

Influence of dissolution on frictional properties of carbonate faults

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Highlights

- Velocity stepping experiments are conducted for a synthetic calcite gouge with an annular shear apparatus to study the effect of the injection of an acid fluid.
- Results show a weakening of the carbonate gouge Rate and State parameters, due to acid dissolution and confining stress.
- This weakening is due to the loss of small particles and not the change of rugosity of the grains.
- A modification of the rate and state friction law is proposed to take into account these effects and determine the conditions of fault reactivation in potential injection sites.

Keywords: Fault stability; Induced seismicity; Chemo-mechanical couplings; Rate and state friction; CO₂ storage; Acid gas injection

24 **Abstract**

25 Velocity stepping experiments have been performed on a simulated calcite gouge using an annular
26 shear apparatus to investigate the effect of dissolution on the frictional properties of a carbonate
27 fault. The tested material was put in contact with hydrochloric acid at different concentration in
28 order to dissolve the grains. Particle size analysis shows that the small grains tend to disappear due
29 to the chemical reaction, whereas the large grains are not much affected. The dissolution process
30 mainly induces a decrease of the fractal number of the grain size distribution. The study of the
31 rate-and-state parameters of the gouge, which enables to characterize the ability of the fault to
32 generate earthquakes, shows a weakening due to the dissolution and the confining stress. This
33 weakening implies that faults can become seismogenic after the injection in a carbonate reservoir
34 of an acid fluid. This effect is explained by the removal of small particles, countering the increase
35 of rugosity of the grains. Based on the experimental results, a constitutive law is suggested, in
36 order to capture the influence of dissolution and confining stress on the frictional properties of
37 fault. It enables to determine in which conditions of maturity and degradation of a fault and at
38 which depth, a seismic slip can be triggered by anthropogenic activities like acid/CO₂ injection.

39

40 **1. Introduction**

41 A variety of human activities can modify the stress state or the material properties of underground
42 rocks and, thus, induce an instability at the origin of an earthquake. The anthropogenic cause of
43 seismicity has been shown for different types of projects. The most commonly reported
44 anthropogenic activities to have induced earthquakes are Mining and water reservoir
45 impoundment, but it can also be caused by fracking, oil and gas extraction, waste water disposal
46 or geothermal projects (Wilson et al. 2017). Human-made tremors have drastically increased in
47 the U.S. since 2001 from a previous average of 21 quakes a year to 188 documented in 2011
48 (Ellsworth 2013). For instance, infrastructures and people in Oklahoma and southern Kansas face
49 potential damages in the next years from induced earthquakes, in a similar level to regions known
50 for their large number of natural earthquakes, like southern California (Schoenball and Ellsworth
51 2017). However, induced earthquakes are usually not a threat for human life, because they have a
52 maximum magnitude of 3-4 and for constructions obeying the actual codes they would not be a
53 root of damages (Zoback and Gorelick 2012).

54 A large number of numerical studies have been devoted to study the mechanisms at the origin of
55 induced seismicity and to evaluate the maximum magnitude of the earthquake a project may induce
56 (Cappa 2012). Most of them consider a hydro-mechanical model and look at the modifications of
57 the stress state at the location of a known fault in a specific site (Baisch et al. 2010; Yehya, Yang,
58 and Rice 2018; Mortezaei and Vahedifard 2015; Cappa 2012). In these studies, fluid injection into
59 a reservoir-caprock system bounded by a fault is modelled with different stress state conditions
60 and assuming various permeability. The time of appearance of the first potential earthquake can
61 be computed and the value of the stress drop permits to estimate the magnitude.

62 Projects like acid gas disposal, enhanced oil recovery or carbon capture and storage involve the
63 injection of a reacting fluid into a reservoir (Khan, Amin, and Madden 2013). The Carbon Capture
64 and Storage (CCS) is one of the methods considered to reduce emissions of CO₂ into the
65 atmosphere (Espinoza 2011). The principle is to capture the CO₂ and separate it from other gases
66 in the combustion smoke of significant plants like cement factories or coal-burning power plants.
67 Once extracted, the gas is compressed and transported with a pipeline to injections sites for long
68 term storage. Acid gas disposal is a method used by oil and gas producer to reduce atmospheric
69 emissions of hydrogen sulphide (H₂S). A mixture of hydrogen sulphide and carbon dioxide (by-
70 product of ‘sweetening’ sour hydrocarbons) is injected into depleted reservoirs or deep saline
71 aquifers (Bachu and Gunter 2004). Enhanced oil recovery, also called tertiary recovery, is a way
72 to improve the extraction of crude oil from reservoir that could not be extracted otherwise
73 (Rubinstein and Mahani 2015). The most common methods are gas or chemical injection that
74 modifies the viscosity of the oil and improve its mobility. In these cases, induced seismicity could
75 produce damage to the well or the caprock and deteriorate the sealing of the reservoirs, leading to
76 the failure of the project.

77 The injection of an acid does not only change the stress state, but also modifies the properties of
78 the storage medium. Some theoretical studies and observations have looked at the influence of
79 chemical reactions on the potential of a fault to create earthquakes. (Yarushina and Bercovici 2013)
80 have looked at the effect of supercritical CO₂ on mafic rocks. The reaction of carbonatation that
81 happens between the minerals and the carbon creates products that will precipitate on the surface
82 of grains leading to an increase of their diameter. In the same time, the pore pressure will decrease
83 because the fluid will be consumed by the reaction. They conclude that all these phenomena will
84 prevent the triggering of earthquakes if the injection rate is not too high. On the contrary, (Collettini

85 et al. 2008) have shown that the presence of CO₂ can destabilize a preexisting fault. After analyses
86 of samples issued from a site in northern Apennines, they discovered that there is a degassing of a
87 trapped bubble of CO₂ under the fault, that operates as a weakening process. In the long term, the
88 fluid reacts with fine grains in the fault gouge to change the mineralogy and produce weak,
89 phyllosilicate-rich fault rocks that deform with a very low friction coefficient, facilitating a seismic
90 slip along the fault.

91 In this work, we perform experiments in carbonate fault gouges at different dissolution degrees
92 and different confining pressures to assess the effect of long-term exposure of a carbonate to an
93 acid fluid. The combined effects of dissolution and confinement are then used to produce a chemo-
94 mechanical enhancement in the classical rate-and-state friction law, and the critical operational
95 conditions to avoid induced seismicity in a carbonate-rich fault are discussed.

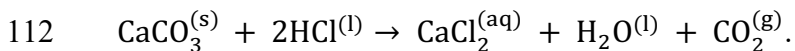
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97 **2. Experimental setup**

98 **2.1. Materials**

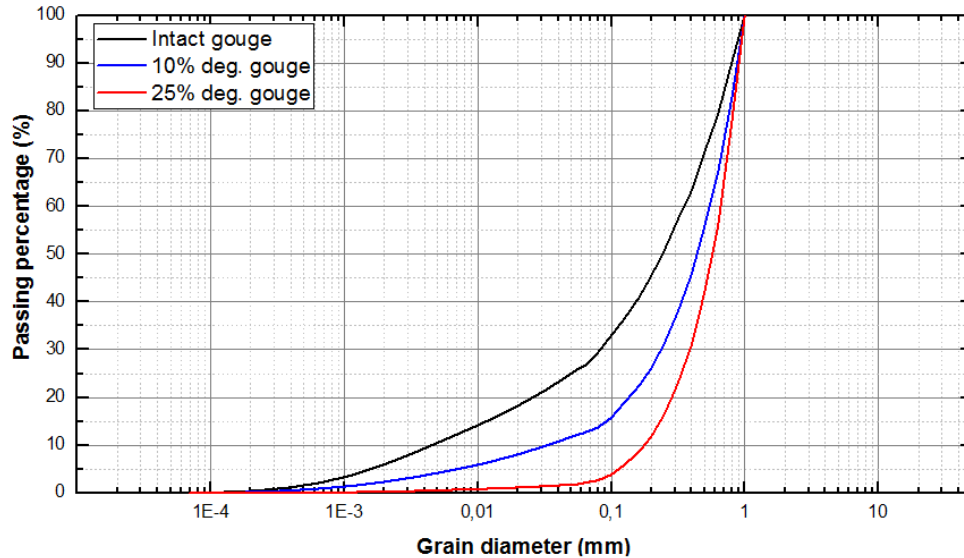
99 The material used for the experiments is a carbonate sand extracted from the Fergues quarry by
100 the company “les Carrières du Boulonnais” located in the North of France. The sand is composed
101 of 99% of calcite (CaCO₃) and show traces of quartz (SiO₂), magnesium carbonate (MgCO₃),
102 Hematite (Fe₂O₃) and Aluminium Oxide (Al₂O₃). The grain size distribution is obtained using a
103 combination of sieves and hydrometer analysis or laser diffraction. Both methods show similar
104 results and the broad size distribution of the material is shown in Figure 1.

