The resin sealed column (RESECO) setup for flow-through experiments on solid rocks under high temperature and high pore pressure conditions

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Abstract

High-pressure flow-through experiments on solid rock samples are commonly conducted with experimental setups using a confining pressure to restrict the flow to the rock. These setups are often spacious, costly, and difficult to replicate by other researchers due to their individual nature. This work presents the RESECO (resin sealed column) setup which allows flow-through experiments on solid rock without a confining pressure. The column setup is only slightly larger than the sample size and has material costs per sample of a few Euros. The fluid flow is confined to the rock by a cast of epoxy resin using a metal column as an outer casing. The functionality was verified by comparing experimental results with a conventional triaxial cell. Four different rock types with varying hydraulic properties were tested and proven compatible with the setup. Additional endurance tests were performed to investigate the physical limits with regard to pore pressure and temperature. The RESECO setup can be operated with pore pressures of at least 40 MPa and temperatures up to 95 °C, and is therefore suitable for many high-pressure, high-temperature experiments, while being easily reproducible.

Keywords Laboratory experiments · Equipment · Hydraulic properties · Low-permeability media · Flow-through

Introduction

Flow-through experiments involving porous and fractured materials are crucial in many scientific and engineering applications such as reservoir characterization, exploitation, or management. The used experimental setups are as diverse as the questions addressed. For high-pressure experiments, a confining pressure on a cylindrical sample is used to confine the flow to the rock, which is the confinement method used in triaxial cells. Those are often specifically tailored to the needs of the experiment and result in quite large experimental setups. One common application for such flow-through experiments is the characterization of geothermal reservoirs. Conducted experiments with pore pressures above 30 MPa include the study of petrophysical parameters (Milsch et al. 2008, 2009; Pei et al. 2014), reservoir mechanics (Atapour and Mortazavi 2020), and permeability evolution due to scaling effects (Orywall et al. 2017).

Experimental setups on solid rocks without a confining pressure are the exception. Ju et al. (2020) used a simple setup to investigate the healing of fractures. They confined the flow to the fracture using silica gel and resin adhesive but water flow was only driven by gravity. Frank et al. (2020b) conducted tracer tests with gravity-driven flow in fractured rock samples isolated by a rubber jacket. Low hydraulic pressures and comparably high permeability allowed for these simple confining methods, but both approaches are insufficient for high-pressure applications like the ones described in the preceding.

Other researchers simplified their experimental setup, voluntarily or by necessity, by using loose material in a column to sidestep the need for confining pressure. Tatamir et al. (2018) did flow-through experiments using glass beads as the matrix to have well-constrained conditions for developing kinetic interface-sensitive tracers, with application to CO₂ storage in reservoirs. Maier et al. (2015) used sand for their experiments on thermosensitive tracers for the study of geothermal reservoirs.

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In both cases, further studies prior to field application would include laboratory flow-through experiments on solid rock.

An alternative confinement method is the sealing of the stone surface with epoxy resin. This method was successfully applied in the past (e.g. Goode et al. 1984; Ettinger and Radke 1992; Jenneman and Clark 1992; Rangel-German and Kovscek 2001) but only for low pore pressure up to 10 MPa. The method has been proven to be low on space demands and is considered to be rather simple in its application; however, a detailed description of a workflow to allow replication of the method was only found in Robertson et al. (1994). Therefore, an in-depth investigation of epoxy as a seal for flow-through experiments on solid rocks is necessary, while extending its range of applicability to reservoir temperature and pressure conditions, and providing a detailed description of the workflow. This work introduces an improved experimental setup, the RESECO setup (resin sealed column), which can be easily replicated with little expense for materials, and which is compatible with state-of-the-art flow-through measurement devices. A conventional preparatory column for HPLC (high-performance liquid chromatography) is used as an outer casing. A solid rock sample is then glued into the preparatory column using epoxy resin. The improved design was applied to determine the permeability of four different rock types and to test the applicable range of temperature and pressure conditions. The experimental findings were verified by comparing the results of the new RESECO setup with those of a conventional triaxial cell.

