Xuehao Liu

Visit to the Australian School of Petroleum, University of Adelaide

September to November 2011

CAGS Report

1. Core Flood Facility at ASP

1.1 Motivation

One of the large uncertainties associated with the injection of CO_2 during the life of a carbon storage project is the effect that chemical reactions will have on the mechanical strength of the formation, both in the short and long term. It has been suggested that these reactions may accelerate mineral dissolution and crack propagation processes thereby altering the mechanical strength and integrity of reservoir and cap rock even on relatively short time-scales.

Thus our motivation to carry out our core flooding experiments was to explore how CO_2-rich fluids react with typical reservoir and cap rock and then to determine whether these reactions have had any observable effect on mechanical properties. One of our goals was to try to determine whether or not mechanical failure of reservoir and cap rock is a possibility during CO2 injection operations and long term storage.

We had planned to use the core flood facility at the University of Adelaide, using experimental conditions of in the pressure range of 10-25 MPa and temperatures of up to 200 °C.

1.2. How far have we come?

Due to continuous technical problems (which still needed repair at the end of my stay at the ASP) we only achieved one very short (a few minutes lasting) core flood. However, I was able to draft a simplified sketch of the core flood facility and set-up, to give future users a simplified guide on how to use the facility.

I was asked to perform a review on core flood experiments and outcomes available in the literature, which is presented in Section 2 of this document, and I also put together a data base, which summarises the main motivations, and set ups used for core flood experiments in Australia and overseas, as well as results. An example “output” of this database is given in Appendix A.

2. Literature Review on Core Flood Experiments

2.1 Background Information on the Literature Review

This literature review focuses on research published on core flood experiments, as well as other experiments to study CO2 -water-rock interactions. It is based on 29 papers mainly published between 2000 and 2011.

2.2 Introduction

In the CO_2-water-mineral reaction system, dissolved CO2 chemical and mechanical alters properties of the rock, which can affect the long term safety of CO2 storage. Thus an improved understanding on the alteration of e.g. the pore structure is needed, as mineral dissolution and/or precipitation processes can significantly alter the porosity and permeability of the rock. Core flood experiments are one way to study these processes, and they are also helpful to understand multiphase flow of supercritical CO2 (with or without impurities) and brine in high pressure and temperature environments.

For the papers included in this literature review, the main reasons to carry out core flood experiments can be summarised as:

*Predict the spatial and temporal distribution of CO2 saturation.

*Improve our understanding on pore (mm) and core-scale (cm) physics, such as the alteration of pore structure during dissolution and precipitation processes, as well as their effect on porosity and permeability.

*Investigate fracture evolution in the tested rocks

*Clarify different mineralogical changes induced by CO2 and then establish a suitable chemistry model for numerical simulation of the CO2 storage process.

2.2. Methods and Experimental Set-Ups

The members of the CO2SINK group in Germany carried out quite comprehensive studies in CO2 -water-rock interactions both on reservoir rock and cap rocks, and obtained considerable experimental results in geophysics and mineralogy, as well as even some insights into microbiological processes. Their "CO2 exposure experiments", were set up as CO2 gas -synthetic brine-sandstone (1-100mD) batch experiments, at experimental conditions of 40°C and 6 MPa. (Amann et al., 2011; Fischer et al., 2010 and 2011; Wandrey et al., 2011a & b; Wigand et al., 2008; Zemke et al., 2010).

The different experimental stages included the following activities:

*Phase I: brine was injected into the dry CO2-saturated sample.

*Phase II: pressure was applied the brine-saturated core.

*Phase III: CO2 was injected into the brine-Saturated core.

*Phase IV: pressure loading after the CO2-flow phase.

At Stanford University researchers carried out core flood drainage experiments using a CO2 fractional flow and synthetic brine. Mainly sandstone rock core samples were used, such as Berea sandstone (USA) and samples from the Otway Basin well CRC-1 (Australia). Pressures of 12.4 MPa and temperatures of 63°C and 50°C were applied to their experimental set up (Krause et al., 2011; Perrin et al., 2009; Perrin et al., 2011). Berea sandstone rock core material was also used by Shi et al. (2009 and 2011) of the Imperial College in London. They used scCO2-saturated brine as well as temperature and pressure conditions of 40 °C and 10 MPa.

Ellis et al. (2011) of Princeton University performed core flood experiments to investigate fracture evolution of a fractured carbonate caprock during simulated leakage of CO2-acidified brine (pH 4.9). Experimental pressure and temperature conditions were 40°C and 10 MPa, which corresponds to an injection at a depth of 1 km.

