

SORPTION OF ANIONS AND FORMATION OF SECONDARY MINERALS AT HYPERALKALINE SPRING IN OMAN

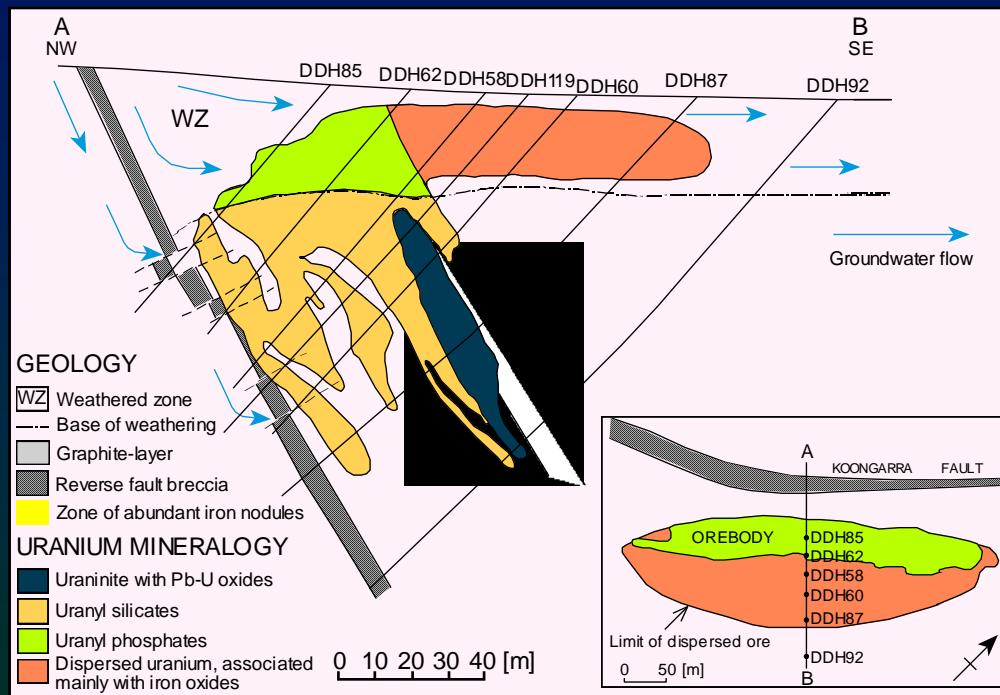


Tsutomu SATO*, Sotaro ANRAKU, Isamu MATSUBARA,
Jun HOSHINO and Tetsuro YONEDA

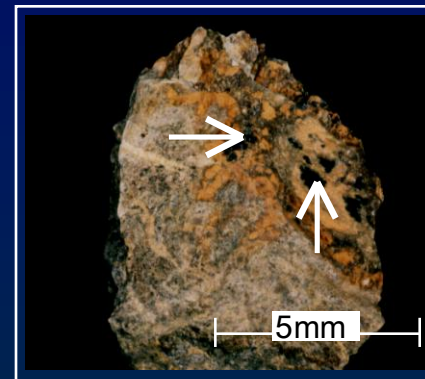
Laboratory of Environmental Geology, Faculty of Engineering,
Hokkaido University, Sapporo, Japan



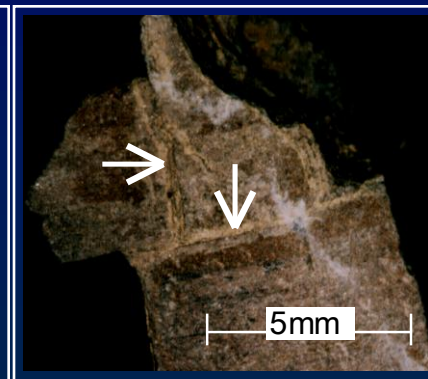
Natural Analogue Study at Koongarra Uranium Mine, Australia



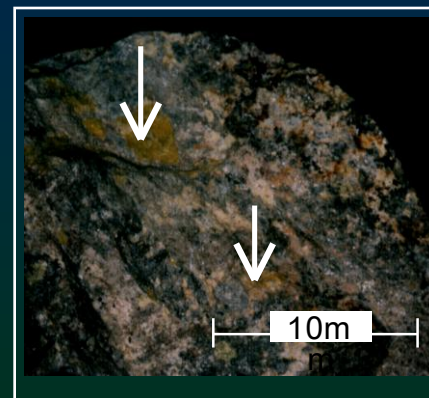
Cross-section and Bird's-eye view of Koongarra uranium deposit (1,800 Ma)



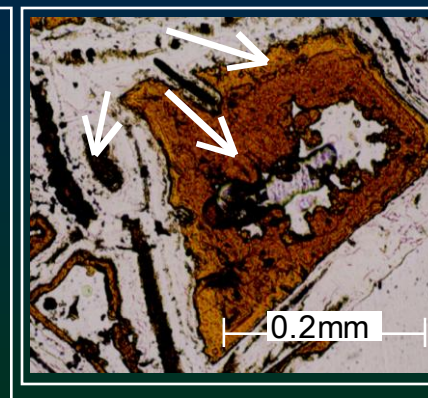
Uraninite



U-silicate

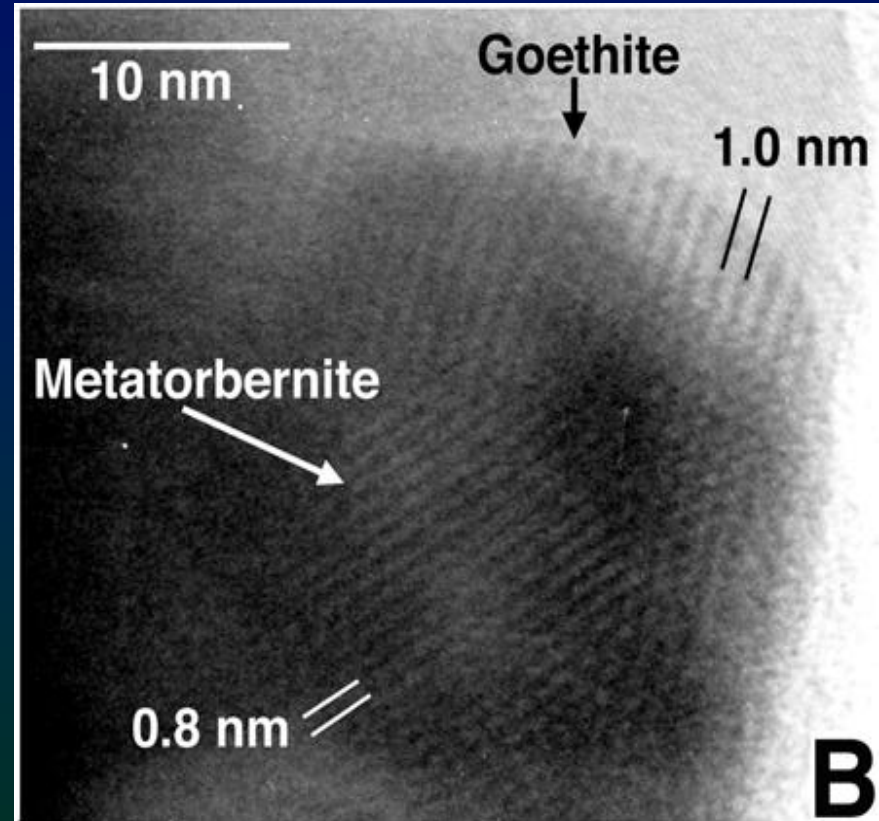
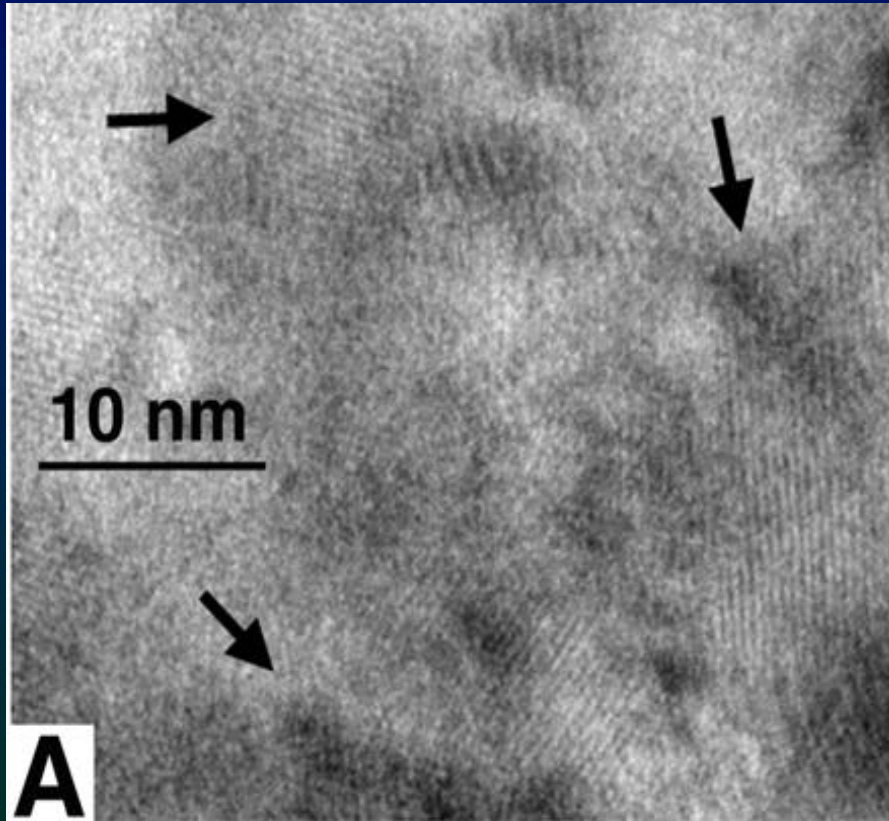


U-phosphate



Iron minerals

Formation of Nano Particles at Post-Adsorption



HRTEM Images of goethite transformed from ferrihydrite adsorbing uranium, phosphate and copper ions (Murakami et al., *Chem. Geol.* 2002)



Event Initiation

- The Fukushima nuclear facilities were damaged in a magnitude 9.0 earthquake on March 11 (Japan time), centered offshore of the Sendai region, which contains the capital Tokyo.
 - ➔ Plant designed for magnitude 8.2 earthquake. An 9.0 magnitude quake is 6.3 times in greater in magnitude.
- Serious secondary effects followed including a significant tsunami, significant aftershocks and a major fire at a fossil fuel installation.



Initial Response

- Nuclear reactors were shutdown automatically. Within seconds the control rods were inserted into core and nuclear chain reaction stopped.
- Cooling systems were placed in operation to remove the residual heat. The residual heat load is about 3% of the heat load under normal operating conditions.
- Earthquake resulted in the loss of offsite power which is the normal supply to plant.
- Emergency Diesel Generators started and powered station emergency cooling systems.
- One hour later, the station was struck by the tsunami. The tsunami was larger than what the plant was designed for. The tsunami took out all multiple sets of the backup Emergency Diesel generators.
- Reactor operators were able to utilize emergency battery power to provide power for cooling the core for 8 hours.
- Operators followed abnormal operating procedures and emergency operating procedures.



Loss of Makeup

- Offsite power could not be restored and delays occurred obtaining and connecting portable generators.
- After the batteries ran out, residual heat could not be carried away any more.
- Reactor temperatures increased and water levels in the reactor decreased, eventually uncovering and overheating the core.
- Hydrogen was produced from metal-water reactions in the reactor.
- Operators vented the reactor to relieve steam pressure - energy (and hydrogen) was released into the primary containment (drywell) causing primary containment temperatures and pressures to increase.
- Operators took actions to vent the primary containment to control containment pressure and hydrogen levels. Required to protect the primary containment from failure.
- Primary Containment Venting is through a filtered path that travels through duct work in the secondary containment to an elevated release point on the refuel floor (on top of the reactor building).
- A hydrogen detonation subsequently occurred while venting the secondary containment. Occurred shortly after and aftershock at the station. Spark likely ignited hydrogen.

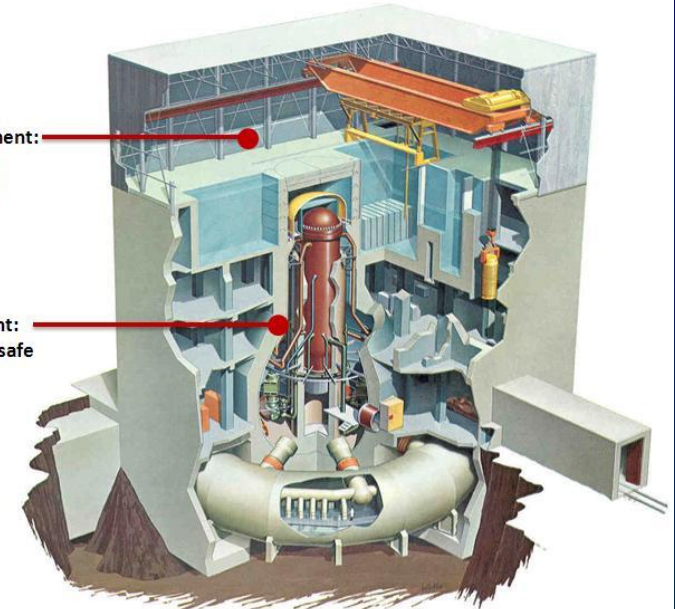


Hydrogen Detonation at Unit 1



Secondary containment:
Area of explosion at
Fukushima Daiichi 1

Primary containment:
Remains intact and safe



Boiling Water Reactor Design



Reactor Building



Fukushima Daiichi Nuclear Power Plant

Okuma, Japan

37 25 18N 141 01 56E



**1 Minute Before Unit 3
Reactor Building Explosion**

**Damaged Unit 1
Reactor Building**

**Unit 3 Reactor
Building Intact**

DigitalGlobe's Panchromatic Satellite Image
March 14, 2011 (02:00 GMT)

DIGITALGLOBE®



Fukushima Daiichi Nuclear Power Plant

Okuma, Japan

37 25 18N 141 01 56E



**3 Minutes After Unit 3
Reactor Building Explosion**

**Damaged Unit 3 Reactor
Building
(Visible Smoke/Dust Plume
Drifting East Approx 2
kilometers)**



DigitalGlobe's Natural Color Satellite Image
March 14, 2011 (02:04 GMT)





Fukushima Daiichi Nuclear Power Plant

Okuma, Japan

37 25 18N 141 01 56E



3 Minutes After Unit 3
Reactor Building Explosion

Damaged Unit 3 Reactor Building
(Roof Blown Off; Panel Walls Blown Out)