Materials and methods

Materials

The RESECO setup was built into a preparatory HPLC column and sealed with epoxy resin. The stainless steel HPLC column from Göhler HPLC-Analysentechnik in Chemnitz, Germany, has a length of 50 mm and an inner diameter of 20 mm. Two endcaps close the column on each end. Between the column and cap, a frit surrounded by a PTFE seal is installed to prevent solid, nondissolved material from entering and leaving the column as well as to disperse the fluid over the complete cross section of the stone. This column has an additional stainless-steel plate containing the connection conus for the fitting, which in other cases is included in the endcap. This design has no advantage or disadvantage for the given purpose. The HPLC column can be connected at inflow and outflow with 1/4 in (0.6 cm) fittings to various instruments and capillary tubes.

The epoxy resin EPO 242 from Schouten group SynTec, Netherlands, was used to glue the stone plug into the column. It has a low viscosity (350–650 mPas) and a high shore hardness (80–85). Casting molds for the epoxy resin were made from polyurethane casting rubber 90 from Schouten group SynTec with shore hardness 90 to avoid deformation during casting. The release agent Pol-Ease 2300 from Schouten group SynTec was applied to all molds before casting. The columns were degreased using tetrachloroethylene from ThermoFischer Scientific in Loughborough, UK and isopropanol from Sigma-Aldrich in Steinheim, Germany. Ultrapure water was taken from a Milli-Q IQ 7003 from Millipore in Molsheim, France.

The stone plugs were drilled with either 30 or 16 mm diameter for the experiments with the triaxial cell or the RESECO setup, respectively. All plugs of one stone type were drilled from one single block. Sandstone samples were drilled orthogonal to bedding, while for the limestones, the bedding orientation could not be determined. The stone plugs for the triaxial cell were sawed and sanded down to 60 mm length, while the plugs for the new experimental setup were not specially prepared before the casting, either in length or smoothness. Then the column preparation for the RESECO setup was done as described in the next section.

Flow-through experiments with the RESECO setup were done using ultrapure water with 25 mg/L of NaCl and a Bio HPLC system consisting of a pump, oven, and detectors from Shimadzu Deutschland in Düsseldorf, Germany. The flow rate was measured by the pump. The pressure difference along the rock sample was measured with electronic pressure transmitters PT5460 from ifm in Essen, Germany. Experiments for comparison were done with a triaxial cell 32-D0553/HC AX from Controls-Group, Italy, and ultrapure water. The flow was measured by a VP12K pump from Vindum Engineering in Sandpoint, USA, and the pressure was measured by a high-pressure transducer from Burster Gernsbach Präzisionsmesstechnik in Gernsbach, Germany.

Preparation of the RESECO setup

Prior to assembling the RESECO setup, casting molds (Fig. 1a) and stone plugs of 16 mm diameter and roughly 7 cm length had to be prepared. The inner surface of the column was roughened to increase the bonding strength between metal and epoxy resin. A continuous rill of 0.2 mm depth and 1 mm distance was lathed into the column for this purpose.

The column was washed, dried, and degreased by overnight immersion (>20 h) in tetrachloroethylene. The remnants of the tetrachloroethylene were allowed to evaporate under an extractor hood prior to further handling. The epoxy resin was mixed and degased in a desiccator for at least 1 h. In the meantime, the rubber molds were sprayed with a release agent. The degreased column and the molds were assembled. Care had to be taken to avoid getting the release agent in the inside of the degreased column. The outside of the assembled mold was degreased using
isopropanol to allow the application of hot glue at the transitions between the column and rubber mold. A stone plug was inserted into the mounting gap in the lower rubber mold and manually centered. The degased epoxy resin was slowly poured into the mold to avoid trapping any air bubbles. The complete assembly for casting can be seen in Fig. 1a. The epoxy resin was then dried at 30 °C for 7 days. Demoulding could be done at the earliest after 2 days. An additional heating up to 80 °C was applied in the last 5 h following manufacturer instructions specific to this epoxy resin. The column protruding cast was removed using a saw and the ends were sanded down to create plane-parallel surfaces. After finishing the column preparation, the stone was saturated by evacuating the open column in a desiccator. The sample was desiccated outside of fluid for 60 min before being pushed over a slide into the saturating fluid for at least 23 h. During column assembly, the PTFE seals and frits of the column were replaced with broader seals with an inner diameter of 18 mm instead of 20 mm and adjusted smaller frits. The complete RESECO setup can be seen in Fig. 1b.