Lin et al. (2008) investigated effects of differently composed fluids and scCO2 on pure minerals such as quartz, biotite and granite. Comparisons were made between water or water vapour or no fluid at 100°C and 10 MPa and after an experimental run time of 96 hours. In contrast Shiraki and Dunn (2000) concentrated on the impact that different brine composition could have on reservoir characteristics. Samples used were derived from the Tensleep Formation, Wyoming, USA. CO2 gas and sandstone interaction were investigated using formation brine and synthetic brine (NaCl), at 80°C and 166 bars.

Rosenbauer et al. (2005) focused on CO2 storage in a carbonate aquifer based on an on-site project, which investigated scCO2-saturated brine (formation water and synthetic brine)-rock (limestone/ sandstones) interactions, at 25 °C and 120 °C and a pressure range of 100 to 600 bar.

Kharaka et al. (2006) also performed much vital work in CO2-water-rock interactions. They used a CO2 gas-formation brine-sandstone set up at 65 °C and 150 bars, conditions that are based on the Frio project in Texas, where 1600 tons of food-grade refineries CO2 were injected into a 24-m sandstone zone of the Oligocene Frio Formation.

In summary, the investigation of CO2-water-rock interactions in core flood experiments usually focuses on reservoir rocks (such as sandstones) and caprocks (e.g. carbonate rocks), to test their integrity or fracture behaviour. Injection or flooding medium in most cases is supercritical CO2. Other gases used are or mixed gas (e.g. SO2, O2), water or water vapour. In addition formation fluid or synthetic brine was both used. The temperature and pressure conditions used range from 25°C (room temperature) to 120°C and pressures of up to 60 MPa. However, most experiment simulate reservoir conditions, with temperatures ranging from 40°C to 65°C and pressures of 10 MPa to 16.6 MPa.

2.3 Results

2.3.1 Geochemistry

Some experiment results tend to provide no clear hints for chemical alteration in CO2-brine-sandrock interaction, because the alteration time even 24 months is still too short (Wandrey et al., 2011a; Lin et al., 2008). However, two major reactions are often described such as the dissolution of cement (carbonate mineral) and framework minerals (feldspar). In some cases dissolution of the carbonate cement such as dolomite and anhydride results in the precipitation of anhydride as secondary phase (Fischer et al., 2010 and 2011). The alteration of framework minerals such as K-feldspar and plagioclase, however, in most cases resulted in the formation of clay minerals (kaolinite) and the precipitation and sometimes albite (as secondary plagioclase). These dissolution processes often caused an increase of dissolved Ca, K, and Mg concentrations in the solution (Wandrey et al., 2011a). In addition, Kharaka et al. (2006) also described a significant increase in dissolved Fe and minor increase in Mn concentrations, which is due to the composition of the sandstone used in this particular case.

Lin et al. (2008) found that an increase of dissolved Ca concentration in the brine is one of the most significant results using granite as CO2 injection target. In this study increased Al concentrations in the brine where another result of silicate weathering, however, concentrations did strongly depend on the pressure conditions applied to the experiments.

Isotopic (δ^{13}C, DIC, δ^{18}O) studies carried out by Assayag et al. (2009) indicated that the quantitative dissolution of carbonate cements tends to neutralise the slightly acidic CO2 bearing brine. This study helped to 'visualise' the mixing of the injected fluid with the formation water as well as cation exchange processes and dissolution of silicate minerals.

While Rosenbauer et al. (2005) studied the effect of differently composed brines during the CO2 injection into limestone, they discovered that low sulphate brine resulted in an increased rock porosity of 2.6% at (at 20˚C and 100 bars). However, alteration effects were much more significant during experiments using high sulphate concentration in brines (with and without scCO2), as they were also characterised by the precipitation of anhydrite and the dolomitisation of the limestone. This effectively resulted in an decrease of porosity of up to 4.5% (at 120˚C and 600 bar).

Other effects on of CO2-water-rock interaction on the brine composition than changes in major ion concentration were summarised by Kharaka et al. (2006). They described an increase in electrical conductivity, a sharp drop in pH (6.5 to 5.7) and high increases in alkalinity (from 100 to 3000 mg/L as bicarbonate) when they simulated the injection of brine into a Frio Formation sandstone reservoir.

The effect of CO2 on the microbiology present in an injection reservoir was studied by Wandrey et al. (2011b). A 16S rDNA (PCR-DGGE) based fingerprinting method was used to identify the dominant species in DNA extracts of pristine sandstone samples. They only found a small number of microbes, which use ferric iron or nitrate as terminal electron acceptors during anaerobic respiration. During CO2 exposure slight changes in the microbial community were observed, however, majority of the microbes were able to adapt to this changed environmental conditions.

