;  $\sigma_r$  denotes the lateral stress;  $\sigma_a$  denotes the axial loading; AE: acoustic emission.

<sup>b</sup> THMC: thermo-hydro-mechano-chemical; EOR: enhanced oil recovery; CBM: coalbed methane; ECBM: enhanced coalbed methane recovery.



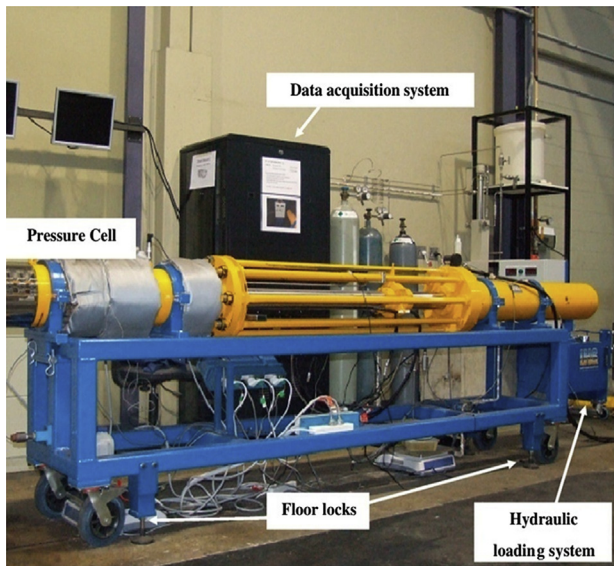


Fig. 6. Core flooding apparatus for investigating CO<sub>2</sub> storage in the coal seams (de Silva and Ranjith, 2013).

#### 4. Research gaps and future work

Having a good knowledge of CO<sub>2</sub>/brine multiphase migration processes in the subsurface is critical for evaluating storage capacity and security of potential CO<sub>2</sub> storage sites (Bachu, 2008; Ghedan, 2009; Wang et al., 2015b). Although there is a fairly large amount of apparatus available for studying scCO<sub>2</sub>-brine-rock interactions and multiphase flow migration in deep saline aquifers, several gaps and challenges in understanding of the sequestration security involved in CO<sub>2</sub> geosequestration projects from a laboratory core-scale perspective still exist, and thus continuous improvement is required in the future (Lv et al., 2015).

Based on existing research and experimental experience, an array of gaps and required future work in the understanding of multiphase core flooding tests are listed as follows:

- (1) Absence of real-time dynamic monitoring devices used for simultaneously capturing the migrating front of CO<sub>2</sub> plume within rock specimens. Laboratory experiments outfitting this device are required. Without the capability to accurately acquire the dynamic information from the CO<sub>2</sub>-brine-rock interaction, under in-situ HPHT conditions, and any consequent chemical and mechanical changes, there can be some uncertainty with regard to the long-term fate of scCO<sub>2</sub>.
- (2) For laboratory core flooding experimental apparatus applied to simulate the process of CO<sub>2</sub> geosequestration in deep saline aquifers or depleted hydrocarbon reservoirs, the corrosivity of the dissolved CO<sub>2</sub> in the rock samples may weaken the material performance and lifetime span, as well as cause the potential leakage of the equipment. More research is thus required in order to elevate the sealing capacity of the core holder to study the experimental material with HPHT resistance, anti-corrosion and long life cycle.
- (3) Better converting the results of laboratory-scale displacement tests and the urgent issues confronted by pilot- and/or field-scale CO<sub>2</sub> geosequestration projects is needed.
- (4) CO<sub>2</sub> geosequestration in deep saline aquifers is an extremely complicated process involving in high temperatures (T), formation water flow (H), mechanical stability (M), chemical reaction (C), biological activities (B) among other factors (e.g. Li et al., 2006; Vilarrasa et al., 2010; Yang et al., 2010; Hou

et al., 2012; Kolditz et al., 2012; Zhou and Burbey, 2014). Although previous studies have focused on one or more factors using numerical modeling, very few studies are conducted on core flooding experiments in view of THMC/THMCB coupling effects. Experimental improvement and theoretical developments to simulate porous media are needed in the future.