105 Some samples of the sand are put in contact with a solution of water and hydrochloric acid, which
106 is a strong acid, to simulate the long-term effect of dissolution by a weak acid like carbonic acid
107 on the grain size distribution. The reason for using this acid, instead of carbonic acid or other acids,
108 corresponding to the fluid injected in the case of acid gas injection or enhanced oil recovery is that
109 the kinetics of the chemical reaction is almost instantaneous. The calcium carbonate (CaCO₃) is
110 dissolved by the hydrochloric acid (HCl) and the products of reaction are carbon dioxide (CO₂),
111 water (H₂O) and calcium chloride (CaCl₂). The equation of the reaction is:



113 CaCl₂ is highly soluble and the specimen are washed several times after the reaction to remove this
114 product of the reaction.

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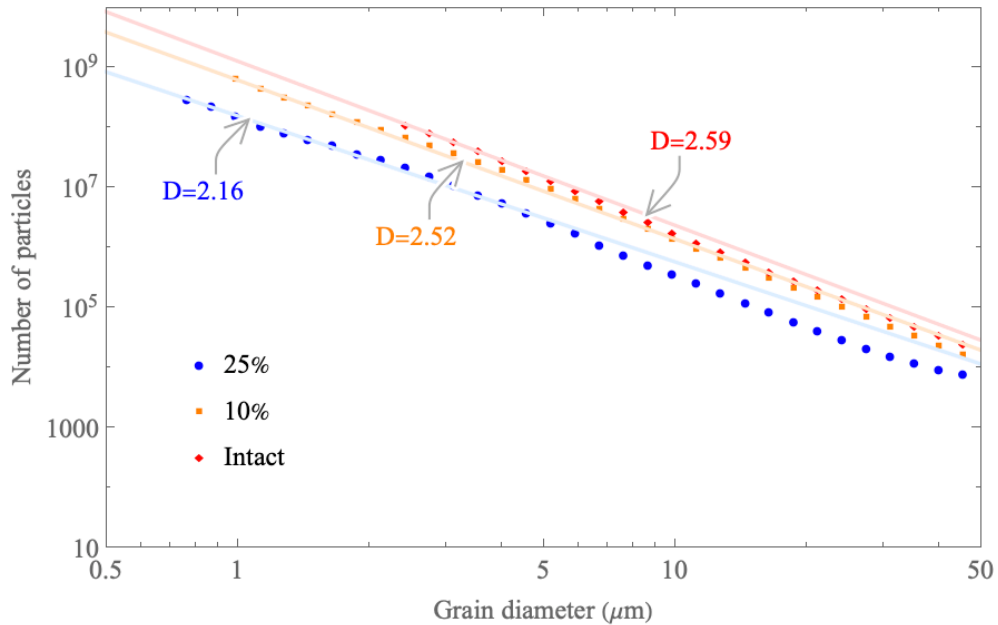
117 *Figure 1. Cumulative grain size distribution of the simulated fault materials showing different level of dissolution*

118 The cumulative grain size distributions of the samples for the initial material, after 10% and 25%
119 of dissolution of the total mass are shown in Figure 1. In particular, the higher the degradation, the
120 more the small particles get dissolved, as shown in the next comparison chart. This phenomenon
121 is due to the greater specific surface that small particles offer to the reaction compared to bigger
122 particles.

123 Considering that our tests are carried out on a material that is supposed to simulate a natural fault
124 gouge, one of the most important material properties to be assessed is the fractal dimension of the
125 grain size distribution. The fractal dimension (D) is defined by
126 $N = r^D$, where N is number of particle of diameter r . (Sammis, King, and Biegel 1987) measured
127 the particle distribution of intact gouge samples retrieved from the Lopez Fault in the San Gabriel
128 Mountains of Southern California. The gouge is composed mainly of feldspars, quartz and
129 Chlorite, with smaller amounts of calcite and other oxides. The analysis has revealed a remarkable
130 degree of self-similarity for the grain size distribution and the authors have found the fractal
131 dimension to be 1.60 ± 0.11 in two-dimensional cross-section, thus 2.60 ± 0.11 in a three-
132 dimensional volume. On the basis of the observations, they proposed a new model, called the

133 comminution model, for the mechanical processes that generate fault gouges. Self-similarity
134 results from repeated tensile splitting of grains and that splitting probability depends largely on the
135 relative size of nearest neighbors: during the fragmentation process, the direct contact between two
136 particles of near equal size will result in the tensile breakup of one of the two. In this way, at the
137 end of fragmentation process, the material will have a particle distribution in which particles of
138 the same size are separated from each other. Such a spatial organization repeats itself at each scale,
139 providing a self-similar grain distribution having a fractal dimension of 2.58, independently of the
140 initial size distribution of the particles. After the development of this model, several authors
141 conducted experiments on natural and simulated fault gouges, showing their tendency to develop
142 a fractal dimension values of about 2.6 supporting the Sammis' theory (Stacey and Sammis 1991;
143 An and Sammis 1994). However, (Storti, Billi, and Salvini 2003) have analyzed gouge samples
144 of a carbonate fault in the Apennines, Italy and have shown that for this material the comminution
145 model is not always verified. Fractal dimensions obtained from gouges varies from 2.61 to 3.49 in
146 strike-slip faults and from 2.93 to 3.13 in extensional faults. They recognized the value 2.6 to be a
147 threshold between a first stage of fault gouge formation dominated by particles fragmentation and
148 a second one dominated by particles abrasion in the case of carbonate materials.

149 In Figure 2, the number of particles as a function of the grain diameter is shown for the different
150 level of dissolution of the grains. Only the grain diameters between $0.5\mu\text{m}$ and $50\mu\text{m}$ are shown,
151 for better visualization. The value of the fractal number of the sand that is not dissolved is in
152 agreement with the comminution theory and this sand can, therefore, be considered as a good
153 analog of a gouge material. The dissolution induces a decrease of the fractal number from 2.59 to
154 2.16. This decrease is due to the removal of the small particles from the grain size distribution, in
155 the same way that abrasion makes the fractal number increases due to the creation of more small
156 particles. An interesting feature of the effect of dissolution on the particle size distribution is that
157 it induces an increase of the mean grain diameter. It can, thus, affect the prediction of models
158 considering the mean grain size explicitly like Cosserat continua (Rattez, Stefanou, Veveakis, et
159 al. 2018).



160

161 *Figure 2. Number of particles as function of the grain diameter for different level of dissolution of the carbonate sand. The*
 162 *fractal number associated with each distribution is also shown.*

163

164 2.2. Experimental set-up

165 The device used for the mechanical tests is an annular simple shear device called ACSA (“Appareil
 166 de Cisaillement Simple Annulaire”), designed in 1993 in the CERMES laboratory of Ecole des
 167 Ponts (Unterreiner et al. 1993) in order to study the behavior of soil-structure interface. It was then
 168 used in the context of fault mechanics by (Chambon, Schmittbuhl, and Corfdir 2006a, 2006b;
 169 Messen, Corfdir, and Schmittbuhl 2013). This device enables to shear the gouge material over
 170 large displacements and with samples that are thicker than other experimental devices. A cut-away
 171 design view of the machine is shown in Figure 3.

172 The internal surface of a ring-shaped sample, which present a square section of 100mm, is sheared
 173 by the rotation of a rough steel cylinder. An optoelectronic rotation encoder is used to monitor the
 174 rotation angle of the steel cylinder, and hence the slip distance. The spacing between the triangular
 175 striation of the inner interface perpendicular to the sliding direction is 1mm, the same size as the
 176 maximum grain diameter of the samples, in order to preclude interfacial slip along the steel-
 177 granular boundary (Koval et al. 2011). The outer boundary of the sample is subjected to a constant
 178 radial confinement σ_e through a 1.5-mm-thick neoprene jacket and applied by a pressure-volume

179 controller. Vertically, the sample is embedded between an upper plate made of duralumin and a
 180 lower plate made of glass. All our experiments have been conducted in nominally dry conditions,
 181 that is with room atmosphere inside the pore space.