2.3.2 Geomechanics

CO2-(water)-mineral interactions can induce the alteration of a rock's porosity. Important experimental findings published by Fischer et al. (2010 and 2011); Rosenbauer et al. (2005); Wandrey et al. (2011a) and Zemke et al. (2010) show that both magnitude and direction (e.g. increase or decrease) of porosity changes are a function of geochemical reactions (e.g. Fischer et al. (2011). Porosities increased from 23% to 25% by a loss of solid sample material in the order of 1 to 2 %). However, the authors listed above concluded, that to be able to build a quantitative relationship between porosity and geochemical reactions more evidence from experimental, field as well as simulation work is needed.

Studies by Perrin et al. (2009 and 2011) focused on the spatial and temporal distribution of CO2 saturation, and found that it is strongly correlated to the variation of porosity and permeability. Besides that it also dependents on flow rate and gravity effects, which should be considered for low injection flow rates in a homogeneous as well as heterogeneous cores. Similar results were published by Shi et al. (2009 and 2011). Those works are based on simulations using computer codes such as TOUGH2 (see Section 2.3.3).

Geochemical reactions can not only change porosity, but can also have a significant effect on permeability, or even change the crack propagation processes. Work by Shiraki and Dunn (2000) and Wollenweber et al. (2010) found that permeability was altered due to kaolinite crystal growth occurring in pore throats and/or the dissolution of authigenic cements. Ellis et al. (2011) show that fracture aperture increased 2.7 times in average, primarily to calcite dissolution (artificially fractured limestone flow for one week at 40°C, 10 MPa and pH of 4.9. A review of studies on CO2 sequestration and cap rock integrity by Shukla et al. (2010) pointed out the need of research on fracture sealing or cap rock strength deterioration in relation to weathering of rock minerals in the long term.

A lot of geophysical properties were also analysed. Fischer et al. (2010 and 2011) carried out CO2 (gas)-brine- sandstone flow experiments for 24 months and found that resistivity and P-wave velocity was dependent of pore fillings, while no significant changes occurred for velocity, shear wave velocity and resistivity after brine only flow experiments.

2.3.3 (Geochemical) Modelling Studies

The injection of dry scCO2 into brine aquifers and salt precipitation due to evaporation effects can impair injection rates, two processes that were confirmed by core flood experiments (e.g. the experimental results published by Wang et al. (2009 and 2010). Using a TOUGH2/ECO2N and a streamline-based simulator Muller et al. (2009) proved that pre-flushing the formation with fresh water for a short period of time prior to CO2 injection is one of the most effective engineering solutions for this problem.

Andre et al. (2006) used FRACHEM, a THC computer code, to simulate on site water–rock interactions on fracture permeability of the deep reservoir near the production well. In line with the above results, it also shows that brine dilution seems to be an efficient method for maintaining the hydraulic properties of the fractured reservoir. As well as calcite dissolved within the first 50m around the injector and towards the production well first, then precipitation of silicates and aluminosilicates near the injection well.

To show temperature and pressure effects as well as to determine the impact of water-rock-gas interactions Gaus et al. (2004) used PHREEQC2.8, to build a geochemical model reproducing the observed on-site effects from Montmiral (Southeast Basin, France, 100°C, 36 Mpa, 2400m) and Messokampos (Florina Basin,Greece, 25°C, 0.5 MPa, 300m). The impact of the reactions on the reservoir mineralogy is much larger in the case of Montmiral than for Messokampos as a result of the elevated temperature, implying that reactions occur orders-of-magnitude faster than in the case of Messokampos.

Wigand et al. (2008) also used PHREEQC to estimate geochemical effects. They found that most of the major (e.g. Ca, Mg, Fe, Mn) and trace elements (e.g. Sr, Ba, Pb) of the sandstone increase in concentration during the reaction with brine and SCCO2. These investigations were supported by SEM which indicates dissolution of dolomite cement, K-feldspar, and albite. However, they also point out that this geochemical model did not yield any precipitation and lacked adequate cation-exchange capacity to explain some compositional (Ca, Mg) trends.

2.4 Conclusions

The studies/papers presented above conclude that geochemical as well as geomechanical effects during CO2-water-rock interactions depend very much on site specific circumstances such as temperature, pressure, brine and rock composition.

3. Natural Analogue Studies for CO2-Storage

Using standard microscopy and Scanning Electron Microscopy (SEM) I was given an overview on the basic geochemical processes to expect during CO2-water-rock interactions (and storage) by Ulrike Schacht. In total 10 thin sections and 4 SEM samples were examined. Samples represented reservoir and cap rocks stemming from the Otway Basin and Gippsland Basin, both SE Australia.