Damaged Unit 1 Reactor Building



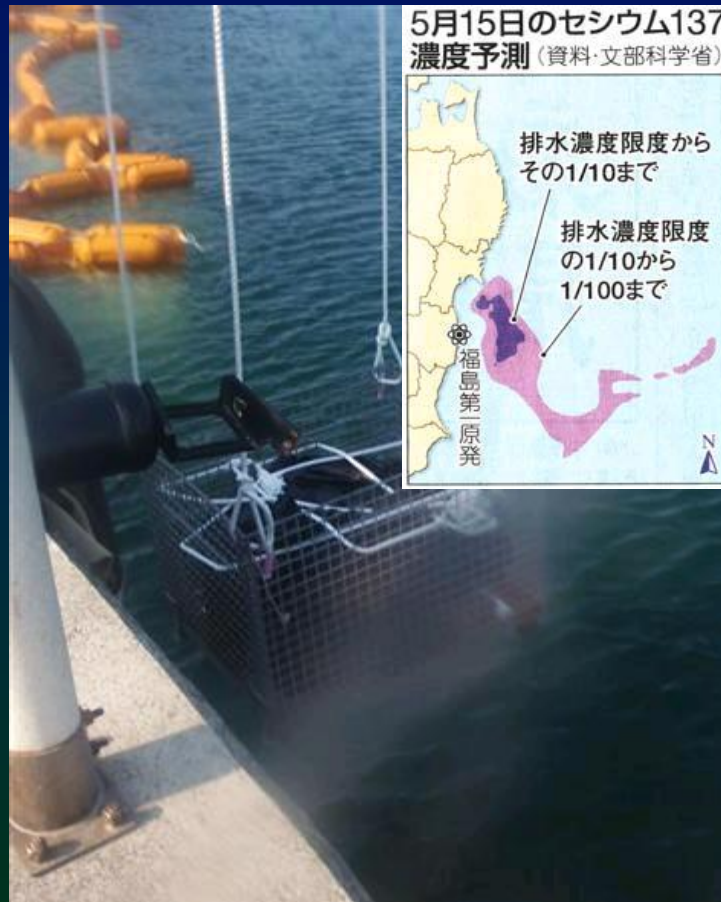
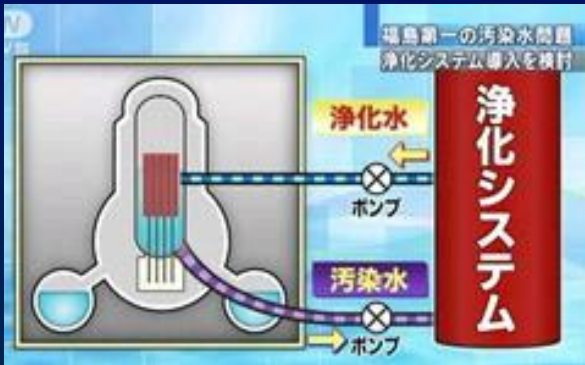
Trucks

DigitalGlobe's Natural Color Satellite Image
March 14, 2011 (02:04 GMT)

There are huge volume of rubbles

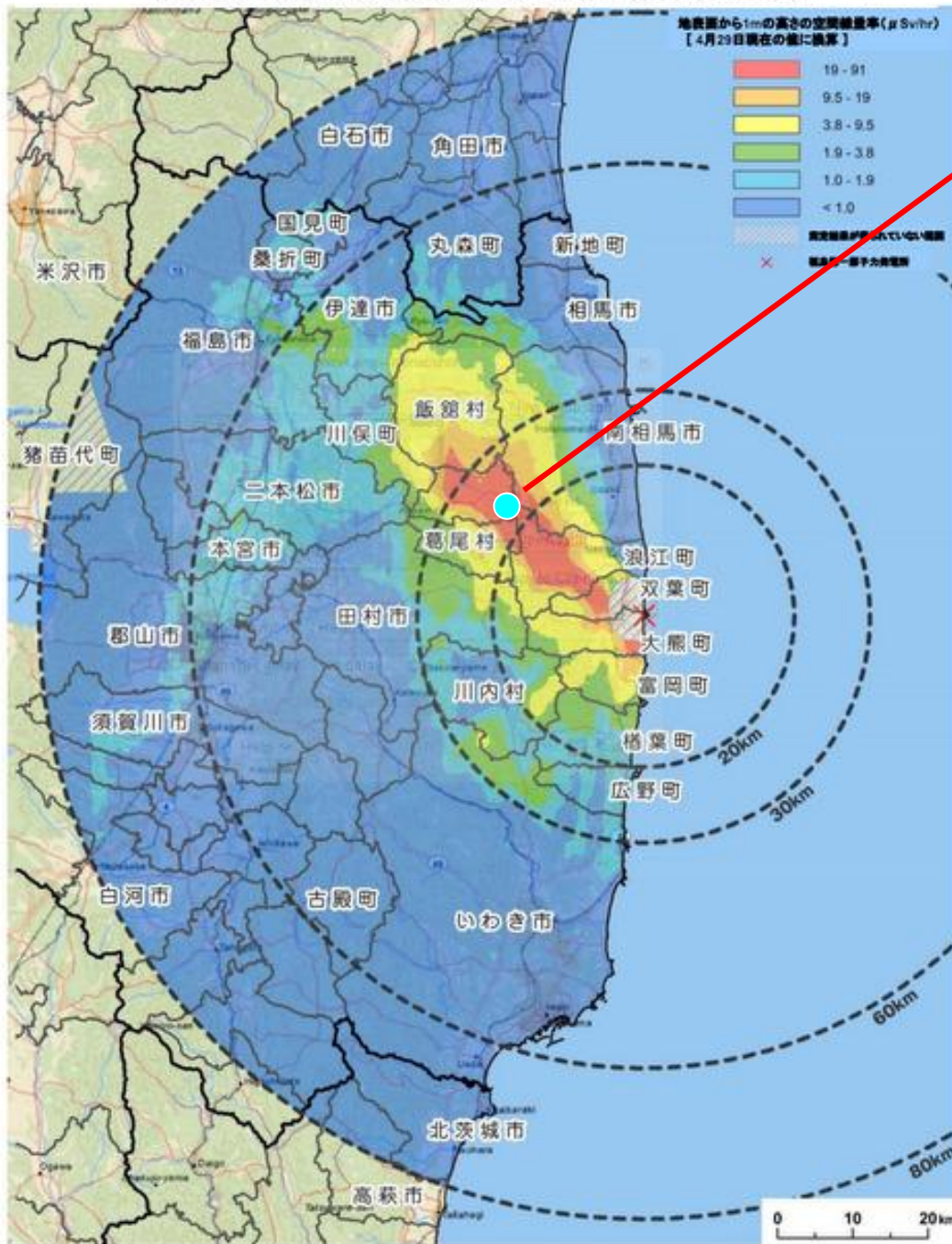


Should Clean Up Water and Soil



小学校の校庭で行われている重機を使った表土除去作業 = 27日午前、福島県郡山市 (時事)

文部科学省及び米国DOEによる航空機モニタリングの結果
(福島第一原子力発電所から80km圏内の線量測定マップ)



313.9 mSv / y

Residents in the 20 km zone around Fukushima Daiichi NPP and red and yellow zone already evacuated.

Soils, water and building in red, yellow and green areas should be remediated.



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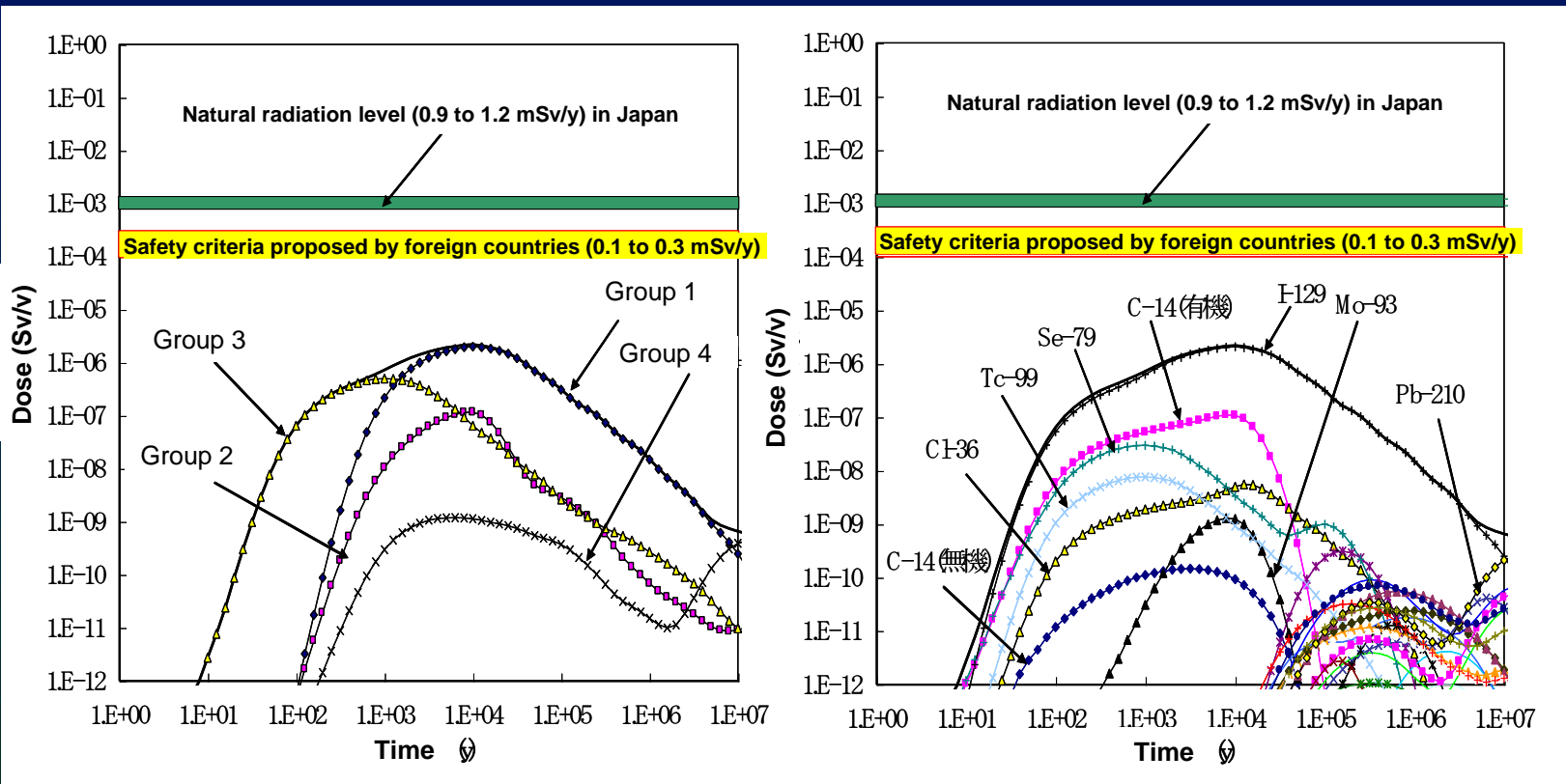


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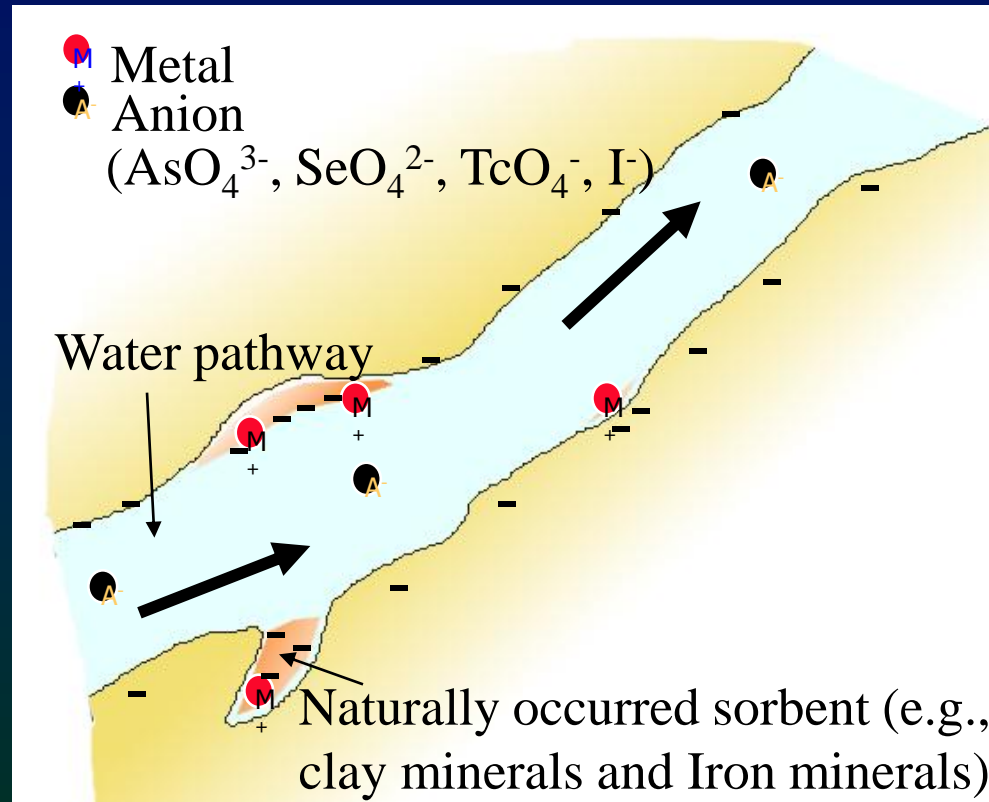
Behavior of anions is a key for safety assessment of TRU waste disposal



According to “The Technical Report on Disposal for TRU Waste in Japan(2005)”, Anions such as I, Cl, Tc, Se are the key nuclide on the safety assessment for the geological disposal of TRU wastes generated from fuel cycle facilities such as reprocessing plants and MOX fuel fabrication plants.



Migration of anion species in geomedia

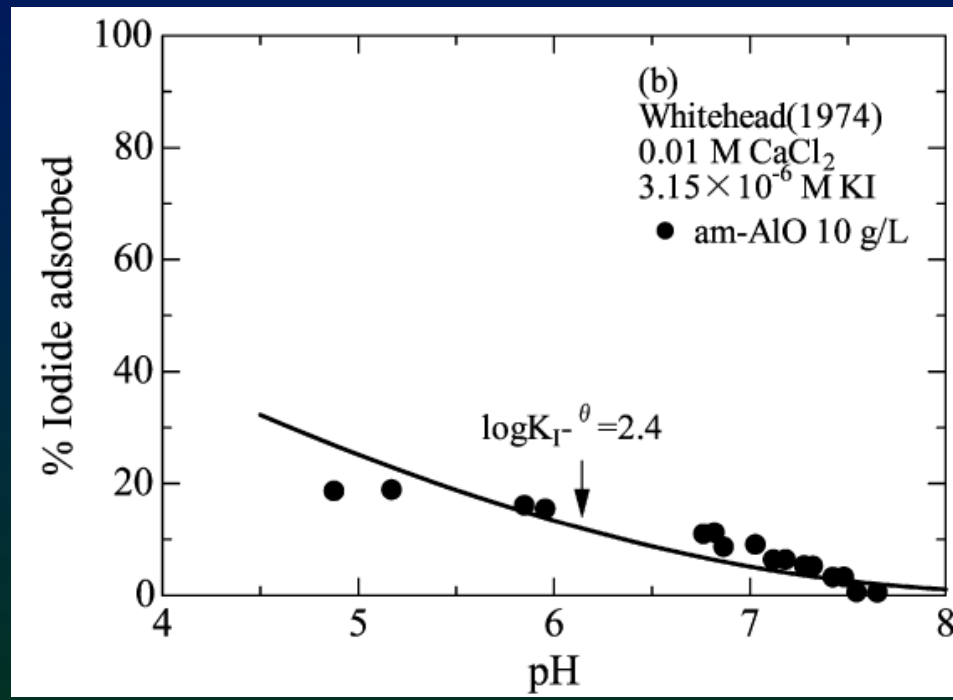
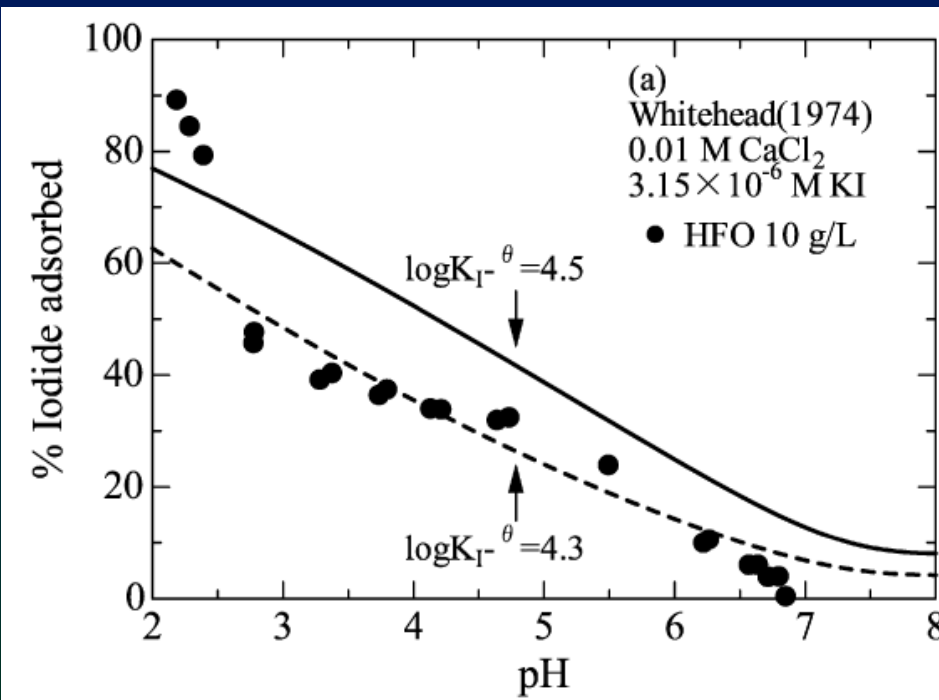


It is difficult to estimate the environmental impact of anion in geomedia, because many anions exhibit complex speciation as a response of solution condition, and are expelled to the common rock-forming minerals of which surface charges are negative in common solution condition and high pH cement water, too.