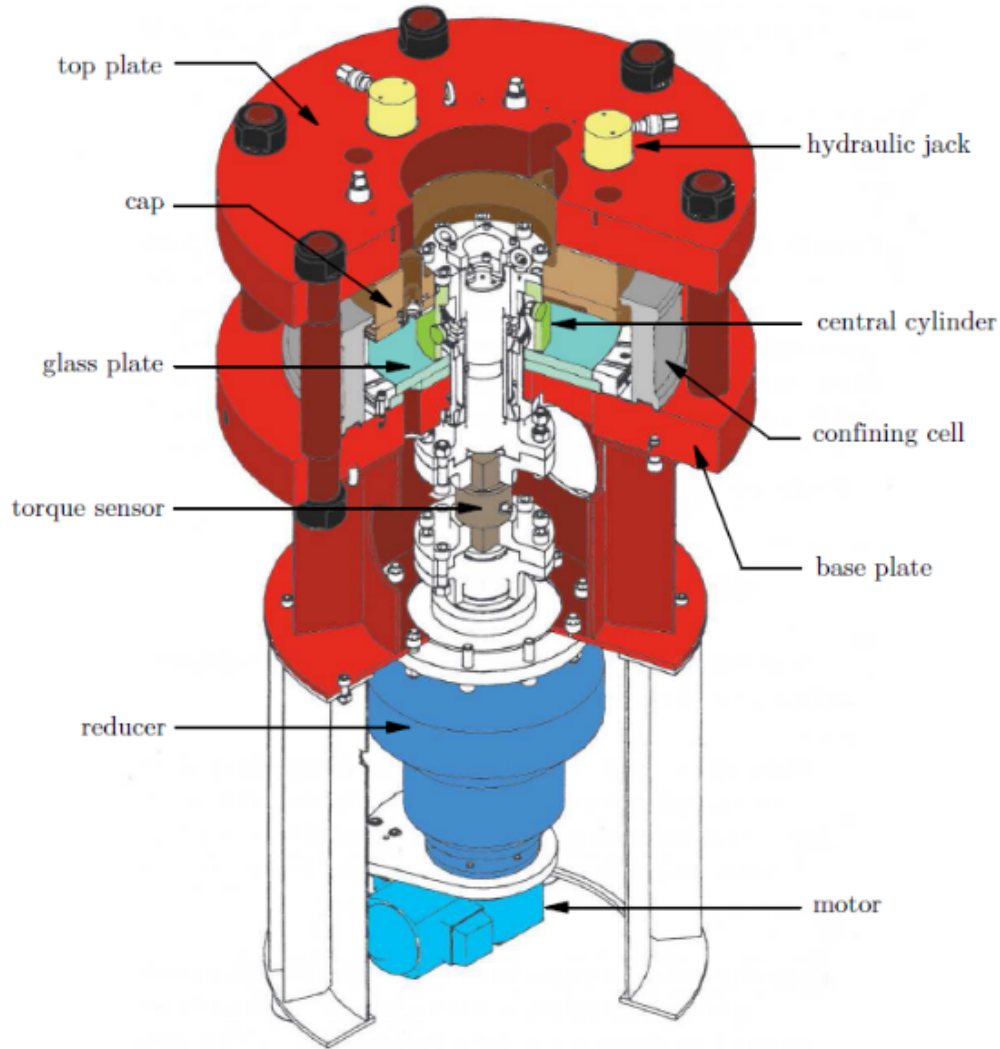


Figure 3. Cut-away design view of ACSA

182
 183
 184 The samples are initially sheared at a constant speed of 2 mm/min for 10 cm, in order to reach the
 185 residual friction coefficient. Then, the shearing velocity is abruptly changed every 2 mm of slip to
 186 apply different velocity steps. The sequence shown in Table 1 is chosen to repeat every speed step
 187 at least two times in order to evaluate the variability of the friction parameters calculated.

188

Stage	1	2	3	4	5	6	7	8	9
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Displacement [mm]	100	2	2	2	2	2	2	2	2
Speed [$\mu\text{m/s}$]	33	10	1.7	10	33	10	1.7	10	33

Table 1. Velocity stepping sequence applied to the sample.

189

190 In Table 2, a list of all the conducted tests with different degrees of dissolution and different
191 confining stresses is presented.

Degradation	Pressure (kPa)	Test name
none	300	Intact0.6
none	500	Intact1
none	800	Intact1.6
10%	300	10deg0.6
10%	500	10deg1
10%	800	10deg1.6
25%	300	25deg0.6
25%	500	25deg1
25%	800	25deg1.6

Table 2 – List of complete set of experiments

192

193

194 2.3. Data acquisition and processing

195 The apparent friction coefficient μ of the sample is calculated as the ratio of the applied shear
196 stress τ to the confining stress σ , $\mu = \frac{\tau}{\sigma}$. The shear stress τ can be found converting the applied
197 torque measurements Γ of the motor:

198

$$\tau = \frac{\Gamma/r}{A} = \frac{\Gamma/r}{2\pi rH} = \frac{\Gamma}{2\pi r^2H} \quad (1)$$

199 where r and H are respectively the radius and the height of the inner cylinder and are both equal
200 to 10 cm. The pressure exerted on the inner cylinder is different from the external pressure applied
201 on the outer boundary of the sample due to the particular geometry of the sample. According to
202 (Chambon 2003), simple geometrical considerations tend to indicate that the inner normal stress
203 σ_i should be equal to twice the applied confinement σ_e , since the external radius of the sample is
204 the double of the internal one; moreover, the same authors conducted some experiments equipping
205 both the smooth and the rough cylinder with five stress sensors, and they found that normal stress
206 on cylinders' surface was strongly varying at the beginning of the test, but it tended to stabilize

207 after few millimeters of slip toward values very close to the double of external pressure. As the
208 shear band is developing near the inner cylinder, the apparent friction is calculated as:

$$209 \quad \mu = \frac{\tau}{\sigma_i} = \frac{\Gamma}{2\pi r^2 H} \cdot \frac{1}{2\sigma_e} \quad (2)$$

210 **2.4.The Rate-and-State Friction (RSF) law**

211 Since Charles-Augustin Coulomb described the frictional behavior of piece of woods, the
212 understanding of complex microscopic and nanoscopic phenomena underlying has made
213 considerable progress. (Reid 1910) was the first to argue after the 1906 San Francisco earthquake
214 that tremors are not created by the emergence of a new crack in the crust, but rather by the sudden
215 slip along an existing fault. After the beginning of slipping the fault can move seismically or
216 aseismically (Scholz 1998), but this depends on the evolution of friction. A model describing the
217 instability along a fault was developed during the eighties by Dieterich and Ruina (Dieterich 1981;
218 Ruina 1983). They introduced new state variables that describe a second order effect on the
219 coefficient of friction. These laws are called “Rate and State”, because the friction depends on the
220 velocity of slipping (Rate) and a state variable θ , which is interpreted as the average lifespan of a
221 set of grain-to-grain contacts in a frictional system. They describe the evolution of the friction
222 coefficient to changes in sliding motions by the equation:

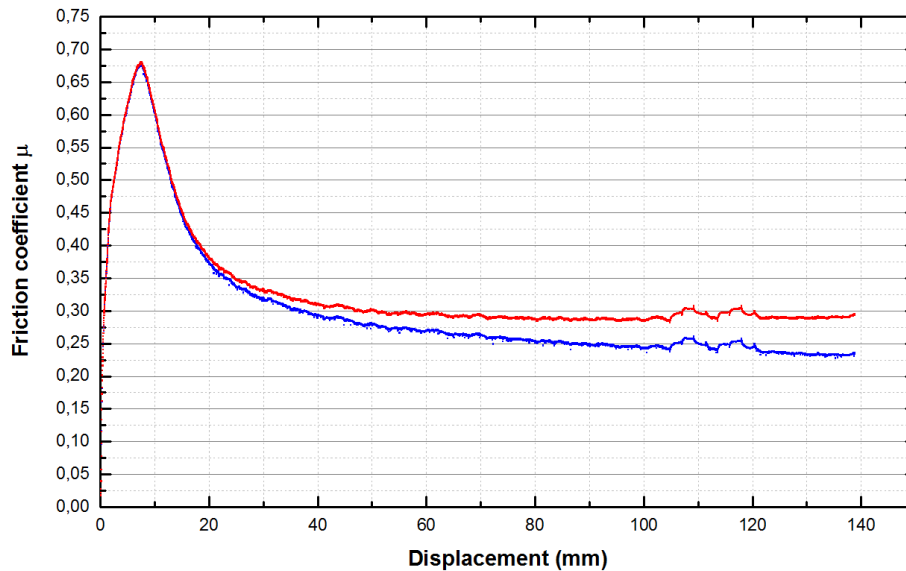
$$223 \quad \mu = \mu_0 + a \ln\left(\frac{V}{V_0}\right) + b \ln\left(\frac{V_0 \theta}{D_c}\right) \quad (3)$$