The reservoir rock examples were sandstone samples derived from the CRC-1 and Haselgrove-2 wells of the Otway Basin as well as the Kipper-2 Marlin-4 wells of the Gippsland Basin. Quartz was the most abundant component within all samples investigated. Feldspar was common but mainly consisted of K-Feldspar, as plagioclase was only detected in Haselgrove-2 samples. Other components included mica (mainly muscovite with some biotite), pyrite and accessory minerals such as heavy minerals (zircon and tourmaline). The most common products of diagenetic reactions were quartz overgrowths as well as kaolinite and traces of illite filling primary pore space. Carbonate cement was present in some samples (e.g. Haselgrove-2), but amounts were only small.

Likely CO2-rock interaction in reservoir rocks as described above will involve feldspar and mica, as well as the dissolution of carbonates. However, CO2-induced diagenetic reactions will be minor, as this type of rock is chemically not very reactive. This is very good for CO2 injection, as geochemical processes will not interfere with this process.

The caprocks investigated, again stem from CRC-1 core of the Otway Basin as well as Kipper and Marlin core of the Gippsland Basin. In contrast to reservoir rocks, caprocks were very fine grained, and mainly consisted out of shale interbedded with silt layers. Pyrite was very abundant in some cases. We could also observe quartz, minor K-feldspar, mica. Carbonate cement mainly consisted of siderite as very fine grained cement.

These caprock observations are very good for CO2-storage. The much lower porosity will slow down any vertical migration of CO2 and the bigger amount of labile minerals (carbonate, clay) will provide the cations necessary for the mineralogical storage of CO2.

These studies helped me to I gain a much better understanding in CO2-water-rock interactions, which will be very helpful for my further research, which is to study coupled chemical-mechanical processes in carbon storage.

4. ‘Footnotes’

Shortly after my arrival to Adelaide and ASP I attended an organised tour for visitors to the school and was given a full overview about the laboratory facilities and the teaching activities at the Australian School of Petroleum. This tour was followed by an introduction to the South Australian Virtual Reality Centre (SAVRC), which is a state-of-the-art fully-immersive 3D visualisation facility, used for analyse and interpret seismic data, well logs, reservoir pressure and rock saturation data simultaneously, "walking through" a 3D representation of underground rock structures. The SAVRC was presented by Ian West.

Throughout my stay at the ASP I participated in the monthly held CO2CRC group meetings – in which all the participating researchers (including myself) give a short overview about work their current research activities and latest achievements.

I also was given an introduction to Mercury Injection Capillary Pressure (MICP) measurements by Ric Daniels and Delise Hollands. This technique is used to analyse capillary pressure and pore network characteristics of reservoirs, cap-rocks, fault seals and intra-formation seals. This technique is very useful for my own geomechanical research, as I am now able to interpret capillary pressure results derived by two different methods: MICP as used at ASP and gas breakthrough experiments as conducted at my home institution IRSM.

Throughout my stay I attended a few University of Adelaide seminars, such as the seminar on:
"Today's opportunity in energy efficiency". The presenter of this seminar, Jim Poss (Founder and Chief Technology Officer of BigBelly Solar), presented a range of economically and environmentally friendly, as well as energy efficient opportunities for fuel production. He also gave an overview on the progress we could make in the next 1-2 decades in using these new opportunities.

During my last week at ASP I gave a presentation to the group of CO2CRC researchers at ASP – summarising the research carried out at my home institution and my own research interest (see Appendix B).

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Appendix A

Example output of the core flood experiment database created during my stay at the University of Adelaide.

Reference	Motivation	Experiments	Experimental / Simulation Conditions	Sample Material	Analytics / Methods / Hypothesis
Amann et al., 2011	caprock sealing efficiency and potential petrophysical and mineralogical changes of caprock integrity due to CO2 exposure are being investigated	the sorption experiments; the fluid flow measurements	*grain size fractions of 125 to 1000 µm for the sorption tests. <25 MPa, 75 °C, He and CO2. * plane-parallel sample plugs, d 28.5 or 38 mm, thickness ranges 10 to 30 mm. Pressures between 8 to 12 MPa	caprock such as shale, mudstone from 5 different place including shell, CO2SINK and Mont Terri.	XRD, TOC/TIC, N2-BET
Andre et al., 2006	FRACHEM, a THC computer code, was developed specifically to predict changes in the geothermal reservoir of the Soultz-sous-Forˆets (EGS) located in Alsace, France.	FRACHEM, a thermo-hydraulic-chemical coupled computer code	initial reservior T set at 200 ◦C, the fluid of a constant 65 ◦C was injected into the modelled fractured zone. Hydrostatic pressure 8MPa	three granites samples,*1) is fresh granite, low fracture density, porosity close to 0 *2)fractured, hydrothermally altered granite, *3)facies is the most altered	*Kinetic model was adopted for the dissolution/precipitation reactions of minerals. * three possible K-Φ models (Kozeny-Carman); *Assumed is no diffusion between the fractured zone and matrix, all the chemical reactions occur in the fractured zone

Appendix B

Handout of the presentation given to the CO2CRC team at the Australian School of Petroleum in November 2011.