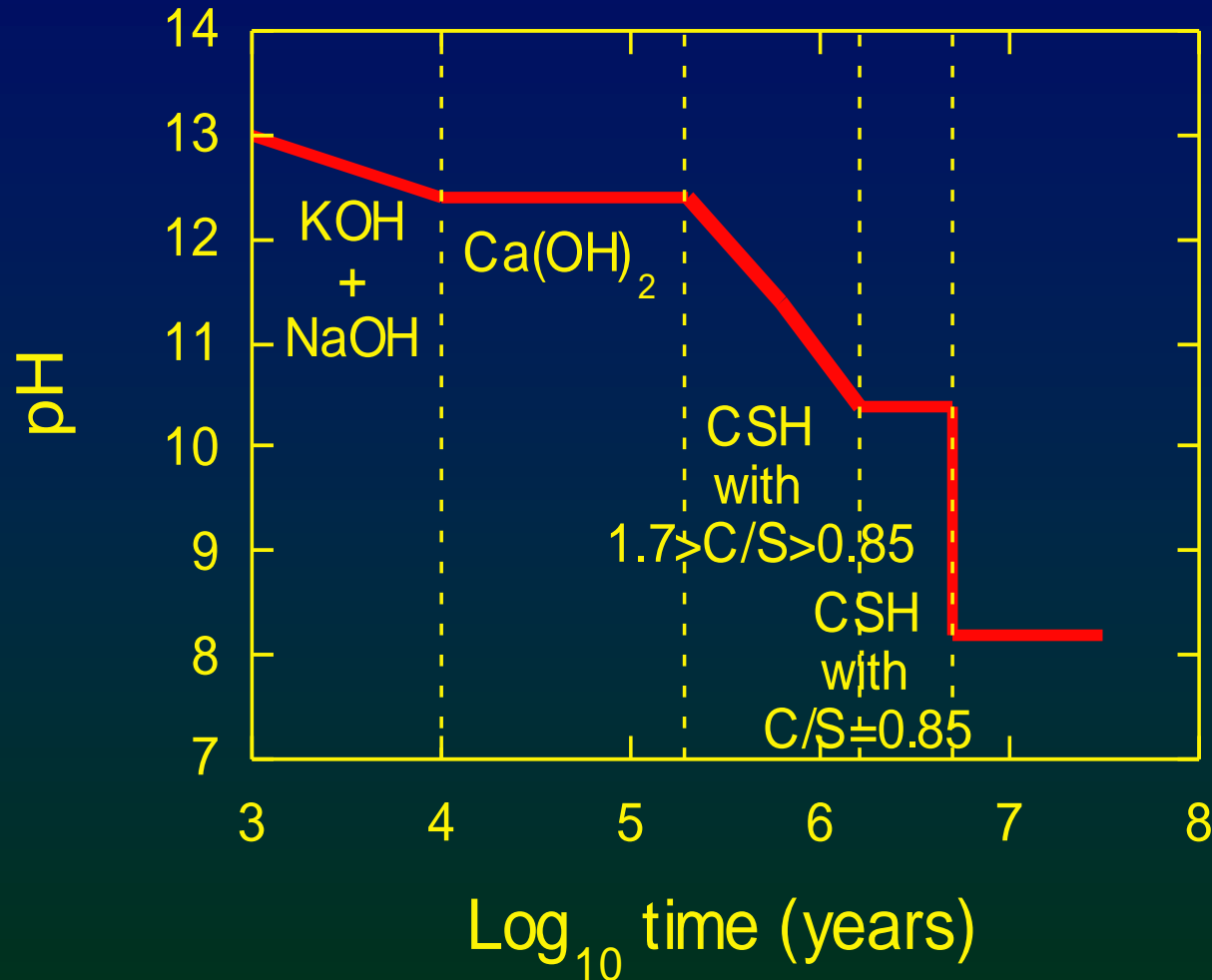
Relative mobility of some essential and potentially toxic elements in different surface conditions (Plant et al., 2001)

		Environmental Conditions		Environmental Conditions	
Relative Mobilities		Oxidising	Top Profile	Acid	Humid
<u>Very High</u>	↑	I		I	
High		Mo U Se F Ra Zn		Mo U Se F Ra Zn Cu Co Ni Hg	
Medium		Cu Co Ni Hg As Cd		As Cd	
Low		Pb Be Bi Sb Ti		Pb Be Bi Sb Ti Fe Mn	
Very Low		Fe Mn Al Cr	Al Cr		
Relative Mobilities		Environmental Conditions		Environmental Conditions	
Relative Mobilities		Reducing	Base Profile	Alkaline	Arid
Very Low	↓	Al Cr Mo U Se Zn Co Cu Ni Hg As Cd Pb Be Bi Sb Ti		Al Cr Zn Cu Co Ni Hg	
Low		Fe Mn		Pb Be Bi Sb Ti Fe Mn	
Medium		F Ra		As Cd	
High		I		F Ra	
<u>Very High</u>		Mo U Se	I		

Iodide adsorption on HFO and am-AlO and ETLM (Nagata et al., 2009)



pH Evolution of Cement Pore Fluids



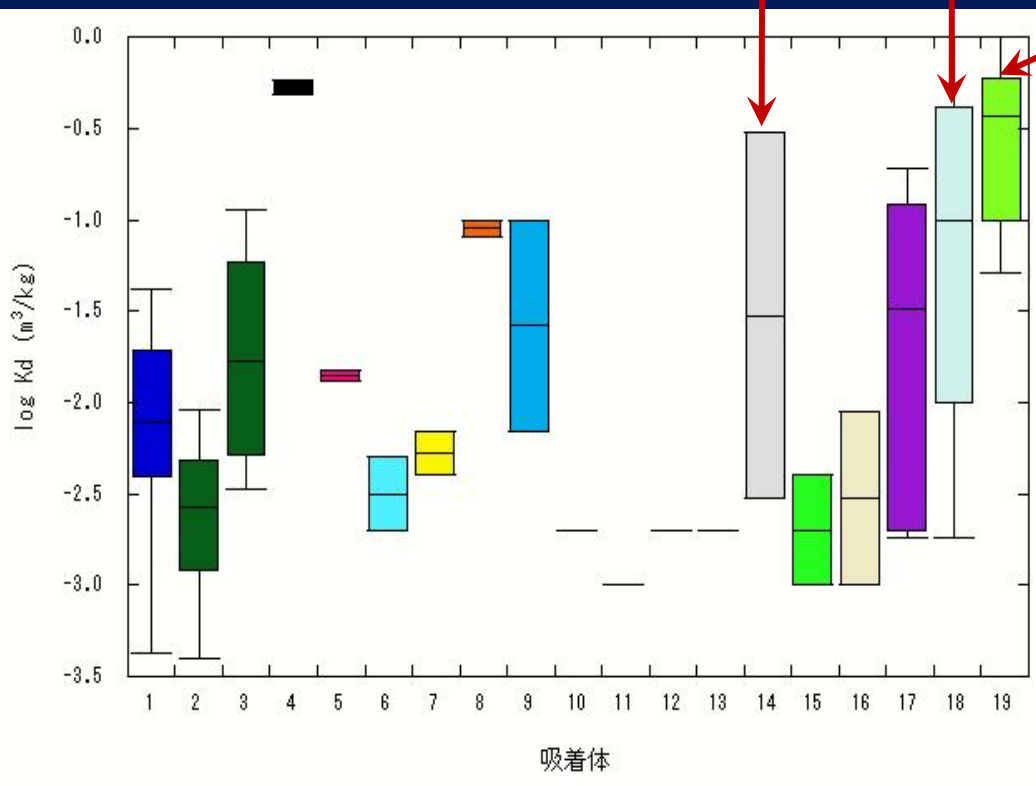
Predicted evolution of the pH within the near-field of the reference case U.K. Nirex Ltd. cementitious low and intermediate-level waste repository with an average cement content of 185 kg/m³ and a water flux density of 10⁻¹⁰ m/s. After 1000 years a pH of 13 will be attained which will gradually decline with time but remain above pH 10 for at least the first one million years (after Atkinson, 1985).



Kd values for cement and cement minerals

Alumina cement
AFt

Monosulfoaluminate



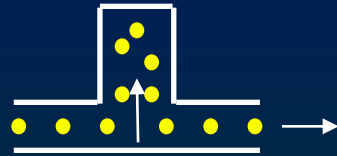
- 1: OPC (Allard et al., 1984; Hoglund et al., 1986)
- 2: 9:1 BFS/DPC (Andersson et al., 1981, 1983)
- 3: Moderate Heat Portland Cement (籍他, 1997)
- 4: CSH (Ca/Si=1.5) (Noshita et al., 2001)
- 5: CSH (Ca/Si=1.2) (Noshita et al., 2001)
- 6: CSH (Ca/Si=1.0) (Noshita et al., 2001)
- 7: CSH (Ca/Si=0.83) (Noshita et al., 2001)
- 8: アフィライト Afwillite (Ca/Si=1.5) (Noshita et al., 2001)
- 9: トバモライト Tobermorite (Ca/Si=0.8) (Noshita et al., 2001)
- 10: ジャイロライト Gyrolite (Ca/Si=0.67) (Noshita et al., 2001)
- 11: ポルトランドライト Portlandite (Noshita et al., 2001)
- 12: ブルーサイト Brucite (Noshita et al., 2001)
- 13: ギブサイト Gibbsite (Noshita et al., 2001)
- 14: AFt (Noshita et al., 2001)
- 15: AFm (Noshita et al., 2001)
- 16: ハイドロガーネット Hydrogarnet (Noshita et al., 2001)
- 17: アルミナセメント+CaSO₄·2H₂O (SO₄/Caモル比=0.0) (Masumitsu, et al., 2000)
- 18: アルミナセメント+Ca(OH)₂ (Ca/Alモル比=0.34) (Masumitsu, et al., 2000)
- 19: モノサルフェート形状カルシウムアルミネート系水和物 (Ca/Al=2, SO₄/Ca=0) (Masumitsu, et al., 2000)

Retardation mechanism that may affect radionuclide transport in groundwater

(McKinley and Hadermann, 1984)

Physico-chemical retardation

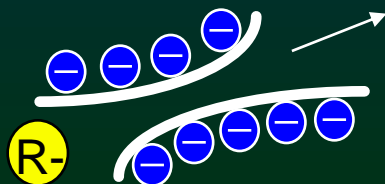
Diffusion into dead-end pores



Molecular filtration

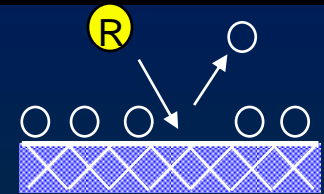


Ion exclusion

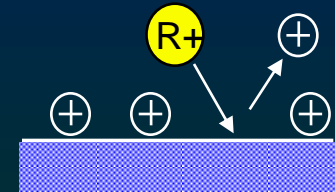


Chemical retardation

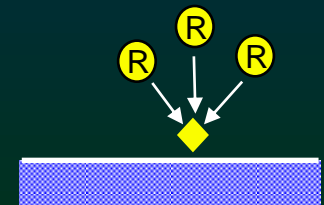
Adsorption



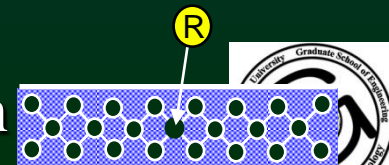
Ion exchange



Precipitation



Mineralization



Conservative setting is depending on scenario

Habitation Scenario

- Solubility · · · Lower
- Distribution coefficient · · · Higher
- Degradation rate of barrier materials · · · Slower

- Permeability of bentonite · · · Lower

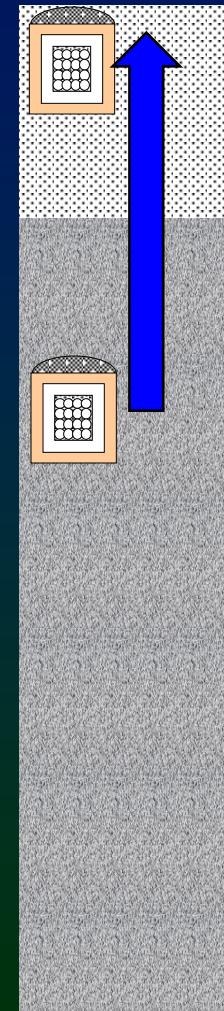


Conservative (pessimistic)



Realistic

exposed elevated



80-100m

Groundwater Scenario

- Solubility · · · Higher
- Distribution coefficient · · · Lower
- Degradation rate of barrier materials · · · Faster
- Permeability of bentonite · · · Higher