224 μ_0 is a reference friction coefficient, V and V_0 are respectively the actual and reference velocities
225 of the fault. D_c is the characteristic distance of accommodation for friction, associated with the
226 state variable θ . a and b are material properties. This empirical law can capture the phenomenon
227 of stick-slip, which is the name of the instability of friction that creates earthquakes. A simple
228 system called the spring-slider model, in which the slider follows the rate and state friction laws
229 and the spring represents the elastic surrounding mass of the fault, is usually applied to model
230 seismic slips. The study of the linear stability of that system (Rice and Ruina 1983) shows a critical
231 value for the stiffness of the spring k_{cr} (if the inertial terms are negligible). Under this value, the
232 system is unstable and over this value, it is partially stable.

$$233 \quad k_{cr} = \frac{N}{D_c} (b - a) \quad (4)$$

234 where N is the normal force applied perpendicular to the direction of sliding.
 235 The parameter $(a - b)$ is therefore fundamental to determine the stability of a fault, because if it
 236 is positive, instability would never happen.

237 The parameter $(a - b)$ can be calculated based on the values of friction before and after a sudden
 238 velocity change. To evaluate the friction parameter $(a-b)$, the general long-term strain trend of the
 239 apparent friction is removed in order to avoid this factor to influence the $(a-b)$ values. It is typically
 240 a softening trend as the one shown in Figure 4. The same procedure was also applied by
 241 (Samuelson and Spiers 2012) and (Tembe, Lockner, and Wong 2010) during their velocity
 242 stepping experiments.



243
 244 *Figure 4. Friction coefficient line: original (blue) vs straightened (red) (test “intact600”).*

245
 246 The $(a-b)$ value of each velocity step can be obtained from the straightened line of friction
 247 coefficient, according to the rate and state friction law:

248
$$(a - b) = \frac{\mu_0 - \mu}{\ln(V/V_0)} \quad (5)$$

249 where μ is the coefficient of friction at the end of a step with velocity V , and μ_0 and V_0 are the
 250 coefficient of friction and velocity immediately prior to the velocity step.

251
 252 **3. Results**

253 In this section, we describe first the frictional data obtained from the mechanical experiments. The
254 global evolution of the apparent friction as well as the rate and state parameters are presented
255 together with their evolution with confining stress and dissolution, Secondly, the specific surface
256 areas of the samples is assessed by two methods in order to estimate the rugosity of the grains.
257 This analysis is completed with observations obtained using a scanning electron microscope to
258 interpret the different mechanical responses of the samples.

259 **3.1.Mechanical results**

260 An example of the apparent friction coefficient evolution with shear displacement is shown in
261 Figure 4. It exhibits a peak for a value between 0.6 and 0.7 for all experiments. This peak is
262 followed by a softening behavior with a characteristic slip distance of a few decimeters, also
263 observed in the experiments performed on glass beads and quartz sand by (Chambon, Schmittbuhl,
264 and Corfdir 2006a).

265 We observe through the glass window at the bottom of the sample a strain localization of the
266 deformation next to the inner cylinders. The shear band has a thickness of approximately 10 times
267 the mean grain size, in accordance with numerical simulations performed using a Cosserat
268 continuum (Rattez, Stefanou, Sulem, et al. 2018; Rattez, Stefanou, and Sulem 2018).

269 Regarding the velocity stepping experiments, the results show that all of the tests performed in this
270 study, regardless of degradation level, pressure, exhibit a velocity weakening behavior. The $(a-b)$
271 values are falling between -0.003 and -0.018; the only exception is the 25deg0.6 test, which
272 exhibits the weakest behavior reaching the value of -0.028.

273 From the slip stability point of view, the general trend is that the dissolution has a weakening effect
274 on the slip stability of tested calcite sand (see Figure 5). All experiments exhibit an unstable
275 behavior ($a-b < 0$), and the dissolution of grains tend to make the values even more negative.
276 Generally, we can see that deg10 results are situated between intact and deg25 ones. It means that
277 a higher degradation induces a higher weakening, and this trend is visible under all pressures.

278 One observation is that even if the deg10 results are situated between intact and deg25, the
279 difference between intact and deg25 is much stronger compared to the difference between intact
280 and deg10. In other words, the slip stability decay is not linear with the percentage of degradation.
281 Moreover, in some velocity steps, we can see that $(a-b)$ values of deg10 tests are equal or even a

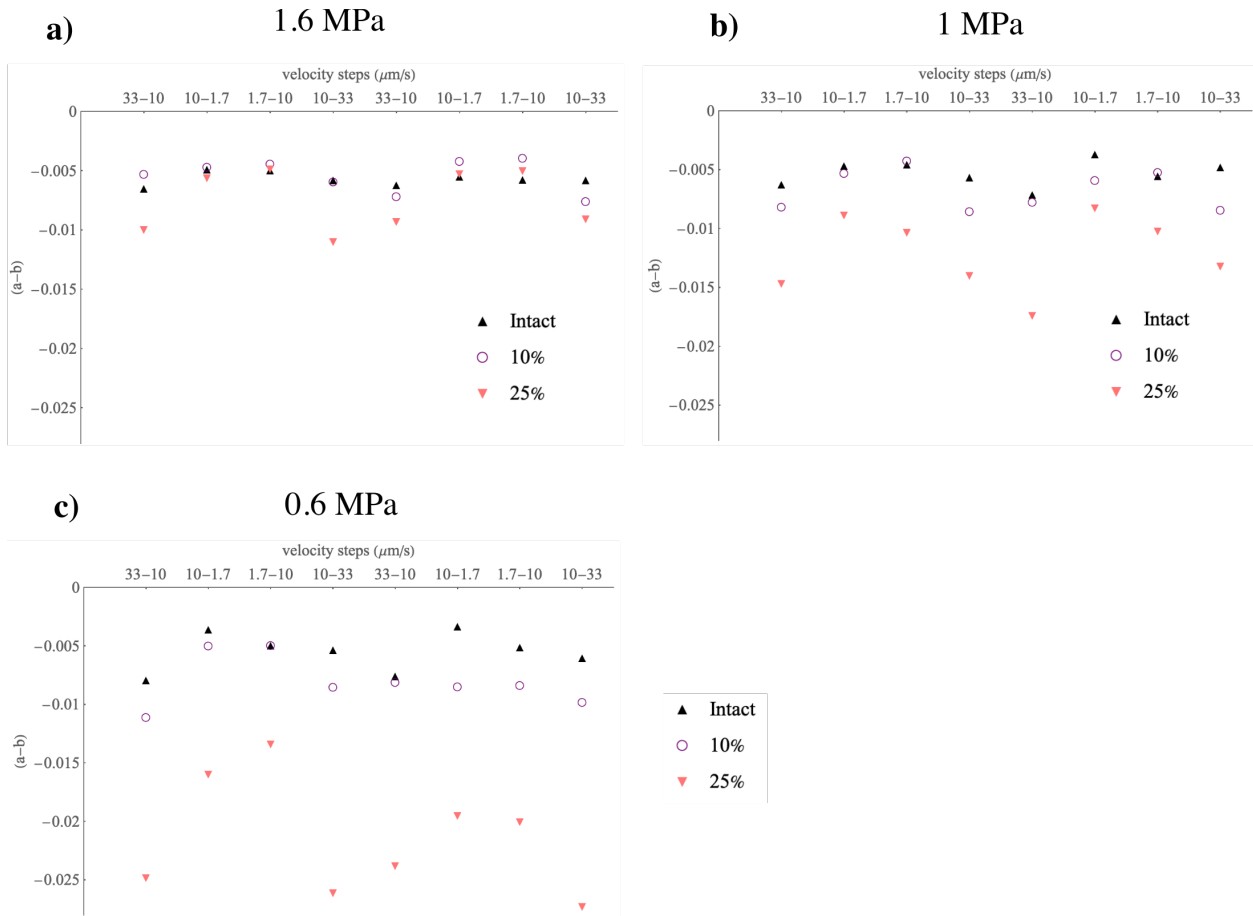
282 bit greater than the intact ones, but the values remain very close, thus it can be considered as due
283 to experimental variability.