IRSM CCS Group Introduction

Institute of Rock and Soil Mechanics
Chinese Academy of Sciences

- Xuehao
- Supervisor: Ulrike Schacht

THE UNIVERSITY OF ADELAIDE AUSTRALIA

- Provided by the CAGS project

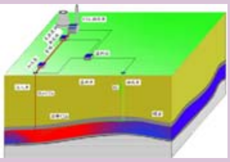
cags China Australia Geological Storage of CO2 中澳二氧化碳地质封存

Institute of Rock and Soil mechanics (IRSM)
*Managed by Chinese Academy of Sciences (**CAS**).

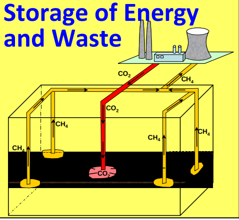
*Focus on geomechanical and technical solutions for environmental geotechnical engineering.

John A Hudson, Xia-Ting Feng, ISRM

CO$_2$ geological storage



Underground Storage of Energy and Waste



Mine slope design

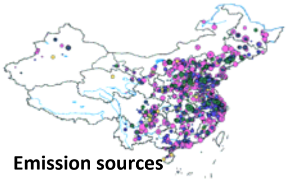
IRSM CCS Group

Prof. Li XC

- **Established in 2005**
- **Staff: 24**
- **Graduate student: 14**
  - Postdoctoral: 2
  - PhD graduates: 6
  - Master's graduates: 6

Prof. Li Qi

The first CCS-GIS Database of China



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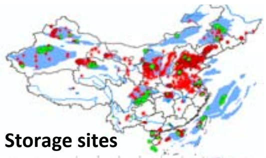

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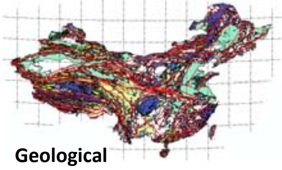
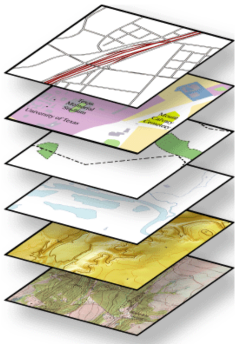
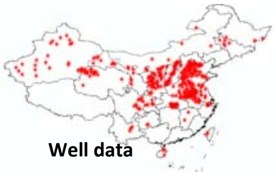


Geological

GIS database sequence structure





Well data

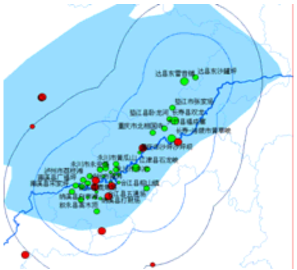
In situ stress

Theoretic capacity → **Source-sink matching** → **Early opportunity**

***Theoretic capacity *Source-sink matching *cost curve**

***Site selection *Safety analysis**

Early opportunity



Chongqing ammonia industries

+ depleted gas fields

*Technical support for Shenhua CCS Demonstration Project 100,000 t/a.

*Site selection for CCS projects from China Power Investment and Total.

Jiangsu IGCC system

+ Jiangsu oil fields

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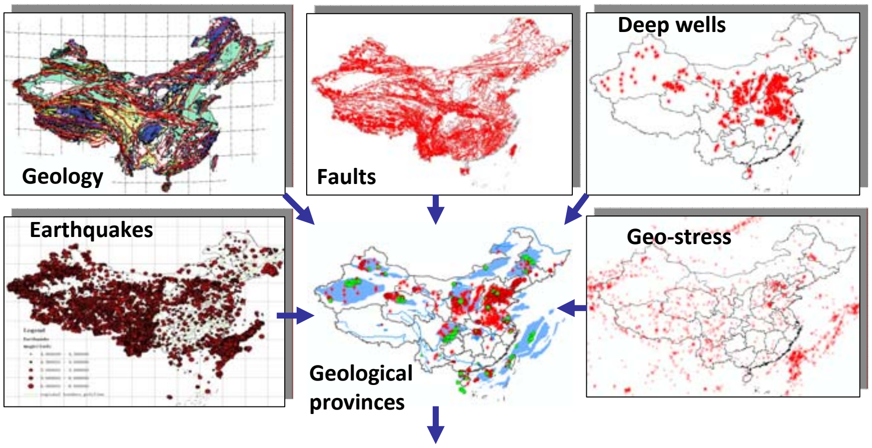
2. Mechanical stability of CO2 storage