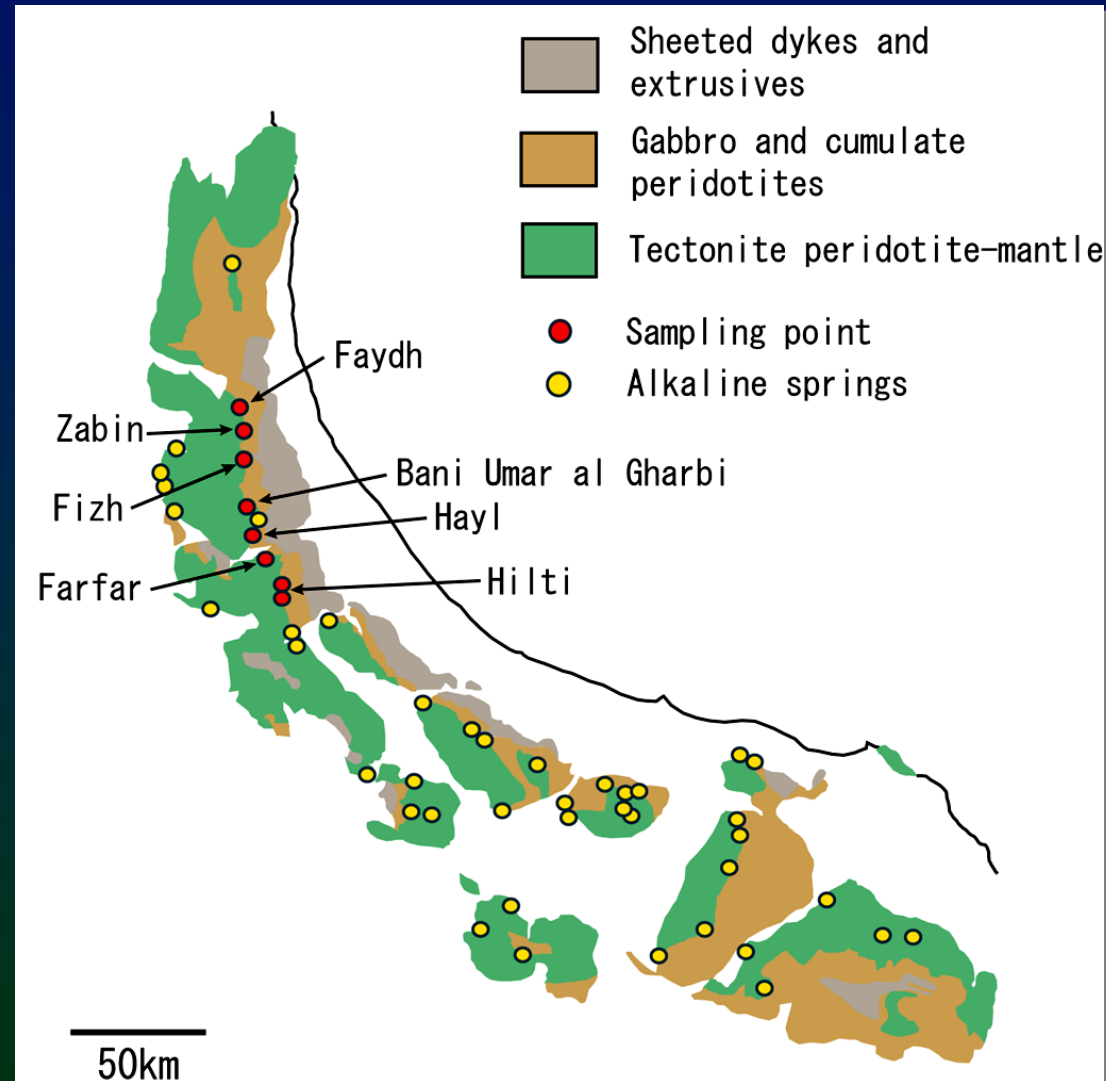
Hyperalkaline springs at Oman ophiolite



- pH11-12
- Ca-rich
- H₂ and CH₄ gas out

(Sano et. al.(1993) ,Neal and Stanger(1983))

Distribution of ophiolites and hyperalkaline springs in Oman ophiolite

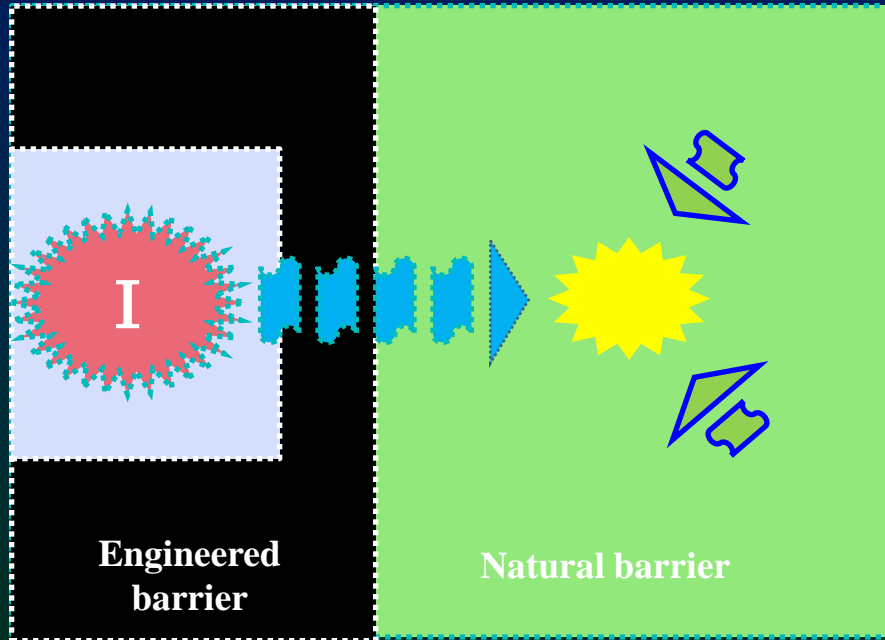


(modified Neal and Stanger, 1985)



Analogy

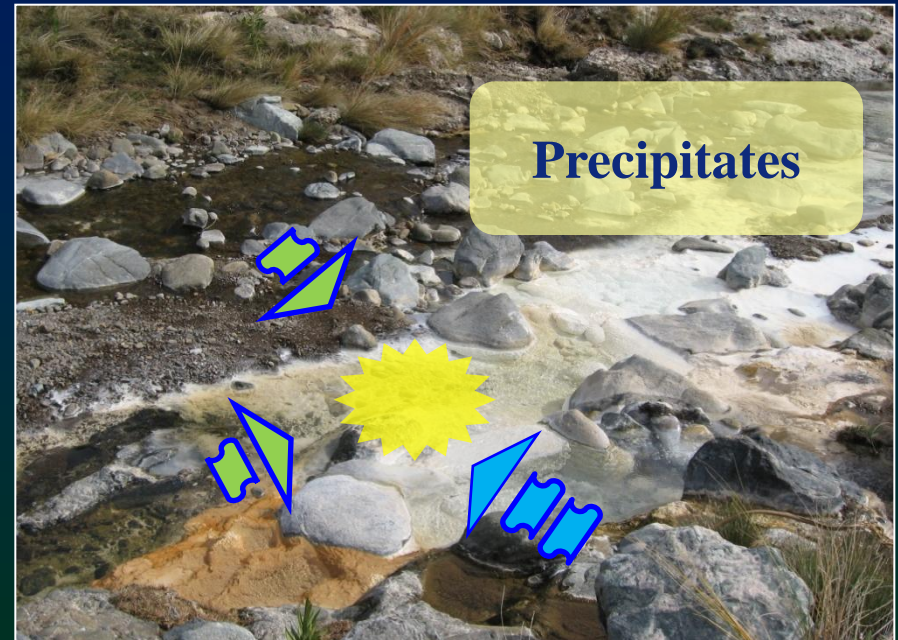
The reaction between...
**Cement pore fluid and
ground water**



Cement pore fluid
High pH Ca-OH

Ground water
Neutral Mg-HCO₃

**Spring water and
surface water in Oman**



Spring water
pH = 11 Ca-OH

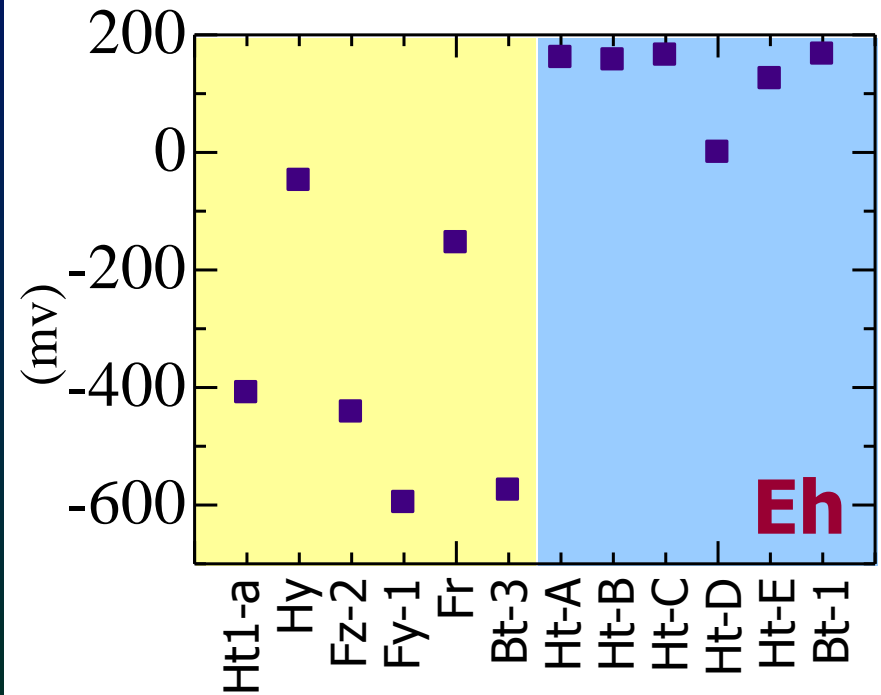
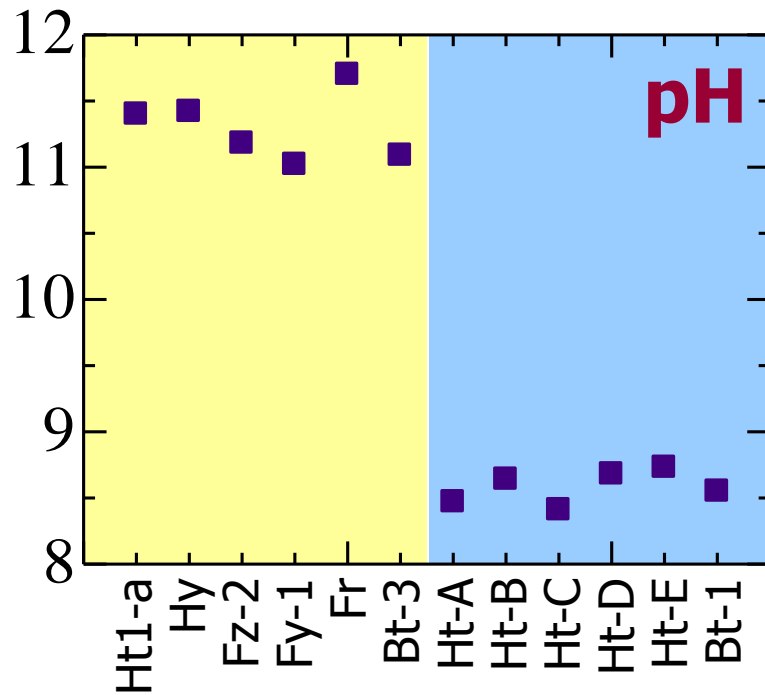
Surface water
pH = 8.5 Mg-HCO₃

Objectives

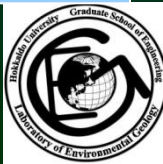
- This study focused on
 - ➔ the sorption behavior of anions such as CO_3^{2-} , H_3SiO_4^- , Cl^- , I^- and formation of their host minerals at naturally-occurring hyperalkaline springs in the Oman ophiolite
 - ➔ to learn how to retard migration of anions from natural processes.



Characteristics of surface and spring water



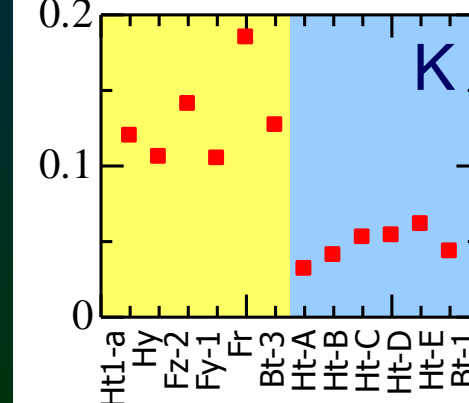
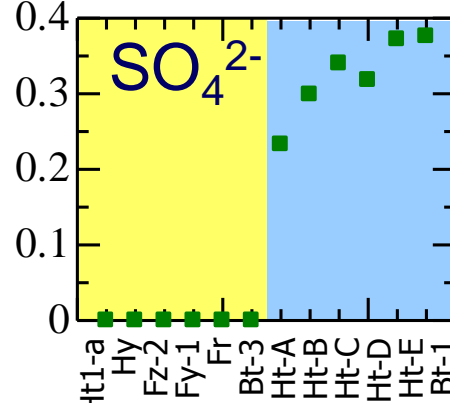
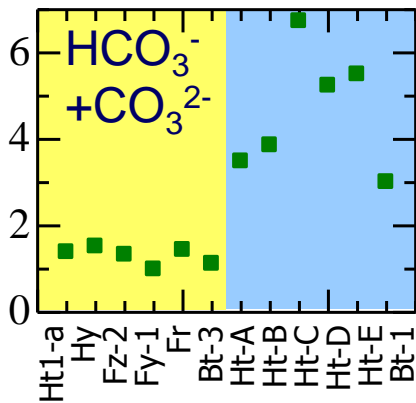
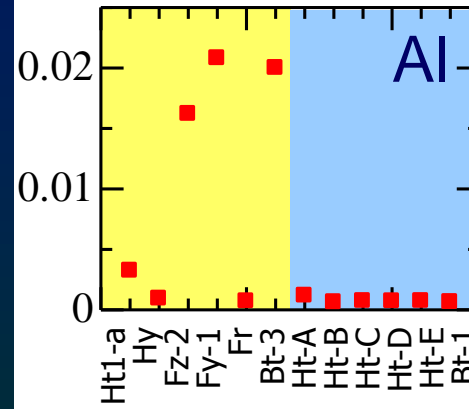
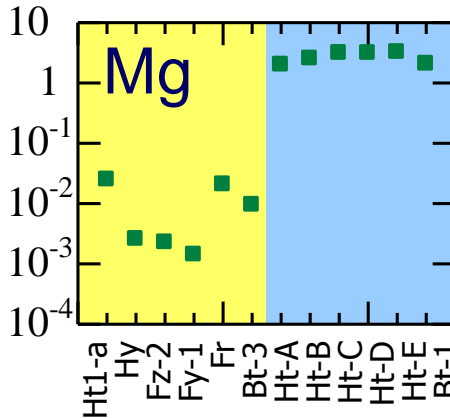
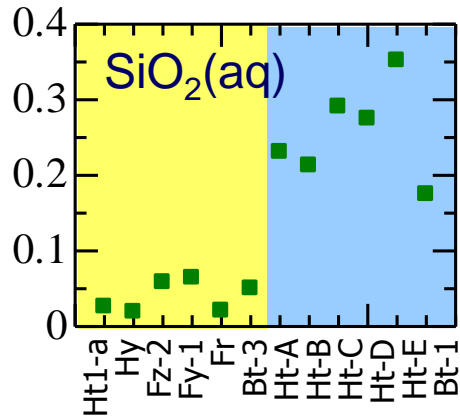
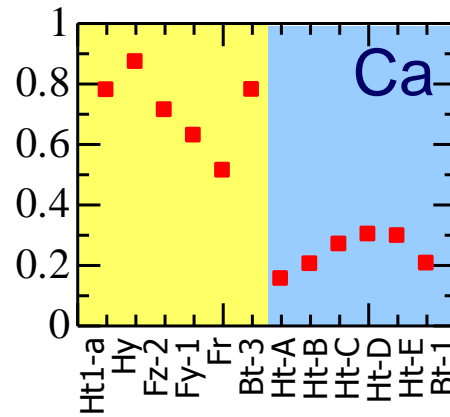
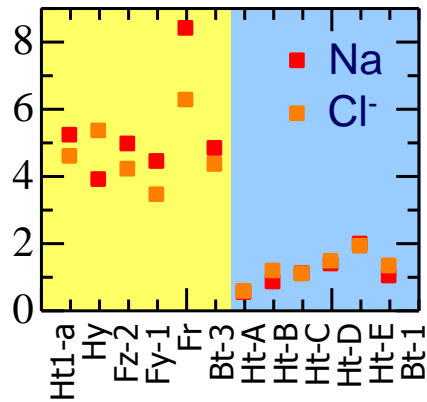
High pH water Surface water