- (5) Traditional measuring instruments such as PZT, strain gage, and electrical sensors are not sufficient to satisfy the required conditions of laboratory displacement testing. Novel measuring devices, such as optical fiber sensor (OFS), that possess the performance of immunity to electromagnetic interference and corrosion, and embedding capability (Sun et al., 2015) could be ideal for elaborate monitoring of the CO<sub>2</sub> flow in the core's pore space and meso/microscale rock fractures.
- (6) Better experimental techniques are needed to investigate the fate of the injected CO<sub>2</sub> reservoir, which can take into account the multiphase flow of CO<sub>2</sub> and brine, the effects of stress on permeability, and the dissolution and chemical interaction of the CO<sub>2</sub> with the rock minerals. New testing techniques associated with conventional geomechanical experimental facilities will be the mainstream direction. For instance, Dr. Ranjith's group (Ranjith and Perera, 2011; Rathnaweera et al., 2014) presented a new high-pressure triaxial apparatus which could provide the high confining and fluid injection pressures and elevated temperatures expected for deep CO<sub>2</sub> geosequestration. This could be coupled with an AE system implanted to identify crack closure, crack initiation and crack damage for each testing condition during the whole deformation process of the specimens.
- (7) Due to the long-term scale and multiscale storage of trapped CO<sub>2</sub>, the temporal and spatial effects may require conducting tests on samples collected from various locations to study the spatial variation of the pore fluids and the host rock induced by CO<sub>2</sub> injectivity, and how they change with time. For example, Dr. Benson's laboratory designed a steady-state 3D core flooding displacements setup with X-ray CT scanner performed in heterogeneous cores, over a range of relevant conditions, to study the impact of sub-core heterogeneity on 3D dynamic CO<sub>2</sub>/brine flow processes (Kuo and Benson, 2015).
- (8) A general lack of a set of appropriate specifications from experimental preparation to final interpretation. Experimental specification is substantially needed to instruct and improve future core flooding testing system. As stated previously from statistical histograms (see Fig. 2), the paper pointed out the optimal intervals of core dimension, pressure and temperature, which will be helpful for reasonable design of future experimental apparatus.

#### 5. Concluding remarks

Owing to potentially reducing CO<sub>2</sub> emissions into the atmosphere, CO<sub>2</sub> geosequestration has attracted a significant amount of research interest. Investigating CO<sub>2</sub>/brine multiphase migration processes is crucial for evaluating storage capacity and potential security of CO<sub>2</sub> geosequestration sites.

Core flooding is a technique used to conduct experiments on core samples simulated in conditions close to the natural environments. Using the results of core flooding experiments, researchers can predict how different fluids or gases would move through the sampled area. This paper presents an overview of laboratory core flooding experimental systems mainly for CO<sub>2</sub>