After the experiments, the columns could be recycled by pushing the epoxy-stone core out using a stamp and a manually moving standing drill. This treatment left the columns intact but created microcracks in the stone plugs.

**Rock samples**

Four different stone types (Fig. 2) were used to apply the method to a variety of materials. Two sandstones and two limestones were chosen as outcrop analogs for geothermal reservoir rocks. They cover a wide range of values for various properties—such as porosity, permeability, surface roughness, and type of porosity (porous and fissured)—in order to check the applicability of the method regarding these parameters of the stones.

**Flechtinger sandstone**

The Flechtinger sandstone (FLE) belongs to the lithostratigraphic unit of the Rotliegend from the Permian period (Heiland 2003). The samples come from the quarry in Bebertal near Magdeburg in Saxony-Anhalt, Germany. The porosity for samples from the same charge was determined as 9.6% ± 0.1% (Frank et al. 2020a) but varies for the Flechtinger sandstone from 5.5 to 11% (Stanchits et al. 2011; Hassanzadegan et al. 2012). The permeability ranges from 1 × 10^{-16} m^2 to 1 × 10^{-17} m^2 (Heiland 2003; Frank et al. 2020a; Stanchits et al. 2011; Hassanzadegan et al. 2012). The Flechtinger sandstone has a light red color and is homogeneous. The bedding is clearly visible and makes the stone anisotropic.

**Remlinger sandstone**

The Remlinger sandstone (REM) belongs to the lithostratigraphic unit of the Upper Buntsandstein from the Triassic period (Frank et al. 2020b). The samples were collected from the quarry Remlingen near Würzburg in Bavaria, Germany (Frank et al. 2020b). Frank et al. (2020b) determined the porosity and the permeability of the Remlinger sandstone to be 12.9% ± 0.3% and 5.50 × 10^{-17} m^2 ± 8.24 × 10^{-18} m^2. The Remlinger sandstone has a red color and is homogeneous and isotropic. No bedding is visible, although the glimmer crystals have a preferred orientation.

**Black Massenkalk**

The black Massenkalk (BMK) samples were taken from the Dorp facies of the Upper Devonian period (Lippert et al.
The samples were collected in the quarry Hohenlimburg near Hagen in North Rhine-Westphalia, Germany. Lippert et al. (2020) determined the porosity of the Massenkalk at this location to be 0.34% and the permeability to range from $1 \times 10^{-20} \text{ m}^2$ to $1 \times 10^{-18} \text{ m}^2$. The Massenkalk has a dark gray to black color and is thick banked. Within the banks, the stone appears isotropic and homogeneous, besides some calcitic veins of different sizes.

Red Massenkalk

The red Massenkalk (RMK) originates from the same location as the black Massenkalk and was sampled some tens of meters away from it. It belongs to the Dorp facies of the Upper Devonian period as well. Balcewicz et al. (2020) determined the porosity of the Massenkalk at this location to be 10.4% and the permeability to range from $1 \times 10^{-15} \text{ m}^2$ to $1 \times 10^{-16} \text{ m}^2$. The dolomitized Massenkalk has a reddish, multicolored appearance. The high amount of veins makes the stone heterogeneous. Not all fractures are filled or completely filled, which causes higher permeability in comparison with the black Massenkalk. The open fractures have an aperture width of about 100 μm.

Experiments

The aim of the experiments was firstly to prove the functionality of the RESECO setup. Darcy experiments to determine the rock permeability were conducted and compared to Darcy experiments using a conventional triaxial cell. A difference in the results beyond the natural variability of the stone and the slightly different experimental conditions would uncover a surrounding flow in the new experimental setup. Secondly, endurance tests were conducted to test the limits of the RESECO setup with regard to experimental conditions, specifically temperature and pore pressure. Besides the bare functionality under these conditions, the behavior of the RESECO setup with changing temperature and pore pressure was investigated.

Functional tests

Darcy experiments on the bigger stone plugs were done at room temperature with a triaxial cell to provide reference values for the permeability of the stone types. The Darcy experiments were conducted with a confining pressure of 2 MPa and different flow velocities. The flow velocities were increased step by step until a pore pressure close to 2 MPa was reached. Higher flow rates and thus more sampling points were not tested to avoid compression of the stone samples and thereby altering the permeability by the confining pressure.