Chemistry of surface and spring water

High pH water

Surface water



(mmolal)



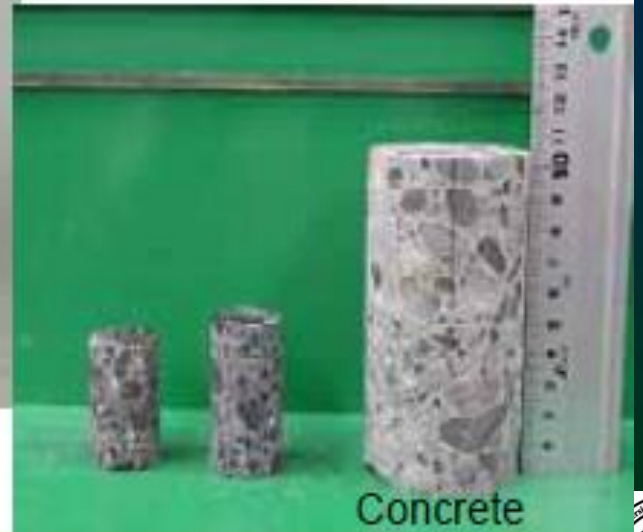
Occurrence of the precipitates



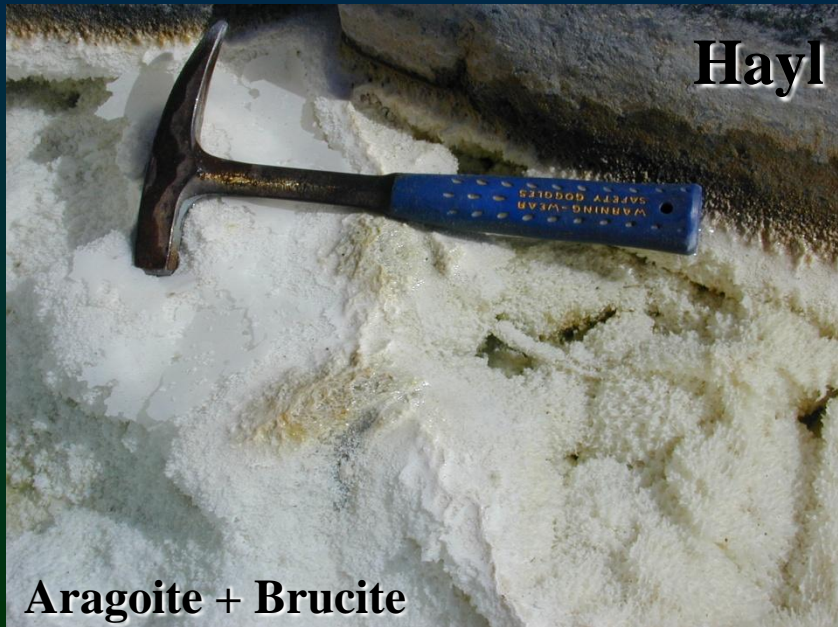
Left FZ1
D 25.3mm H 42.0mm
 $\gamma=2.58$

Right FZ2
D 25.3mm H 50.6mm
 $\gamma=2.61$

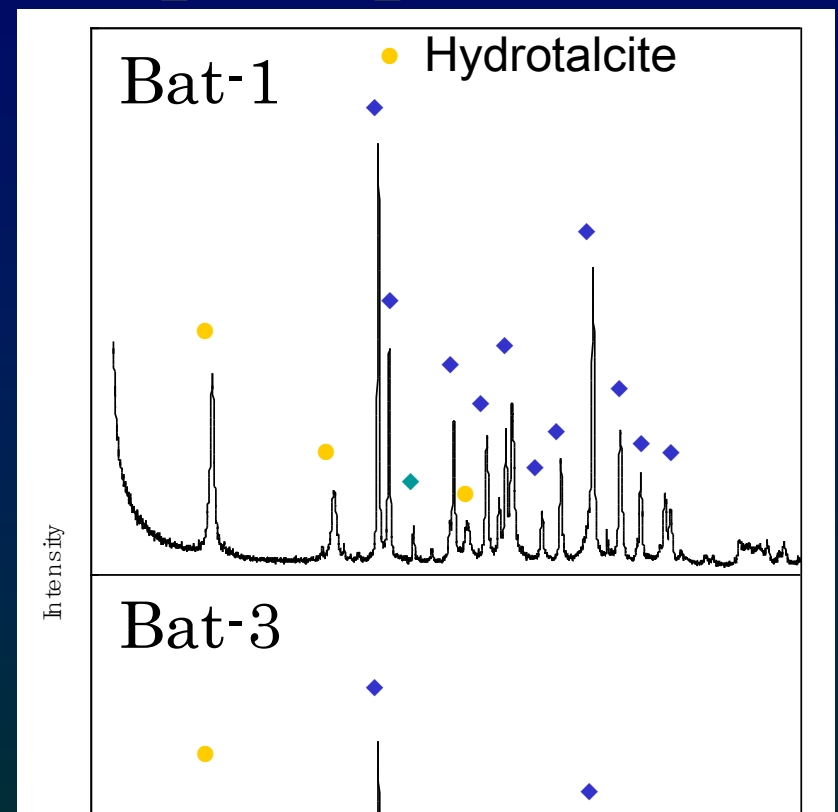
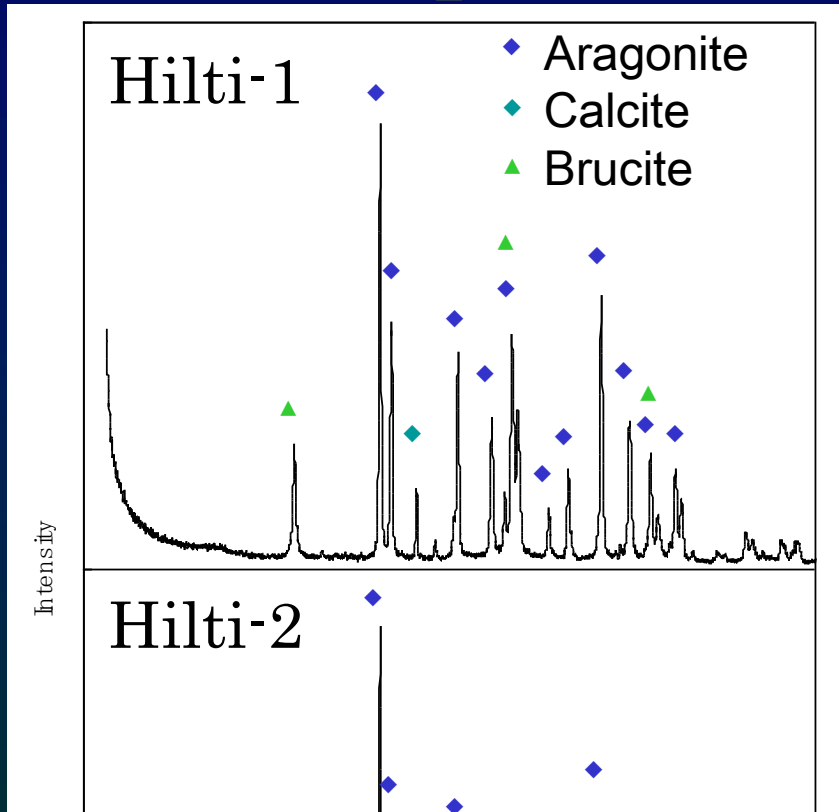
Fizh200912



Minerals formation at hyperalkaline springs



XRD patterns of the precipitates



Minerals

Hydrotalcite like compound (HTLc)

Aragonite

Calcite

Brucite

Chemical Formulae

$\text{Mg}_6\text{Al}_2\text{X}(\text{OH})_{16}\cdot 4\text{H}_2\text{O}$ **X : anions**

CaCO_3 **(with Mg)**

CaCO_3 **(without Mg)**

$\text{Mg}(\text{OH})_2$

Morphology vs. mineralogical composition

Sample Name	Form	Mixing ratio of SW to HW	鉱物名				
			Aragonite	Calcite	HTlc	Brucite	Hydro-magnesite
t_Fizh①	Ice	I	○	◎			
t_HayI-a	Ice	I	○	◎			
t_Hilti①-b	chimney	*	◎	○	○		
t_Hilti①-c	terrace	*	◎	○		○	
t_Hilti②-a	Ice	*	◎	○		○	○
t_Hilti②-b	mousse	*	◎			○	
t_Hilti②-c	mousse	*	◎			○	
t_HayI-c	mousse	*	◎	○		○	
t_Fizh②-a	chimney	*	◎	○	○		
t_Faydh②-a	Ice	*	◎	○	○		
t_Fizh②-b	chimney	*	◎	○	○		
t_Faydh②-b	needle	*	◎	○	○		
t_Faydh②-c	mousse	*	◎	○	○		
t_Farfar-a	Ice	*	○	◎		○	
t_Farfar-b	mousse	*	◎	○		○	
t_Farfar-c	mousse	*	◎	○		○	○
t_Bat③-a	terrace	*	◎	○	○		
t_Bat③-b	terrace	*	◎		○		
t_BaniA-a	mousse	*	◎	○	○		
t_Hilti②-d	mousse	***	◎				
t_Hilti②-e	mousse	***	◎				
t_HayI-b	needle	***	◎	○			
t_Bat③-c	chimney	***	◎				
t_BaniA-b	chimney	***	◎	○			

I . . . Isolated from surface water

* mixed with small amount of surface water

** * . . . mixed with large amount of surface water

◎ dominant in XRD pattern

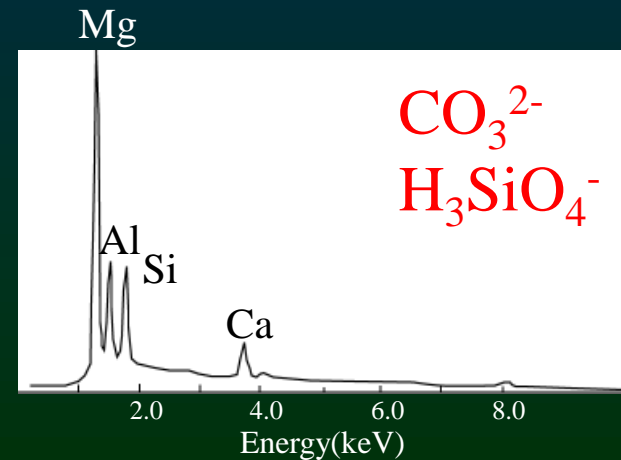
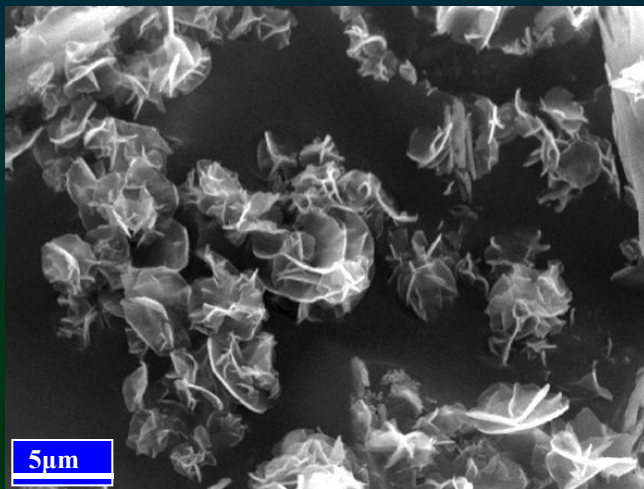
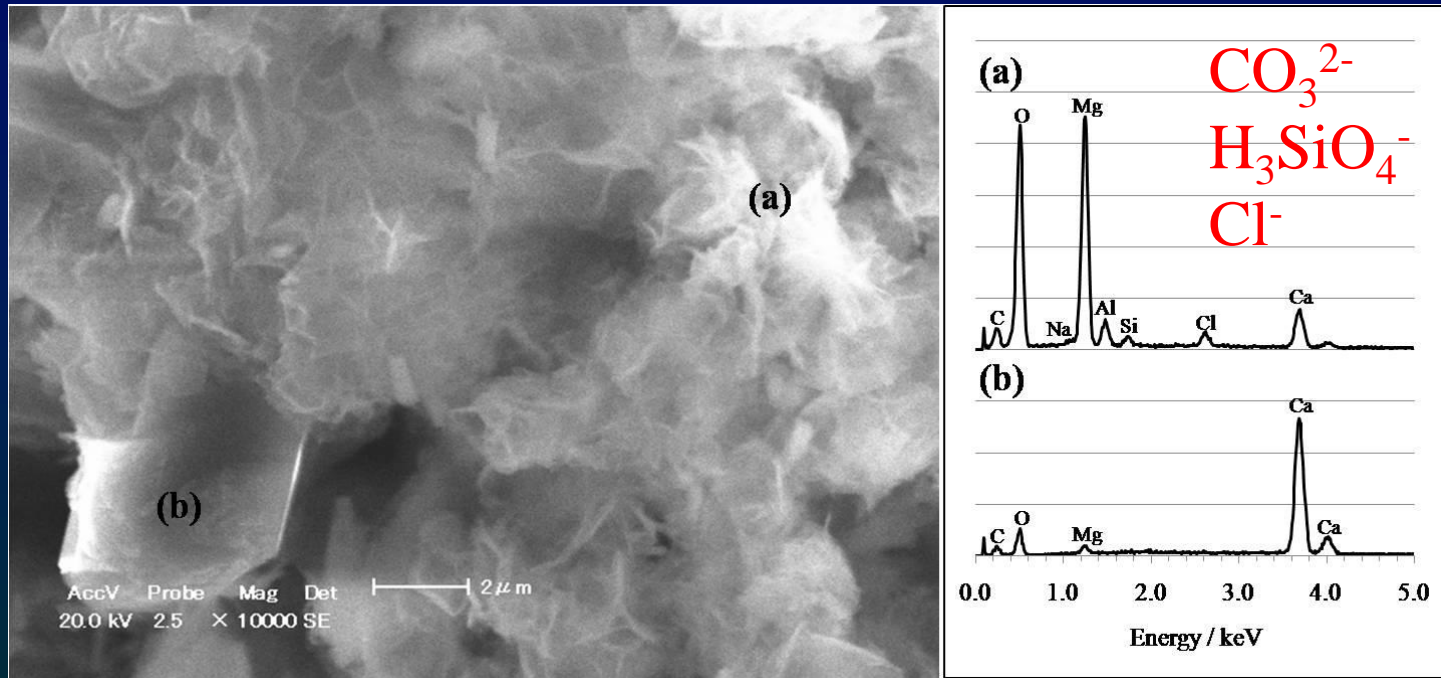
○ detected in XRD pattern