284 The value of the friction μ is, on the other side, not much affected by the dissolution (see Table 3.
285 Values of the residual friction coefficient for different confining pressure and level of dissolution).
286 In fact, we can see that in the 600 kPa tests, the three peak values are nearly the same ($\mu=0.63$ –
287 0.67), as well as the residual values ($\mu=0.22$ –0.25). The same happens in the 1 MPa tests where
288 the peak is exactly the same and the intact residual value is only a bit higher than the degraded ones
289 ($\mu=0.20$). Finally, the 1.6 MPa run exhibits only a small difference in the value of the residual
290 friction.

	600 kPa	1 MPa	1.6 MPa
intact	0.24	0.24	0.26
dissolution 10%	0.21	0.20	0.21
dissolution 25%	0.24	0.20	0.19

291 *Table 3. Values of the residual friction coefficient for different confining pressure and level of dissolution*

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Figure 5. Effect of the dissolution on the rate and state friction parameters ($a-b$) for the different velocity steps and for different confining stress.

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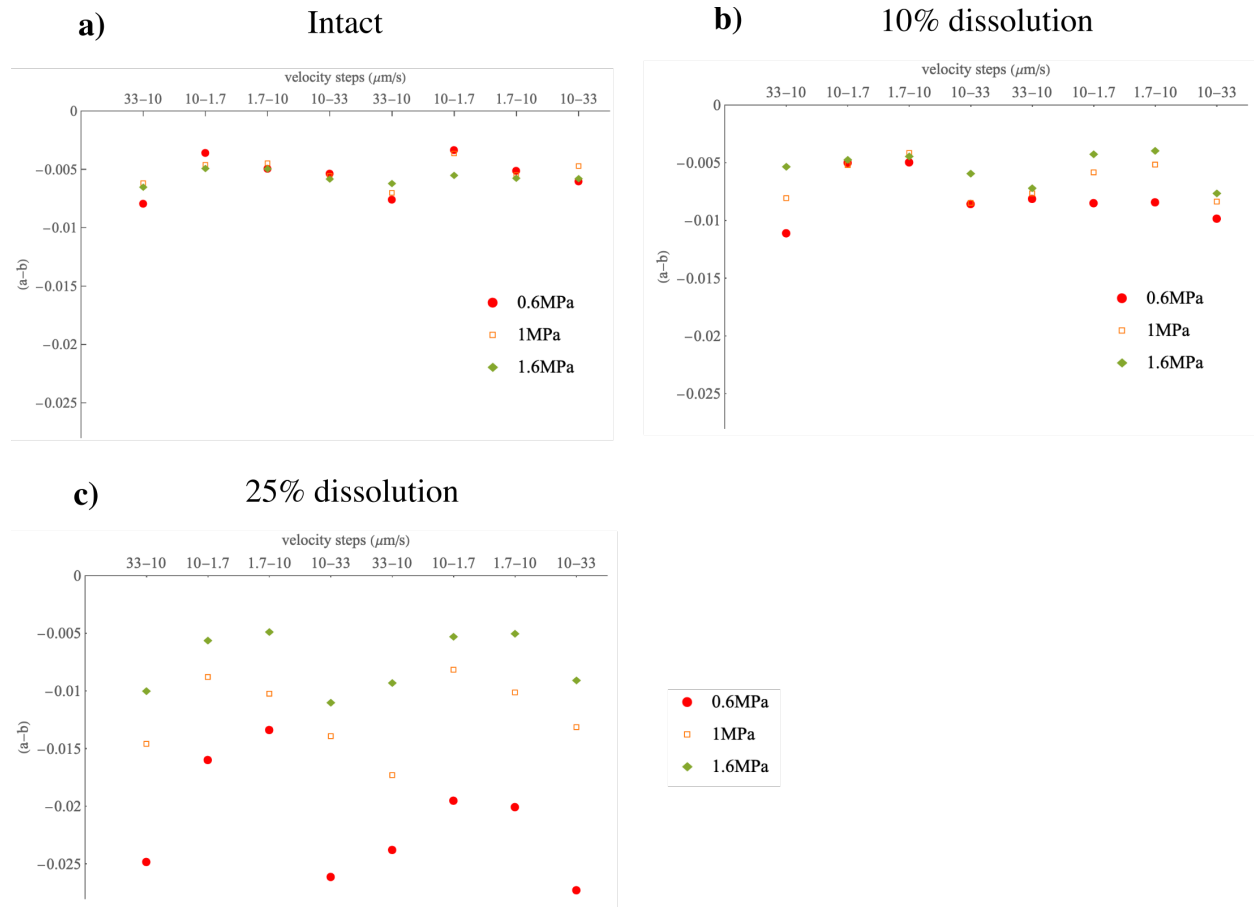
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In Figure 6, we plot the values of the friction parameters ($a-b$), as a function of the confining pressure for the different degrees of dissolution. For the initial sand, the rate and state parameters are not affected by the confining stress but more by the velocity steps considered. The transition from 10 to 33 $\mu\text{m/s}$ induces more weakening than the transition from 1.7 to 10 $\mu\text{m/s}$. This effect is also observed for the degraded sand. However, the sand that underwent 10% and 25% dissolution shows a clear effect of the confining stress on the rate and state parameters. For these tests, the ($a-b$) values get closer to zero when the confinement pressure is increased.



304

305
306

Figure 6. Effect of the confining pressure on the rate and state friction parameters (a-b) for the different velocity steps and for different rate of dissolution.

307

308 3.2. Particles roughness

309 The dissolution of the calcite through contact with an acid solution affects the grain size
 310 distribution but also the shape of the particles. The shape and in particular the roughness of the
 311 grains could influence the mechanical response of the sample (Anthony and Marone 2005). Thus,
 312 we characterize the roughness and shape of the particles using the specific BET surface area (SA
 313 3100 BET surface area and pore size analyzer; Beckman Coulter, U.S.), sieves analysis and a
 314 Scanning Electron Microscope (SEM).

315 The roughness of a given sample is defined as the ratio between the surface areas obtained from
 316 the BET method and the particle size analysis (considering spherical particles). The BET method
 317 is a way to calculate the specific surface area of a sample based on gas adsorption. Nitrogen is
 318 pumped into the sample at a given pressure, with constant temperature (corresponding to the

319 boiling point of liquid Nitrogen) and the adsorption process is measured volumetrically. The
 320 isotherm data obtained from this procedure enable to determine the specific surface area based on
 321 the theory developed by (Brunauer, Emmett, and Teller 1938). This value of the specific surface
 322 area is accurate and can be compared with one obtained from the particle size distribution (PSD).
 323 The latter is obtained by assuming that the mass of material obtained for each size correspond to
 324 spherical particles with the diameter of the mesh size (or the size of the volume equivalent for laser
 325 diffraction). The results are reported in Table 4.

326

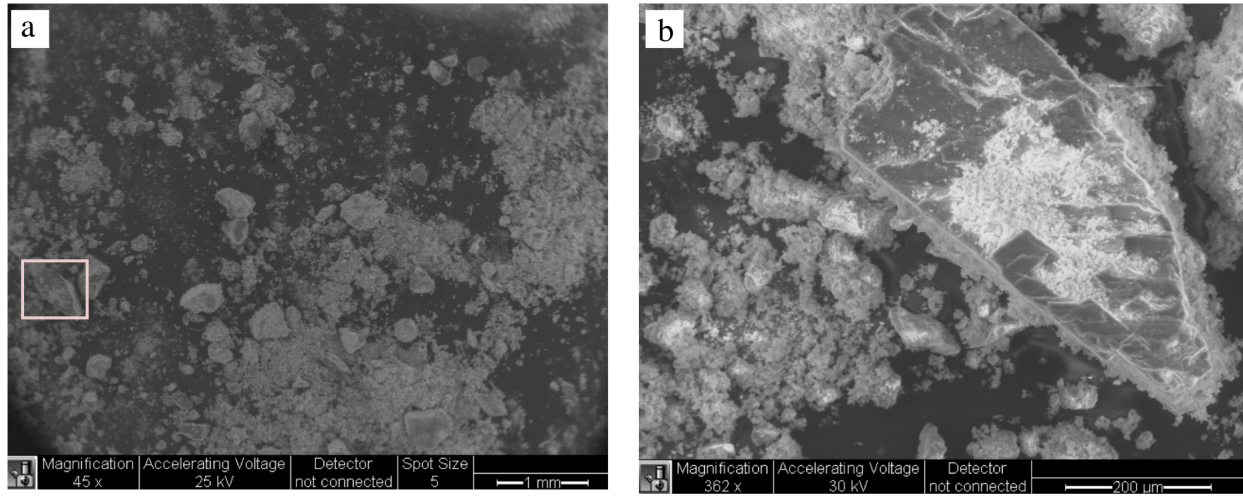
Sample	S_{PSD} (m²/g)	S_{BET} (m²/g)	Rugosity
Intact	5.34×10 ⁻²	0.96	18.03
10%	3.29×10 ⁻²	0.83	25.32
25%	9.48×10 ⁻³	0.99	104.32