Up to three columns of the new experimental setup for each stone type were prepared as described in section ‘Preparation of the RESECO setup’. Darcy experiments were then conducted using small flow rates to avoid any pressure effects, which were investigated during the following endurance tests. The lower limit for the flow rate was a resulting pore pressure of at least 3.5 MPa to ensure proper functionality of the HPLC pumps. The RESECO setup was placed in the HPLC oven at 40 °C for the Darcy experiments. The thermal expansion due to the difference between assembling temperature and experimental temperature was a safeguard for leaking at the capillary connections.

The permeability $k$ of the stones was then calculated using Darcy’s law

$$Q = \frac{kA}{\mu L} \Delta p$$  \hspace{1cm} (1)

The flow $Q$ was set during the experiments and the back pressure $\Delta p$ was measured. Due to the abrasive step during column preparation, the length $L$ of the columns varied slightly and was measured, as was the length of the stone samples used in the triaxial cell. The cross-sectional area $A$ was measured after drilling the stone plugs. Then for the RESECO setup it was corrected for the intruding epoxy resin using photographs from the top and bottom of the prepared columns (Fig. 3). The harmonic mean of those two areas was used to calculate the permeability. Further cross-sections on exemplary cores confirmed the linear trend of penetration depth.
between top and bottom of the column. The viscosity $\mu$ of the water was corrected for the respective pressure and temperature conditions as taken from literature values provided by the Python library CoolProp (Bell et al. 2014). A comparison of the calculated permeabilities of the two experimental setups was used to determine the functionality of the RESECO setup.

**Endurance tests**

For each rock type, endurance tests for (1) high pressure at 40 °C, (2) high temperature at a fixed flow rate, and (3) high pressure at high temperature were conducted. Table 1 lists the experimental conditions of these endurance tests. One column at a time was connected to the HPLC system and placed in the oven at 40 °C. Then, the pressure endurance test at 40 °C or 95 °C, respectively, or the temperature endurance test, was conducted. For the pressure endurance tests the flow rate was gradually increased, with the step size depending on the stone type, up to failure or 40 MPa at maximum, due to the limits of the technical devices used. The pressure level for each flow rate had to be constant for at least 5 min to signal pressure equilibrium before the next higher flow rate was used. For the temperature endurance test, the temperature was gradually increased from 40 to 95 °C in 5 °C steps with 40 min in between to reach temperature equilibrium. The endurance tests were terminated at maximum pressure or temperature values, respectively, or by system failure. The temperature was limited to 95 °C due to the open character of the system, as the fluid should not evaporate in the low-pressure part of the system to avoid two-phase flow. After the respective endurance test, the initial experimental conditions were restored to identify any permanent changes in permeability. The pressure readings of each experimental step were converted to permeability using Darcy’s law as described for the functional tests.

**Results and discussion**

**Functional tests**

The permeabilities measured with the RESECO setup were always slightly lower than the permeabilities of the triaxial cell. The permeabilities for the same flow velocity using the red Massenkalk were $1.74 \times 10^{-17}$ m$^2$ for the triaxial cell and $1.44 \times 10^{-17}$ m$^2$ for the RESECO setup. The permeabilities for Remlinger sandstone were $1.43 \times 10^{-16}$ m$^2$ for the triaxial cell and $0.95 \times 10^{-16}$ m$^2$ for the RESECO setup. An error analysis was done using Gaussian error propagation. The relative error for the permeability estimation using the RESECO setup was around 2% (e.g. $4.13 \times 10^{-16}$ m$^2 \pm 4.44 \times 10^{-18}$ m$^2$ for a Remlinger sandstone plug). The relative error using the triaxial cell was in the same order of magnitude. A small difference in the error between the two experimental setups was only due to the precision of the used pump and pressure measurements and was not intrinsic to the experimental setups.