pH and aluminum content vs. mineralogy

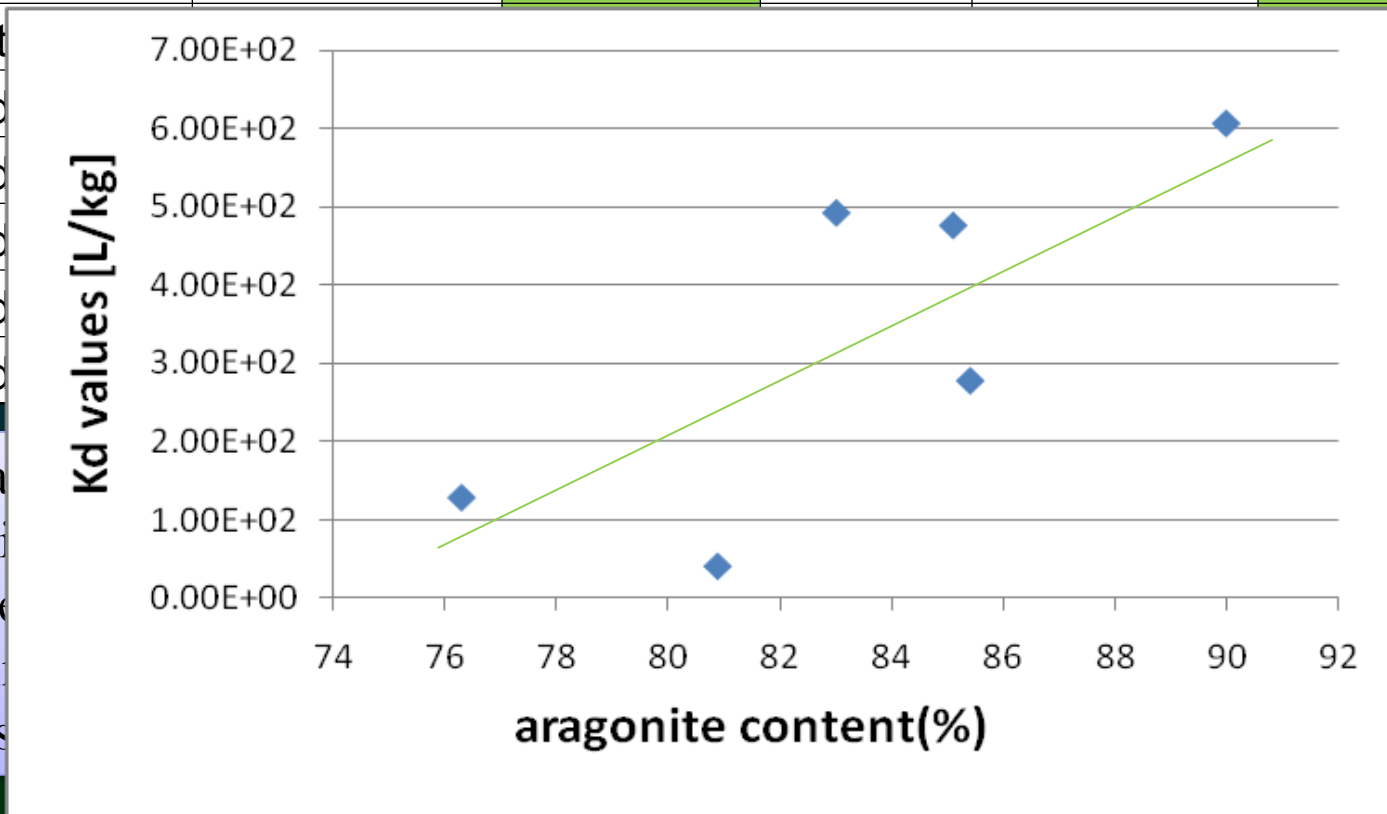
Area	Vent No.	pH	Al ($\mu\text{mol/L}$)	HTlc	Brucite
Feydh	4	9.41	4.3	X	X
Feydh	1	9.36	5.9	X	X
Feydh	6	10.38	1.0	X	X
Feydh	2	11.1	11.2	O	X
Feydh	7	10.96	31.4	O	X
Jaja	2	11.04	25.9	O	X
Fizh	2	10.88	25.8	O	X
Hilti	1	11.63	0.4	X	O
Hilti	8	11.43	0.5	X	O
Bani	1	11.18	0.5	X	O

SEM images and EDX spectra



Constituent minerals and Kd value of iodine for the precipitates

sample name	constituent minerals			Distribution coefficient	
	hydrotalcite	aragonite	calcite	Si [L/kg]	I [L/kg]
Bath-P2	○	○	○	1.52E+04	9.45E+01
Bath-P4		○		4.42E+01	4.76E+02



Relationship between aragonite content and Kd value of iodine

Kd va
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Synthesis experiment in the field

Spring Water



River Water

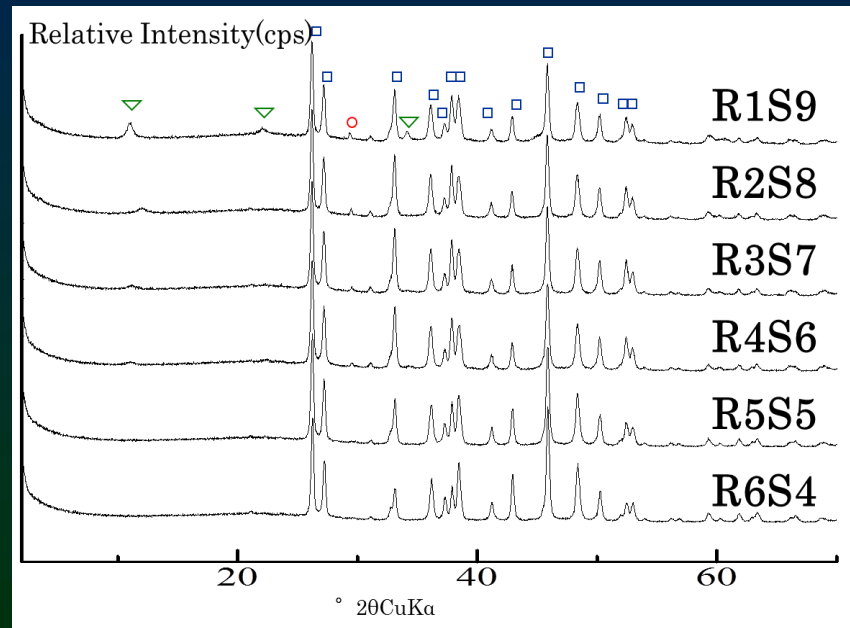


KI

- 0.5mM
- 0.05mM
- 0.005mM



Spring water and river water were mixed in particular ratio to reveal the relationship between the mixing ratio and the secondary mineral phases. Iodide reagent also added in deferent concentration to know the dominant phase of iodide uptake.



Calcite crystal growth rate -Mg inhibition model-

Langmuir adsorption model involving Mg^{2+} and Ca^{2+}

Calcite crystal growth rate (Lin *et al.*, 2009)

$$\frac{R_i}{R_0} = \frac{K_{Ca} [Ca^{2+}]}{K_{Ca} [Ca^{2+}] + K_{Mg} [Mg^{2+}]}$$

The concentration of calcite surface site occupied by Ca^{2+}

The concentration of calcite surface site occupied by Ca^{2+} and Mg^{2+}

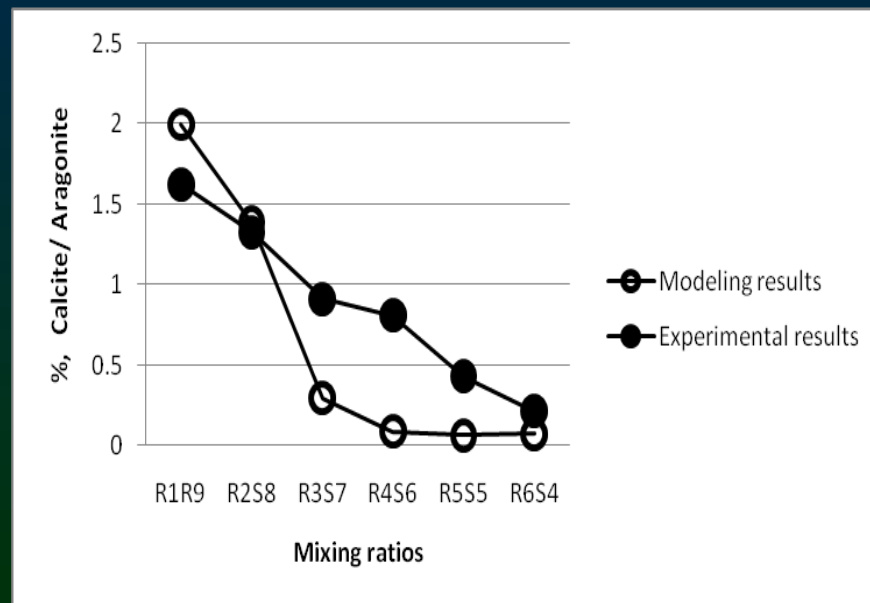
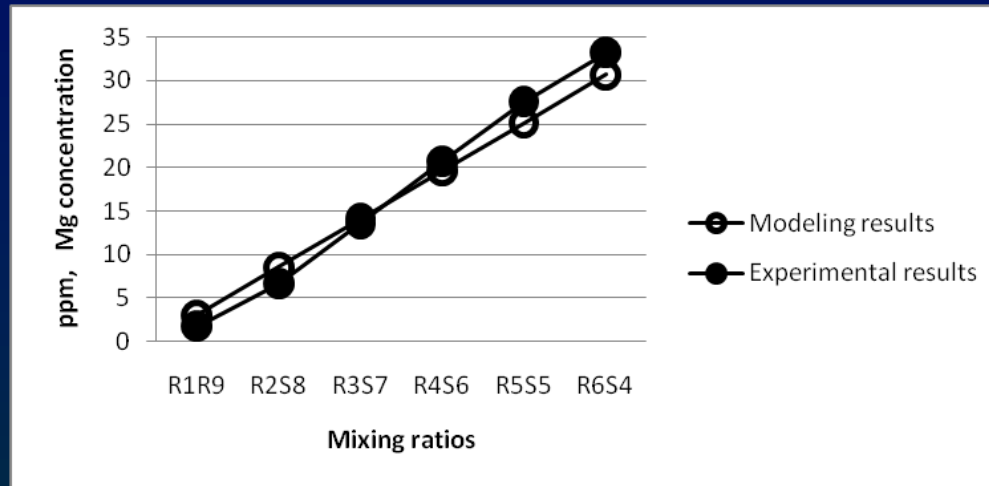
R_i and R_0 are the calcite crystal growth rates in the presence and absence of Mg^{2+} , respectively

K_{Ca} and K_{Mg} represent the respective adsorption constants for Ca^{2+} and Mg^{2+} on the calcite surface in the competitive adsorption system.

$$R_i = R_0 \times \frac{[Ca^{2+}]}{[Ca^{2+}] + 1.56 \times [Mg^{2+}]}$$

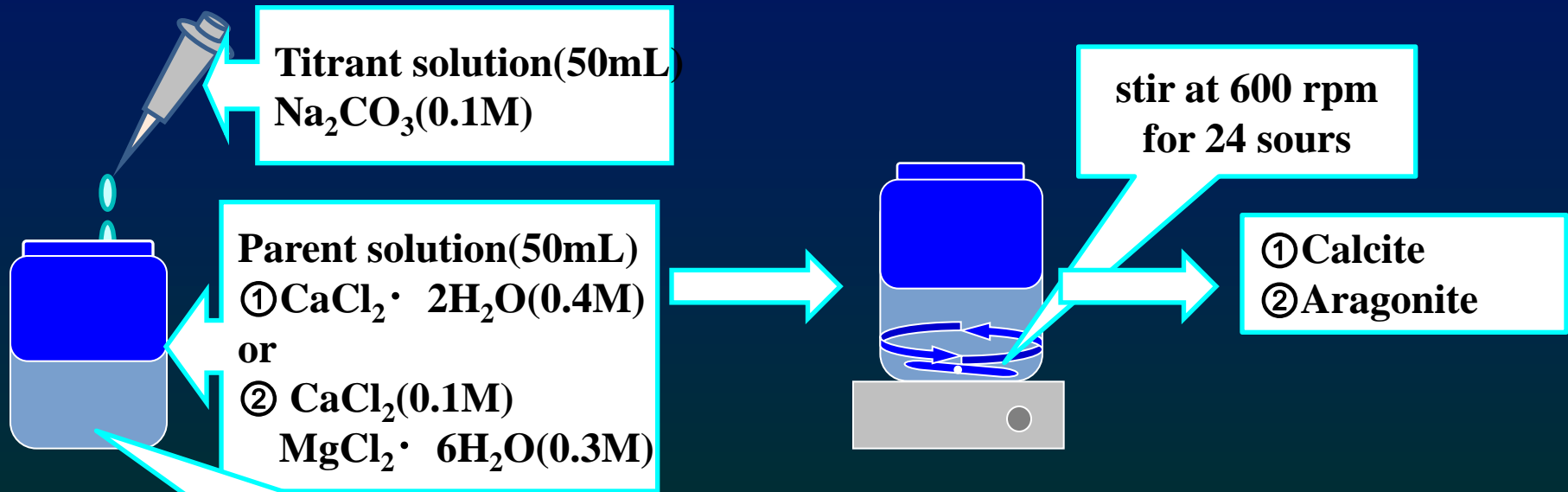
$$K_{Mg/Ca} = \frac{K_{Mg}}{K_{Ca}} = 1.56$$

Comparison between measured and calculated Mg concentration and calcite / aragonite ratios



Batch co-precipitation experiments in lab. to determine the K_d values of calcite and aragonite

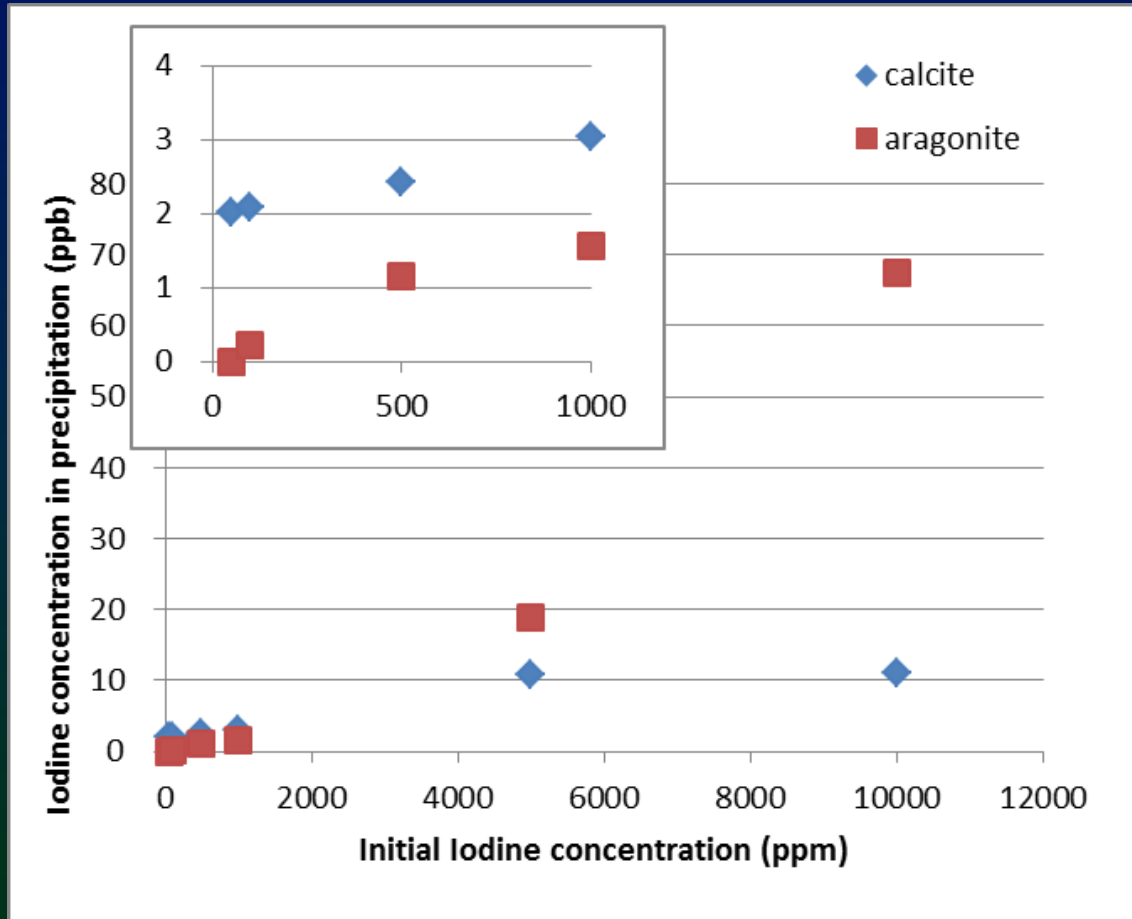
✓ Synthesis method



I^- 、 IO_3^- concentration
50, 100, 500, 1000, 5000, 10000ppm
added by sodium salt