327 *Table 4. Values of the specific surface area obtained from the BET method (SBET) or the particle size distribution (SPSD) and the*
 328 *rugosity for different level of dissolution*

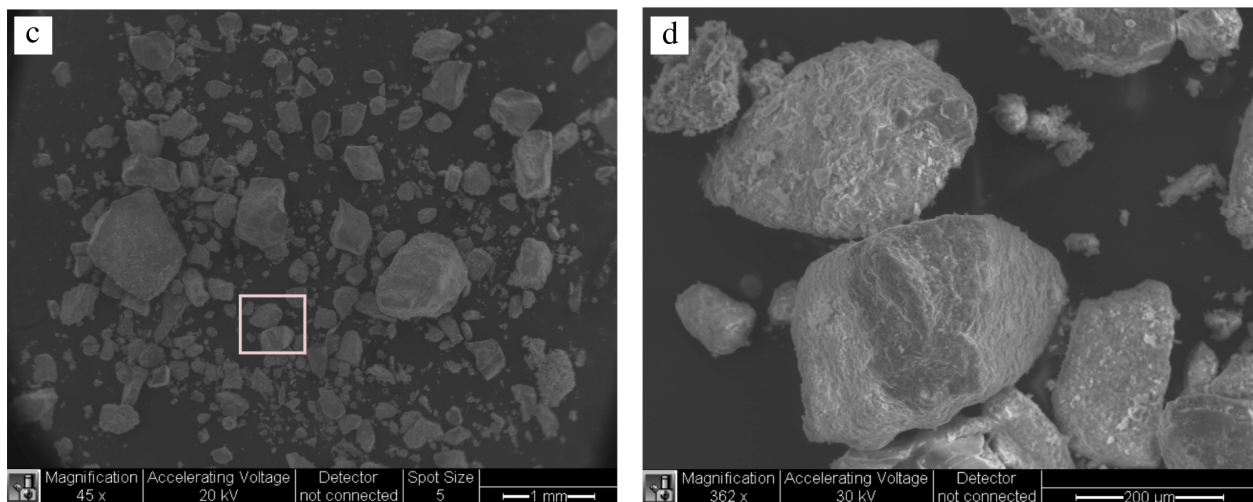
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330 The specific surface area obtained from the PSD exhibits a decrease with the dissolution. It is due
 331 to the elimination of the small particles that contributes substantially to the value of the specific
 332 surface. However, the values obtained from the BET method are not notably affected by the
 333 dissolution. Consequently, the roughness increases with the dissolution rate. To understand these
 334 results, observations of the grains have been performed using a Scanning Electron Microscope
 335 (FEI XL30 SEM) with variable pressure. The specimens have been coated with gold film prior to
 336 be placed in the microscope. Images of the intact sand and the sand with 25% degradation are
 337 shown in Figure 7. The intact sand exhibits much more fine particles attached to each other or to
 338 larger grains than the samples which experienced dissolution. Moreover, grains with a diameter
 339 larger than 10µm in the intact sand (Figure 7. b) present an external surface that is smoother than
 340 the one in the dissolved sand (Figure 7. d).

Intact sand



Dissolved sand 25%



342

343 *Figure 7. Scanning Electron Microscope images of the intact sand (a and b), and the sand with 25% dissolution (c and d)*

344

345 **4. Discussion and implications for potential fault reactivation due to acidic fluid** 346 **injection**

347 In this section, we integrate the mechanical data and the microstructural investigations from our
348 experiments to discuss the role of the confining stress and the dissolution on the velocity
349 weakening behavior of carbonate bearing faults. An empirical law is proposed to account for these
350 phenomena on the rate and state friction parameters. This law is then used to determine in which

351 conditions the injection of an acidic fluid could induced potential seismic slips in projects like
352 carbon storage and acid gas disposals.

353

354 **4.1. Empirical law for the evolution of rate and state friction parameters**

355

356 Our results show that the mechanical behavior of simulated carbonate-bearing faults strongly
357 depends on the applied normal stress and is modulated by the imposed slip velocity and the
358 chemical dissolution of the grains, characterized by the fractal number of the grain size
359 distribution.

360 A number of experimental studies have focused on the behavior of carbonate materials (Carpenter
361 et al. 2016; Smith, Nielsen, and Di Toro 2015; Chen, Verberne, and Spiers 2015) under different
362 range of temperature, velocities and confinement using double direct shear and rotary shear
363 experiments. They observed a strong resistance and a friction coefficient at the peak between 0.6
364 and 0.7, consistent with our observations. However, the steady state friction coefficients they
365 obtained in their experiments stay close to the peak value, exhibiting thus almost no weakening or
366 strengthening. This discrepancy is due to the size of the sample used in the ACSA as explained in
367 (Chambon, Schmittbuhl, and Corfdir 2006a). This macroscopic slip-weakening operating over
368 decimetric distances is induced by a progressive mechanical decoupling between the interfacial
369 layer and the bulk of the samples, as shown by image analysis during shearing.

370 Experiments conducted at typical velocities consistent with earthquake nucleation (a few
371 micrometers per seconds like here) exhibits a velocity neutral or velocity strengthening behavior
372 (Carpenter et al. 2016; Verberne et al. 2014) for normal stresses corresponding to those
373 encountered in injection sites ($\sigma < 20$ MPa). The order of magnitude of the $(a-b)$ coefficient, we
374 have obtained (10^{-2}) is in agreement with previous studies on friction in gouge materials.
375 Nevertheless, our experiments reveal a velocity weakening behavior. This weakening has also
376 been observed by (Chambon 2003) for monodisperse quartz sand and glass beads. Friction in
377 gouge material displays a peculiar behavior compared rock to rock interface. The latter exhibits a
378 velocity softening for sub-seismic velocities whereas, the gouge a velocity strengthening
379 (Chambon 2003). The difference comes from the volumetric variations in granular materials that
380 can affect the shear behavior (Marone 1998; Beeler and Tullis 1997) and the large displacements

381 applied in our experiments to the gouge before applying the velocity steps. The apparent friction
382 is a gouge can be expressed as:

$$383 \quad \mu = \mu_g + \frac{d\phi}{d\gamma} \quad (6)$$

384 where μ_g is the grain to grain friction and $\frac{d\phi}{d\gamma}$ is the volume change (dilatancy rate) with increasing
385 shear displacement γ . In a granular assembly, even if the grain to grain friction is velocity
386 weakening, the properties of the apparent friction is dominated by the dilatancy rate for the usual
387 sizes of the sample. Yet, the rate of dilatancy increases with velocity (Marone, Raleigh, and Scholz
388 1990). (Chambon, Schmittbuhl, and Corfdir 2006b) have observed a low rate of volumetric
389 variations with the ACSA. Therefore, in this device, the behavior of the gouge is controlled by the
390 grain to grain friction, which explains the velocity weakening. The discrepancy between our results
391 and previous studies is due to the difference of the samples' thickness.

