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Expanding the Limits of Experimental Fluid-Rock Interaction: The Supercritical Laboratory at ESNZ

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ABSTRACT

The Experimental Geochemistry Laboratory (EGL) at Earth Sciences New Zealand is advancing the study of fluid-rock interactions under extreme conditions through experimental work at temperatures up to 700°C and pressures up to 500 bar, encompassing both the subcritical and supercritical conditions. Using continuous flow reactors, EGL has investigated basalt-seawater and greywacke-fluid interactions, as well as the effects of CO_2 on fluid-rock interactions. Detailed measurement of mineral solubilities of quartz and anhydrite under supercritical conditions have been completed. The acquisition of the Geothermal Process Simulator expands EGL's capability to study fluid-rock interactions, scaling, and corrosion, supporting both fundamental geochemical research and the sustainable development of geothermal energy operations.

1 INTRODUCTION

The study of water-rock interactions under high temperature and pressure conditions has long been a cornerstone of geochemical research, particularly in the context of geothermal energy exploration and production. As the demand for sustainable energy sources grows, the need for advanced experimental techniques to replicate and understand chemical reactions that occur within reservoirs and geothermal power facilities has become increasingly important.

The Experimental Geochemistry Laboratory (EGL) at Earth Sciences New Zealand (ESNZ) has taken a significant advance in this field by expanding its capabilities into the supercritical pressure-temperature region. With the acquisition of state-of-the-art equipment, the EGL now supports experimental work at temperatures reaching up to 700°C and pressures up to 500 bars. The laboratory's enhanced capabilities allow experimental simulation of geochemical processes under supercritical conditions.

To date, the EGL has conducted a series of studies focused on water-basalt interaction, water/brine-greywacke interaction, CO_2-brine co-injection, corrosion, and mineral solubilities at supercritical conditions. By simulating these extreme environments, the EGL contributes not only to the advancement of geochemical understanding but also to the development of practical solutions for the geothermal industry.

2 EXPERIMENTAL WATER-ROCK INTERACTION

2.1 Basalt-Seawater Interaction

A new experimental method has been developed to investigate basalt-seawater interactions under near supercritical conditions (400°C/500 bar) using one of EGL's continuous flow reactors (Passarella, 2021). This approach, the first to achieve continuous seawater flow through rock at such extreme conditions, used a custom-built *temperature gradient* pressure vessel (Preheater Vessel) that heats the seawater from ~40°C to 377°C as it descends (Figure 1). During heating, retrograde solubility minerals such as anhydrite, caminite, and brucite precipitate within the Preheater, preventing blockage of the narrow Ti tubing and the basalt in the second pressure vessel (Reactor Vessel). Zirconia beads (~2 mm) provided surfaces for mineral deposition. This approach allowed identification of distinct alteration fronts including glass dissolution, chloritization, and anhydrite precipitation. These would not be discernible using a batch reactor approach.