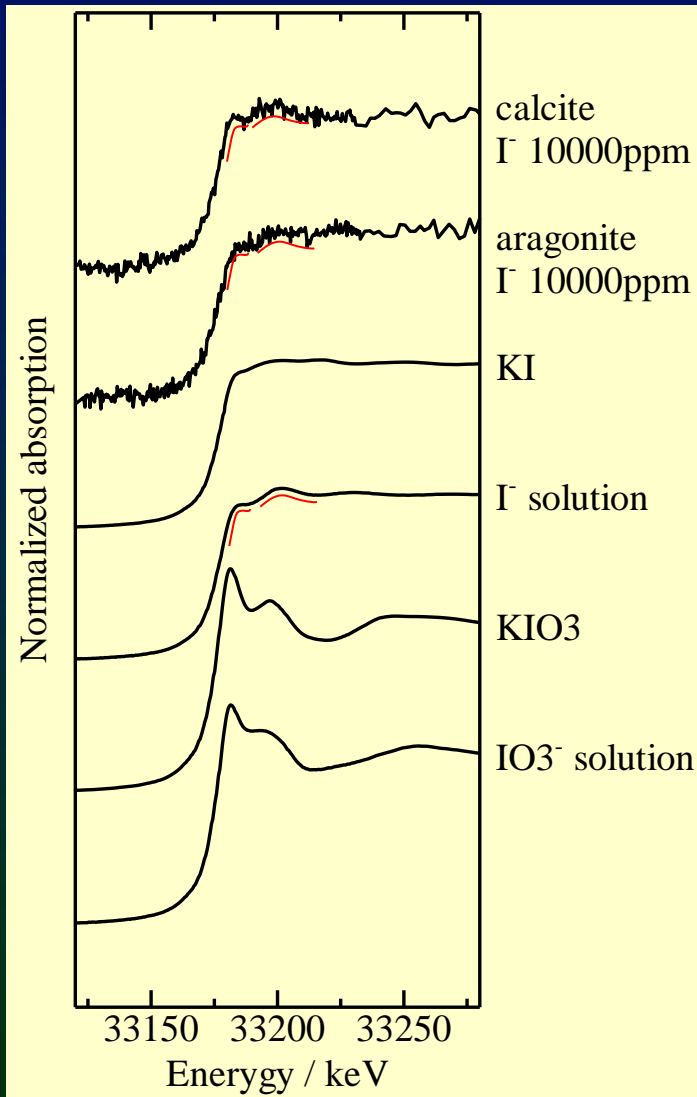
Calcite and Aragonite were synthesized
by the difference of Mg content in the
reaction solution

Result of iodide* (I^-) uptake as function of initial iodide conc.



Aragonite has greater uptake capacity of iodide at the range of higher iodide concentration (5000-10000 ppm).

Results of I *K*-edge XANES measurements



The pattern fitting of I *K*-XANES spectra of synthesized calcite and aragonite (initially 10000 ppm iodide added) indicated chemical shift from iodide to iodate is not existed.

The two weak peaks at around 33182 and 33199 eV to the formation of an octahedral complex through coordination of six water molecules with one I⁻ (Tanida et al. 2003).

These results support that I⁻ adsorbed to calcite and aragonite such as an outer-sphere complex in its hydratio



Previous study on halogen anion distribution to calcite and aragonite

(Kitano & Okumura, 1973)

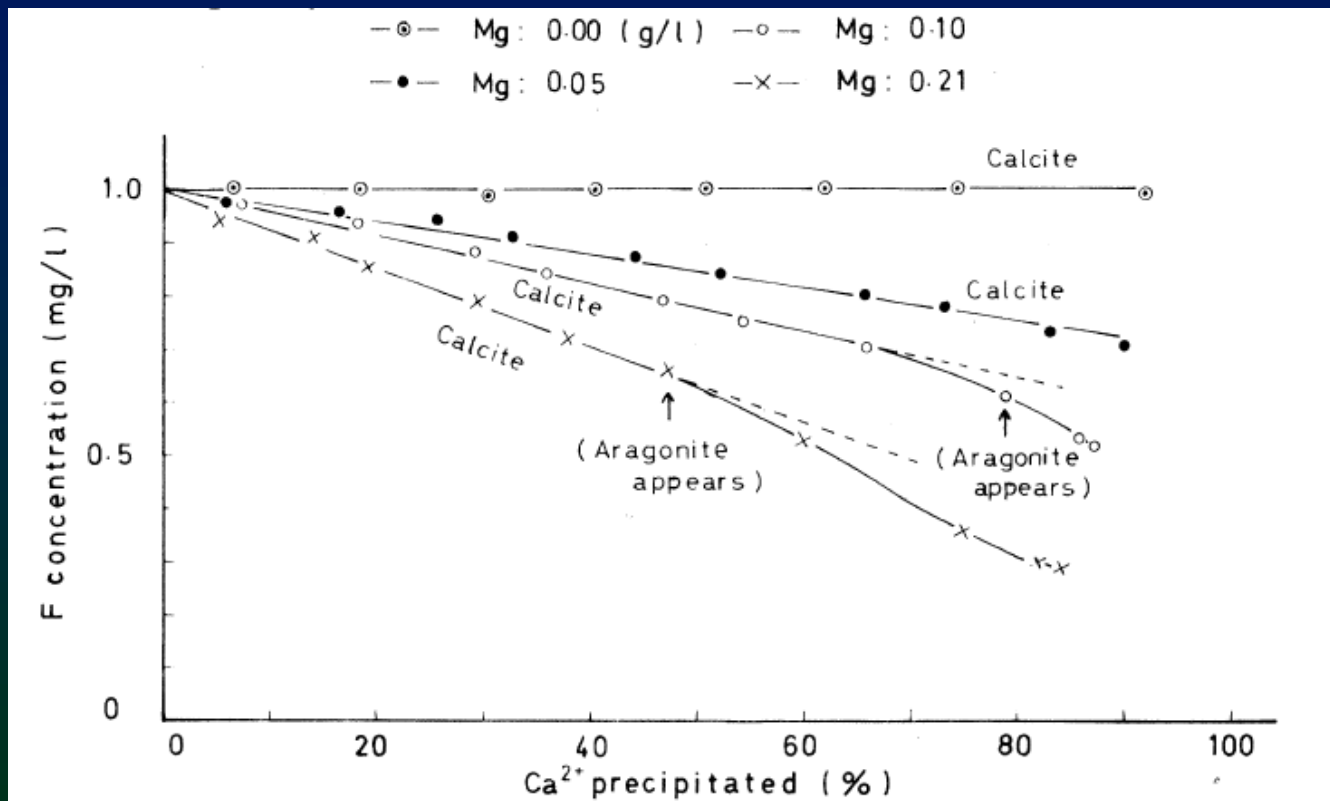


Fig.2. Change in fluoride concentration in a parent solution with per cent calcium precipitated ($25 \pm 1^\circ\text{C}$) in the system:
 $\text{Ca}(\text{HCO}_3)_2 + \text{MgCl}_2 + \text{NaF} \rightarrow \text{calcite} + \text{aragonite}; \text{ or calcite}$

Correlation between total iodine* and carbonate content at Callovian Oxfordian argillite (Claret et al. 2010)

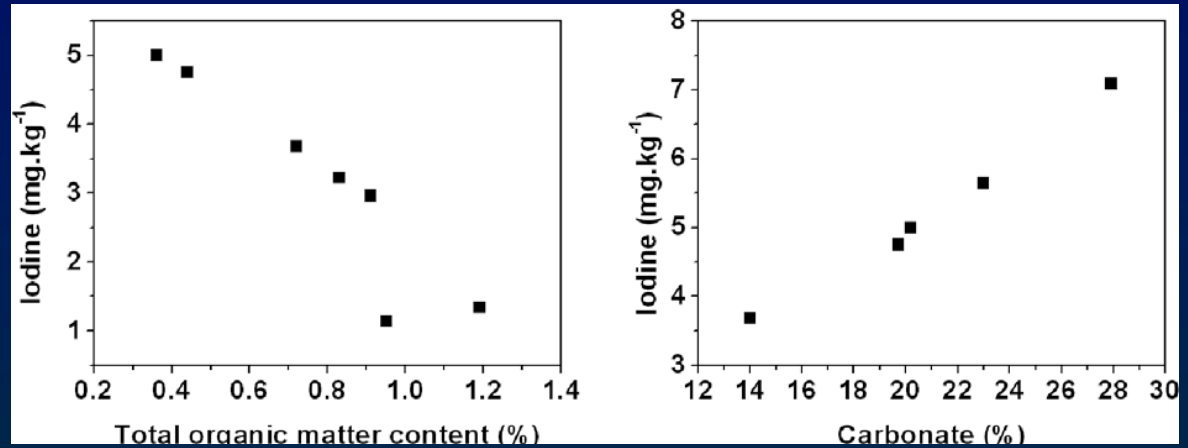
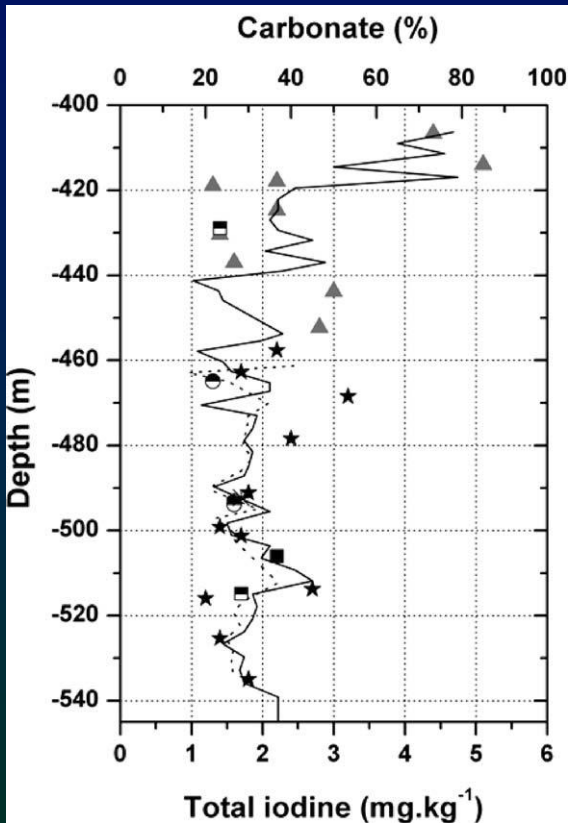


Fig. Total iodine* content as a function of organic matter content (left) and carbonate content (right) of the reference COx sample.

This finding is potentially in **conflict with** the usual assumption that iodine* is mainly associated with **the organic matter** of rock or sediment (Schlegel et al. 2006; Tournassat et al. 2007).

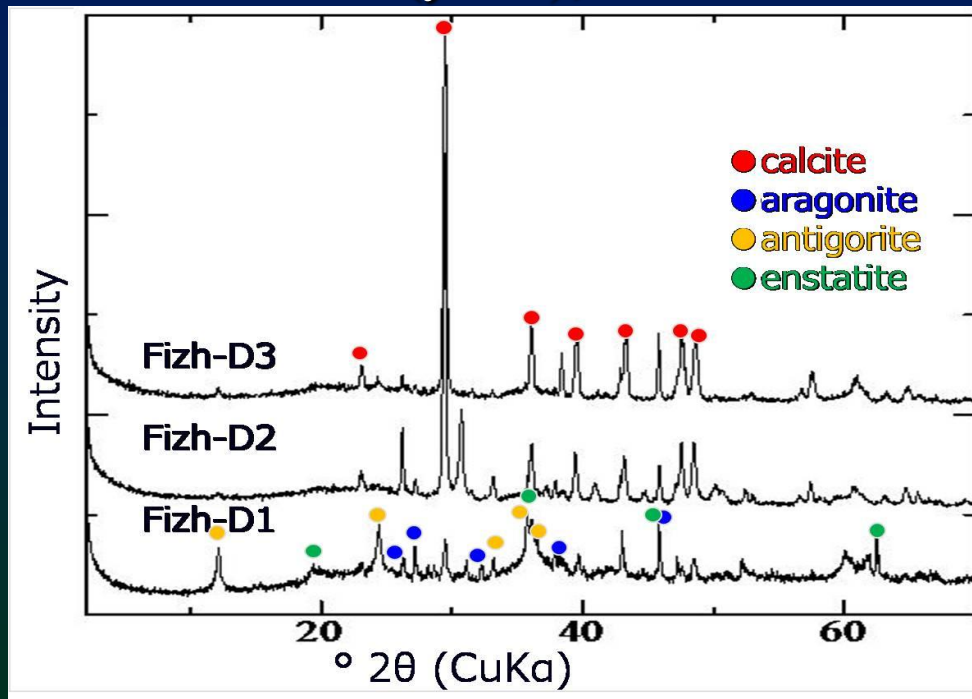
Fig. Total iodine* content (symbols, measured after HNO₃ digestion at 90 C overnight) and carbonate content (lines) as a function of depth.



Age determination of carbonate samples in the vein of river terrace

Conventional radiocarbon age

D2: 13900 ± 110 (y BP), D3: 12750 ± 70 (y BP)



Aragonite transformed to calcite. How is iodine sorbed in the aragonite for long-term?

Summary

- Aragonite was dominant phase in the observed precipitates at the mixing points of the springs and surface water, due to high Mg content in the surface water .
- Hydrotalcite (Mg-Al hydroxides) was also observed as accessory minerals when the hyperalkaline springs was comparatively rich in Al.
- During formation of the minerals at the mixing points, HCO_3^- in the surface water was mainly fixed as carbonate minerals such as aragonite and calcite.
- H_3SiO_4^- and Cl^- in the surface water were fixed into interlayers and/or surface of hydrotalcite.
- Iodide in the springs and surface water was mainly associate with aragonite.



Low temperature present-day-serpentinization
can provide some research fields such as

- Sciences at extremely hyperalkaline condition
- Natural and abiotic H_2 and CH_4 gas generation



Carbon Sequestration and Concrete Longevity



In situ carbonation of peridotite could consume **> 1 billion tons of CO₂ per year** in Oman alone, affording a low-cost, safe, and permanent method to capture and store CO₂ (Kelemen and Matter, 2008)



Faydh 20071223



Garra barreimiae



Why can the fish live in the hyperalkaline springs?

No biologist know fish surviving at such hyperalkaline pond.

Questions from biologist....