392 In Figure 6, our results show an increase of the $(a-b)$ parameter with the confining stress. This
393 evolution is in agreement with the results of (Carpenter et al. 2016) for the shearing of powdered
394 Carrara marble. They observed an increase of $(a-b)$ for confining stresses below 50 MPa and then
395 a decrease of this parameter. They argue that this change in the evolution is due to a transition
396 from a brittle to a semi-brittle mechanism for the deformation of the gouge. We can plot (see
397 **Error! Reference source not found.**) the evolution of the rate and state parameter $(a-b)$ as a
398 function of the confining stress for the different ratio of dissolution. The values for a given step is
399 defined as the mean of the different values obtained and the error bars are estimated by the
400 maximum deviation from the mean value of the different values of the coefficient obtained for the
401 same velocity step in the same conditions. The evolution of the rate and state parameter is well
402 described by a logarithmic law as follows:

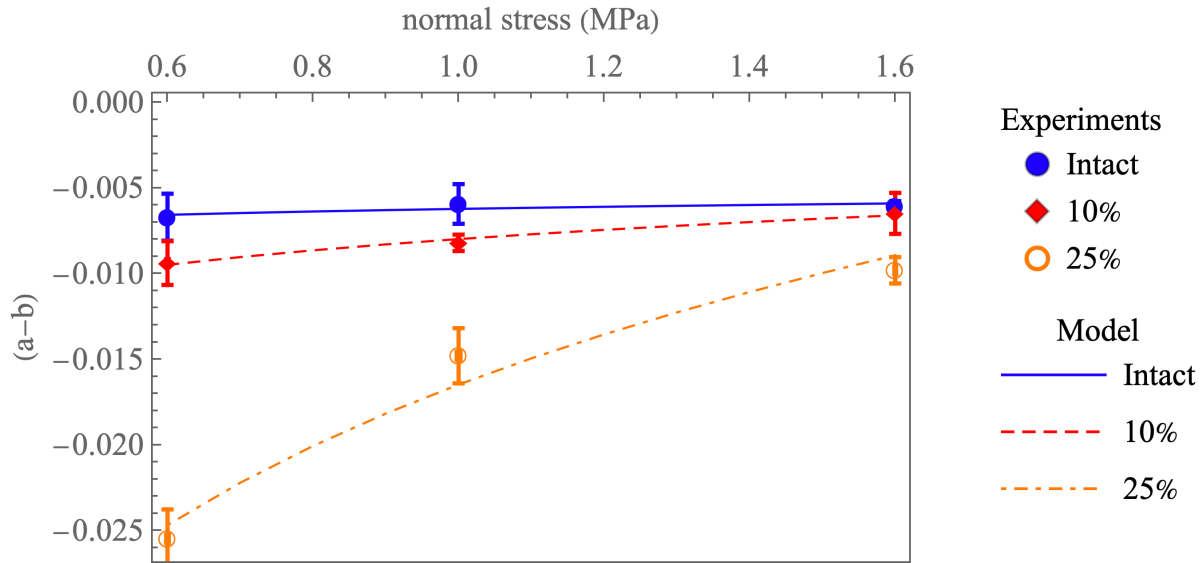
$$403 \quad (a - b) = A_1 \log\left(\frac{\sigma}{\sigma_0}\right) \quad (7)$$

404 where σ_0 is a reference confining stress, and A_1 is a parameter that depends on the velocity step
405 considered. The experimental values and the evolution of the model with parameters obtained from
406 a fitting with the least-squares method are represented together on Figure 8 for the velocity step
407 10-33 $\mu\text{m/s}$. The coefficient of determination exhibits values above 0.99 in all cases.

408 The analysis of the rugosity of the particles exhibit an increase with the dissolution. However,
409 previous studies on simulated gouges have shown that an increase of the roughness in a frictional
410 contact tends to induce a strengthening with velocity (Marone and Cox 1994; Anthony and Marone
411 2005). Therefore, the evolution of the rate-and-state parameters with the dissolution cannot be
412 interpreted as an effect of the rugosity of the particles, but rather on the evolution of the particle
413 size distribution. In the same way as the confining stress, we can plot the evolution of the rate and
414 state parameter ($a-b$) as a function of the degree of dissolution, represented by the fractal number
415 of the particle size distribution, for the different confining stresses (Figure 9). It is also well
416 described by a logarithmic law as follows:

$$417 \quad (a - b) = A_2 \log\left(\frac{D}{D_0}\right) \quad (8)$$

418 Where D_0 is a reference fractal number, and A_2 is a parameter that depends on the velocity step
419 considered. These empirical laws enable to capture the evolution of the parameter ($a-b$) in our
420 experiments, but previous studies have shown the triggering of complex multi-physical
421 mechanisms with increasing velocities and normal stresses (Di Toro et al. 2011; Verberne et al.
422 2014; Carpenter et al. 2016) that would limit the application of this law. However, for conditions
423 of the shallow crust ($\sigma < 20$ MPa and $T < 100^\circ\text{C}$) and for velocities corresponding to the nucleation
424 of a seismic slip, the mechanisms involved in the deformation of a gouge are the same as our
425 experiments.

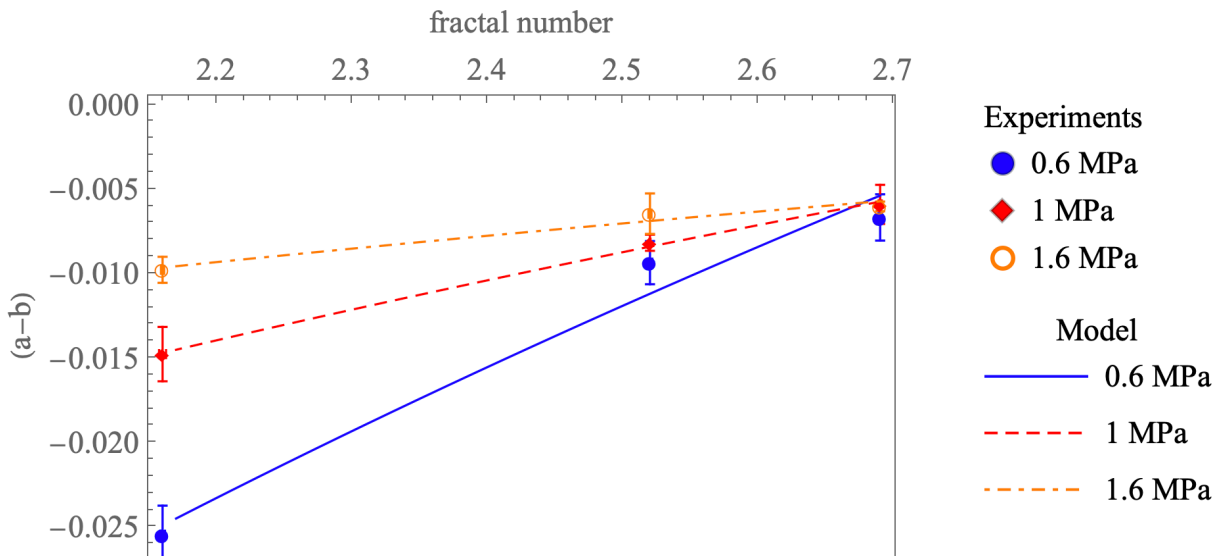


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Figure 8. Comparison of the model and the experimental data for the evolution of the rate and state parameters (a-b) as a function of the normal stress (velocity step 10^{-33} /s).

429



430

431
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Figure 9. Comparison of the model and the experimental data for the evolution of the rate and state parameters (a-b) as a function of the fractal number of the grain size distribution (velocity step 10^{-33} /s).

433

434 4.2. Implications for fault reactivation

435

436 The study of carbonate bearing faults is of key importance as a great number of earthquakes are
437 triggered in such lithologies. Noteworthy examples of seismic slips triggered and propagated
438 through a fault composed of calcite are the magnitude 7.9 Wenchuan (China) earthquake in 2008
439 (Chen et al. 2013), the magnitude 6.2 Aigion (Greece) earthquake in 1995 (Bernard et al. 1997),
440 or the magnitude 7.6 Chi-Chi (China) earthquake in 1999 (Boullier et al. 2009). Carbonate rocks
441 are particularly ubiquitous in Italy and in the Apennines where the tectonic activity produce
442 number of seismic events, like in the Amatrice and Norcia areas for the 2016-17 seismic sequence
443 (Pizzi et al. 2017), or in L'Aquila for the 2009 earthquake (Valoroso et al. 2014). Moreover, many
444 reservoirs in the world and potential sites for injection of CO₂ storage or acid gas disposal are
445 composed of carbonate materials (Bjorlykke 2010) and present faults that may induce leakage
446 from the storage if they would be reactivated.