The results showed that the surrounding flow of the rock sample could be effectively prevented even at high pressures using epoxy resin without the need for active confinement. Considering temperature dependence of the permeability as

| Rock | $Q$ [μl/min] | $T$ [°C] | Variable | $\Delta$ | Max. $p$ [MPa] |
|------|--------------|-----------|-----------|---------|---------------|
| FLE  | 130          | 40        | $Q$       | 20–250  | 3150          | 40.82        |
| REM  | 50           | 40        | $Q$       | 10–500  | 2750          | 40.94        |
| BMK  | 10           | 40        | $Q$       | 5–200   | 2850          | 40.82        |
| RMK  | 25           | 40        | $Q$       | 5–50    | 245           | 40.99        |
| FLE  | 16–20        | 40        | $T$       | 5       | 95            | 8.63         |
| REM  | 120          | 40        | $T$       | 5       | 95            | 7.99         |
| BMK  | 10           | 40        | $Q$       | 5       | 95            | 27.17        |
| RMK  | 50           | 40        | $T$       | 5       | 95            | 39.99        |
| FLE  | 100          | 95        | –         | –       | –             | 37.92        |
| REM  | 50           | 95        | $Q$       | 10–50   | 230           | 40.71        |
| BMK  | 10           | 95        | $Q$       | –       | 650           | 41.11        |
| RMK  | 50           | 95        | –         | –       | –             | 39.99        |

Besides the rock type and the starting conditions of flow rate $Q$ and temperature $T$, the varied property, its step size $\Delta$, and maximum value are listed as well as the maximum pressure $p$ reached during the experiment.
described in the literature (e.g. Cheng and Milsch 2020), the differences in permeability determined by the two experimental setups could be explained by the 20 °C difference in temperature. This most probably caused the slightly lower permeabilities measured in the RESECO setup (see the discussion of the temperature dependence of the permeability in section ‘Behavior of the stones’ and Fig. 5). Also, heterogeneity in the stone blocks from which the plugs were drilled, as well as different sample sizes, probably caused differences in the calculated permeabilities.

The results of the functional tests could only be achieved for the column preparation as described in section ‘Preparation of the RESECO setup’. Other tested methods of sealing the sample included a heat shrinking tube, silicon, methyl methacrylate as glue instead of epoxy resin, roughened surface, or a primer for the epoxy resin without additional sealing and direct heat shrinking of the metal column onto the stone plug. All of these approaches showed surrounding flow during the functional tests. An 18 × 1 mm o-ring at the column inflow could effectively replace the broader PTFE seal, but a small nut in the epoxy resin was necessary for its placement; hence, this sealing was discarded due to the necessity of the additional work step while not providing any benefits. Besides these alternative general approaches, other methods of surface roughening of the inner column surface were tested but proved insufficient. These methods included roughening with sandpaper (grit size 40), grit sandpaper (grit size 1000), a rasp, grit blasting, and a corundum grindstone. Since the roughness of the metal surface proved to be important for the strength of the bond between the epoxy resin and the metal, stones with different surface roughness were tested. However, the differences in the surface roughness of the stone plugs had no visible effect on the performance of the experimental setup.

For the evaluation of the functional tests, namely calculation of the permeability using Darcy’s law, the corresponding cross-sectional area had to be known. Pictures of the prepared column tops and bottoms showed different penetration depths of the epoxy resin into the stones ranging from 1.20 mm for the Flechtinger sandstone, to 0.95 mm for the Remlinger sandstone, to 0.33 mm for the red Massenkalk. The epoxy resin did not visibly penetrate into the black Massenkalk. When correcting the cross-sectional area for the penetration depth, a differentiation between the stone types was necessary. The differences between the penetration depth along the length of the sample as well as between the individual samples were small enough such that the error of the permeability estimation did not increase when using averages for each stone type. For example, the determined permeability for a Remlinger sandstone plug was $9.25 \times 10^{-17}$ m² using the epoxy-corrected area at the top, $9.66 \times 10^{-17}$ m² using the area at the bottom, and $9.83 \times 10^{-17}$ m² using the average epoxy penetration depths of the three Remlinger stone plugs. The estimated permeability from the cross-sectional area of the stone without considering the penetration depth of the epoxy resin ($7.60 \times 10^{-17}$ m² for a REM plug) was in the same order of magnitude as the corrected one ($9.45 \times 10^{-17}$ m²).