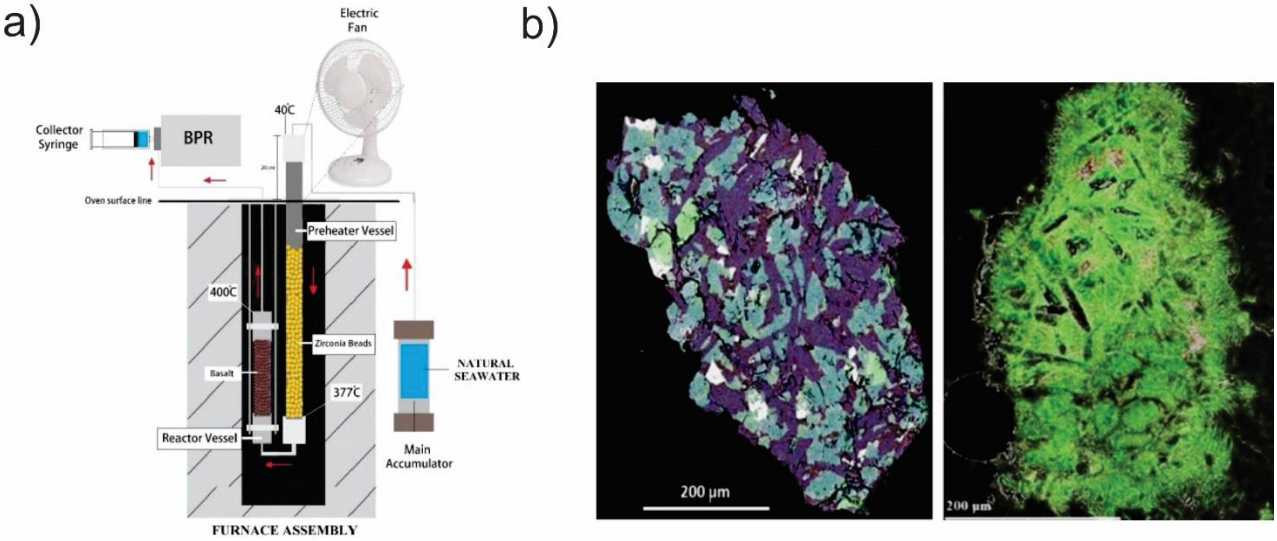


Figure 1. a) Continuous flow temperature gradient pressure vessel (Preheater Vessel) with second pressure vessel (Reactor Vessel) containing basalt. b) unreacted (left) and reacted (right) basalt fragments.

2.2 Greywacke – Fluid Interaction

2.2.1 Greywacke – Distilled Water

The basement rocks underlying the Taupo Volcanic Zone (TVZ) are composed of Mesozoic-aged interbedded mudstone and sandstone, collectively known as greywacke. These rocks are frequently intersected in conventional geothermal wells and serve as the primary host for brine reinjection. At greater depths, they are also expected to host potential supercritical geothermal fluids within the TVZ.

To investigate water-rock interactions under such conditions, a series of high T-P experiments was conducted to examine the evolution of mineralogy and fluid chemistry over time. A representative New Zealand greywacke was reacted with fluids of differing compositions - distilled water, geothermal brine, and geothermal brine with added CO_2 – under conditions ranging from 350 to 500°C and 200 to 500 bars, simulating deep subcritical to supercritical environments.

Under supercritical conditions (400°C/500 bar) with distilled water, quartz dissolution from the greywacke dominates. This is expected as fluid initially contains negligible silica. Effluent SiO_2 concentrations range up to 1450 mg kg^{-1}, consistent with quartz saturation at these conditions. Other dissolved components (Na, K, Ca, Mg, Al, and Fe) remain at low concentrations (<10 mg kg^{-1}). However, SEM imaging of reacted rock surfaces reveals significant secondary mineral growth including albite, chlorite, and unidentified phases that are likely amphiboles (e.g. actinolite) (Figure 2a). This indicates considerable element mobility at the rock surface, despite low bulk fluid concentrations. The low cation concentrations can be attributed to supercritical water's reduced density (0.63 kg m^{-3}), lower dielectric constant (~27), and its weak hydrogen bonding network.

At 500°C/200 bar, effluent silica concentrations are significantly lower, reaching a maximum of ~12 mg kg^{-1}. The steady state concentrations of other dissolved components are all near or below detection limit. SEM imaging reveals a distinctly different mineralisation pattern, with the rock grains coated by a semi-continuous layer of cristobalite (Figure 2b). This suggests that at this temperature and lower fluid density, only SiO_2 exhibits appreciable mobility, whereas other rock-forming elements remain immobile in the low-density (0.09 kg m^{-3}) and low dielectric constant (~6) fluid.