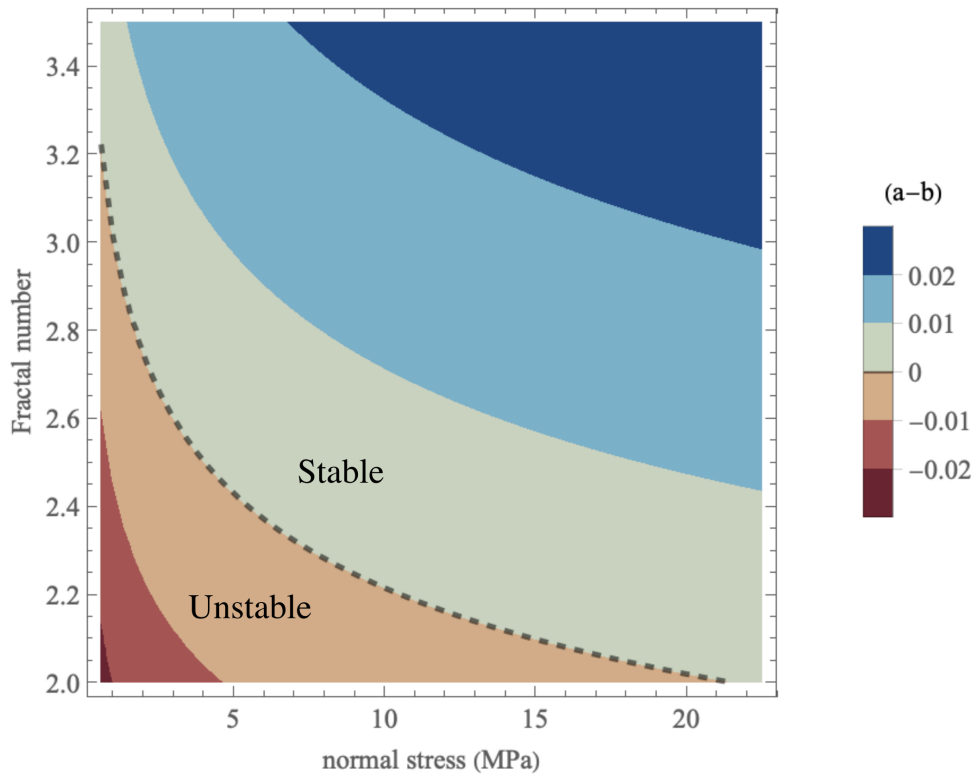
447 Sliding along an existing fault favors grain comminution and abrasion and thus, an increase of the
448 fractal number (Storti, Billi, and Salvini 2003). Our results show that this increase of the fractal
449 number would tend to stabilize the fault. On the other side, injection of an acid fluid tends to
450 decrease this fractal number by dissolving the small grains. This dissolution would make the fault
451 more velocity weakening and promote seismic slips. To determine in which conditions a fault
452 located in a carbonate reservoir can be reactivated by a change in its frictional properties, we merge
453 the two evolutions of the rate and state properties with the confining stress and the fractal number
454 into a single law:

$$455 \quad (a - b) = A_1 \log\left(\frac{\sigma}{\sigma_0}\right) + A_2 \log\left(\frac{D}{D_0}\right) \quad (9)$$

456 The coefficient of determination for this law and the two velocity steps considered in our
457 experiments are 0.95 and 0.98. We plot, in Figure 10, the value of $(a-b)$, as a function of the normal
458 stress and the fractal number. It enables us to define the region of stability for a fault in a carbonate
459 reservoir.

460 In our experiments, the maximum ratio of mass decrease due to dissolution of calcite that we have
461 considered is 25%. This value is similar to the decrease of calcite content obtained in (Bakker
462 2017) after 10,000 years, based on geochemical simulations of the long term effect of CO₂ on the
463 mineral composition of a fault gouge, considering a residence time of 1000 years. In this study,
464 the authors considered a carbonate bearing claystone and have investigated the effect of long-term

465 exposure to CO₂ on the frictional properties of the fault by changing its mineral composition
 466 according to the geochemical model and different scenarios.



467

468 *Figure 10. Contours of the values of (a-b) as functions of the normal stress and the fractal number. The dashed line represents*
 469 *the limit between the velocity stable and unstable regions.*

470

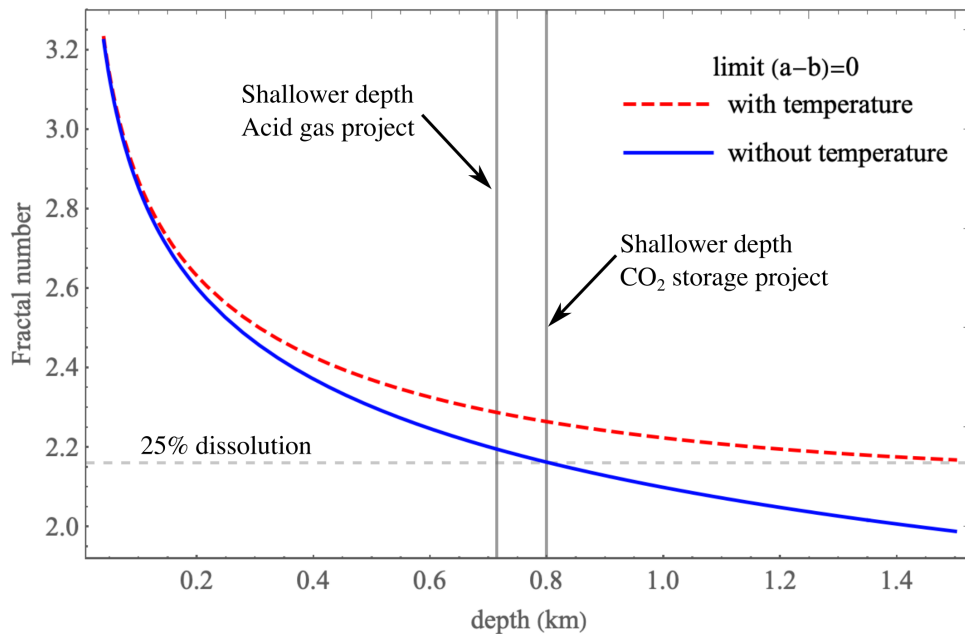
471 The temperature plays also an important role in the values of the rate and state friction parameters.
 472 (Verberne et al. 2014) conducted velocity stepping experiments on carbonate materials at
 473 velocities similar to this study and highlighted the effect of temperature. From their experimental
 474 data on CaCO₃, we can interpolate the dependency of the (a - b) parameter using a linear law
 475 between 20 and 100°C. This dependency of the rate and state parameter in temperature is then
 476 integrated into the previous logarithmic law (see Eq. 10).

$$477 \quad (a - b) = A_1 \log\left(\frac{\sigma}{\sigma_0}\right) + A_2 \log\left(\frac{D}{D_0}\right) - A_3 T \quad (10)$$

478 Where A_3 is a parameter describing the influence of temperature on (a-b). In Figure 11, we plot
 479 the diagram representing the limit of region of stability of the fault (a-b=0) considering the effect
 480 of temperature on the value of (a-b). Compared to the previous limit of stability, the one that

481 considers the effect of temperature is shifted upwards and it tends to destabilize the fault. The
 482 effect of temperature is integrated considering a geothermal gradient of 30°C/km and the normal
 483 stress at a given depth is calculated using a submerged unit weight of 15 kN/m³. In this diagram,
 484 we also highlight the shallower depth of a CO₂ storage project, in order to keep the CO₂
 485 supercritical, which is 800 meters (Nakanishi et al. 2009) and the shallower depth of acid gas
 486 injection project in western Canada, which is 705 meters (Bachu and Gunter 2002). We observe
 487 that the injection of an acid in a reservoir can lower the fractal number by dissolving the small
 488 grains and reach a value around 2.16 if 25% of the mass is dissolved and the initial fractal number
 489 is close to the value of a self-similar distribution (2.6). In this case, the (a-b) parameter of a fault
 490 gouge can present a transition from positive to negative, and therefore, the fault can become
 491 unstable and slip seismically at typical depth considered for acid gas and carbon storage.

492



493

494 *Figure 11. Plot of the limits of the stable and unstable zone for the friction parameters obtained from the logarithmic law in the*
 495 *depth-fractal number space taking (or not) into account the effect of temperature on (a-b).*

496

497 5. Concluding remarks

498 In this paper, we investigate the effect of the injection of an acid fluid on the frictional properties
 499 of a fault located in a carbonate reservoir. The rate and state parameters (*a-b*) of a simulated calcite

500 gouge are evaluated using an annular shear apparatus and conducting velocity stepping
501 experiments. We observe that the long-term exposure to an acid fluid can weaken the fault by
502 dissolving the small particles and changing the fractal number of the grain size distribution.
503 Moreover, the $(a-b)$ parameter increases with the normal stress applied to the fault. These two
504 dependencies are described using a logarithmic law and it enables us to estimate that chemical
505 reactions could be at the origin of a seismic slip in a storage site by changing the frictional
506 properties of the fault.

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672