Nevertheless, the error introduced by the cross-section estimation was significantly larger than the experimental error with $6.79 \times 10^{-18}$ m² for this example. Both were insignificant compared to the permeability variation due to temperature ($9.56 \times 10^{-17}$ m² to $2.58 \times 10^{-17}$ m² for REM from 40 to 95 °C) or pore pressure conditions ($1.19 \times 10^{-17}$ m² to $4.13 \times 10^{-16}$ m² for REM from 15.85 to 40.94 MPa) as shown in the following section. The microfractures in the red Massenkalk allowed deeper penetration of the epoxy resin in comparison with the black Massenkalk. However, no complete closure of the microfractures occurred as proven by the functional tests, demonstrating the feasibility of the RESECO setup for porous as well as microfractured rocks.

### Endurance tests

#### Behavior of the stones

The permeability of the stones was dependent on the experimental conditions. The permeability in both experimental setups increased with increasing flow rate $Q$ and increasing pore pressure (Fig. 4). This increase varied for the different stone types and was reversible. After an initial steep increase in permeability, the slope of the curve flattened for higher flow rates. Such a pore pressure dependence of permeability has been previously observed (e.g. Yasuhara et al. 2011).

The permeability was also dependent on temperature with a decrease in permeability for increasing temperature for all samples (Fig. 5). This permeability decrease was not fully reversible. The Flechtinger sandstone showed only weak temperature dependence of the permeability. The Remlinger sandstone and the red Massenkalk showed similar trends, though at different levels of permeability. The black Massenkalk showed a temperature dependence up to 65 °C, after which nearly no change in pressure due to temperature increase was measured. As there was a 20 °C temperature difference between the triaxial tests and the RESECO experiments, the permeability values determined by the two methods were subjected to the stone-specific temperature dependence. The inverse relation between temperature and permeability is well documented for sandstones (e.g. Guo et al. 2017) and limestones (e.g. Homuth et al. 2015) as is the incomplete recovery of the permeability (Rosenbrand et al. 2015) and the flattening of the curve due to smaller thermal expansion coefficients for higher temperatures (Hassanzadegan et al. 2012). Further, the rock samples are subjected to a natural variability in permeability, which was assessed through testing of multiple samples using the triaxial cell (Fig. 5).
The documented pressure and temperature dependence of the rock permeability show one of the possible applications of the RESECO setup because it offers the possibility to study the pore pressure effects without the disturbing influence of the confining pressure. As it is beyond the scope of this study to describe the permeability dependence on thermal and hydraulic boundary conditions in detail, the interested reader is referred to the cited studies.

**Performance of the setup**

The experimental setup did not fail during the endurance tests up to 95 °C and 40 MPa. The calculated permeabilities before and after each endurance test are listed in Table 2. For all four stones, the permeability values before and after the pressure endurance test were nearly the same. The permeabilities after the temperature endurance tests were lower than before for all stones but the black Massenkalk. For this stone, as well as the red Massenkalk, the temperature test was also the pressure endurance test at high temperature because the pressure increase due to the temperature increase was high enough to reach the HPLC system limit of 40 MPa. Their results in Table 2 are thus the same. The permeabilities after the pressure endurance test at high temperature were mostly higher than before, with the exception of that of the red Massenkalk, which was lower than before. The epoxy resin was seemingly intact after the pressure or temperature endurance test, but after the pressure endurance test at high temperature, the epoxy resin at the outer rim in contact with the metal column seemed to be crushed and some of the material was loose.

**Fig. 4** The dependence of permeability $k$ on the flow rate $Q$ for all stone types. The open circles show the recovery of the permeability after the pressure endurance test (filled circles)
The endurance tests proved the stability of the experimental setup up to a temperature of 95 °C and pore pressures up to 40 MPa. The physical limits of the system were not reached under those conditions and are probably somewhat higher. Even the double strain of high pressure and temperature did not result in immediate failure, but reuse of the cast for new experiments is not recommended. Opening the column showed the mechanical failure of the epoxy resin under these high-pressure, high-temperature conditions.

The physical limits of the RESECO setup were not reached yet. It is expected that the temperature limit of the RESECO setup is lower than for the rock itself. Samples of epoxy resin changed color at 200 °C and became ductile at 350 °C. The pressure limit of the setup cannot be reliably estimated at this point, but the RESECO setup proved functional up to 48 MPa using equipment with a higher pressure limit.