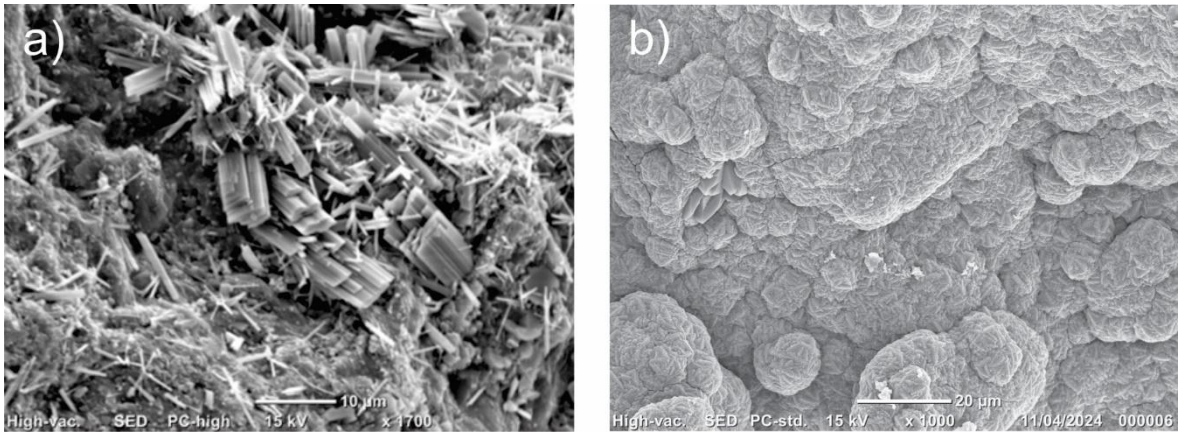


Figure 2. SEM images of greywacke reacted with distilled water at a) 400°C/500 bar and b) 500°C/200 bar. At 400°C there has been extensive remobilization of rock components while at 500°C the rock is covered with a monomineralic coating of cristobalite.

2.2.2 Greywacke – Geothermal Brine

Two experiments were conducted at 350°C/300bar and 400°C/250 bar, in which the greywacke was reacted with a reinjection brine sourced from a New Zealand geothermal power station. In these experiments the brine is expected to dominate the exchange of components with the rock due to its high solute concentrations. Such conditions could occur if a conventional re-injection brine were introduced into a geothermal reservoir at near-supercritical or supercritical conditions.

At 350°C/300 bar, the dominant reaction was quartz dissolution, consistent with the reinjection brine being undersaturated with respect to quartz at these conditions. SEM analysis revealed that the rock surfaces were coated with newly formed secondary minerals, primarily albite (Figure 3a). At this temperature and pressure, the fluid density (0.7 kg m^{-3}) is sufficient to sustain relatively high solute concentrations.

At 400°C/250 bar, the rock surfaces were coated with newly formed quartz crystals, albite, and at an unidentified phase, probably an amphibole (Figure 3b). Under these conditions, the fluid density (0.3 kg m^{-3}) is considerably lower, and quartz becomes supersaturated in the entering fluid, resulting in its precipitation.

These results indicate that introducing a conventional reinjection brine into a supercritical system is not advisable, as the large changes in fluid density and dielectric constant can initiate extensive precipitation of secondary mineral phases, potentially impairing permeability and reservoir performance.

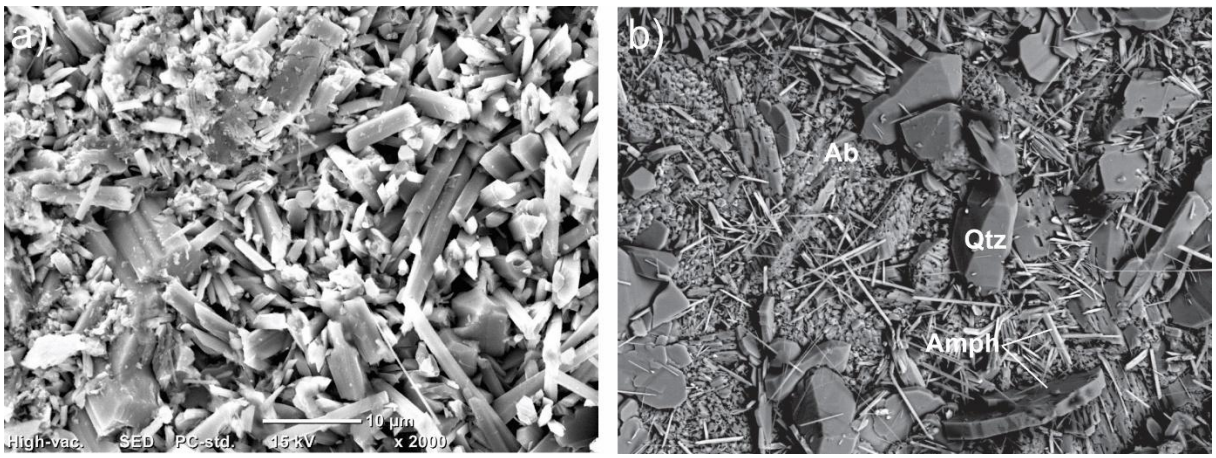


Figure 3. SEM images of greywacke reacted with geothermal brine at a) 350°C and b) 400°C. At 350°C the rock is covered with a coating of primarily albite, while at 400°C, quartz (Qtz), albite (Ab), and an unidentified mineral (Amph), most likely an amphibole, are found.