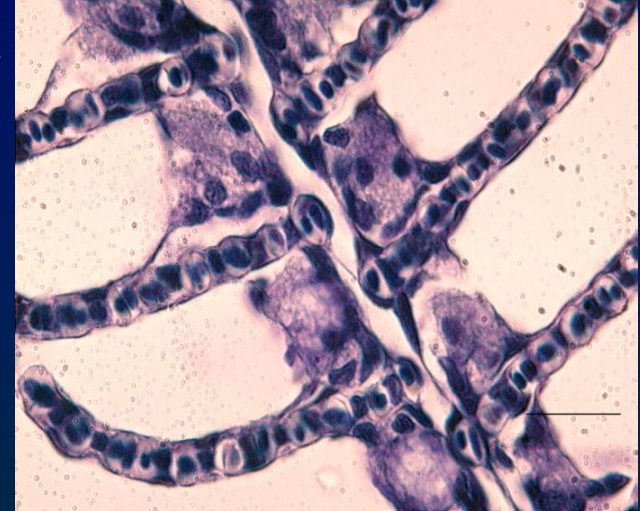
How does the fish control pH in their body fluid and blood?

How does the fish get a resistance capacity against hyperalkaline condition?

What is their skin structure?

What is their cell structure?

Why does the fish have so much cartilage (jiggling bone) ?

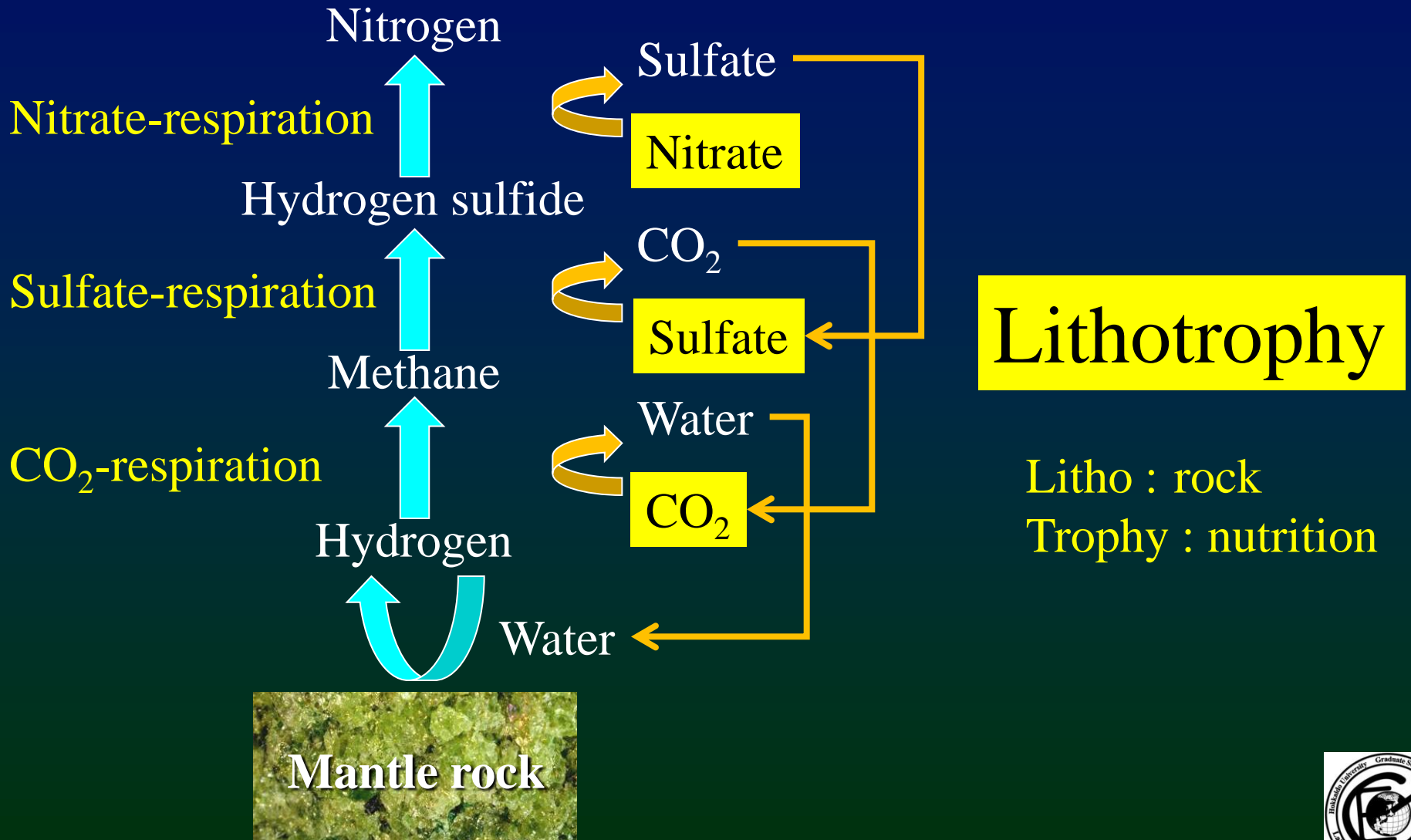


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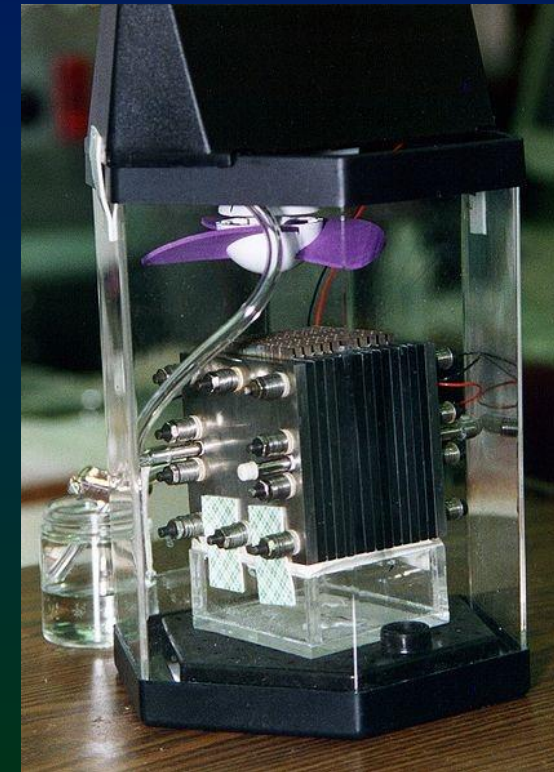
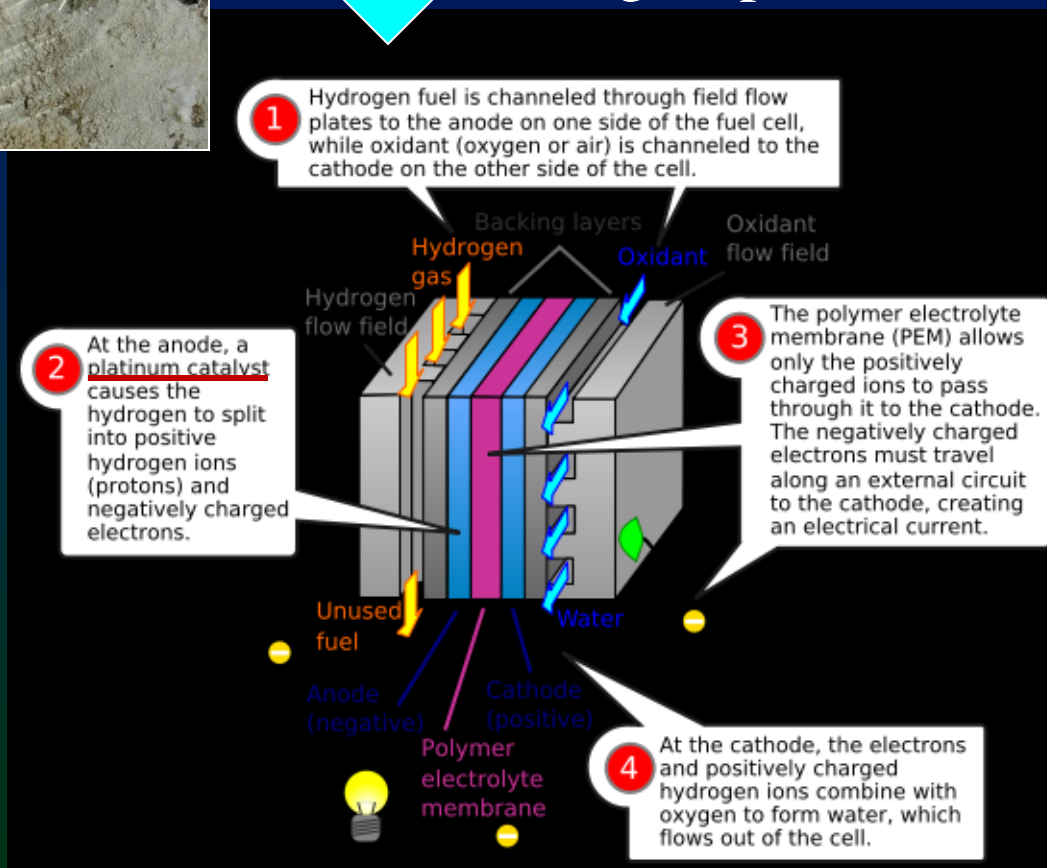
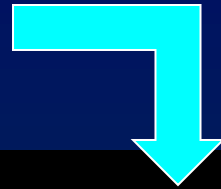
Food Chain Starting from Hydrogen Generated by Serpentinization



Hydrogen and Methane Generation

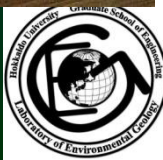


One cubic meter of olivine can deliver approximately 500 moles of hydrogen during serpentinization (Fisher, 2005).

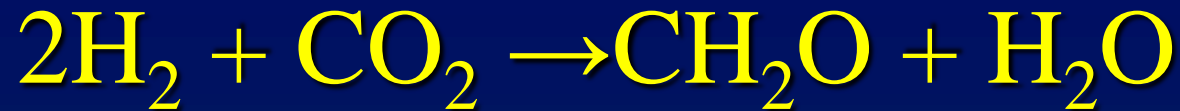


Awaruite
 FeNi_3

Hydrogen and hydrocarbon fuel cell



Petroleum Formation by Serpentinization



Some hydrocarbons may be produced by serpentinite reactions which is a key argument for the theory of abiogenic petroleum origin.



Olivine + Water + Carbonic acid → Serpentine + Magnetite + Methane

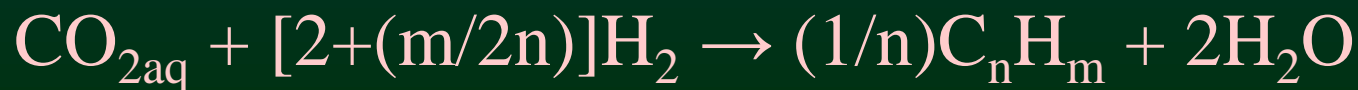
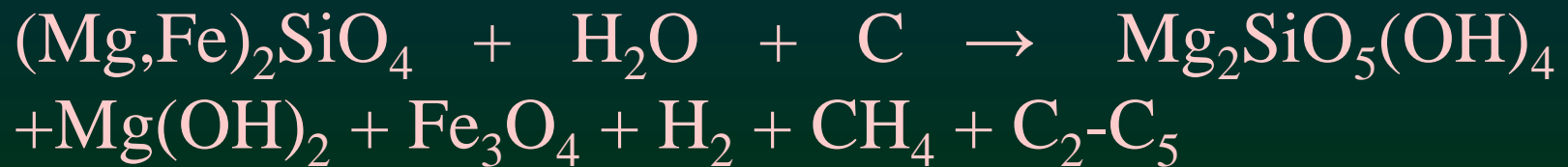
(“Petroleum formation by **Fischer-Tropsch synthesis** in plate tectonics” Peter Szatmari, AAPG bulletin, 73, 989-998, 1989)

Abiogenic petroleum have been suggested, originally by Robinson (“Duplex origin of Petroleum”, Nature, 199 113-114, 1963)



What is Fischer-Tropsch Type Reaction?

a well know chemical engineering process whereby millions of tons of hydrocarbon oil resembling petroleum (ex. Fischer-Tropsch Diesel) are produced annually from carbon monoxide or carbon dioxide and hydrogen gasses reaction on a metallic iron or iron oxide catalyst.

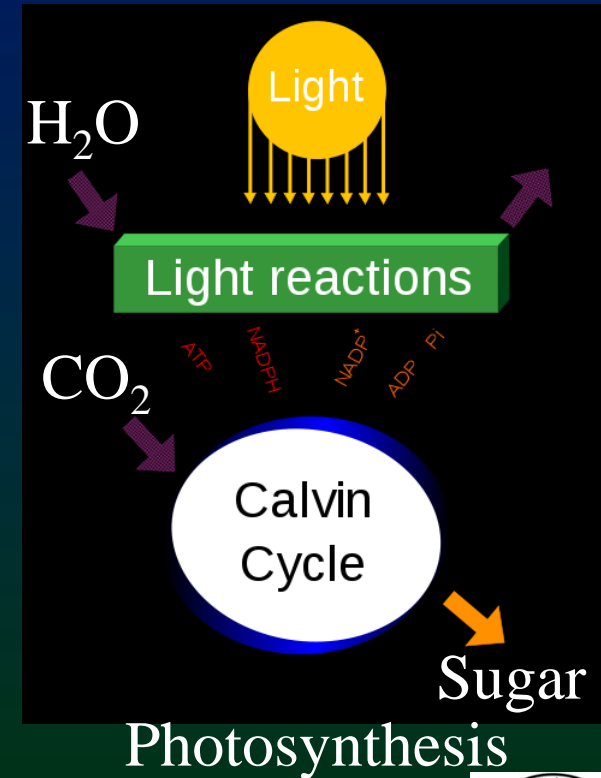


(Martin et al., 2008)



Not only hydrocarbon but also pentose (sugar), particularly ribose

- The high pH condition from serpentinization may promote abiotic formation of pentose, particularly ribose.
- Ribose is a constituent of RNA, and the related deoxyribose of DNA.



Low temperature present-day
serpentinization teaches us

Intelligent Earth Engineering Technology

for

Sustainable Geo- and Bio-resources

Utilization and Development



New Project (2009-2011)

on

Geo- and Bio-resource Science and
Technology Learnt from the Processes
in Hyperalkaline Springs at Oman

(オマーンに湧出する高アルカリ泉から学ぶ
アルカリ環境の地球・生物資源科学)



Based on the Concept of Holonc Path

creates the whole system by integrating autonomous sub-systems.

Research Background

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Workshop

Field Survey

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Geochemistry G.

Engineering G.

Microbiology G.

Physiology G.

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Engineering G.
Microbiology G.
Physiology G.

Ministry of Commerce and Industry of Oman
Person in charge :
Dr. Durair A'Shaikh
Japanese Embassy in Oman

Leader: **Tsutomu Sato**



Research Team at Oman



Natural process at hyperalkaline springs in Oman teaches us

- There are possibilities of anion retention by various mechanisms even at hyperalkaline conditions.
- There are many candidates as host minerals with different properties to remove anions from contaminated water.
- We can control the mineral composition if we can control the water chemistry in pores and fractures.
- Aragonite is a candidate for iodine (other halogen anions) retention by addition of Mg source (such as blast furnace slag) to the cement.
- Hydrotalcite is a candidate for silica (probably other oxyanions) retention by addition of Al source (such as aluminum sulfate) to the cement.



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- 21st COE Program, Hokkaido University, Japan

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- Prof. Miyashita and Dr. Adachi of Niigata Univ.
- Prof. Michibayashi of Shizuoka Univ.
- Drs. Yanase of JAEA
- Our students of Kanazawa and Hokkaido Univ.