Conditions and mode of instability



Understanding initial stability and conditions of instability by projects and natural analogy

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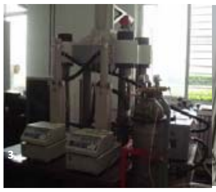
The host of the State Key Laboratory of Geo-mechanics and Geo-technical Engineering

- **Fracture pressure of cap rock**
- **Capillary pressure testing apparatus**
- **Flow-reaction-mechanics coupling testing apparatus   Core Flooding System**

- Mechanical testing apparatus (such as TTT)
- Micro-scope mechanics-flow testing system
- Improved pulse decay permeability testing apparatus and so on.


1


2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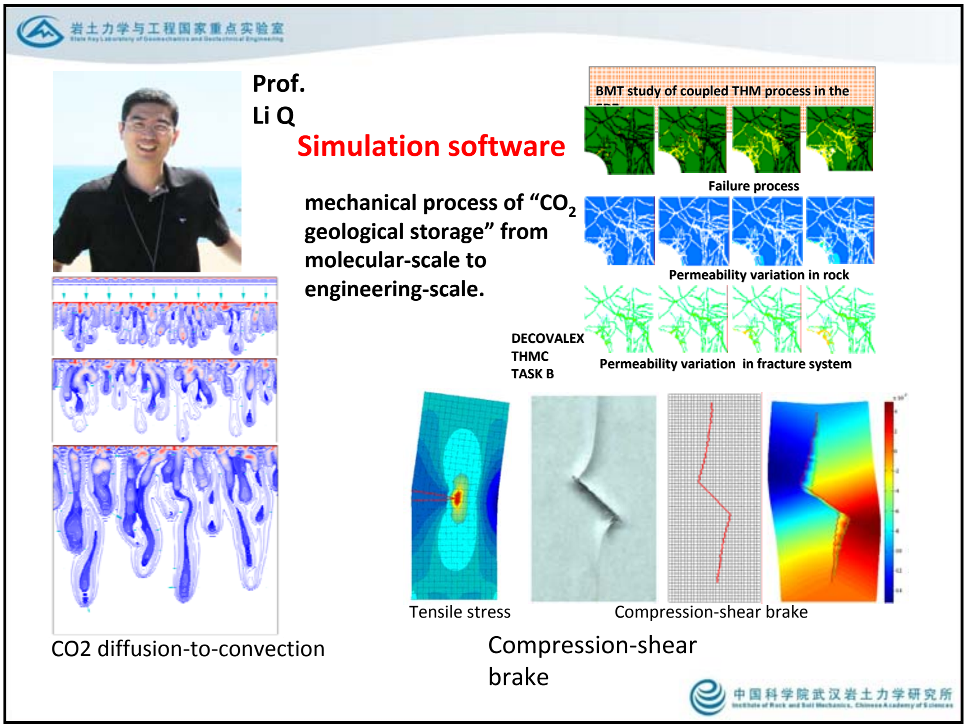