2.2.3 Greywacke – Brine – CO_2 interaction

A single experiment was conducted at 400°C/250 bar to simulate the re-injection of a conventional geothermal brine containing added CO_2 at a concentration of 2000 mg kg^{-1}. The purpose of this experiment was to evaluate the potential for CO_2 sequestration within greywacke under supercritical conditions.

SEM examination of the reacted rock surface showed extensive dissolution and restructuring of rock components (Figure 4). The newly formed minerals included albite, anhydrite, and an unidentified phase, likely an amphibole. Quartz was not observed as a secondary mineral phase in this experiment. This suggests that the introduction of elevated CO_2 concentrations, lowers the fluid pH, significantly enhancing the mobility of rock components. Consequently, SiO_2 was consumed in the precipitation of other aluminosilicate phases, leaving the fluid undersaturated with respect to quartz.

No secondary carbonate phases were identified; however, extensive mobilisation of calcium was evident from the presence of anhydrite. These results indicate that under supercritical conditions, CO_2 sequestration via carbonate mineral formation in greywacke is not expected to be an active mechanism.

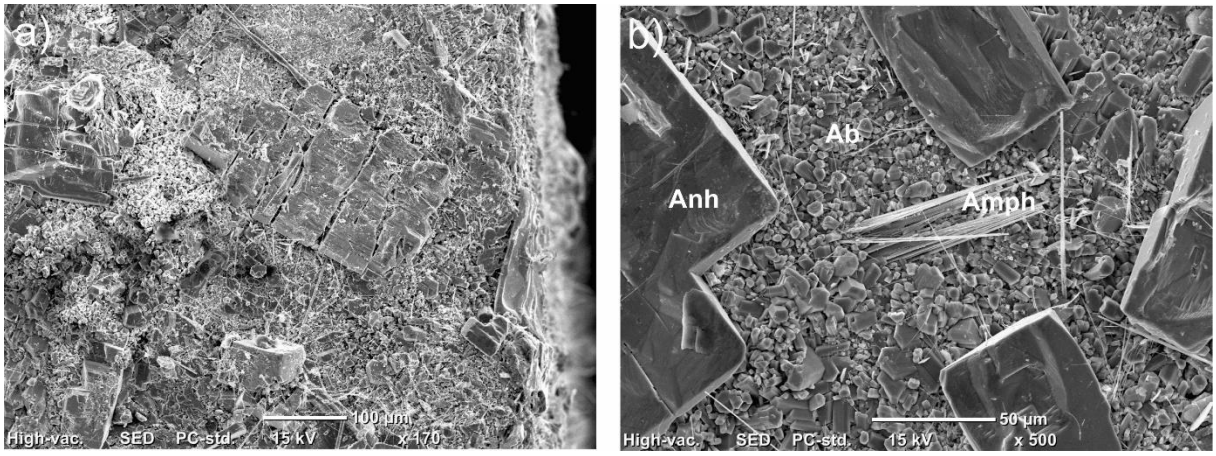


Figure 4. SEM images of greywacke reacted with geothermal brine + CO_2 at 400°C/250 bar. In a) extensive corrosion of the rock surface is observed. In b) newly formed phases include albite (Ab), anhydrite (Anh), and an unidentified phase (Amph).

3 MINERAL SOLUBILITIES

The measurement of the thermodynamic parameters of key minerals such as quartz and anhydrite under supercritical conditions is critical for the design and operation of next-generation geothermal power stations. At the EGL, detailed studies have been conducted on the solubility behaviour of these two minerals at elevated temperature and pressure conditions relevant to supercritical geothermal systems.

3.1 Quartz

The solubility of quartz in distilled water has been measured over the temperature range of 375 - 600˚C and pressure range of 200 - 270 bars (Rendel and Mountain, 2023). These conditions were selected to reproduce the conditions expected to be found in deep geothermal systems in the TVZ. The experiments were conducted using EGL's supercritical continuous flow reactor.