The RESECO setup has only the means to conduct flow-through experiments. No mechanical tests can be conducted and no overburden pressure can be simulated. The type of flow-through experiments that can be conducted with the RESECO setup is not limited to permeability measurements. Tracer tests and chemical alteration experiments were already successfully conducted using this setup. Limiting factors are the type of analysis planned after the flow-through experiments due to the covering in epoxy and the small amount of mechanical damage from column removal, as well as the slightly irregular and unknown stone geometry due to the intruding epoxy resin. This problem can be addressed using varnish spray to prevent intrusion of the resin into the stone. This is also useful for stones with very high porosities to prevent complete saturation of the stone with epoxy resin. In contrast to other means like heat shrinking tubes, the varnish spray does not introduce a new weak spot. The bonding between varnish and stone as well as varnish and epoxy is sufficient to prevent water flow in between. For all experiments, the compatibility of the epoxy with the circulating fluids should be checked, but epoxy resin is in general very chemical resistant.

### Conclusion

The new experimental setup for flow-through experiments on solid rock presented in this work was successfully tested.

- The RESECO setup is only slightly larger than the sample itself. Of course, the more analytical equipment that is used, the bigger the setup will get. However, the sometimes quite big facilities to produce the confining pressure are rendered unnecessary for the confinement of the flow. Also, the material cost is low. A rough estimate considering the reusability of the columns, the casting molds and the tetrachloroethylene, gives material costs below 10 € per cast.
- The RESECO setup proved its feasibility with solid rock samples of different kinds. The experimentally determined rock permeabilities from functional tests were compared to those of a conventional triaxial cell experiment and proved to be in accordance.
- Due to the successful confinement of the flow to the rock samples, flow-through experiments of different sorts can

### Table 2 Calculated permeability values ($k, m^2$) before and after (recovery) the respective endurance tests

| Endurance test | FLE    | REM    | BMK    | RMK    |
|----------------|--------|--------|--------|--------|
| p-test Before  | $3.26 \times 10^{-17}$ | $1.19 \times 10^{-17}$ | $1.57 \times 10^{-18}$ | $2.82 \times 10^{-18}$ |
| After          | $3.08 \times 10^{-17}$ | $1.24 \times 10^{-17}$ | $1.41 \times 10^{-18}$ | $2.74 \times 10^{-18}$ |
| T-test Before  | $8.50 \times 10^{-18}$ | $9.56 \times 10^{-17}$ | $2.72 \times 10^{-18}$ | $2.27 \times 10^{-17}$ |
| After          | $7.67 \times 10^{-18}$ | $4.24 \times 10^{-17}$ | $4.57 \times 10^{-18}$ | $1.63 \times 10^{-17}$ |
| p-T-test Before| $5.24 \times 10^{-17}$ | $1.11 \times 10^{-16}$ | $2.72 \times 10^{-18}$ | $2.27 \times 10^{-17}$ |
| After          | $7.01 \times 10^{-17}$ | $1.59 \times 10^{-16}$ | $4.57 \times 10^{-18}$ | $1.63 \times 10^{-17}$ |
be conducted. This includes permeability measurements as shown above as well as tracer tests and more. First experiments with aggressive fluids to alter the rock samples were conducted and the RESECO setup was not affected.

- The RESECO setup works fine with the pressure and temperature conditions of reservoirs. A maximum of 95 °C and 40 MPa was tested without reaching the limits of the setup. This covers a wide range of the common experimental conditions for reservoir experiments.

The possible applications of the RESECO setup are manifold, especially with respect to reservoirs of different kinds. They include the study of permeability dependence (e.g. Yasuhara et al. 2011), scaling experiments (e.g. Frank et al. 2021b), alteration studies (e.g. White et al. 2017), heat transfer experiments (e.g. Frank et al. 2021a), and others. The advancement of the RESECO setup will include experiments with other sample geometries, the application to macrofractures and the incorporation of further analytical equipment. The addition of other measurements like electrical conductivity or seismic wave velocity should be possible since there is some space in the epoxy ring around the stone sample.

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Declarations

Competing interests The authors declare that they have no known competing interests.

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