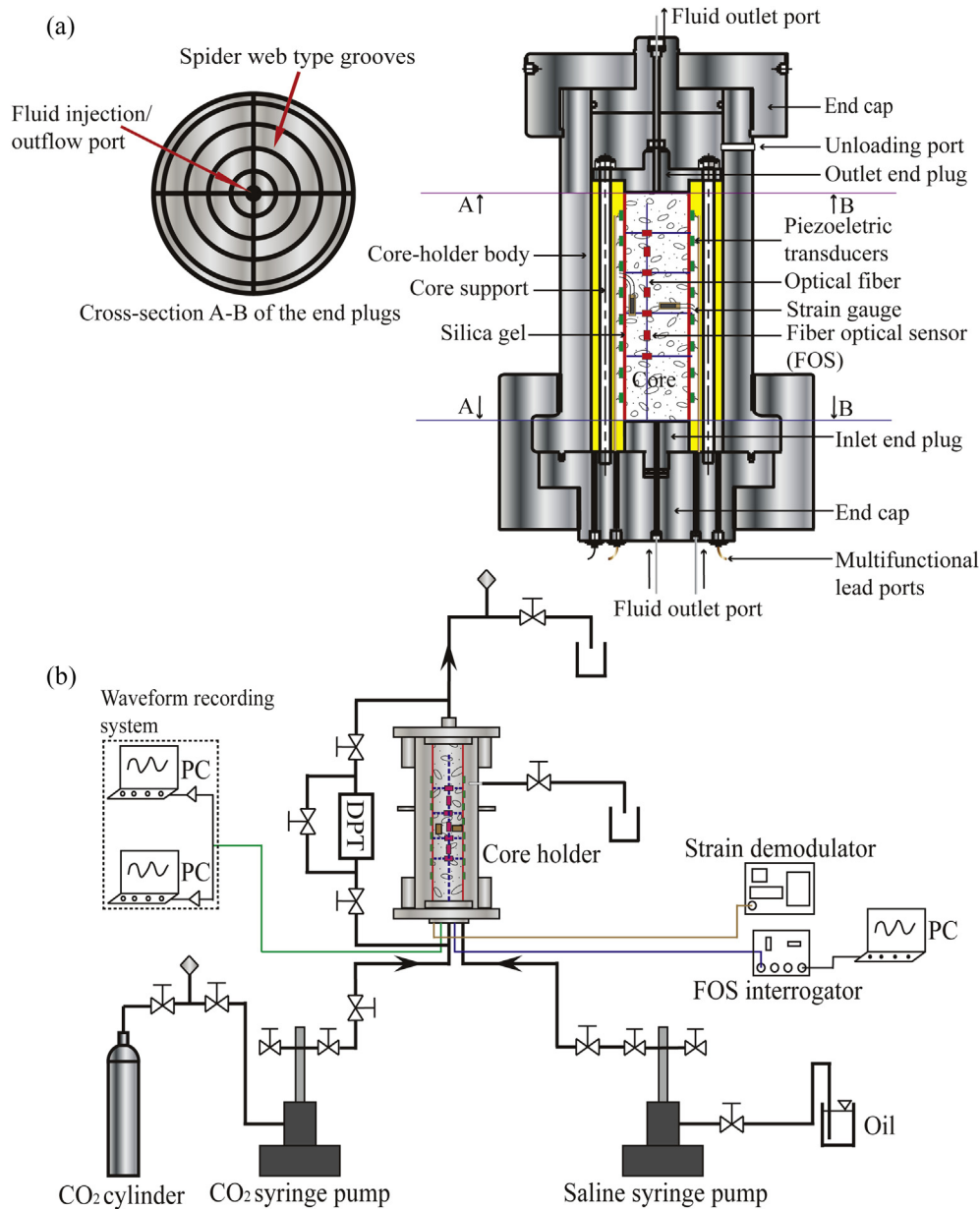


Fig. 7. Schematic diagram of core-holder assembly (a) and conceptual sketch of core flooding experimental apparatus (b) in the Institute of Rock and Soil Mechanics (IRSM), Chinese Academy of Sciences, Wuhan, China.

geosequestration, including various trapping mechanisms involved, with a summary of three kinds of up-to-date displacement apparatus reported.

In order to implement more accurate studies of CO<sub>2</sub> migration in saline aquifers, comprehensive experiments using elaborate tools should be conducted under the conditions representative of natural reservoir conditions. For example, coupling OFS and AE probe with the specimen in the HPHT core holder or triaxial setup can be used to investigate core flooding experimentally. Furthermore, 3D imaging techniques such as photo luminescent volumetric imaging, magnetic resonance imaging (MRI) (e.g. Ma et al., 2013) and X-ray CT (e.g. Zhang et al., 2013) are desirable for measuring steady and unsteady multiphase flows in porous media.

Overall, understanding multiphase fluid flow in multi-scale reservoir formations is a challenging issue for geoscientists and

engineers in laboratory experiments and field projects across the world.

#### Conflict of interest

The authors wish to confirm that there are no known conflicts of interests associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

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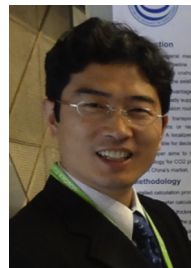
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