The resulting dataset provides significantly improved constraints on quartz solubility under these supercritical conditions, an area that was previously poorly defined. The data were used to refine the empirical equation of Fournier and Potter (1982), improved predictive capability for quartz solubility in geothermal reservoirs operating within or near the supercritical region.

3.2 Anhydrite

The solubility of anhydrite ($CaSO_4$) in distilled water has recently been measured in the temperature range 380 – 625°C and pressure range 220 – 270 bar (Rendel et al. 2024). These experiments represent the first comprehensive dataset for anhydrite solubility at these extreme conditions. Prior to this work, anhydrite solubility in the supercritical region was poorly constrained. The new experimental data were used to refine the empirical parameters of the Dolejš and Manning (2010) solubility model.

Results show that anhydrite solubility is extremely low under supercritical conditions, reaching only about 1 mg kg^{-1} at 380°C (Figure 5a). The solubility decreases, with increasing temperature, to below 10 µg kg^{-1} (Figure 5b). The results also demonstrate that anhydrite dissolution under supercritical conditions is incongruent, indicating the formation of one or more secondary Ca-bearing phases.

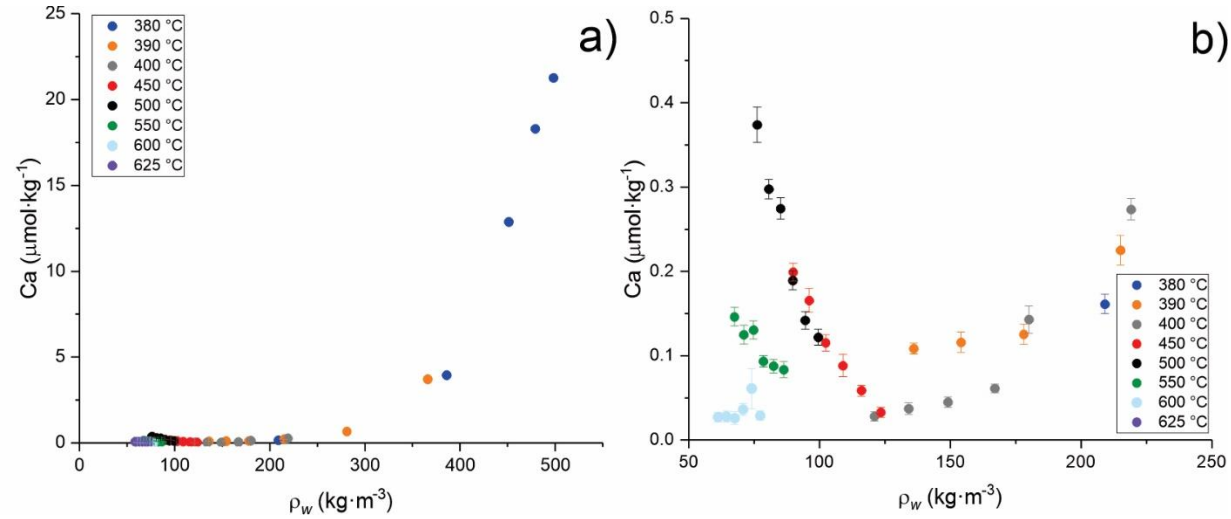


Figure 5. The solubility of anhydrite at supercritical conditions in terms of Ca concentration a) 0 – 25 µmol kg^{-1} (0 – 1 mg kg^{-1}) and b) 0 – 0.5 µmol kg^{-1} (0 – 20 µg kg^{-1}).

4 FUTURE RESEARCH

The Experimental Geochemistry Laboratory (EGL) at ESNZ has recently acquired a new flow-through simulator (Figure 6), which we have named the *Geothermal Process Simulator (GPS)*. This instrument enables continuous flow experiment reacting aqueous fluids with rocks, minerals, alloys, and polymers, at temperatures up to 600 °C and pressures up to 500 bar. The system provides the capability to reproduce and monitor geothermal processes both in surface infrastructure and in the subsurface, up to and including supercritical conditions. The new instrument will be used to investigate mineral dissolution and precipitation kinetics, scaling and corrosion behaviour, tracer transport, and fluid–rock interactions relevant to geothermal energy production and reservoir sustainability.



Figure 6. The Geothermal Process Simulator (GPS).

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