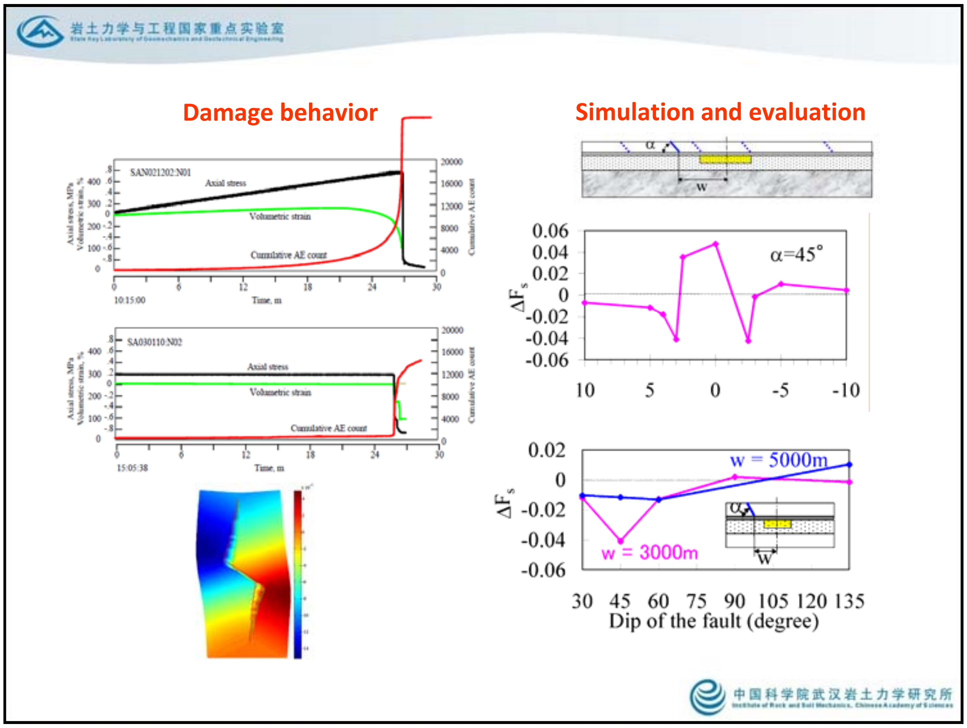
Prof.
Li Q

Simulation software

mechanical process of “CO_2 geological storage” from molecular-scale to engineering-scale.

CO2 diffusion-to-convection

Compression-shear brake



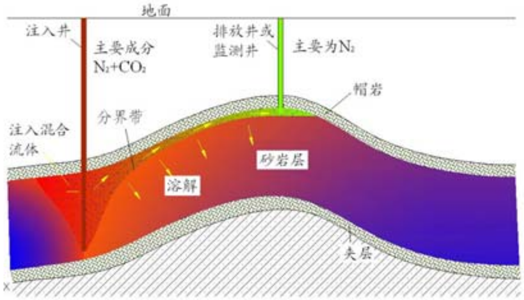
Damage behavior

Simulation and evaluation

3. Impure CO_2 storage in deep saline aquifer

Principle and motivation

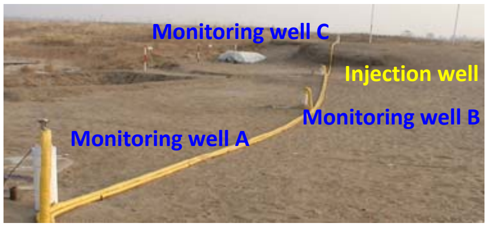
- **Principle: During the flow process, CO_2 is gradually dissolved in the water, N_2 separated from the injected gas due to low solubility and then released from the emission well.**
- **Motivation: Reduce the cost of separation by reducing the required concentration**

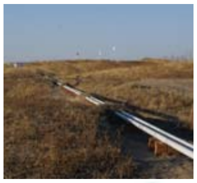


For pulverized coal power plant , the cost can be reduced by 30%.

Pilot test

- ➢Tongliao, Inner Mongolia
- ➢200t CO_2 injected, concentration: 80-95%



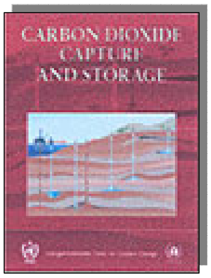


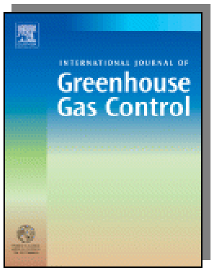
International Honour

- 《Science》 of 2009 CCS reviews the research of our group.

- Host the 3rt CAGS Workshop and Summer School at Sep 2010.

Award for the CSLF Third Ministerial Conference





4. My research Interests

Host: CO2CRC/University of Adelaide
Visiting Student: Xuehao Liu, Institute of Rock and Soil Mechanics, CAS

Working Program:

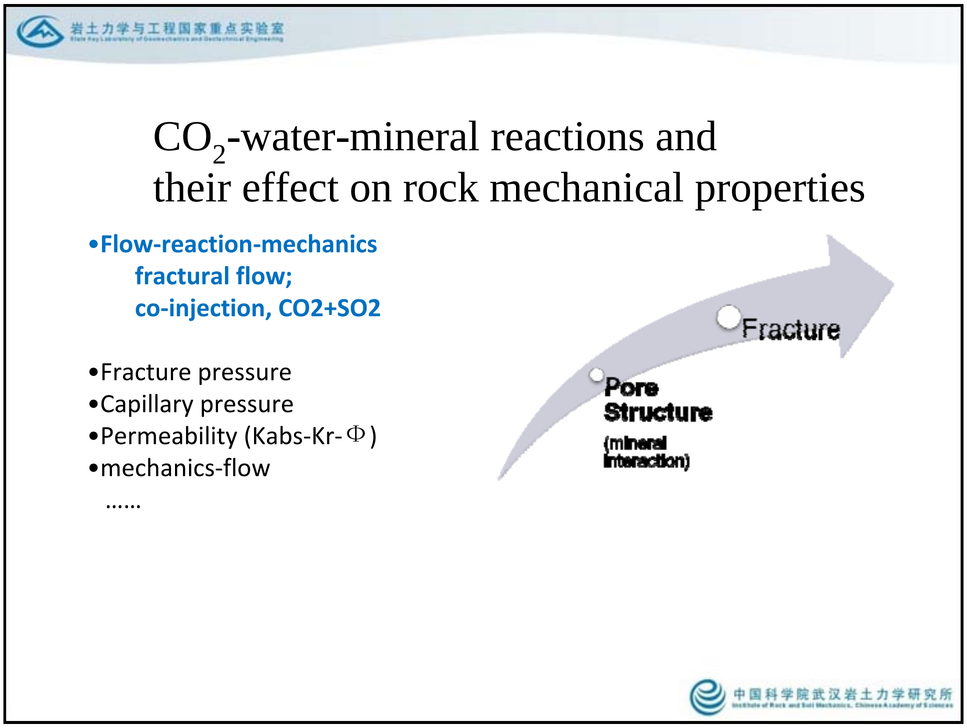
[CO_2-water-mineral reactions and their effect on rock mechanical properties]

Objectives:

- Develop an experimental programme to determine how CO_2-fluid-rock interactions affect the mechanical strength of reservoir rocks.
- Determine whether or not mechanical failure of reservoir and cap rock is a possibility during CO_2 injection operations.

cags China Australia Geological Storage of CO_2
中澳二氧化碳地质封存

Australian Government
Geoscience Australia

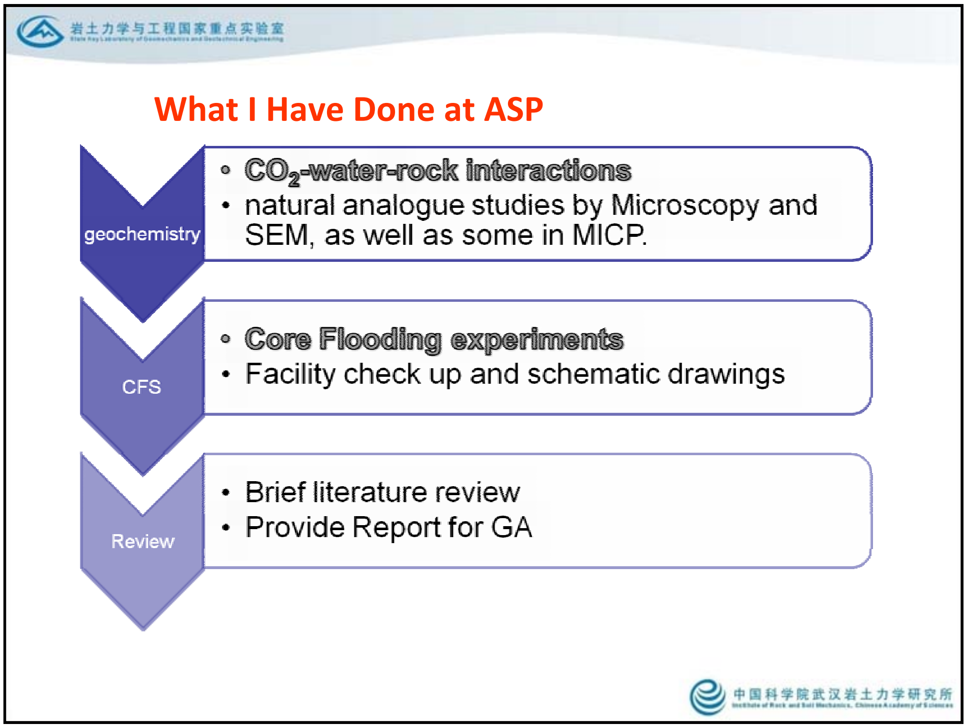


CO_2-water-mineral reactions and their effect on rock mechanical properties

- **Flow-reaction-mechanics**
  - **fractural flow;**
  - **co-injection, CO2+SO2**

- Fracture pressure
- Capillary pressure
- Permeability (Kabs-Kr-Φ)
- mechanics-flow

……



What I Have Done at ASP

geochemistry

- **CO_2-water-rock interactions**
- natural analogue studies by Microscopy and SEM, as well as some in MICP.

CFS

- **Core Flooding experiments**
- Facility check up and schematic drawings

Review

- Brief literature review
- Provide Report for GA

Thanks for your attentions!

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中國科學院
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